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TRANSFORMATION CHARACTERISTICS OF Si-Mn TRIP STEELS AFTER THERMOMECHANICAL PROCESSING

by

ABBASS ZAREI HANZAKI

Department of Mining and Metallurgical Engineering McGill University Montreal, Canada

A Thesis Submitted to the Faculty of Graduate Studies and Research in partial fulfillment of the requirements for the degree of *Doctor of Philosophy*

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February, 1994



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TRANSFORMATION CHARACTERISTICS OF Si-Mn TRIP STEELS AFTER TMP

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February, 1994

In the name of God the compassionate and the merciful

> To my wife, who always believed in me,

and to my parents, to whom I owe everything.

ABSTRACT

Thermomechanical processing, which is a combination of deformation and heat treatment, is an optimum method to control the microstructural evolution and, accordingly, to generate the desired mechanical properties of materials. TRIP (Transformation-Induced-Plasticity) behavior is a powerful mechanism with which to improve mechanical properties. The basis of TRIP behavior is the retention of austenite with optimum characteristics (volume fraction, stability, size, morphology and composition) at room temperature. The transformation of retained austenite to martensite during deformation can lead to TRIP-enhanced properties. This work deals with the effects of thermomechanical processing parameters on the microstructural characteristics of TRIP steels, primarily from the point of view of the retained austenite condition in Si-Mn and Si-Mn-Nb bearing TRIP steels. Initially, a new test technique based on continuous cooling compression (CCC) testing was developed to find the critical temperatures of thermomechanical processing (*TMP*). A major finding from the CCC test is the ability to determine the Ae₃ (equilibrium austenite-to-ferrite transformation) temperature. In the CCC test, the Ae_3 appeared to be associated with an increase in the rate of increase in flow stress with decreasing temperature. In order to further evaluate this result, neutron diffractometry at high temperatures was used to monitor any crystallographic changes associated with the metastable region. The results revealed an increase in the rate of contraction of the austenite lattice as the temperature decreases through the metastable state (below Ae_3), compared with that observed as the temperature decreases through the stable austenite region. Having defined the critical *IMP* temperatures, the effects of processing parameters on the state of the retained austenite were examined by changing the thermomechanical processing conditions. These effects were more fundamentally considered by taking into account the influence of other micro constituents (austenite transformation products), present in the microstructure, on the retained austenite characteristics. The approach involved a systematic investigation of the effects of TMP parameters on the state of the retained austenite and finally, for the important cases, evaluating strength and ductility. In this respect, for example, it was shown that the prior austenite grain size obtained by different TMP (RCR or DRCR) has opposing effects on the retained austenite characteristics when a microalloying element (e.g., Nb) is added. In addition, if the dynamically recrystallized austenite substructure is retained to transformation temperatures, a higher volume fraction of retained austenite is obtained.

RĖSUMĖ

Les traitements thermomécaniques combinant déformation et traitement thermique offrent une méthode efficace pour le controle de l'évolution de la microstructure d'un alliage, ce qui permet l'obtention des propriétés mécaniques désirées dans le matériau. Le comportement TRIP (Transformation-Induced-Plasticity) est un mécanisme puissant permettant d'améliorer les propriétés mécaniques. Le principe du comportement TRIP repose sur la conservation de l'austénite avec des caractéristiques optimales (fraction volumique, stabilité, taille, morphologie et composition) à la température de la pièce. Lors de la déformation, la transformation de l'austénite résiduelle en martensite peut ammener des propriétés améliorées par TRIP. Ce travail se rapporte aux effets des paramètres de traitement thermomécanique sur les caractéristiques microstructurales des aciers TRIP, surtout du point de vue des conditions de conservation de l'austénite dans les acier Si-Mn et Si-Mn-Nb. Une nouvelle méthode d'essais basée sur un refroidissement continu en compression, la méthode CCC, (Continuous Cooling Compression) a été développée pour déterminer les températures critiques de traitement thermomécanique. L'avantage de la méthode CCC repose sur la capacité de déterminer la température Ae_3 (température d'équilibre de la transformation austénite-ferrite). Dans les essais CCC, la température Ae_3 apparait, lors de la diminution en température, comme un accroissement du taux d'augmentation de la contrainte d'écoulement. Pour permettre une évaluation plus complète de ces résultats, la diffractometrie de neutrons à haute température a été utilisée pour suivre les changements crystallographiques associés au régions métastables. Les résultats révèlent une augmentation du taux de contraction de la maille austénitique lors d'une baisse de température dans la zone métastable (en dessous de Ae_3), ceci comparé avec ce qui est observé quand la température diminue dans la zone où l'austénite est stable. Ayant défini les températures critiques de traitement thermomécanique, les effets des paramètres de traitement sur l'état de l'austénite résiduelle ont été examinés en changeant les conditions de traitement thermomécanique. Ces effets furent considérés suivants les caractéristiques de l'austénite résiduelle obtenue, en tenant compte de l'influence des autres microconstituents (produits de transformation de l'austénite) présents dans la microstructure. L'approche utilise une étude systématique de l'effet des paramètres de traitement thermomécanique sur l'état de l'austénite résiduelle et pour les cas les plus important, la résistance mécanique et la ductilité sont évaluées. Il fut, entre autre, démontré que la taille des grains austénitique initiaux, obtenus par différents traitements thermomécaniques (laminage controle par recristallisation ou laminage controle par recristallisation dynamique (RCR et DRCR)), a des effets opposés sur les caractéristiques de l'austénite résiduelle lors de l'ajout d'un élément de microalliage, comme le niobium. De plus, si la structure d'austénite obtenue par recristallisation dynamique est retenue jusqu'à la température de transformation, un plus grand volume d'austénite résiduelle est obtenu.

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CHAPTER 1

INTRODUCTION

When quenched and tempered low alloy steels are heat treated to produce higher tensile and yield strengths, the true stress-true strain curve shapes are similar, but necking begins at lower strains ⁽¹⁾. The start of plastic instability at lower strains in quenched and tempered steels indicates that the rate of strain hardening produced by dislocation interactions is inadequate to compensate for the increase in stress in the region of the neck. Therefore, effects stronger than increases in dislocation density must be introduced during plastic straining if the onset of necking is to be delayed, so that the ductility, as indicated by the reduction of area, can be increased by increasing the region of uniform strain. Such barriers must be introduced *during* plastic straining, not before, otherwise the yield strength would be increased without necessarily increasing the strain hardening rate.

It is known that deformation-induced martensite can significantly strengthen the deforming alloy $^{(2)}$, thus increasing the strain to necking. This is the principle of *TRansformation Induced Plasticity* (TRIP) steels. The phenomenon of strain-induced transformation (*SIT*) of retained austenite to martensite, also results in higher toughness properties through localized TRIPing or crack tip blunting.

The role of the transformation in postponing necking was first recognized by *Banerjee et al.* ⁽³⁾. They suggested that, when necking takes place, the retained austenite in the neck region transforms to martensite in response to the applied deformation. The transformed region possesses a much higher flow stress than the adjacent untransformed regions. Thus, the progress of necking stops, and the regions immediately adjacent to the neck begin to deform, in turn, transforming to martensite. It has been clearly demonstrated that the mechanical properties of such metastable austenite (austenite which is retained at room temperature) are dependent on the formation of deformation-induced martensite, and that the production of this martensite is, in turn, dependent on deformation parameters ⁽⁴⁾. For a given set of deformation parameters, the retained austenite characteristics (volume fraction, morphology, chemical composition, size and distribution) are the factors which dictate the final mechanical properties of TRIP steels.

The appropriate microstructures, to attain such properties, have been obtained through the application of heat treatment (mostly intercritical annealing), as has been well established in the majority of previous investigations. On the other hand, thermomechanical processing (*TMP*), which is a combination of heat treatment and deformation, is also a powerful method to control the microstructure, but has not gained general acceptance in the processing of TRIP steels.

In order to produce a TRIP steel, it is clearly necessary to generate a microstructure which has retained austenite. Alloying elements (e.g., Mn, C, etc.) can stabilize the austenite, by decreasing the M_s temperature (martensite start temperature) to below room temperature. The other important factors affecting the austenite retention are austenite particle size (particles of austenite to be retained) and the flow strength of austenite. These factors are changed by both alloying and TMP.

In order to establish the effectiveness of *TMP* in the processing of TRIP steels, the effects of *TMP* on the transformation characteristics of TRIP steels must be first considered, since these ultimately control the retained austenite characteristics. Consequently, the first object of this work was to verify the transformation characteristics of TRIP steels during *TMP*. The latter was carried out with the help of a novel method based on continuous cooling deformation testing. Since the quantity, morphology and composition of the retained austenite have major roles in the final mechanical properties of this class of steel, the second part of the work was to study the effect of thermomechanical processing parameters on the state of the retained austenite.

A literature review of the fundamentals of TRIP steels is presented in *Chapter 2*. TRIP steels are divided into three classes and their compositional/microstructural differences are explained. After demonstrating the various effects of processing, the microstructural characteristics and their related mechanical properties are described. The peculiarities of various transformations during cooling from single phase austenite to room temperature are then highlighted. *Chapter 2* is closed by referring to different modes of deformation-induced martensite formation.

In Chapter 3, the experimental materials and methods are presented. TMP was performed by hot compression testing. Optical microscopy, image processing, scanning electron microscopy, transmission electron microscopy, x-ray and neutron diffractometry were used to characterize the microstructural variations of Si-Mn bainitic TRIP steels. A small-scale punch test, was used to establish basic mechanical properties.

Chapter 4 deals with the analysis of continuous cooling compression (CCC) data, for characterization of transformations during TMP. The results and analysis of fourteen different grades of steel, (including five different compositions of Si-Mn bainitic TRIP steels) are described in this chapter.

The results of thermomechanical, microstructural and mechanical tests, in connection with the retained austenite characterization, are given in *Chapter 5*. This part of the study is presented in the following sequence:

Thermomechanical processing in the austenite region : effect of dynamically and statically recrystallized austenite effect of pancaked austenite
The characteristics of thermomechanically formed ferrite: effect of kinetics of ferrite reaction effect of morphology of ferrite effect of size and quantity of ferrite
The characteristics of thermomechanically formed bainite: effect of kinetics of bainite reaction effect of kinetics of bainite reaction

These results are discussed and rationalized in *Chapter 6*. From this, the general concepts which can be used to control the properties of Si-Mn TRIP steels, will be established.

Finally, conclusions, recommendations and suggestions for future work are given in *Chapter 7*.

CHAPTER 2

LITERATURE REVIEW

In principle, it is possible to design a metastable austenitic steel whose M_s (martensite transformation start on cooling) and M_d (the upper limit of temperature at which plastic deformation can initiate a martensite transformation) temperatures would be below room temperature prior to forming processes, but whose M_d temperature would be above room temperature after processing. In such a steel, any subsequent formation of deformation-induced martensite would enhance strength and ductility, as will be seen later. Because of the enhanced ductility caused by the deformation induced transformation, the grades of steel designed around these concepts have been given the acronym "TRIP" which stands for "TRansformation Induced Plasticity" (2).

Since the object of this work is to investigate the effect of thermomechanical processing (*TMP*) parameters on the state of the retained austenite in a new class of TRIP steels, the general aspects and behaviour of TRIP steels will be reviewed in the following sections.

2.1 Classification of TRIP Steels

2.1.1 Metastable Austenitic Steels

This class of TRIP steels encompasses metastable austenitic stainless steel grades. It has long been known that if a stainless steel, such as Type 301 (0.15%C, 2.00%Mn, 1.00%Si, 16.00-18.00%Cr and 6.00-8.00%Ni), is plastically deformed at room temperature, some of the austenite in the most severely strained portions of the specimen will transform to martensite by the action of the applied strain ⁽⁴⁾. Martensite produced during straining can prevent early failure by necking via stabilization of the neck through increases in the rate of strain hardening. This, coupled with the suppression of early necking, leads to an overall increase of both tensile strength and elongation ⁽²⁾. The structure, properties and processing of this group have been reviewed by many workers ⁽²⁻⁹⁾.

It is useful to note that a variety of compositions [Table 2.1] and processing steps can be employed to produce classical TRIP steels ⁽⁹⁾. An example of a processing sequence is as follows:

- 1. Solutionizing anneal followed by quenching
- 2. Straining above M_d to deform the austenite and to produce fine carbide precipitation
- 3. Cold form at room temperature (below M_d) to transform some austenite to martensite

The excellent combinations of yield strength and total elongation obtainable with this kind of processing are shown in Fig. 2.1. However, because of the cost of the alloys, the difficulty of applying large plastic deformations at low temperature, and problems with joining, such steels would only be used in special applications.

Table 2.1	Typical	composition	of
	austenitic	TRIP steels	

Composition	(1)	(2)	(3)
С	0.25	0.25	2.00
Mn	2.50	1.70	2.00
Si	2.00	2.00	2.00
Cr	9.00	10.00	13.50
Ni	9.00	8.80	8.80
Мо	5.00	5.50	3.00

2.1.2 Dual-Phase and HSLA Steels Containing Retained Austenite

Research on dual-phase steels began, through the demands of the automotive industry for steel sheet with high formability combined with high work-hardening rate (*i.e.* high strength requirements), with *Cairns* and *Charles* (10,11). Interest in these steels intensified with the work of *Hayami* and *Furakawa* (12). They showed that continuous



Fig. 2.1 Ranges of yield strength and total elongation to failure characteristic of high strength steels.

annealing of cold-rolled Si-Mn sheet steels in the intercritical temperature region, *i.e.* the $\gamma + \alpha$ two phase region, produced steels with a ferrite-martensite microstructure, which had ductilities superior to that of normal precipitation-hardened or solid-solution-hardened HSLA sheet steels, [Fig. 2.2].

Basically, dual-phase steels are a class of high-strength lowalloy steels characterized by a microstructure consisting of a dispersion of about 20% of hard martensite particles in a soft, ductile ferrite matrix. The term "dual-phase" refers to these two phases, although small amounts of bainite, pearlite and retained austenite may also be present. These steels have a number of



Fig. 2.2 Relation between tensile strength and total elongation for various HSLA sheet steels.

unique properties, which include: continuous yielding behaviour (no yield point), a low

0.2% offset yield strength (~340 MPa), a high tensile strength (~690 MPa), a high work hardening rate and an unusually high uniform and total elongation ⁽¹³⁾. Typical compositions of dual-phase steels are given in Table 2.2.

As was mentioned above, dual-phase steels consist of about 80% retained and transformed ferrite, with the balance being a mixture of martensite and retained austenite after an intercritical heat treatment. It was found, surprisingly, that the amount of retained austenite could be quite significant, about 10% in some dual-phase steels (14-16). The characteristics of the retained austenite were

Table 2.2	Typical	compositions	of	dual
	phase sto	eels		

Composition	(1)	(2)	(3)
С	0.11	0.12	0.06
Mn	1.43	1.55	0.90
Si	0.58	0.61	1.35
Cr	0.12	-	0.50
V	-	0.06	-
Мо	0.08	-	0.35

studied in detail by transmission electron microscopy (*TEM*), where the morphology, composition and size were shown to be substantially variable and inhomogenous (17). The microconstituents present make the yielding and deformation mechanism in dual-phase steels quite complex, and this has been the subject of many studies (18-23).

Turning now to high-strength, low alloy (HSLA) steels, these are usually low C grades, with relatively low levels of alloying addition which impart improved mechanical properties. These steels can be produced in the as hot rolled condition with yield strengths in the range of 290-550 MPa and ultimate tensile strengths of 415-700 MPa ⁽²⁴⁾.

The presence and the effect of retained austenite in HSLA steels have received little attention in the literature. Early work was done by *Rigsbee* and *VanderArend* (14) on Nb and V bearing HSLA steels. The retained austenite characteristics and its effects on the mechanical properties of some HSLA steels have been studied in a few other investigations (17,25). Such studies focussed on the influence of prior microstructure and other intercritical annealing parameters, as will be discussed later. The findings of these investigations have been compared to those obtained from dual phase grades, despite differences in the austenite formation characteristics during intercritical annealing.

2.1.3 The Si-Mn Bainitic TRIP Steels

This latest version of TRIP steels has a chemical composition range of about 0.15-0.4%C, 1-2%Si and 1-2%Mn. They were developed as a result of the observation that a significant amount of austenite can be retained in steels containing about 2% silicon, when

cooled to room temperature through the bainite transformation region ^(26,27). Their microstructure consists of a combination of ferrite, bainite, retained austenite and a small amount of martensite, depending on the treatment and process variables. The different microstructures and mechanical properties of these steels, mostly due to intercritical annealing, have been related to retained austenite characteristics. This has been reviewed by many workers ⁽²⁸⁻³⁷⁾, and will be discussed in more detail later. Before this, the processing aspects will be covered.

2.2 Processing of TRIP Steels

Apart from the case of metastable austenitic stainless steels (classical TRIP steels), in order to retain austenite and to produce the desired microstructure, TRIP type steels have usually been subjected to intercritical annealing (heating up to and holding in the two phase $\alpha + \gamma$ region). Thermomechanical processing (*TMP*), which is a powerful method to control microstructures, has not found general acceptance in the processing of TRIP steels. In the following, the main aspects of both thermal (*i.e.* intercritical annealing) and thermomechanical processing techniques, with respect to TRIP steels, will be discussed.

2.2.1 Intercritical Annealing

The most important parameters which dictate the characteristics of the microstructural constituents after intercritical annealing are prior microstructure, annealing conditions (temperature and time) and cooling rate after annealing.

2.2.1.1 Prior microstructure

As will be discussed later, the austenite particle size plays a significant role in whether or not it will be retained. Thus, if the growth of austenite particles that nucleate upon heating to intercritical temperatures is restricted to a very small size (about 1 μm or less), higher amounts of austenite are expected to be retained. Since austenite formation is a nucleation and growth type of reaction, a reduction in the average size of austenite particles during reaustenitization can be brought about by increasing the nucleation rate of austenite, and by inhibiting the growth of these nuclei.

It is fairly well established ⁽³⁸⁾ that the austenite nucleation rate can be significantly increased if the starting microstructure consists of a large number of fine carbide particles (which provide a ready source of carbon) at ferrite grain boundaries. This is due to the creation of triple junctions in the vicinity of the carbide/grain boundary interfaces, which

result in a lowering of the interfacial energy for heterogeneous nucleation of austenite. It has been suggested $^{(39)}$ that the growth of austenite particles during reaustenitization can be severely restricted by the presence of martensite lath boundaries (*MLB*). A starting microstructure consisting of martensite, or more preferably tempered martensite, seems to satisfy the above condition $^{(23)}$.

Due to the absence of *MLB* in an (F+P) structure, the austenite which nucleates at ferrite/pearlite, pearlite/pearlite and ferrite/ferrite boundaries grows rapidly, and only a few austenite particles which could not grow are retained, resulting in low retained austenite volume fractions. It should be noted that, within the aforementioned (F+P) type boundaries, changing the ferrite/pearlite initial microstructure to ferrite/cementite (*e.g.*, spheroidized pearlite by prolonged aging) can result in still less retained austenite (³⁰).

2.2.1.2 Annealing temperature and time

It is generally believed that there are optimum values for annealing time and temperature, which depend on whether the treatment after intercritical annealing involves quenching to room temperature (14,23), or quenching into the bainite region (28,34). In the former, the optimum temperature falls between the Ae_1 and Ae_3 (the equilibrium

temperatures of austenite to pearlite and austenite to ferrite transformation_ respectively), Fig. 2.3, whereas in the latter, it is close to the Ae_1 . More specifically, temperature two regimes can be defined; regime I from the Ae_1 to the optimum, in which the amount of retained austenite increases, and regime II, from the optimum to the Ae_3 , which is accompanied by a decrease in retained austenite. The existence of these regimes can be explained as follows.



Fig. 2.3 Variation of the retained austenite volume fraction as a function of annealing temperature.

By annealing at temperatures in regime I, more and more austenite particles nucleate with increasing temperature up to the optimum (for a constant annealing time) without undergoing any significant growth. Thus, most of these will tend to be retained because of their very small size. However, at temperatures in regime II, significant growth of austenite particles takes place after their nucleation. Therefore, although the total volume of austenite formed in this temperature regime increases with increasing temperature, the amount of austenite retained decreases since the austenite particles are, on average, larger. In addition, the initial carbon content of larger amounts of austenite formed during annealing at higher intercritical annealing temperature would be lower (*i.e.* higher M_s).

The effect of annealing time on austenite retention is analogous to that of annealing temperature, in that the austenite formation at any temperature is dominated by nucleation kinetics up to a certain time, after which growth becomes dominant.

In the case of quenching into the bainitic region, decreasing the annealing temperature to just above the Ae_1 , leads to a lower critical cooling rate necessary to avoid pearlite formation. Increasing the annealing temperature without changing the cooling rate encourages pearlite formation, which in turn leads austenite consumption to and a lowering of its hardenability. The effect of annealing temperatures on the retained austenite and, accordingly, on the mechanical properties of a Si-Mn TRIP steel is shown in Fig. 2.4 (36). As can be seen, a decrease in the annealing temperature significantly affects the amount of retained austenite and hence the percentage of uniform elongation.



Fig. 2.4 Changes in mechanical properties and retained austenite volume fraction with holding time in bainite transformation range after annealing in the single austenite or intercritical range.

Chapter 2 LITERATURE REVIEW

Finally, it should be noted that the annealing conditions affect the subsequent transformation kinetics of austenite. As can be seen in Fig. 2.5, bv changing the annealing temperature, the volume fraction and the type of microstructural constituents change. This, in turn, can result in different amounts of retained austenite



Fig. 2.5 Changes in the volume fraction of microstructural components with annealing temperature. (The samples were held 5 min at annealing temperature and then cooled fast to 400°C and held for 5 min.)

2.2.1.3 Cooling rate

Another important processing variable in the production of TRIP steels is the cooling rate after intercritical annealing, which affects: (i) the isothermal (nonmartensitic) decomposition products, and (ii) interstitial solute partitioning. Fast quenching essentially suppresses both these reactions. With slower cooling rates, decomposition products at high temperatures cannot be suppressed, and part of the austenite transforms to ferrite, pearlite and/or bainite.

There is evidence to suggest that there is a minimum cooling rate below which austenite retention is basically negligible (17,28,36). It should be mentioned that changing the cooling rate not only changes the volume fraction of retained austenite, but also alters the morphology of retained austenite. For example, the change from oil quenching to air cooling leads not only to a decrease in the amount of retained austenite, but also results in a shift from interlayer films between laths of martensite to blocky particles enclosed by ferrite and martensite (17).

2.2.2 Thermomechanical Processing

An industrial *TMP* schedule is usually divided into four stages, these being: (i) deformation in the high temperature austenite region to obtain grain refinement via repeated deformation and recrystallization; (ii) deformation in the non-recrystallized austenite region to increase nucleation sites for ferrite grains via deformed austenite; (iii) deformation in the austenite-ferrite two phase region to increase ferrite strength; and (iv) accelerated cooling during the austenite-to-ferrite transformation in order to produce a fine
ferrite structure mixed with fine bainite or martensite islands (40-43). The microstructural characteristics resulting from *TMP* that affect the subsequent austenite transformation are the prior austenite condition (grain size, dislocation density and morphology), finishing temperature (which may dictate the state of prior austenite, ferrite grain size and ferrite strength) and coiling temperature (*i.e.* the temperature at which the hot rolled steel is coiled and effectively held for a long time).

An initial study of the effects of TMP parameters on the as-hot deformed mechanical properties of TRIP steels was performed by Kawano et al. ⁽²⁹⁾. In a steel of 0.2%C, 1.44-1.46%Si and 1.45-1.60%Mn, they found that increasing the ferrite fraction and refining the ferrite grain size effectively increased retained austenite. For the ferrite fraction, an optimum of 60-65% was determined. They showed that, as the finishing temperature was decreased towards the Ar_3 , the fraction of retained austenite increased. This result was interpreted as an effect of ferrite refining, due to the lower finishing temperature, on increasing the diffusion path for carbon to move to the remaining austenite.

Another investigation on processing of Si-Mn bainitic TRIP steels by means of *IMP* was performed by *Tsukatani et al.* ⁽³⁵⁾. They also demonstrated that finish rolling temperature and coiling temperature appeared to be important factors in terms of introducing large amounts of retained austenite. Once again, the finishing temperature which gave rise to a high volume fraction of retained austenite, nearly coincided with the Ar_3 temperature.

2.3 Microstructure and Mechanical Properties

The microstructural parameters which determine the mechanical properties of TRIP steels are not only determined by the state of the retained austenite (quantity and stability), but by the characteristics of all the other microstructural constituents. The latter in turn, are a function of processing. The stability of the retained austenite, which will be discussed in detail later, is dictated by its morphology, size and solute partitioning aspects.

2.3.1 Mechanical Properties

The various mechanical properties (*e.g.*, formability, strength, fracture toughness, fatigue properties, etc.) of TRIP steels resulting from different states of retained austenite, have been investigated extensively (31,32,35,44,45).

With regard to formability, the product of tensile strength and total elongation ($TS \times T. EL$) can be considered as an index of formability. Values as high as 30000 MPa·% (1000 MPa×30 %) have been reported for Si-Mn and dual phase TRIP aided steels ⁽³⁴⁾. *Matsumura et al.* ⁽³²⁾ investigated the effects of retained austenite on press-formabilities of austempered (austempering is the isothermal transformation of austenite to bainite) 0.4C, 1-1.5Si and 1.5Mn sheet steels, with tensile strengths of more than 1000 MPa. The results showed convincing relationships between formability and initial volume fraction of retained austenite (V_{RA}). As V_{RA} increased, the height of stretch forming increased linearly to a maximum at 20% V_{RA} . Bendability, as well as hole expandability of mechanically ground holes, were gradually improved, and were optimized at 15%-20% V_{RA} . All of these formability indexes decreased rapidly beyond 15%-20% retained austenite, probably because of the reduction in the retained austenite stability.

It has been clarified that strain induced transformation (SIT) of retained austenite to martensite tends to improve the fatigue properties of the steels containing retained austenite, such as in all types of TRIP steels, carburized and carbonitrided steels, etc. ⁽³¹⁾. For instance, the low cycle fatigue behaviour of 0.1~0.4%C-1.5%Si-1.5%Mn TRIP-aided steels (intercritically annealed at 780°C for 1200s and ausformed (low temperature thermomechanical treatment of austenite) at 400°C for 10 to 1000s) containing 6 to 20% retained austenite was measured at room temperature. The role of retained austenite on this behaviour was investigated in terms of the measured bauschinger stress by Yasuki and co workers (31). Low cycle fatigue of these steels was generally characterized by severe initial fatigue-hardening and successive small fatigue hardening or fatigue softening. The amount of initial fatigue hardening increased with increasing initial retained austenite content and total strain amplitude. The fatigue properties were superior to those of conventional ferrite-martensite dual-phase steels. The fatigue-hardening was principally ascribed to the increased internal stress arising from a misfit strain between soft and hard phases (retained austenite and newly formed martensite, respectively). Retained austenite enhanced the internal stress due to the increase in fresh martensite produced by the strain induced transformation.

It has been pointed out that the phase transformation is equivalent to a mode of plastic deformation. As such it is capable of absorbing strain energy, during a fracture toughness test, that might otherwise have gone to crack extension, thereby resulting in potential toughness increases (6.46). Antolovich and Singh (8) noted that the room temperature fracture toughness of metastable austenitic TRIP steels (0.6%C, 9%Cr, 8%Ni and 2%Mn) was about 105 (MPa. \sqrt{m}) at a yield strength of 1723 MPa. This is an extraordinary toughness when it is considered that fracture takes place by cleavage through martensite. This high toughness was attributed to the TRIP phenomenon. They also investigated the plane strain fracture toughness of TRIP alloys as a function of temperature. Two distinct regimes were observed: a high temperature regime (100 °C to 200 °C) where no martensite formed during fracturing and a low temperature regime (-196 °C to 20 °C) where martensite formed during fracturing. The fracture toughness of the low temperature regime at room temperature was much higher than the extrapolated high temperature predicted values, indicating that the austenite to martensite phase transformation makes a significant contribution to the room temperature fracture toughness of TRIP alloys.

The presence of retained austenite in M2 high-speed-steel ⁽⁴⁷⁾ and in low-alloy ultrahigh-strength steels, such as AISI 4340 ⁽⁴⁸⁾, has been found to be beneficial for the toughness and the plane strain fracture toughness (K_{IC}), respectively. Two mechanisms have been postulated to explain this beneficial effect of retained austenite on toughness ⁽⁴⁹⁾: (i) localized TRIP and (ii) crack tip branching or blunting.

2.3.2 Microstructure and the Stability of the Retained Austenue

The transformation of retained austenite to martensite will serve as a source of dislocations which will further enhance the rate of work hardening. This indicates that, to obtain a significant effect, the transformation should take place gradually with deformation, beginning at strains somewhat lower than the maximum uniform elongation. Hence, the extremes of the retained austenite stability and its effects on properties should be defined. This has been reviewed by *Sangal et al.* ⁽²²⁾ through the development of a model. As is shown in Fig. 2.6, for very unstable and moderately stable retained austenite, the *UTS* (ultimate tensile strength) increases almost linearly with increasing volume fraction of retained austenite. On the other hand, whereas ε_{μ} (uniform strain) increases almost linearly with increasing volume fraction of a moderately stable retained austenite, it decreases for an unstable one. For a highly stable retained austenite, the *UTS* increases modestly and ε_{μ} decreases only marginally with increasing volume fraction of this phase.

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In the case of unstable austenite, the rate of strain-induced transformation is so rapid that most of the retained austenite transforms to martensite at less Therefore, the than 8% plastic strain. beneficial effect of strain-induced transformation on ductility apparently cannot be obtained with an unstable retained austenite. The probable reason for this is that SIT contributes to the work-hardening only in the initial stage of straining. Once necking starts, martensite is already present and decohesion of the ferrite/martensite interface, or martensite particle fracture, results in early fracture, leading to a lower overall ε_{ν} .

On the other hand, if the rate of strain-induced transformation is so



Variation of uniform strain and ultimate tensile strength as a function of the retained austenite volume fraction of different mechanical stability.

sluggish that retained austenite does not transform to martensite (*i.e.* in the case of stable retained austenite) then an increase in ε_u or UTS is not observed. In this case, the work-hardening rate continuously decays up to fracture. The significant increase of ε_u , in the case of the steel containing retained austenite of moderate stability, is due to the fact that a gradual transformation of retained austenite to martensite during the entire process of deformation results in an additionally higher dislocation density, and therefore increased rate of work-hardening up to the later stages of deformation. Such a sustained work hardening delays the process of necking, resulting in much higher values of ε_u and UTS.

Fig. 2.6

As was mentioned earlier in this section, the parameters which dictate the stability of the retained austenite are: particle size, morphology and solute partitioning. The increase in stability with decreasing particle size can be explained by the probable higher level of chemical partitioning and by the increased effectiveness of the interface (relative to the volume of the particle) in constraining the particle against the required particle shape changes accompanying a martensitic transformation ⁽⁵⁰⁾. However, it has been emphasized that the role of particle size on the stability of the retained austenite is more important than solute partitioning (14.25). Thus, the role of plastic constraint is more effective than solute partitioning.

The morphologies of the retained austenite in TRIP steels have been classified into three groups (14,16.37,51-54). One is an *interlath* type which is found along martensite or bainite lath boundaries (37,52). Another is the *isolated* type which is observed around ferrite grain boundaries and isolated from martensite or bainite particles (14,16). The last, which is usually found inside martensite particles, has no particular shape and has been designated as *capsulated* type (52-54). The isolated type of retained austenite transforms at low strains (a few percent of elongation) (51) since it lies mainly around the ferrite grain boundaries, where much higher stresses can be developed due to stress gradient effects between grains of different orientations (55). The situation is quite different for the interlath and capsulated type since they would be protected from the imposed stress by the surrounding martensite, which would bear much of the load. Consequently, the capsulated and interlath types of retained austenite are much more stable than the isolated one and, therefore, can transform at much higher total strains.

The last effective parameter which controls the stability of retained austenite is solute partitioning, *i.e.* C and Mn enrichment of the retained austenite $(^{56})$. The higher the solute enrichment, the higher the stability (*i.e.* higher hardenability and lower M_s temperature). One further point which should be noted, is the effect of test conditions

(e.g., temperature in a tensile test) on the stability of retained austenite. For instance, Sachdev (21) pointed out that increasing the tensile test temperature resulted in a shift in the retained austenite to martensite transformation strain to higher values (*i.e.* higher stability, or lower M_s temperature, of retained austenite).

The presence and the effect of retained austenite in HSLA steels have received little attention in the literature. By applying intercritical annealing on Nb and V bearing HSLA steels followed by heated oil quenching, the morphology,



Fig. 2.7 X-ray analysis of normalized retained austenite volume fraction as a function of tensile strain for steel of: 0.072%C, 1.26%Mn, 0.30%Si, 0.077%Nb and 0.08%V.

amount and stability of retained austenite have been studied. *Rigsbee* and *VanderArend* ⁽¹⁴⁾, in their research on Nb and V bearing HSLA steels, rationalized the stability of retained austenite more on the basis of particle size rather than solute partitioning. Their findings regarding changes in the retained austenite volume fraction by straining are shown in Fig. 2.7. As can be seen, during the first five percent strain, slightly less than 50% of the retained austenite transformed to martensite. The rate of loss of retained austenite decreased with increasing strain, and almost 20% of the retained austenite remained untransformed at 20% strain (probably because of increasing stability of the retained austenite due to plastic constraint by the newly formed martensite). These findings are also supported by the work of *Long et al.* ⁽²⁵⁾.

2.4 Chemical Composition and TRIP Behaviour

One of the most important factors affecting the characteristics of the remaining austenite, which gives rise to TRIP behaviour, is the alloy content. Some of the alloying elements (such as C, Mn, Cr, Ni, etc.) have a direct effect on austenite retention and therefore TRIP properties. If sufficient alloying elements of this kind are added, the M_s and M_f temperatures (start and finish temperatures of martensite transformation, respectively) are lowered to a marked degree. For example, a 1% increase in C, Mn, Cr, Mo and W content produces a 50%, 20%, 11%, 9% and 8% increase, respectively, in the as-quenched retained austenite volume fraction.

These alloying elements may also have a direct effect on TRIP properties, as pointed out by *Ludwigson* and *Berger* ⁽⁷⁾. They proposed a model of the plastic behaviour based on contributions of austenite strain hardening, transformation, and the strength of the resulting martensite. The model was quantitatively verified by the load-strain data collected on a series of 11 stainless steels, based on type 301, with different C, Mn, Ni, Cr, and N levels. They pointed out that, of the compositional variables, carbon and nitrogen had the greatest effects on plastic behaviour. Additions of these interstitial elements strengthened both the parent austenite and any martensite formed from it. Substitutional elements such as Mn, Ni and Cr generally had much smaller effects on plastic behaviour than the interstitial elements.

Other alloying elements, such as Si, P, etc., have an indirect effect on TRIP properties via control of the microstructural evolution (*e.g.*, inhibiting the carbide precipitation by Si). In this respect, *Tsukatani et al.* (35) showed that silicon additions

greater than 1.0% resulted in a significant increase in the volume fraction of retained austenite, due to the change in second phase from bainite-pearlite to bainitic ferrite. The maximum volume fraction of retained austenite, and the resulting optimum combination of tensile strength and ductility, was obtained in a 0.2%C, 2.0%Si and 1.5%Mn steel. They also reported that martensite increased with increasing manganese content, with only a small change in the volume fraction of retained austenite (35).

In the case of Si-Mn TRIP steels, the transformation from austenite to bainitic ferrite was assumed to be highly encouraged at temperatures of 350 to 400°C. If it is supposed that the bainitic transformation is a diffusionless transformation, then the growth of a bainite sheaf occurs by the martensitic propagation of bainite subunits. Redistribution of carbon from these subunits to the remaining austenite mainly occurs after the actual transformation has taken place. Consequently, austenite is retained due to a lowered M_s temperature by the enrichment of carbon (35). A decrease in manganese content was assumed to bring about instability of austenite and thus accelerate ferrite formation during the hot rolling and cooling process (35).

The indirect effect of alloying elements such as Si and Mn (57,58) was also studied by *Sakuma et al.* (36). They showed that annealing a sheet steel with 0.2%C in the intercritical range produced very fine particles of retained austenite. These were moderately stabilized by subsequent holding in the bainite transformation range, due to C enrichment. It was shown that the optimum cooling rate from the intercritical region was reduced with an increase of Mn content but was not influenced by Si content. Higher Mn levels increased V_{RA} although the uniform elongation remained the same. In this case, the product of tensile strength and total elongation was increased due to an increase in the tensile strength. In contrast to Mn, Si did not affect the retained austenite level but improved the uniform elongation by increasing its stability.

2.4.1 Effects of Si

The effect of silicon is covered here in some depth because it is the element that makes it feasible to retain austenite after processing. In fact, Si is essentially a ferrite stabilizer, but there are several different effects attributable to Si addition which lead to increases in retained austenite volume fraction. These are reviewed below.

(i) Silicon increases the activity of C, and hence promotes the ductility of ferrite (59,60). Wada and co-authors (61) agree with the first statement, adding that the extent of the increase is a function of both the silicon and carbon contents. The second effect is, however, an indirect one. As Si is a ferrite stabilizer, it helps reject C from solution, promoting segregation to the grain boundaries and sub-boundaries (62). In this way, it scavenges the transformed ferrite of other elements (62-67), and thus reduces the yield strength. This may also be linked to the effect of Si, a fast diffusing species in Fe, on the diffusivity of C (as implied from it's effect as a ferrite stabilizer). An increase in the latter would promote overaging, and therefore the elimination of the yield point.

(ii) It has been concluded that Si inhibits carbide, particularly Fe_3C , formation at the ferrite-martensite interface (59,60). On the other hand, *Imamura et al.* (62) consider that Si "cleans up" the ferrite in terms of C, promoting the segregation of C to the grain boundaries. However, according to other authors (64,66), Si delays the formation of pearlite and accelerates the ferrite reaction. The ejected C enriches the austenite (63-66,68-76) helping to form martensite as a final transformation product. A further alternative is that the Si addition simply increases the solubility of C in the ferrite, decreasing the driving force for carbide formation in this way.

Silicon is believed to enhance Mn segregation to austenite grain boundaries. Because of its inhibiting effect on Fe_3C precipitation, the carbon content in the untransformed austenite is increased and the sympathetic nucleation rate of α_b is hence, decreased ⁽⁶⁹⁾.

(iii) Si may contribute to solid solution strengthening.

(iv) Since it is a ferrite stabilizer, Si raises the transformation temperature (59,60,62-64,66,73,75,76). There is no disagreement about the high values of Ac_3 (and consequently Ae_3) found in steels with a high (> 1%) silicon content.

(v) Si accelerates the polygonal ferrite transformation in low carbon steels (59, 62, 64, 66, 73, 74, 76), and refines the austenite microstructure (59, 64, 75, 77). Figure 2.8 shows the findings of *Kato et al.* (59) which indicate that the steel with the highest Si concentration led to the smallest austenite grain size at all reheating temperatures.

2.4.2 Effect of C

According to *Kirkaldy et al.* ⁽⁷⁸⁾, C, Mn, Ni and Cr (< 8 wt.%) fall into the category of austenite stabilizers. Thus, they decrease the Ae_3 temperature, assuring a reduction in the driving force for transformation at any temperature.

There is unanimous agreement that C is the most powerful enhancer of the hardenability of austenite. The detrimental effect of C on the ductility is associated with the way in which the microstructure changes with increasing %C, as shown in Fig. 2.9⁽⁷²⁾. The restriction imposed on the growth of polygonal ferrite and the increase in the amount of "granular bainite" (a carbide-free massive bainitic structure) are the important drawbacks more associated with increasing the carbon concentration (63,72).



Fig. 2.8 Effects of chemical composition and austenitizing temperature on austenite grain size.



Fig. 2.9 Variation of microstructural constituents with carbon content of optimum Mn-Mo-Si-Cr as hot rolled dual phase steels.

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2.4.3 Effect of Mn

In addition to being a recognized austenite stabilizer, Mn is also reported to lower the activity coefficient of carbon $^{(61)}$, the decrease being proportional to its concentration. According to *Kato et al.* $^{(59)}$, electron probe microanalysis carried out on both the ferrite and martensite phases in an as-hot-rolled dual-phase steel indicated no partitioning of Mn, Si or Cr between the two phases. This was probably because there was insufficient time for partitioning of the substitutional solutes to occur during austenite decomposition. On the other hand, *Rigsbee* $^{(79)}$ found that both the local Mn and C concentrations were greater in the martensite/austenite particles than in the ferrite matrix. He explained this as being due to the formation of austenite on reheating in the regions where Mn is already present in above average concentrations. Since Mn is a relatively slow diffusing element in ferrite, the segregation produced during casting is likely to persist, even in the austenite, after the heat treatments involved in industrial practice (⁸⁰).

It is widely accepted that the Mn improves the hardenability of austenite and therefore has a marked effect on the mechanical properties. For example, as the manganese content is increased, the ultimate tensile strength is raised (59,68,70,81-83). However, some other very important microstructural effects are also associated with its addition. More specifically because it expands the austenite field and simultaneously depresses both the Ac_3 and Ac_1 lines, it reduces the width of the intercritical region (84).

2.5 Austenite-to-Ferrite Transformation

The formation of proeutectoid ferrite from austenite is diffusional ⁽⁸⁵⁾ and occurs by a nucleation and growth reaction, nucleating in a heterogeneous manner on the crystalline defects in the austenite. It is well known that the ferrite grain size decreases with an increasing ratio of the rates of nucleation to growth ⁽⁸⁶⁾.

When ferrite forms from austenite, it nucleates mainly at the austenite grain boundaries. When the supercooling is small, polygonal ferrite forms and many adjacent ferrite grains with similar orientations coalesce into one grain. However, one austenite grain usually gives rise to several ferrite grains. Thus, the smaller the austenite grain size the finer the resulting ferrite. When the cooling rate is fast (accelerated cooling), and supercooling is large, very fine ferrite is formed. However, when the supercooling is increased beyond a critical value, acicular or widmanstatten ferrite forms. In low alloy steels, the ferrite grain size can be reduced using one or all of the following: (i) addition of microalloying elements (87-91); (ii) decreasing the rolling temperature (87.89.91) and increasing the deformation strain (87,91); (iii) lowering the ferrite transformation temperature by either increasing the cooling rate (91-94) and/or moving the Ccurves in a TTT (temperature-timetransformation) diagram to a longer time (i.e. increasing the hardenability of steel) (91,95,96) An example of the influence of composition and rolling conditions on ferrite grain size is shown in Fig. 2.10 ⁽⁸⁶⁾.



Fig. 2.10 The effect of finishing temperature, Nb addition and total reduction on the ferrite grain size of a 0.17%C-1.6%Mn steel.

2.5.1 The Morphological Classification of Ferrite

Ferrite morphology can be classified into different categories, based on the Dubé (97-99) system, as follows.

2.5.1.1 Grain boundary allotriomorphs

The term allotriomorph means that the phase is crystalline with regard to atomic packing, but not in the outward form. In other words, the symmetry of its internal structure is not revealed by the morphology of the phase, implying that the limiting surfaces of the crystal are not regular ⁽⁹⁸⁾. Thus allotriomorphic ferrite, which nucleates at prior austenite grain boundaries, tends to grow along the austenite boundaries at a rate faster than in the direction normal to the boundary plane. Therefore, its shape is strongly influenced by the presence of the boundary and hence does not necessarily reflect its internal symmetry. Of course, allotriomorphic ferrite does not form only at austenite grain boundaries, but it invariably does so, presumably because these are energetically favourable nucleation sites. It should be mentioned that deformation bands and twins, when present, can also act as nucleation sites ...

2.5.1.2 Idiomorphic ferrite

The term, idiomorphic, implies that the phase concerned has faces corresponding to its crystalline form. In steels, idiomorphic ferrite is taken to be that which has a roughly

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equiaxed or polygonal morphology. Idiomorphic ferrite usually forms intragranularly ⁽⁹⁹⁾, presumably at inclusions or other heterogeneous nucleation sites.

2.5.1.3 Widmanstätten ferrite (sideplates, sawteeth and intragranular needles)

The formation of widmanstatten ferrite is accompanied by a change in the shape of the transformed region. The shape change due to a single wedge of widmanstatten ferrite consists of two adjacent and opposing invariant-plane strain (IPS) deformations. These IPS deformations each have large shear components (~0.4) and imply the existence of an atomic correspondence between the parent and product phases as far as the iron and substitutional solute atoms are concerned. Interstitial atoms, such as carbon, can diffuse during growth without affecting the shape change or the displacive character of the transformation. The co-operative growth of a pair of adjacent mutually-accommodating crystallographic variants allows the elastically accommodated strain energy accompanying plate formation to be rather small, of the order of 50 J/mole. This is consistent with the low undercoolings at which widmanstatten ferrite forms and with the wedge morphology which arises because the adjacent variants have slightly different habit planes. The shape change indicates that the widmanstatten ferrite/austenite interface is glissile and the plates therefore grow at a constant rate controlled by the diffusion of carbon in the austenite ahead of the plate tip. Finally, it is noted that, when widmanstatten ferrite nucleates from grain boundary allotriomorphs of ferrite, it is called a widmanstatten ferrite side-plate, but when it nucleates directly from the austenite boundaries, it is referred to as a widmanstatten ferrite primary side-plate (100).

2.5.1.4 Massive ferrite

This structure is not a fundamental morphology but results from the impingment of crystals of other morphologies and of grain growth within the aggregate. This leads to bulky, roughly equiaxed polycrystalline masses in the microstructure.

2.5.2 Ferrite Transformation in Si Containing Steels

As was mentioned previously, Si is a ferrite stabilizer and therefore its first effect is to shift the Ar_3 to higher temperatures. This can clearly be seen in Fig. 2.11 which illustrates that, for the same cooling rate, the higher the Si level, the higher the Ar_3 . Figure 2.12 shows the rate of ferrite nucleation in steels with different Si content, and shows that lowering the Si level lowers the nucleation rate (101).



Fig. 2.11 Effect of silicon content on the Ar_3 .

Fig. 2.12 Effect of silicon content on nucleation rate of ferrite.

2.5.3 The Effect of Nb and Nb(C,N) on the Ferrite Transformation

Generally, it has been considered that the onset of the austenite-to-proeutectoid ferrite transformation can be delayed with the addition of Nb and a maximum retardation occurs at some intermediate Nb content (102,103).

At low Nb supersaturations (<0.049%), part of the Nb may form fine matrix precipitates during cooling from the solutionizing temperature to the onset of proeutectoid ferrite formation; however, a large portion of the Nb remains in solution. According to the solute drag effect (104), Nb in solution, because of its affinity for C and large atomic size mismatch with respect to the matrix, should segregate to austenite grain boundaries, decreasing the activity of C in austenite. This means that Nb locks the carbon at austenite grain boundaries (locally increases the hardenability of austenite and hence retards ferrite formation).

At higher Nb supersaturations, the competing effect of increased coarsening rates of the Nb(C,N) precipitates occurs during cooling. The Nb(C,N) particles, because of their large lattice mismatch with respect to austenite, provide high energy interfaces conducive for nucleation of ferrite. The formation of large Nb(C,N) particles also locally reduces the C concentration, which assists ferrite formation and reduces the Nb concentration at the austenite grain boundaries, thus diminishing the strong solute drag effect.

2.6 Austenite-to-Bainite Transformation

The word bainite was coined in 1934 after *E. C. Bain* to describe the nonlamellar (*i.e.* nonpearlitic) aggregate of ferrite and carbide which forms in steel by the decomposition of austenite at temperatures above that of martensite formation, but below that of fine pearlite formation, through either isothermal transformation or continuous cooling (105). The well accepted differences in carbide distribution and ferrite morphology, between bainite formed at high and low temperatures, has led to the classification of upper and lower bainite (27), respectively.

Upper bainite forms at temperatures between 350°C and 550°C and is often characterized by a feathery structure. Upper bainitic ferrite laths are practically free of carbon (<0.03% C) and can nucleate coherently, in general, at the austenite grain boundaries. Consequently, with the growth or thickening of ferrite laths, the remaining austenite becomes enriched in carbon and finally reaches such a level that cementite begins to precipitate at the α (lath)/ γ boundaries (106,107). (According to some workers, enrichment of the remaining austenite is not directly relevant because the carbon concentration in austenite at the α/γ interface is not a function of time.) However, the amount of carbide precipitated is low. In this way, nucleation of bainitic ferrite and cementite is repeated several times in order to produce "sheaves" consisting of groupings of parallel intragranular ferrite subunits (108), with cementite precipitated along the bainitic ferrite/austenite boundaries. As the transformation temperature is decreased, or the amount of carbon is increased in the upper bainite range, the carbon content of the enriched austenite increases more quickly and the laths increasingly form adjacent to each other in parallel groups by increased rates of sympathetic nucleation. (Sympathetic nucleation may be defined as the nucleation of a phase, whose composition is different from that of the matrix, at an interphase boundary (109,110), e.g. ferrite nucleation at austenite-ferrite boundaries.)

The temperature of transformation, separating the two types of bainite, depends upon the carbon concentration ⁽¹¹¹⁾, as shown in Fig. 2.13. Lower bainite frequently forms as individual plates rather than lath colonies ⁽¹¹²⁾. Here, the carbide tends to precipitate as smaller particles, in parallel arrays, which appear as cross-striations, making an angle of about 55° with the longitudinal axis of the ferrite plate ⁽¹¹³⁾. The precipitates in lower bainite can be either ε -carbide ($Fe_{2.4}C$) or cementite (Fe_3C), depending on both the transformation temperature and the composition of steel.

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During isothermal transformation, bainite formation ceases well before the carbon content of the remaining austenite has attained the level represented by the extrapolated paraequilibrium Ae_3 and Ae_1 phase boundaries. (At high temperatures, where diffusion rates are reasonable, the substitutional alloying elements can redistribute during transformation if equilibrium demands such redistribution. In these circumstances. the transformation is said to occur under "orthoequilibrium" conditions. This compares with "paraequilibrium" transformation, in which the substitutional alloying elements are unable to partition during the time scale of the experiment, although carbon, which is a fast diffusing





interstitial element, redistributes between the phases and reaches equilibrium subject to this constraint.) In other words, the bainitic transformation becomes increasingly incomplete as the B_s temperature (bainite transformation start) is reached, finally being entirely absent at and above this temperature ⁽¹¹⁴⁾. The quiescent period between B_s and Ae_I is termed 'stasis' to signify the incomplete reaction phenomenon. The latter has been attributed to the interaction between Mn and Si ⁽¹¹⁵⁾.

2.6.1 Bainite Transformation in Si-Mn Steels

Bainite in steels is usually a nonlamellar aggregate of ferrite and carbide. However, it is now well known that, in the carbon steels containing about 2% Si, carbide free bainitic ferrite forms in the initial stage of transformation (26,27,116,117).

The *TTT* diagram of these steels consists of two C-curves. The upper C-curve is hardly affected by carbon content, whereas the lower C-curve markedly shifts to a longer time with an increase in carbon content. It has been confirmed that the upper C-curve corresponds to the proeutectoid ferrite and pearlite transformation and the lower C-curve to the bainite transformation (35,117). The bainite morphology appears to be in the form of

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upper bainite (ferrite laths) above 350°C. Below this temperature the carbide precipitation characteristics of bainitic ferrite changes to lower bainite.

2.7 The Influence of Si and Mn on the Kinetics of the Bainite Transformation

As has been mentioned, Mn reduces the activity of carbon in austenite. Therefore, the segregation of Mn to α/γ interfaces diminishes the driving force for, and hence the rate of, ferrite growth ⁽¹¹⁴⁾. This behaviour has been termed the solute drag-like effect (*SDLE*) ⁽¹⁰⁴⁾. Silicon, however, raises the activity of carbon in austenite and, thus, does not cause a *SDLE*. The ferrite growth rate is expected to be rapid, thus leading to a rapid rate of formation of bainite in *Fe-C-Si* alloys ⁽¹¹⁸⁾. But it is worth mentioning that, in a *TTT* diagram, the initiation of the bainite transformation moves to the right (longer times) either by increasing Mn concentration at constant Si concentration, or with increasing Si concentration can be related to effects on both the nucleation and growth rates of the bainitic ferrite ⁽¹¹⁸⁾. From the above discussions, it is apparent that, not only does the mechanism of the effect of Si and Mn in combination is different from that of Si and Mn alone (*i.e.* the effects of Si and Mn on bainite formation are not simply additive).

In order to rigorously explain the above behaviour, segregation of these solutes should be considered more precisely. According to *Enomoto et al.* (119), the segregation of the solutes produces a significant effect on nucleation kinetics through the attendant reduction in grain boundary energy. In view of the unsatisfactory knowledge of the austenite grain boundary structure and its influence on the segregation of solutes, use of the change in grain boundary energy is a convenient approach in determining the effect of segregation on grain boundary nucleation.

It is clear that the formation of nonequilibrium segregation depends upon the combination of vacancy-solute pair diffusion toward the grain boundary and the reverse diffusion of the solute ⁽¹²⁰⁾. Since the equilibrium concentration of vacancies increases with increasing temperature, the degree of nonequilibrium segregation to the prior γ grain boundaries should increase with increasing austenitizing temperature ⁽¹¹⁸⁾. According to the model of equilibrium segregation in multicomponent systems, formulated by *Guttmann*

and *McLean* ⁽¹²¹⁾, the energy changes associated with interactive segregation of interstitial (carbon) and substitutional (Si and Mn) solutes can be expressed as follows:

$$\Delta G_{1} = \Delta G_{1}^{0} + \alpha' (X_{2}^{B} - X_{2}^{M}) \qquad Eq. \ 2.1$$

$$\Delta G_2 = \Delta G_2^0 + \alpha' (X_1^B - X_1^M)$$
 Eq. 2.2

- where $\alpha' =$ the excess interaction coefficient between solutes 1 and 2,
- $\Delta G_1, \Delta G_2$ = the segregation energy of solutes 1 and 2, respectively,
- $\Delta G_1^0, \Delta G_2^0$ = the segregation energy of solutes 1 and 2, respectively, when they dissolve alone in the base element, and
- X = the concentration of solutes. The notations B, M, 1 and 2 express grain boundary, matrix, solute 1, and solute 2, respectively.

From these equations, it can be seen that an attractive C-Mn interaction, $\alpha'>0$, results in the enhancement of their respective levels of segregation. On the other hand, a repulsive C-Si interaction ⁽¹²²⁾, $\alpha'<0$, and preferential carbon segregation may repel Si from the boundary. From these considerations, it is expected that adding Si to ternary *Fe*-*C-Mn* alloys results in enhancement of segregation of Mn and C to the boundaries.

Mn has been observed to segregate to the prior austenite grain boundaries and the regions adjacent to them, in Mn containing Fe-C alloys ⁽¹¹⁸⁾. Higher Mn concentrations increase the segregation of Mn, thereby increasing the inhibiting effect on the nucleation of α_b (bainitic ferrite). Accordingly, it is evident that the bainite fraction formed within the austenite grains increases with increasing Mn concentration. It is known that the formation of ferrite is often initiated preferentially at austenite grain boundaries. However, as discussed by *Balluffi* ⁽¹²³⁾, not all the sites within the austenite grain boundaries are suitable for the nucleation of α_b . If half the appropriate sites become unsuitable because of the segregation of the solutes, the initiation curve of the *TTT* diagram will move a distance to the right which is twice as far as the nonsegregated one.

Once Si exerts a suppressing effect on the cementite precipitation, the formation of ferrite will lead to carbon enrichment of austenite adjacent to the ferrite plates. The higher carbon concentration of austenite lowers the driving force for the subsequent precipitation of ferrite plates, and the sympathetic nucleation rate of ferrite decreases ⁽¹²⁴⁾. In *Fe-C-Si* alloys, since Si increases the activity of carbon in austenite and produces an antisolute

drag-like effect on the growth of ferrite, Si does not decrease the overall kinetics of bainite reaction to any degree.

Bainitic ferrite formation is thought to result from the inhibition of carbide precipitation due to the presence of silicon. The prevention of carbide formation correspondingly leads to a high concentration of carbon in residual austenite. The presence of silicon is needed for the stabilization of austenite. Silicon is also known to inhibit cementite formation during the tempering reaction in steels. This is generally explained by the relative insolubility of silicon in cementite¹ requiring the diffusion controlled ejection of silicon at the transformation front, which, in turn results in a silicon concentration build-up during an early stage of growth. This locally increases the activity of carbon so that the carbon flux is reduced and further development of the cementite embryos is inhibited ⁽¹²⁵⁾.

2.8 Austenite to Martensite Transformation

The name martensite was first used in honor of the German metallurgist Adolf Martens who identified the very hard, platelike or acicular constituent produced in many steels rapidly quenched from the austenitic state. Generally, a martensitic transformation involves the coherent formation of one phase from another without change in composition by a diffusionless (absence of long range diffusion), homogenous lattice shear (126). Christian coined the term 'military transformation' for this type of reaction, in which an orderly and highly coordinated or disciplined atomic rearrangement occurs where the relative atomic positions do not change, but the individual atoms move by a fraction of an interatomic distance (127,128).

In ferrous alloys, there are three kinds of martensites with different crystal structures depending on alloying elements and compositions ⁽¹²⁹⁾, these being α' (BCC or

¹It can be assumed that the silicon content of the cementite is small compared with that of the matrix, causing a build up of silicon in front of advancing interface. Then, for precipitation to continue, silicon must diffuse from the interface to the matrix at the same time as carbon is diffusing in the opposite direction. Since the activity of carbon increases in the presence of silicon, there will be an interaction between the two diffusion processes. It seems that for low silicon concentrations, carbon diffusion is the controlling factor, but on increasing the silicon content control transfers to diffusion of the latter.

BCT) martensite, ε (HCP) martensite and FCT martensite. The most common martensite in ferrous alloys is α' formed in Fe-C, Fe-Ni alloys, etc. The ε martensite is formed in ferrous alloys with a low stacking fault energy of austenite, such as Fe-Cr-Ni and Fe-high Mn alloys. FCT martensite has been found only in Fe-Pd and Fe-Pt alloys.

The morphology of Fe-C martensite can be broadly classified into two types: (i) lath martensite with high dislocation density with or without twinned regions $(^{130})$ and (ii) plate martensite containing an internal substructure of twins with or without dislocations $(^{131})$. These morphologies depend upon the carbon content of the alloy. The lath martensite forms in low carbon and medium-carbon steels up to 0.6% C; mixed lath and plate martensite is found in steels with C levels between 0.6 and 1.0% C; and plate type martensite predominates above about 1.0% C.

2.8.1 Mechanically Induced Phase Transformations and Transformation Plasticity

Deformation can stimulate the kinetics of solid-state phase transformations through both the thermodynamic effect of applied stress and the production of new defects by plastic strain. The flow stress, in these cases, is influenced by both the *static hardening* contribution of the transformation product, and the *dynamic softening* effect of the transformation as a deformation mechanism ⁽¹³²⁾. These combined effects can distort the true stress-strain curve to an upward curving shape approximating an ideal exponential hardening behaviour, which imparts maximum stability of the macroscopic flow behaviour.

The macroscopic plastic strain accompanying phase transformations (e.g., austenite-to-martensitic) under stress is defined as transformation plasticity. The strain can arise from the transformation shape change (preferential formation of favored crystallographic variants) and/or plastic accommodation processes which occur around martensitic islands as they form (*i.e.* from biasing of accommodation slip, triggered by both the volume change and the transformation shear) (4,8,132-137). The large transformation plasticity accompanying martensitic transformation allows substantial alteration of mechanical behaviour under monotonic as well as cyclic loading conditions.

Transformation plasticity provides a significant departure from the classical view of structure/property relationships in materials. The traditional role of transformation kinetics is in the control of materials processing to achieve desired properties associated with the

structure of the transformation product. Via transformation plasticity, metastable materials can be designed to exploit the properties of the transformation itself ⁽¹³²⁾.

2.8.2 Deformation Induced Transformation

Since the kinetics of martensitic transformations are nucleation controlled, two modes of deformation induced transformation can be distinguished according to the origin of the nucleation sites which initiate transformation. Nucleation on the same initial nucleation sites which are triggered on cooling, but assisted thermodynamically by the applied stress, is defined as *stress assisted* nucleation. *Strain-induced* nucleation arises from new nucleation sites generated by plastic strain. The condition under which each mode of transformation dominates can be illustrated in a stress-temperature diagram [Fig. 2.14]. For an alloy exhibiting apparently athermal transformation kinetics, no stress is required for transformation at the M_s temperature (represented by point A in Fig. 2.14). At higher temperatures, the stress required for stress-assisted transformation on the same



Fig. 2.14 Schematic representation of the stress and strain induced regimes for mechanically induced transformation. Initial yielding is controlled below M_s^{σ} by transformation plasticity, and above M_s^{σ} by slip in parent phase.

nucleation sites follows the line ABC. At point C, the yield stress for slip in the parent phase is attained, defining a temperature M_s^{σ} which can be regarded as the highest temperature at which transformation can be induced by elastic stress. Owing to the effect

of transformation plasticity below M_s^{σ} , the observed macroscopic yield stress will follow the curve ABCD, such that the M_s^{σ} temperature can be identified by a reversal of the temperature dependence of the yield stress.

Above M_s^{σ} , plastic flow occurs before the stress for stress-assisted nucleation is reached, and the production of the new strain-induced nucleation sites contributes to the transformation kinetics. The stress at which martensite is first detected then follows the path CE. Due to the potency of the strain-induced nucleation sites, this curve can at first follow the curve CD for the initiation of parent-phase slip. M_d is the upper limit of temperature at which plastic deformation can initiate a martensite transformation.

In the vicinity of M_s^{σ} , a mixture of the two transformation modes operates, whereas, further away from M_s^{σ} , the behavior of each mode can be observed independently. Although the differing origins of the nucleation sites can give different macroscopic kinetic behavior, it is believed that the basic nature and mechanism of operation of these sites is the same in both cases.

2.8.3 The Effect of Austenite Strengthening on Martensitic Transformation

The substructure strengthening of prior austenite may be generated by plastic deformation (work hardening) (138), alloying and even transformation (139). Such defects and substructure apparently can encourage or suppress martensitic transformation. The former occurs at small strains (140-144), and at large strains, the austenite becomes more stable (138,143-147). (The magnitude of these small and large strains depends on the chemical composition. For example, for an *Fe-26%Ni-0.4%C* alloy, the M_s temperature was reported (148) to increase up to 10% reduction and, beyond that, to decrease.) Small strains may align the defects for cooperative martensite nucleation, while large strains cause work hardening of austenite, thus inhibiting the transformation by resisting shear (139,149-151). Other workers have identified a decrease in the M_s temperature with an increase in the austenite yield strength (*i.e.* the substructure) (151).

The origin of this dependence of M_s temperature upon prior austenite flow strength and dislocation substructure can be interpreted on the basis of either the *i*) driving force, *ii*) ease of nucleation or *iii*) ease of growth in the austenite-to-martensite transformation.

2.8.3.1 Driving force

Following the concepts of homogeneous nucleation, it was proposed (152) that the thermodynamic condition for the martensitic reaction to occur is:

$$G_{Chem}^{\gamma} - G_{Chem}^{\alpha'} \ge \Delta G_{Strain} + \Delta G_r + K.E.$$
 Eq. 2.3

where G_{Chem}^{γ} and $G_{Chem}^{\alpha'}$ are the chemical free energies of austenite (γ) and martensite (α') respectively, ΔG_{Strain} is the strain energy brought about by the volume change, ΔG_r is the increase in interfacial energy per mole of martensite product of size r and K.E. is the kinetic energy of the martensite plates. The latter term was added to show all the irreversible factors, such as heat, during the formation of martensite plates. Rewriting the above:

$$\Delta G_{Chem}^{\gamma \to \alpha'} + \Delta G_{Non-Chem}^{\gamma \to \alpha'} < 0 \qquad \qquad Eq. \ 2.4$$

where $\Delta G_{Chem}^{\gamma \to \alpha'} (= G_{Chem}^{\alpha'} - G_{Chem}^{\gamma})$ is the chemical free energy change associated with the formation of a unit volume of martensite and is negative below T_o (which is the metastable equilibrium temperature between austenite and martensite), and $\Delta G_{Non-Chem}^{\gamma \to \alpha'}$ is a non-chemical free energy change combining all the other terms in Eq. 2.3.

Considering these thermodynamic concepts, Koistinen and Marburger (153) showed that, in pure Fe-C alloys, the relationship between the extent of transformation at any temperature, T, and the transformation temperature, M_s , can be described by the following relationship (153):

$$(1-f) = \exp\left[-0.011\left(M_{s}-T\right)\right]$$
 Eq. 2.5

where f is the fraction transformed. This relationship was originally used to describe the extent of transformation as a function of decreasing temperature below a fixed M_s , the only driving force being due to net chemical free energy change. Subsequently, it has been shown that it is applicable when the M_s temperature of a given alloy is varied by work hardening the austenite. In addition, the slope of the volume fraction of austenite transformed to martensite vs temperature curve can be correlated to the M_s temperature (154). An example of this correlation, for a series of Cr and Ni alloy steels, is shown in Fig. 2.15. As can be seen in the top curve, where the M_s temperature was varied by changing the composition, the slope (volume fraction of transformed austenite per °C) changed with the M_s temperature. However, the slope did not vary with the M_s

temperature when the M_s was changed by prior deformation (138). On the other hand, it was also shown that the gradient proportional was directly to the temperature derivative of the driving force $(\partial \Delta G^{\gamma \to \alpha'} / \partial T).$ transformation for Consequently, the $\partial \Delta G^{\gamma \rightarrow \alpha'} / \partial T$ is neither function of the prior austenite а deformation nor of the resultant variation of the M_s temperature for a given alloy composition.



Fig. 2.15 Percent transformation per °C as a function of M_s temperature.

2.8.3.2 Nucleation

It is widely accepted that nucleation of martensite is mainly determined by certain structural heterogeneities existing in the austenite (151). Various proposals have been made as to the nature of these nucleation sites or embryos and their mode of propagation. Based on these theories, the martensitic transformation kinetics can be discussed in terms of the dislocation density of parent austenite. This has been done firstly by referring to the propagation (*i.e.* nucleation and growth) of embryos and secondly by considering the possible size and number of embryos.

Propagation of embryos. This may be considered in general terms without specifying a particular physical model. Referring back to Eq. 2.4, the $\Delta G_{Non-Chem}^{\gamma \to \alpha'}$ term, which is positive and acts as a restraining force for the transformation, emanates from the fact that the austenite is being exposed to both elastic and plastic deformation when an embryo propagates. Therefore, the lowering of the M_s temperature with an increase of austenite yield stress (due to dislocation density increase and dislocation configuration changes) can be interpreted as being due to an increase in the resistance to elastic and plastic deformation.

Embryo propagation may also be influenced by dislocation substructure of the parent austenite in a manner suggested by *Kaufman and Cohen* (155). They proposed that imperfections in the path of the interface may serve to reduce the close coupling between the austenite and martensite lattices, and also the interface mobility. The dislocation

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density may also affect the embryo shape and hence the restraining force opposing propagation.

Embryo size and number. The substructure in austenite can also affect the size and number of martensite nucleation sites. Based on the embryo model proposed by Cohen (155), the embryos in the austenite can be considered to originate from interaction between certain pairs of dislocations, generating regions geometrically similar to martensite. These may be viewed as being bounded by loops of dislocations, embryo growth occurring by the propagation of the interface made up of dislocations. In this model, the embryo was assumed to be an oblate spheroid in shape. A minimum value of $\Delta G_{Non-Chem}^{\gamma \to \alpha'}$, Eq. 2.4, for a given volume of martensite embryo can then be calculated, and it was shown that this decreases with increasing embryo size. The most probable embryo size at any temperature in the austenite range above T_o was calculated, assuming that the association of dislocations into embryo interfaces might lead to a reduction in free energy of the austenite. Embryo size, in terms of semi-thickness, c, and radius, r, is given by:

$$c^* = \frac{\frac{6\Gamma_s}{d} - 4\sigma}{5\Delta G_{Chem}^{\gamma \to \alpha'}} \qquad \qquad Eq. \ 2.6$$

$$\Gamma_s = \frac{Gb^2}{4\pi} \left[Ln \frac{r_1}{r_0} - 1 \right] \qquad \qquad Eq. \ 2.8$$

where Γ_s is strain energy per unit length of random dislocation, d is the spacing between screw dislocations; σ is the specific interfacial free energy of the austenite/martensite interface, A is an elastic constant, G is the shear modulus, b is the Burger's vector, r_0 is the radius of dislocation core and finally r_1 is one half the mean distance between dislocations. An increase in dislocation density should decrease Γ_s , via a decrease in r_1 , which would reduce the values of c and r, thereby lowering the M_s temperature.

Finally, *Kajiwara* in his recent model (156) considered plastic accommodation in austenite to be a very important step in the nucleation event. Based on this model, in order for a martensite plate to be brought into existence from a potential nucleation site, three kinds of deformation must take place; these are, the *"lattice deformation"* which generates a martensite lattice from austenite, the *"lattice invariant deformation"* which produces an

interface with no average distortion between the two phases (*i.e.*, an invariant habit plane), and the deformation which accommodates the shape strain of the martensite plate. The shape strain of nucleating martensite (such as the shape strain of martensite plates during growth) must be accommodated by plastic deformation in austenite. Therefore any factors impeding the plastic accommodation, such as work hardening and small austenite particle size (section 6.3.3), suppress the transformation.

2.8.3.3 Growth

Generally, the austenite to martensite transformation has been shown to require a non-chemical free energy change of approximately 300 cal/mole (157). The contribution of both the elastic strain and surface energies of small martensite embryos was estimated to be less than 100 cal/mole. The volume change accompanying the transformation is not accounted for by the lattice invariant strain. It requires considerable elastic or plastic deformation of the surrounding austenite during the transformation in order to accommodate this volume strain. The energy necessary for the deformation of surrounding austenite can represent a substantial part of the non-chemical part of the free energy required for transformation. For example, for the situation that 275 MPa is needed for the motion of a flat martensite-austenite interface, the free energy for deformation was estimated to be 180 cal/mole (138). Accordingly, the flow strength of austenite, which can be varied by work hardening, can determine the ease of martensite growth.

The most important conclusion of this section is that, regardless of the mechanism, prestraining (beyond a critical small strain) or work hardening of austenite increases the stability of austenite by lowering the M_s temperature.

CHAPTER 3

EXPERIMENTAL TECHNIQUES

3.1 Experimental Materials

For the purpose of investigating the effect of thermomechanical treatments (TMT) on the state (quantity and quality) of the retained austenite, a series of four Si-Mn TRIP steels was cast and hot rolled at BHP, Melbourne labs. The chemical compositions of these steels are shown in Table 3.1. For the first time Nb has been added to the Si-Mn TRIP steels (steel A) in order to enable the full execution of TMP (in particular pancaking). Steel B, which has the same chemical composition as steel A apart from Nb, was chosen as a reference. The other two steels, which have different levels of Si and C (*i.e.* the two elements which most strongly influence the state of the retained austenite), were selected to investigate the effects of these alloying elements on the behaviour of this class of TRIP steels.

Composition	С%	Si%	Mn%	Nb%	Ti %	Al%	N(ppm)
Steel A	0.22	1.55	1.55	0.035	-	0.028	20-40
Steel B	0.19	1.50	1.54	-	-	0.024	20-40
Steel C	0.145	1.55	1.50	-	-	0.027	20-40
Steel D	0.18	0.93	1.50	-	-	0.024	20-40

Table 3.1 Chemical composition of experimental steels

3.2 Mechanical Testing Equipment

All the mechanical experiments were carried out on a computerized materials testing system, adapted for hot compression/tension tests, [Fig. 3.1]. Basically, the equipment is composed of an MTS automated testing machine coupled with a radiant furnace and a tool geometry that allows for the quenching of specimens at any time.



Fig. 3.1 Schematic diagram of the compression testing equipment.

3.2.1 MTS Automated Testing System

The equipment used in the compression tests is composed of an MTS automated testing machine (Model 510 MTS), which consists of a frame rated for a maximum load of 25KN, a hydraulic power supply and closed loop servohydraulic and computerized outer loop systems. [The compression mode was used because it avoids heterogeneous flow due to necking, as would occur in the case of tension tests, so that relatively large strains (up to 0.9, depending on the temperature) could be applied.] A hydraulic actuator controlled by a servovalve generates the force and the linear displacement, which are measured by the load cell and *LVDT* (linear variable differential transformer), respectively. The outer control loop consists of a PDP-11 computer running under the RT-11 operating

system and interfaced with the servohydraulics through a 486 processor unit. The computerized outer loop system mainly performs the function of command generation, data acquisition and real time decision making. To control the mechanical tests, programs are written in MTS BASIC version 02.09. This version is similar to the usual BASIC language but with added features giving access to various MTS functions.

3.2.2 Compression Tools

Since all the hot compression tests are carried out at high temperatures, the compression anvils are heated together with the specimen in the chamber. Therefore, the alloy used for the anvils must withstand the combination of high temperatures and flow stresses required to perform a hot deformation process. Candidate materials require a combination of high strength, good creep resistance and high melting point. Such an alloy is TZM, a molybdenum-base alloy containing a dispersion of 0.5% Ti and 0.08% Zr oxides. This alloy fulfills the above mentioned requirements as well as having other key characteristics, such as the absence of a phase transformation in the solid state and high resistance to recrystallization or softening (158,159). Another important property is good machinability and, in this respect, TZM is similar to that of medium carbon alloy steel at a hardness of 30-35 HRc. At temperatures over 550°C TZM oxidizes very rapidly in air or oxidizing atmospheres, but it has a long life in high vacuums and is also inert in a pure argon atmosphere at all temperatures.

3.2.3 Furnace and Temperature Control

In order to attain high temperatures, a Research Incorporated radiant furnace, equipped with an 16 kW power supply, was utilized. The furnace is mounted on the columns of the MTS load frame and linked to a *Micristar* digital controller/programmer. The heat, generated by the four tungsten filament lamps, is reflected to the center of the furnace (where the specimen is located) by four mirror finished elliptical reflectors of aluminum, positioned symmetrically about the center. To minimize oxidation, the specimen and anvils were enclosed by a quartz tube sealed with O-rings in which a high purity argon atmosphere was maintained.

The temperature measurement was performed by a chromel-alumel (K-type) closed tip thermocouple in contact with the sample. In some tests, a second thermocouple was installed at different positions on the sample in order to confirm the temperature readings of the first one. This also indicated that the temperature difference between the two ends of the compression sample was within ± 3 °C.

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Quenching the samples was done by a lever through a hole which was made in the lower support between the inside wall of the quartz tube and the lower anvil. This passage was kept closed during testing and was only opened a few seconds before initiating a quench. At the desired time, the specimen was pushed off the anvil (by the lever), so that it fell down the quartz tube into the quenching medium. Using this system, it was possible to quench deformed specimens in about 1 sec.

3.3 Experimental Methods

3.3.1 Sample Preparation

The compression samples were machined from the as-received plates, with their longitudinal axes parallel to the rolling direction. The sample height was 11.4 mm and the height-to-diameter ratio was kept constant at 1.5. These dimensions were chosen in consideration of the load capacity of the MTS machine and load cell, as well as of the strain rate used. This aspect ratio also promotes homogeneous deformation (160,161), which is the first requirement in mechanical testing for the results to be reproducible and comparable to other tests. In previous work (162,163) such specimens were grooved at the top and bottom surfaces to retain a glass lubricant, which can help maintain uniform deformation during high temperature compression. However, in this work, thin (50 to 80 μm) sheets of mica were successfully used to reduce the friction between the smooth (without any grooves) faces of the specimen and the compression tools.

3.3.2 Constant Strain Rate Testing

All the compression tests were carried out under constant strain rate conditions. This is because materials are rate sensitive at high temperatures, and testing at constant cross-head speed makes it difficult to determine if metallurgical changes (*e.g.*, dynamic recrystallization, precipitation, transformation, etc.) are taking place. In a compression test, an increment of true strain in a specimen deformed from an initial height h is given by Eq. 3.1.

$$d\varepsilon = \frac{dh}{h} \qquad \qquad Eq. \ 3.1$$

By differentiating with respect to t, the true strain rate in compression is obtained (Eq. 3.2), where dh/dt is the rate of change of specimen height.

$$\dot{\varepsilon} = \left(-\frac{1}{h}\right) \frac{dh}{dt} \qquad \qquad Eq. \ 3.2$$

To run a constant true strain rate test, the sample must be deformed at a rate which is proportional to its instantaneous height (Eq. 3.3). By integrating Eq. 3.3 and setting $h=h_0$ at t=0, Eq. 3.4 is obtained.

$$\frac{dh}{dt} = -\dot{\varepsilon} h \qquad \qquad Eq. \ 3.3$$

$$h = h_0 \exp(-\dot{\varepsilon} t) \qquad \qquad Eq. \ 3.4$$

With a computer controlled system, true strain rate tests are produced by dividing the entire deformation time into *i* small intervals of Δt (Eq. 3.5). This equation was employed in the program listed in *Appendix I* to build an array of 50 intervals. Each point in the array corresponds to a position of the actuator, in such a way that Eq. 3.4 is followed in practical terms:

$$h_i = h_0 \exp(-\dot{\varepsilon} \cdot i \cdot \Delta t) \qquad \qquad Eq. \ 3.5$$

3.3.3 Thermal Cycles and Deformation Schedule

An austenitizing heat treatment was the first step of each separate test, primarily to ensure dissolution of the Nb(C,N) and AlN precipitates present in the initial microstructure, so that they can reprecipitate during later cooling and/or processing. Dissolution temperatures for precipitates were estimated from the formulas given by Leslie et al. (164) and Lakshmanan and Kirkaldy (165).

$$Log([%A1][%N]) = -\frac{6770}{T} + 1.03$$
 Eq. 3.6

$$Log([%Nb][%C]^{0.87}) = -\frac{7920}{T} + 3.4$$
 Eq. 3.7

where the alloying concentration is in wt% and the temperature is in Kelvin. The holding times were selected to produce about the same starting austenite grain size in all specimens.

Since this work dealt with the effects of different *TMP* parameters on the state of the retained austenite in Si-Mn TRIP steels, different deformation processes and thermal cycles were used. The thermal cycle typical of all the tests is shown in Fig. 3.2. As can be



Fig. 3.2 The schematic thermal cycle and deformation processing.

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seen, all hot deformation tests were initiated from a reference temperature (1050°C). After that different processes (such as changes in the temperature, strain rate, cooling rate, etc. in order to attain microstructures which were statically and dynamically recrystallized, pancaked, etc.) were followed. The thermal cycle and deformation schedule associated with each of these tests will be discussed separately later in the corresponding section.

3.4 Microstructural Investigation

The tested specimens were initially cut into two halves along the compression axis with an Isomet slow speed diamond saw operated with coolant, to protect the samples from overheating. One half was used for optical microscopy (OM), scanning electron microscopy (SEM) and x-ray diffraction (stage I, see below). The other half was sliced into several thin samples, 400 to $600\mu m$, (again by means of a very low speed diamond saw to prevent the deformation induced transformation of retained austenite to martensite) in order to make thin foils (stage II, see below) for transmission electron microscopy (TEM).

3.4.1 Sample Preparation

Stage I. After cutting, one half of each sample was mounted in bakelite and then ground using 240, 400, and 600 grit silicon carbide papers in sequence. The next step involved electropolishing the surface of the samples in percholoric-acetic acid (60%-40\%) electrolyte. The electropolishing was performed to remove at least a layer of $50\mu m$ thickness of the materials from the surface. In this way, not only were all the surface inhomogeneities eliminated, but the further transformation of the retained austenite to martensite by mechanical polishing was also prevented.

Stage II. The slices of 400 to $600\mu m$ thickness were ground to a thickness of 120 to 150 μm using 600 grit silicon carbide paper. Disks with a diameter of 3 mm were then cut from them using a punch, and subsequently thinned to perforation by a *Temupol-3*, jet-electropolisher. The various choices of jet-electropolisher thinning parameters (*e.g.*, voltage, photosensitivity of the perforation detector, flow rate of the electrolyte, and, the most important one, the choice of electrolyte) depends on the chemical composition, thermomechanical processing, and heat treatment history of the materials. In this work 3mm disks were thinned to perforation in a solution of 8% perchloric acid, 2% 2-butoxyethanol and 90% methane! at <-25°C and 40V potential, with a high photosensitivity and medium electrolyte flow rate. This solution and its related conditions

were found to thin ferrite, bainite, retained austenite and martensite at virtually identical rates.

3.4.2 Qualitative Metallography

3.4.2.1 Optical microscopy

All the polished samples were chemically etched using 2% nital. Using this, the distribution, size and morphology of ferrite (as a light component) can be distinguished from those of second phases (as darker components). A *Neophot 21* optical microscope was utilized for the microscopic studies.

Tint etching. In order to perform qualitative examinations on some special microstructural constituents (e.g., dispersion. overall morphology, etc. of retained austenite and bainite), an improved tint etching method was exploited. In this technique, selection of the temperature of etchants, etching time and temperature, and the humidity of the air used to dry the specimens are important factors in the attainment of а satisfactory delineation of different phases.

The etchant is a mixture of three ingredients the compositions and mixing ratio of which are given in Table 3.2. After 5-7 sec holding in freshly



An example of color-etched specimen, polygonal ferrite (F), upper bainite (UB), lower bainite (LB) and retained austenite or martensite (R).

mixed etchant, retained austenite and martensite are colored white, ferrite gray and bainite brown ⁽¹⁶⁶⁾, [Fig. 3.3].

Fig. 3.3

Solution I		Solution: II	Solution III		
$Na_2S_2O_3$, 5H ₂ O +	7 g	(NO ₂) ₃ C ₆ H ₂ OH	5 g	HNO3	4 ml
H ₂ O	100 ml	C ₂ H ₅ OH	100 ml	C ₂ H ₅ OH	96 ml
24 ml		24 ml		5 ml	

Table 3.2 The compositions and mixing ratios of etchant ingredients

3.4.2.2 Scanning electron microscopy (SEM)

Those parts of the microstructural investigation which needed higher resolution than optical microscopy, were carried out using a high resolution scanning electron microscope (*JEOL JSM-840A*). The samples were either etched by nital, to resolve very small microconstituents such as pearlite, or by using an etchant ⁽¹⁴⁾ of 2 gms (NH₄)₂S₂O₈, 2 ml HF, 50 ml CH₃COOH and 150 ml H₂O to distinguish the ferrite, bainite, retained austenite and martensite by differing degrees of etching ⁽¹⁶⁷⁾. The microscope was operated in secondary electron (SE) mode at 20 kV accelerating voltage for almost all the examinations.

3.4.2.3 Transmission electron microscopy (TEM)

TEM investigation on thin foils was performed using a Phillips CM20 transmission electron microscope at an accelerating voltage of 200 kV. Selected area diffraction patterns (SADP) were utilized to distinguish the ferrite, bainite, retained austenite and martensite. Morphological studies on microconstituents were performed by comparing bright and dark field images.

Energy dispersive x-ray spectroscopy (EDX). Qualitative and quantitative chemical microanalysis on the microstructural constituents in the thin foils was performed using a EDAX-9900, energy dispersive x-ray spectroscopy, incorporated in the TEM. The EDAX-9900 system detector was equipped with a beryllium window. In the case of quantitative analysis the Thin Section Quantitative Software Package, which has been developed for the microanalyst using a TEM, was applied. This software takes into account the unique nature of x-ray generation in a TEM due to a highly focused electron beam, large accelerating voltages (200 kV), and small specimen thickness. For quantification of the phases, the Ratio Model, which relates the ratio of measured intensities of any two elements to the ratio of their respective concentrations, was utilized.

3.4.3 Quantitative Metallography

3.4.3.1 Image analysis

Image analysis, which is a technique of processing an image in order to extract quantitative information, was utilized to quantify the size, quantity and distribution of some phases (e.g., ferrite). In this work the *LECO L2002* image analysing system was used. The quantity of the phase of interest was measured by area percent, which is the result of the ratio of total detected phase area to image frame area. In order to avoid the errors of image frame sampling, an automatic stage pattern facility was utilized (for each sample at least 50 fields were quantified). The grain size measurement was based on the average of ferets (straight line measurements made between tangents at various angles) in 8 different directions (0°, 22.5°, 45°, 67.5°, 90°, 112.5°, 135°, and 157.5°).

3.4.3.2 X-ray analysis

Many different techniques have been applied successfully to the measurement of retained austenite in martensitic and ferritic structures. Quantitative optical microscopy is generally satisfactory, as long as the austenite content is high (168-172). However, optical microscopy becomes unsatisfactory below about 15% in many steels due to etching and resolution difficulties. A new etchant containing CuSO₄ in a slightly acidified water solution has been developed (173) which is claimed to provide good resolution of austenite down to 2%, but it seems very sensitive to the polishing technique.

One of the most accurate retained austenite measurement techniques is x-ray diffraction. It is essentially absolute and independent of external calibration and can be used to easily measure low austenite contents (>2%) with excellent precision (174). The outstanding features of the method are its simplicity, the use of automatic equipment and the short times required for a single determination. The details of the retained austenite calculation are given in *Appendix II*.

Other worthwhile techniques employed in the past for retained austenite measurement are: electrical resistivity (168,175), magnetic permeability (169,172), dilatometry (170), and thermal analysis (171). All of these are, however, more amenable to measurement of the austenite present during transformation than that retained in the final microstructure.

Measurement of the retained austenite volume fraction and corresponding carbon content was carried out by step scanning in a *Phillips RSX11M* x-ray diffractometer system using Ni-filtered Cu K α radiation. The scanning speed was 0.2° per min and the apparatus was operated at 40kV accelerating voltage and 20mA beam current. The retained austenite content was calculated from the x-ray intensities. The effects of preferred orientation or texture were taken into account in accordance with the work of *Jatczak et al.* (174) by utilizing up to four austenite and three ferrite peaks and applying the following equation:

$$V_{RA} = \frac{\frac{1}{n_{RA}} \sum_{0}^{n} \left(\frac{I_{RA}^{hkl}}{R_{RA}^{hkl}} \right)}{\frac{1}{n_{RA}} \sum_{0}^{n} \left(\frac{I_{MA}^{hkl}}{R_{MA}^{hkl}} \right) + \frac{1}{n_{RA}} \sum_{0}^{n} \left(\frac{I_{RA}^{hkl}}{R_{RA}^{hkl}} \right)} \qquad Eq. 3.8$$

where I_{RA} and I_M are the integrated intensities of retained austenite and martensite, respectively, and R_{RA} and R_M are their related R values (*Appendix II*). It should be noted that the x-ray diffraction data were verified using two standard samples (containing 2% and 5% retained austenite) obtained from the National Bureau of Standards.

The x-ray diffraction data were also used to determine the carbon concentration in the retained austenite. The austenite lattice parameter extrapolation method [Fig. 3.4], together with the following empirical expression, was employed to calculate the carbon content ⁽¹⁷⁶⁾.

$$a_{o} = 3.578 + 0.044\% C$$
 Eq. 3.9



Fig. 3.4 Extrapolation of measured lattice parameters.

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3.5 Mechanical Properties Measurement

In order to reveal the effects of the different microstructures produced by TMP on the room temperature mechanical properties, a small specimen test technique was used, since the compression tested samples were too small for the usual mechanical property measurements. A number of different approaches have been taken in developing small specimen test techniques. These range from relatively straightforward scaling down of some standard specimen geometry (such as miniaturization of tensile and fatigue specimens) to developing a technique which can be used on a preexisting, small volume specimen. The analyses which accompany these tests are also varied, and range from empirical correlations between small and large specimen data, to finite element analyses of small specimen deformation. Table 3.3 provides a brief summary of the various specimen types, the volume of the corresponding specimen, the type of the analysis carried out, and the type of information obtained (177).

Test	Approximate Avg. Specimen Volume (mm ³)	Nature of Raw Data	Type of Properties	Nature of Analysis
Tersile	15	Load-Displacement	σ=f (ε)	Direct
Pressurized Tube	350	Diameter-Time	Creep Rate Rupture Time	Direct Direct
Microhardness	17	Land/Arma	Strength	Empirical
Ball	1.7	Load/Area	$\sigma = f(\varepsilon)$	Semiempirical
Punch				
Ball	1.7	Load-Displacement	Ductility, $\sigma = f(\varepsilon)$	Analytical Analytical Semicontical
Shear	1.7	Load-Displacement	Strength, Ductility	Empirical
Bend	10	Load-Displacement	Strength	Analytical
Fracture	370	Load-C:ack Growth	Fracture Toughness	Direct
Impact	200	Load-Time-Temp.	Energy-Absorbed Strength	Direct Analytical
Fatigue				
S-N FCP	1000 245	Load-Cycles Crack Length-Cycles	S-N Crack Growth-∆K	Direct Direct

Table 3.3 Small specimen tests and characteristics

From all of these techniques, the shear punch (178-180) test was selected to measure strength and ductility. The reasons for this selection were: (*i*) small average specimen volume and therefore high sensitivity to the microstructure, (*ii*) nature of raw data,
properties and analysis (*i.e.* they can be easily related to tensile properties), (*iii*) measurements are not as localized as those of microhardness tests and (*iv*) the production of 3mm *TEM* disc after each separate test. The details of the method and the method of calibrating the empirical equation relating the load and displacement to stress and elongation have been given in *Appendix III*.

3.6 Neutron Diffractometry

Lattice parameter variations during cooling from high temperatures (e.g., the solutionizing temperature) to a temperature below the Ar_3 were followed using a neutron diffractometer system (Dualspec Powder Diffractometer) at AECL (Atomic Energy of Canada, Limited), Chalk River laboratories. This was equipped with a vacuum furnace to enable beam scanning and therefore diffraction data collection at elevated temperatures. The furnace programming and data acquisition system are fully computerized. This implemented the accurate temperature control of the furnace during cooling and the rapid data collection at any time during scanning the neutron beam on the sample.

CHAPTER 4

METHOD DEVELOPMENT

Synopsis

In the processing of steel, the design of a heat treatment and/or thermomechanical processing schedule, to obtain a given microstructure after any type of austenite transformation (ferritic, pearlitic, bainitic and martensitic), is greatly facilitated by the knowledge of the no-recrystallizaton temperature (T_{nr}) , and the start (Ar_3) and finish (Ar_1) temperatures of the austenite-to-ferrite (plus acicular structure) transformation characteristics.

In the past, isothermal and continuous cooling tests have been used in the laboratory to create *TTT* (Temperature-Time-Transformation) and *CCT* (Continuous-Cooling-Transformation) diagrams, respectively, which have then been the source of transformation data. The problem with such information is that it is only truly applicable to one particular microstructure, usually one resulting from a simple reheating cycle in the austenite region. Furthermore, all the aforementioned critical temperatures are not addressed in these diagrams. Most industrial steel processing operations additionally involve several stages of high temperature deformation. Any changes in the design of the hot deformation schedule will lead to changes in the microstructure which emerges from the final pass. Such variations may include austenite grain size, degree of recrystallization,

level of pancaking and amount of precipitation. Clearly, standard *TTT* and *CCT* data generated from a specimen that has merely been reheated will not be precisely applicable to such diverse microstructures.

In order to account for the aforementioned (industrial) situation, a novel laboratory method for the determination of the transformation characteristics, based on continuous cooling deformation testing, has been developed. A major attraction of this test technique is that the specific microstructure, for which the transformation characteristics are required, can be generated by hot deformation, and then immediately evaluated by continuous cooling deformation. Such a technique is particularly useful in the light of the proliferation of run out table strategies to improve the final microstructure directly after hot rolling (accelerated cooling, direct quenching, etc.). To maximize the effectiveness of these approaches, the transformation behaviour of the microstructure which has been generated at *the final deformation stage* must be precisely known.

In this chapter, the basic continuous cooling deformation test technique and general methods of data analysis are illustrated, using results from several different grades of steel.

4.1 Introduction

The aim of thermomechanical processing (TMP) is to improve the mechanical properties of materials by combining deformation and heat treatment ⁽¹⁸¹⁾. To obtain the desired mechanical properties, *i.e.* microstructures, the critical temperatures of *TMP* must be identified ⁽¹⁸²⁾. The first of these temperatures is the "*no-recrystallization temperature*" or T_{nr} . This is the temperature below which the deformed austenite will not undergo recrystallization before the execution of the next pass, therefore allowing strain to accumulate in the microstructure from pass to pass. Laboratory techniques to characterise the recrystallization behaviour include compression testing ⁽¹⁸³⁾, laboratory scale hot rolling and multi-stage torsion testing ⁽¹⁸⁴⁾.

Another critical temperature is the start of the austenite-to-ferrite transformation (Ar_3) . The practical significance of the Ar_3 in *TMP* is rooted in the accumulation of strain between the Ar_3 and T_{nr} , leading to pancaked austenite, which subsequently transforms to fine ferrite. In addition, deformation in the intercritical region between the Ar_3 and Ar_1 (end of the austenite-to-ferrite transformation) will work harden the ferrite phase and

further pancake the remaining austenite. These effects lead to a microstructure of fine equiaxed ferrite and work hardened ferrite, respectively, both contributing to a higher room temperature strength (184).

The measurement of the Ar_3 has been performed by a variety of laboratory techniques, including thermal analysis ^(185,186), dilatometry ^(187,188), microstructural changes ⁽¹⁸⁹⁾ and texture intensity measurements ⁽¹⁹⁰⁾.

The use of torsional physical simulation to generate the three critical rolling temperatures, *i.e.* the T_{nr} , Ar_3 and Ar_1 , developed by *Boratto* and co-workers ⁽¹⁹¹⁾, was a major step towards examining the transformation characteristics of an austenite microstructure produced during industrial hot rolling. In the latter, the critical temperatures were evaluated from an analysis of the variation of the mean flow stress with temperature. However, the Ar_3 and Ar_1 generated by this technique is only valid for a schedule involving deformation stages *through* the austenite transformation temperature, into the two phase region. The majority of industrial schedules finish hot deformation *above* the Ar_3 . To address this problem, a continuous cooling, continuous deformation test was developed.

It should be noted that, with regard to industrial rolling schedules, the continuous cooling deformation test is intended to be used immediately after executing a physical simulation of the desired rolling schedule, so that the relevant microstructure can then be evaluated. Thus, in the ideal case, torsion testing (for example) would be used to generate the specific as-hot rolled austenite microstructure, and the transformation behaviour of this structure would then be evaluated by continuous cooling *torsion* testing. In the present work, in order to clearly illustrate the principles of the test, the steels have simply been reheated. These microstructures have then been subjected to continuous cooling *compression (CCC)* testing in order to examine their transformation behaviour. Compression testing is, in principle, more straightforward to analyze than torsion data, since deformation is inhomogenous in the latter. Nevertheless, the same concepts are applicable to torsion testing.

4.2 Experimental Procedures

In order to develop the test technique, and to validate the analysis of the CCC data, fourteen different steels, the compositions of which are given in Table 4.1, were investigated.

Composition	С%	Si%	Mn%	Nb%	Ti %	Al%	N(ppm)
Steel A	0.22	1.55	1.55	0.035	-	0.028	20-40
Steel B	0.19	1.50	1.54	_	-	0.024	20-40
_Steel C	0.145	1.55	1.50		-	0.027	20-40
Steel D	0.18	0.93	1.50	-	-	0.024	20-40
Steel E	0.21	1.10	1.50	-	-	0.027	20-40
Steel F	0.05	0.26	1.50	0.040	-	0.050	20-40
Steel G	0.061	0.20	1.20	0.039	0.021	0.032	20-40
Steel H	0.07	0.10	0.43	-	_	0.046	80-90
Steel I	0.078	0.35	1.39	-	-	0.016	75-85
Steel J	0.22	0.08	0.87	_	-	0.046	40-60
Steel K	0.45	0.29	0.85		-	0.044	60-80
Steel L	0.51	0.30	0.83	-	-	0.040	100-120
Steel M	0.89	0.24	0.81	-	-	0.034	50-70
Steel N	0.83	0.26	0.83	-	-	0.001	40-60

Table 4.1 Chemical composition of experimental steels

The steels were received as-hot rolled, from which specimens were machined. It should be noted that steels A, B, C, D and E, which contain high Si levels, are *TRIP* grades. Steels F to N were added essentially to obtain data from compositions of more conventional Si content. The grades chosen were expected to exhibit a range of microstructural variations during hot deformation.

To reveal the contraction behaviour of the compression anvils during CCC testing, and hence its effect on the stress-strain curves, a constant load was maintained on an alumina specimen subjected to continuous cooling over the range of test temperatures used in this work. Since alumina does not deform over this temperature range, any change in strain is basically indicative of anvil contraction behaviour. The resulting curve is shown in Fig. 4.1. As was expected, the anvil contraction was linear with temperature.



Fig. 4.1 The contraction behaviour of the Fig. 4.2 The heating and deformation schedule compression anvils in the test of CCC testing. temperature range.

Figure 4.2 shows the thermal cycle and deformation schedule of a CCC test. After solutionizing at T_1 , the temperature is reduced to T_2 in order to have a reference starting temperature for different tests on different grades (T_2 , for example, could be the finishing temperature of a torsional simulation of hot rolling). The specimen is continuously deformed, using a constant strain rate, as the temperature is decreased from T_2 to a temperature below the Ar_3 or Ar_1 ; this generates the basic CCC data. Such tests were performed for each of the above grades at a strain rate of 0.0003/sec., cooling from 1050° C to 550°C, with a cooling rate of 0.5°C/sec.

4.3 Expected Stress-Strain Behavior of CCC Tests

Referring to the general form of the constitutive equation (192) and the expression for the calculation of high temperature flow strength (193), when the temperature changes, the stress can be defined by the following equation:

$$\dot{\varepsilon} = A \sinh (\alpha \sigma)^{n'} \exp(\frac{-Q}{RT})$$
 Eq. 4.1

rearranging, gives:

$$\sigma = \frac{\sinh^{-1}(X)}{\alpha} \text{, where } X = \left[\frac{\left(\dot{\varepsilon} \exp(\frac{Q}{RT})\right)}{A}\right]^{1/n'} \qquad Eq. 4.2$$

Here σ and $\dot{\varepsilon}$ are stress and strain rate, respectively, R is the gas constant, and A, n' and α are effectively constant for a given composition, although they will probably change slightly with temperature.

The above relationship indicates that, in a *CCC* test, the stress will increase smoothly in response to the decreasing temperature [Fig. 4.3], providing the microstructure does not change. Therefore, if any deviation from this behaviour appears in an actual *CCC* curve, it may be related to a microstructural change. This is the basic idea behind the use of *CCC* testing to determine the austenite transformation characteristics.



Fig. 4.3 The expected stress-temperature (strain) behaviour of CCC tests.

4.4 Results and Discussion

4.4.1 Basic Stress Strain Curve of CCC Test

The CCC curves of some of the steels examined in this work are shown in Fig. 4.4. All except steel H, which is lean in solute atoms, follow almost the same pattern. This is illustrated in more detail in Figure 4.5, which shows the CCC curve of steel C and the superimposed linear regression of each portion of the curve between two deviations. As expected from the preceding analysis, the flow stress generally increases with decreasing temperature and increasing strain. However, the curves exhibit several deviations from the ideal behavior. To define the exact position of each deviation quantitatively, *i.e.* in terms of strain and, therefore, temperature, a linear regression was first performed on each



Fig. 4.4 The true stress-true strain CCC curves of grades A to F, (cooling rate and strain rate are 0.5° C/sec and 3×10^{-4} /sec, respectively).

portion of the curve between two deviations (which may be considered to be approximately linear), and all the data points were then processed to calculate a value, X_{new} , which is defined in the following manner ⁽¹⁹⁴⁾:



Fig. 4.5 The CCC true stress-temperature curve of steel C with superimposed linear regression lines.

$$X_{new} = Strain - \frac{Stress}{Regressed Slope}$$
 Eq. 4.3

Therefore, by plotting stress vs X_{new} , the deviation is revealed at the point when X_{new} deviates from a constant value close to zero, exaggerating the deviation in this way [Fig. 4.6].

In the following sections, it will be demonstrated that any specific deviation observed in Fig. 4.5 corresponds to a microstructural change. In order to help confirm these structure-deviation correlations, where possible, *CCC* tests were interrupted and quenched before and after each deviation, and optical metallography was performed to reveal the microstructure. The interval between test interruption and quench was a maximum of 1s.

4.4.2 Microstructural Changes in the Single Phase Austenite Region

In this section the microstructural changes in the parent austenite revealed by CCC testing will be discussed. Since these features control the structure, substructure, morphology and grain size of the parent austenite and, accordingly, the grain and subgrain structures of the subsequent austenite transformation products, it is important to identify them.



Fig. 4.6 The CCC true stress-true strain curve of steel A with superimposed differentiating method.

4.4.2.1 First deviation after yielding

The CCC curve appears to exhibit a hump at the first deviation. This kind of behaviour is characteristic of dynamic recrystallization (195). A frequent consequence of dynamic recrystallization is grain refinement or coarsening, with the size distribution becoming more inhomogeneous. In order to reveal the microstructure resulting from this deviation, one sample was quenched before starting the CCC test and another was interrupted and guenched at the first deviation [Fig. 4.5]. A salt bath held at a temperature in the lower range of two phase region (i.e., close to Ar_1) was used as the quench medium. In this way, ferrite would decorate prior austenite grain boundaries to reveal the austenite microstructure. The resulting austenite-plus-ferrite microstructure was then quenched into water to transform the remaining austenite to the martensite. This heat treatment was designed to reveal the features of the austenite at these interruptions, by nucleating ferrite at features characteristic of the austenite microstructure which existed at the point of cessation of loading. Although the subsequent microstructure is not directly related to the state of the austenite at the point of interruption, it is indirectly associated and can therefore be used to indicate the possible prior austenite microstructure. Moreover, such a microstructure is far easier to reveal metallographically than prior austenite grain boundaries quenched into the martensite structure. In other words, the directness of quenching the austenite at the point of interest has been sacrificed for a much



Fig. 4.7 The prior austenite grains of steel C; (a) at 1050°C, (b) at 980°C. (The prior austenite grain boundaries are decorated by ferrite.)

The prior austenite grains of steel C at 930°C. (The prior austenite grain boundaries are decorated by ferrite.)

clearer microstructure. It was anticipated that the latter would outweigh the indirect nature of the approach.

As can be seen [Fig. 4.7], there are some very small prior austenite grains (which have subsequently been transformed to martensite) in the microstructure of the sample interrupted and quenched after this deviation, which have formed during dynamic recrystallization. Figure 4.8 shows the microstructure of the sample interrupted and quenched before the second deviation. As is visible the prior austenite grains have grown, presumably by DRX or strain induced grain growth. The first deviation is very sensitive to the conditions of the *CCC* tests (*e.g.*, cooling rate, strain rate and starting microstructure), and its position in the *CCC* curves therefore depends on the steel composition and *CCC* conditions. Accordingly, this deviation would not necessarily appear in all cases.

Generally, metals which exhibit dynamic recrystallization are those that undergo a relatively low degree of dynamic recovery during high temperature deformation. Retardation of recovery is usually attributed to relatively large separations of partial dislocations, making climb, cross-slip and node detachment difficult. The low rate of dynamic recovery gives rise, in turn, to a dense dislocation substructure, which promotes nucleation of recrystallized grains. This latter effect occurs in FCC metals of low and moderate stacking fault energy (e.g., Ni, Co, Cu and austenitic Fe) (196-198). The capacity for dynamic recrystallization also depends on the ease of grain boundary migration, which increases as the purity of the metal is increased. In solid solution alloys, the addition of solute tends to diminish the ability of the metal to recover, thus increasing the tendency towards dynamic recrystallization. However, the solute may also hinder boundary migration, slowing the rate of dynamic recrystallization. The presence of dispersed second phase particles tends to stabilize the substructure and prevent or restrain grain boundary migration. Such inhibitation or retardation of dynamic recrystallization has been observed in austenite containing solute Nb and Nb(CN) ⁽¹⁹⁹⁾. The latter is consistent with the CCC results of steels A, F and G.

4.4.2.2 Second deviation

This deviation is delineated by a sudden increase of the slope of the curve, suggesting a significant decrease in the rate of dynamic restoration. To confirm this, a sample was interrupted and quenched into the salt bath from a temperature 30°C (~880°C) below the second deviation (this temperature was selected to be sure of attaining the corresponding microstructural changes). As can be seen in Fig. 4.9b, there is no change in the austenite grain size and morphology compared to the microstructure which was interrupted and quenched from 10°C (~930°C) above the second deviation (Fig. 4.8). This means that the observed grain growth, between the first and second deviation, did not continue below the second deviation. It should be noted that, since Nb exerts both solute drag and precipitation effects on the restoration stop processes, this deviation is more pronounced in steels A, F and G than the others. This is clearly illustrated in Fig. 4.9, which shows the microstructures of the samples interrupted and quenched from 10°C above and 30°C below the second deviation into the salt bath. Since the quenching process (cooling rate and holding time in the salt bath) was the same for all cases, then, as was the case of steel C, both the samples should undergo almost the same nucleation and growth rates during austenite-to-ferrite transformation. However, as can be seen in Fig. 4.9, the sample interrupted and quenched 30°C below the second deviation temperature has more ferrite. This is only possible, if some of the strain after the second deviation is retained and accumulated in the microstructure in the vicinity of the prior austenite grain boundaries. The latter can stimulate the rate of the austenite-to-ferrite transformation, *i.e.* increase the rate of ferrite formation.

In order to further confirm these conclusions, another CCC test was performed at a higher strain rate ($\dot{\epsilon}$ =0.001/sec.). This was anticipated to introduce more accumulated strain in the samples interrupted 10°C above and 30°C below the second deviation. If the second deviation was representative of restoration а retardation point (i.e. the recrystallization stop temperature) in the CCC tests, then there should be some differences in the ferrite nucleation sites. Tests were interrupted before and after the second deviation, and the specimens were then cooled $(0.5^{\circ}C/sec)$ to a given temperature in the two phase





region. The resulting austenite-plus-ferrite microstructure was then quenched into water to transform the remaining austenite to martensite.

The differences between the austenite plus ferrite microstructures before and after the second deviation in steel A are illustrated in Fig. 4.10. After the deviation, the microstructure shows the existence of deformation bands which were not present prior to the deviation. These deformation bands divide individual austenite grains into several parts. Such features may also be due to twin boundaries, which apparently can lose coherency and severely corrugate, leading to features similar to deformation bands (^{200,201}). These findings are in agreement with results in the literature (^{200,201}). All of the above phenomena increase the number of ferrite nucleation sites markedly (²⁰¹⁻²⁰³). The presence of this deformation structure (deformation and twin bands) indicates that strain has accumulated in the matrix, because of recrystallization retardation. It should be mentioned that this deviation was not observed for steels H and I. The most probable reason for this is the lower alloying level in these steels, as will be discussed in more detail later.



Fig. 4.10 The microstructures of steel A interrupted from CCC testing, (a) at 940°C and (b) at 880°C.

4.4.3 Flow Stress Changes in the Parent Austenite Between T_{nr} and Ar_3

In this section, the flow stress changes in the parent austenite revealed by CCC testing between the T_{nr} and Ar_3 will be discussed. The CCC results reveal a strengthening mechanism in addition to that originating from recrystallization retardation. The origin for this will be discussed in the following.

4.4.3.1 Third deviation

As is shown in Fig. 4.5, the third deviation after yielding, which was invariably observed in all steels tested (Table 4.1), is characterized by an increase in the rate of strengthening. By using *Andrew's* formula ⁽²⁰⁴⁾ as a first approximation for the evaluation of the Ae_3 , it appears that this deviation is in the proximity of the Ae_3 temperature (Table 4.2). Metallography of the samples interrupted from *CCC* testing and quenched 10

°C above and below this temperature revealed no microstructural changes. On the other hand, as can be seen in Fig. 4.11, changes in steel composition significantly alter the temperature of this deviation.

To further characterize the nature of this deviation, the effect of changes in thermomechanical processing (TMP) parameters was examined. Thus, a series of CCC tests with different cooling rates (0.25 to 1°C/sec),

 Table 4.2
 The temperature of the third deviation measured by different methods

Grade	Ae₃ (°C)					
<u> </u>	Calcul.	ссс	Neut Dif.			
Steel A	899	860±5	850±5			
Steel B	863	840±5	850±5			
Steel C	890	<u>855±5</u>	850±5			
Steel D	885	890±5	900±5			
Steel E	805	790±5	800±5			
Steel F	763	750±5	780±5			

austenite grain sizes (80µm to 250µm) and strain rates (0.0003 to 0.0012/sec) were



Fig. 4.11 Comparison of the points of the third deviation of two different steels.

performed [Fig. 4.12]. None of these changes altered the temperature of the third deviation significantly. Since such parameters essentially affect the *kinetics* of any transformation during TMP (or CCC testing), the negligible influence of these variables indicates that this deviation does not correspond to a conventional diffusional nucleation and growth phase transformation. Since the Ae_3 is affected by thermodynamic and not kinetic considerations, it is another indication that the third deviation is associated with this temperature.



Fig. 4.12 The effect of thermomechanical processing parameters (austenite grain size, cooling rate and strain rate) on the point of deviation for steel C.

In order to further confirm that this deviation is an indication of the attainment of the Ae_3 , a specimen of steel C was interrupted from cooling 10°C below its corresponding deviation temperature (850°C), isothermally held for one hour and then quenched into water. The microstructure at the end of this process is shown in Fig. 4.13. As can be seen, there is a trace of ferrite at the prior austenite grain boundaries, thus further demonstrating that this deviation apparently corresponds to the Ae_3 temperature.

Assuming that this third deviation is an indication of the Ae_3 , from these results, it may also be tentatively concluded that the flow stresses of stable and metastable austenite (*i.e.* in the temperature region between the Ae_3 and the Ar_1) differ. Possible reasons for flow stress changes include:

- 1. Variations in dislocation movement (glide, cross-slip, jogging and climbing) (162).
- 2. Differences in the *Peierls-Nabarro* force.
- 3. Changes in the strain induced precipitation rate.
- 4. Changes in solute segregation characteristics

All of the above mechanisms can be affected by, or can lead to, changes in crystallography and/or new phase formation. To detect such changes, a series of high temperature neutron diffraction experiments was performed. With neutron diffraction, any changes in the austenite lattice parameter and the formation of new phases could be detected.



Fig. 4.13 The microstructure of steel C after CCC interruption at 840°C, continued with isothermal holding.

4.4.3.2 Neutron diffraction

The variation of lattice parameter with temperature is shown in Fig. 4.14. As is well known, the position of atoms in a lattice, which determines the lattice constant, is dictated by two opposing factors: the repulsive force of neighbouring atoms and their thermal motion. The latter is very sensitive to temperature and decreases with decreasing



Fig. 4.14 Lattice parameter of austenite vs temperature; (a) curves of steel A and C, (b) curves of steel H, J and K. The indices indicate the specific *(hkl)* planes.

temperature. Therefore, theoretically, the value of austenite lattice parameter should decrease with decreasing temperature. This lattice contraction was expected to be linear in the austenite single phase region. However, as can be seen, the rate of lattice parameter shrinkage is not linear and becomes more pronounced below a temperature which is more or less identical with the third deviation in the corresponding *CCC* curve.

To examine the possibility of the occurrence of a new phase, the diffraction patterns at each temperature were examined. Figure 4.15 shows the intensity vs 2θ curves of steel A at six different temperatures. The statistical quality of these diffraction patterns is sufficient to detect minority phase fractions down to a volume fraction of 0.1%. The lack of any new peaks in the curves of any temperature between the Ae_3 and Ar_3 implies no large scale precipitation, (which of course could have increased the rate of strengthening). It is, however, likely that, in the case of steel A, small amounts of Nb(C,N) precipitates, which are not detected by these relatively rapid neutron diffraction tests, may be responsible for the increase in the rate of strengthening in the corresponding CCC curve. It can be argued that the depletion in austenite of carbon, in response to Nb(C,N) formation, coincides with the increase in the rate of austenite lattice parameter contraction. However, the change in the rate of austenite lattice parameter shrinkage is also seen in the other steels which contain no strong carbide forming elements.

Since the formation of ferrite leads to carbon rejection into the remaining austenite, an increase in the austenite lattice parameter corresponding to the appearance of ferrite is expected. This increase competes with the lattice contraction due to the decreasing temperature, leading to a plateau. This plateau, which is obtained after formation of a given amount of ferrite, is observed for all the steels.

There is a trace indication of the presence of ferrite formation immediately after the Ae_3 temperature in steel C [Fig. 4.16], in response to the relatively slow cooling rate during neutron diffraction tests. As explained above, the formation of ferrite should lead to a retardation in the rate of decrease of the austenite lattice parameter. However, the contraction behaviour of the lattice parameter still maintains the increased rate of decrease that was realised at the Ae_3 . This suggests that the appearance of ferrite is not related to the changes observed in the metastable region.



Fig. 4.15 Typical diffraction patterns of steel A at six different temperatures.



Fig. 4.16 Typical diffraction patterns of steel C at eight different temperatures.



Fig. 4.15 Typical diffraction patterns of steel A at six different temperatures.



Fig. 4.16 Typical diffraction patterns of steel C at eight different temperatures.

parent phase. Since, during deformation, the density of dislocations is much higher than other imperfections, it is logical to assume that the stabilization of carbon clusters is achieved mostly by dislocations. This, in turn, can lock some mobile dislocations and increase the intensity of mobile and sessile dislocation interactions which consequently lead to the higher rate of strengthening.

An alternative rationalization tentatively links the change in lattice parameter, which emanates from carbon clustering, to a change in the *Peierls-Ncbarro* force in the following qualitative manner. The metallic bond is usually described as an attractive force between an array of positive ions and an intervening 'gas' of free electrons. The bond between two atoms is due to a net attractive force which depends upon their separation (209). For a pair of atoms, a potential energy, V, can be assigned to them which is a function of the distance (interatomic distance, r) between them. Thus (210):

$$V = -\frac{C_1}{r^n} + \frac{C_2}{r^m}$$
 Eq. 4.4

where, following the usual convention for potential energy, the term arising from an attractive force is given a negative sign and that from a repulsive force a positive sign. In this expression, C_1 , C_2 , n and m are constants, and their actual values will depend upon the nature of the crystal bonding forces and on the crystal structure itself ⁽²¹⁰⁾.

The net force, F, between the atoms is given by:

$$F = -\frac{dV}{dr} = -\frac{nC_1}{r^{n+1}} + \frac{mC_2}{r^{m+1}} \qquad Eq. \ 4.5$$

and this force will be zero at some critical separation (the equilibrium separation) r_{o} , so that:

$$0 = -\frac{nC_1}{r_0^{m+1}} + \frac{mC_2}{r_0^{m+1}}$$
 Eq. 4.6

These equations are illustrated graphically for arbitrary values of n and m in Fig. 4.17; they are known as the *Condon-Morse* curves ⁽²¹⁰⁾. Knowing these relationships, the effect of atomic separation on *Young's* modulus can be defined ⁽²¹¹⁾. For a single atom in a crystal, during elastic deformation, the stress and the strain can be defined as: $\sigma \approx \Delta F / r^2$ and $\varepsilon \approx \Delta r / r_0$ (where Δr is the displacement of the atoms from their equilibrium positions and r_0 is the equilibrium interatomic distance). Young's modulus E is equal to σ / ε , and as the displacement is small during elastic deformation, $r \approx r_0$. Therefore:

With respect to the above equations and the equilibrium state:

$$\left(\frac{\partial F}{\partial r}\right)_{r=r_0} = \frac{n(n+1)C_1}{r_0^{n+2}} - \frac{m(m+1)C_2}{r_0^{m+2}} = \frac{nC_1(n-m)}{r_0^{n+2}} \qquad Eq. \ 4.8$$

and substituting into (Eq. 4.7) the Young's modulus in terms of r can be obtained:

$$E = \frac{nC_1(n-m)}{r_0^{n+3}}$$
 Eq. 4.9

This equation is only approximate because it relates to two atoms only, ignoring the influence of the other neighbouring atoms, but it illustrates the relationship between Young's modulus and the atomic parameters, particularly the interatomic spacing. As can be seen from the above equation, the value of Young's modulus, and, consequently, the atomic bond strength, increases with a decrease in r_{o} . The lattice constant decreases with the decrease in temperature, as was indicated by neutron diffraction in the



Fig. 4.17 The Condon-Morse diagram of force F as a function of interatomic spacing r.

case of the austenite lattice parameter. Thus, changes in the rate of lattice parameter variations below the Ae_3 temperature can result in associated changes in the Young's modulus variations towards higher values.

With this in mind, and assuming the *Peierls-Nabarro* force (τ_p) is a significant factor in the flow behaviour in a *CCC* test, it can be concluded that any changes in the *Young's* modulus will affect the flow behaviour. This can be clearly illustrated by referring to the *Peierls-Nabarro* equation ⁽²¹²⁾:

$$\tau_{p} = \frac{E}{(1-v^{2})} \exp\left(-\frac{2\pi a}{(1-v)b}\right) \qquad Eq. \ 4.10$$

where v is *Poisson's* ratio and a and b are the distances between slip planes and atoms in the slip direction, respectively. However, an approximate calculation using typical values shows that the changes in *Young's* modulus, based on the austenite lattice parameter changes, cannot be the only reason for austenite flow stress variations in the metastable region.

Finally, increases in internal stress and dislocation density, which are the inevitable result of the plastic constraint exerted by lattice parameter contraction, are other factors which may contribute to the observed increase in the flow stress. More work is necessary to rationalise the observed coincidence of this deviation in *CCC* curves with lattice parameter deviation in the metastable region.

4. Detection of the equilibrium austenite-to-ferrite transformation temperature

It has been demonstrated that both the CCC and neutron diffraction techniques are capable of detecting changes in the austenite phase prior to the actual formation of ferrite. These changes are likely due to the inhomogenous redistribution of carbon in solid solution. Thus, the onset of the changes in flow stress and lattice parameter do not exactly coincide with the thermodynamically defined Ae_3 because carbon diffusion still requires a However, the activation energy for carbon diffusion is relatively low. finite time. Furthermore, it is likely that carbon clustering involves a much lower free energy increase than that required for ferrite nuclei to form, because clustering would probably not involve the formation of an interface. It is therefore expected that the carbon clustering would be detectable at temperatures much closer to the Ae_3 . This explains why changes in thermomechanical processing (TMP) parameters, which would strongly affect the kinetics of a conventional nucleation and growth diffusional transformation, had little effect on the 'Ae₃' deviation in the CCC curves. It also accounts for the reasonably close correspondence between calculated Ae_3 temperatures and the temperatures coinciding with the onset of the flow and lattice parameter changes. As a practical approach to the determination of the Ae₃, the techniques used in this work are thus viable.

4.4.4 The Variation of Flow Stress During Austenite Transformation (213,214)

The following section deals with the flow stress variations during austenite-toferrite and -pearlite transformation.

4.4.4.1 The start of the austenite-to-ferrite transformation

As shown in Fig. 4.18 after initial yielding, there is a decrease in the slope of the stress-strain curve at $755\pm5^{\circ}$ C. The microstructures from tests interrupted and quenched 10°C above and below the first deviation are superimposed on this curve. In the former,



Fig. 4.18 The microstructures of grade A, above and below the start of austenite-to-ferrite transformation point.

the structure is entirely martensitic (*i.e.* purely austenitic just prior to quenching). In the latter, a small volume fraction (less than 5%) of polygonal ferrite can be seen at the prior austenite grain boundaries in the form of thin films. From this, it is clear that this deviation can be attributed to ferrite formation, *i.e.* it occurs at the austenite-to-ferrite transformation start temperature (Ar_3) .

4.4.4.2 Acicular ferrite formation

The deviation indicated in Fig. 4.19 is very clearly characterized by a negative slope in the stress-strain curve starting at $625\pm5^{\circ}$ C, *i.e.* the material has become very soft.



Fig. 4.19 The microstructures of grade A, above and below the transition temperature of ferrite formation morphology from polygonal to polygonal+acicular.

The corresponding metallography before and after this deviation reveals that the morphology of the ferrite being formed changes from a polygonal to a needle or acicular structure (intragranular fine Widmanstätten plates).

As is well known, small degrees of undercooling below the Ae_3 lead to ferrite nucleation on austenite grain boundaries, which grows in a 'blocky' manner to form what are known as grain boundary allotriomorphs, or polygonal ferrite ⁽²¹⁵⁾. At higher levels of undercooling, there is an increasing tendency for the ferrite to grow from the grain boundaries, or within the grains, as plates, which become finer with increasing undercooling ⁽²¹⁵⁾. The larger undercooling and higher mobility of curved interfaces correspond with a higher rate of transformation, through increased nucleation and growth rates, respectively. Therefore, the kinetics of acicular ferrite formation are faster than those of polygonal ferrite. Consequently, acicular ferrite formation will occur rapidly at this point. It is this increased transformation rate that is responsible for the drop in flow stress which signals this deviation.

In addition, the microstructure after the second deviation reveals that the boundaries of deformation bands and twins in the parent phase (*i.e.* austenite), have become preferential sites for ferrite nucleation, also contributing to a rapid and large degree of ferrite nucleation. These deformation bands and twins had been generated at earlier stages, and then subjected to subsequent work hardening, apparently increasing the interface energies in this way, until they became favourable ferrite nucleation sites. Such a hypothesis is in agreement with *Tanaka's* suggestion that, if recovery of the dislocation structure is allowed to occur within deformation bands, the potential for ferrite nucleation at these bands is greatly decreased (216).

4.4.4.3 The finish of the austenite-to-ferrite transformation

The deviation before the final one, at $590\pm5^{\circ}$ C for steel C [Fig. 4.5], is characterised by the change back to a positive slope in the stress-strain curve. From the very rapid increase in the rate of strengthening after the third deviation, it may be postulated that this signifies the end of the austenite-to-ferrite transformation or, for this particular cooling rate (0.5°C/s), the start of pearlite formation. Unfortunately, this was not clearly ascertained by observing the microstructures of the samples quenched from above and below the deviation point, because pearlite was observed in both cases. In the case of the specimen interrupted before the deviation, pearlite is observed because the remaining austenite passes through the pearlite region during quenching.

4.4.4.4 The start of pearlite spheroidization

The final deviation appears to coincide with a decrease in the rate of strengthening [Fig. 4.20]. In fact the material starts once again to become softer.



Fig. 4.20 The CCC curves of steels B, C, E, K and M in the lower range of test temperature. The superimposed linear regressions indicate the start of softening.

Theoretically, the only mechanism which can change the microstructure to a softer one, in this temperature range and to this extent, is the spheroidization of pearlite. Spheroidization of pearlite, which generally reduces strength and increases the ductility, was confirmed by metallography of the samples interrupted below the last deviation [Fig. 4.21].

4.4.4.5 Verification of CCC analysis

To further substantiate the findings of the CCC test, dilatometric tests were performed. The resulting length change vs temperature curve for steel A is given in Fig 4.22. Some of the deviations previously noted in the CCC test can also be observed in the dilatometry curve. The corresponding temperatures from the CCC test are also shown on this diagram. As can be seen, there is very good agreement between the two tests for the austenite-to-ferrite start temperature, and the formation of acicular ferrite temperature. In both cases, the CCC test registered slightly higher temperatures, possibly indicating a small effect of deformation. The temperature signifying the end of the austenite-to-ferrite transformation, (*i.e.* the occurrence of pearlite) is 20°C higher in the CCC case, perhaps due to a stronger effect of deformation on this transformation.



(a)



Fig. 4.21 The microstructures of; (a) and (b) steel C, above and below the corresponding spheroidization deviation, respectively, and (c) steel N below the spheroidization deviation.





Fig. 4.22 The length change vs temperature curve for steel A subjected to dilatometry test.

4.4.4.6 Effect of composition on CCC curves

Because the deviations of the flow curve are the result of the various stages in the transformation of austenite during cooling, it is to be expected that chemical composition will influence the position of the deviations. The results from the 14 grades of steel tested generally confirm this concept. However, apart from the anticipated changes in the temperature of these deviations, there are other significant variations in the flow curve behaviour. In fact, in this series of steels, two types of *CCC* curve were identified.

The first type of behaviour (designated *Class I*) is the one described above for steel A, and was observed in steels A to G and also J to N, as can be seen in Fig. 4.4. The deviation temperatures for this series of steels are listed in Table 4.3. Within *Class I* curves, there are obvious differences in terms of temperature of the deviations, as well as flow stress levels, which reflect the various compositional changes of these steels. For example, steels A and F contain Nb, and exhibit the highest flow stresses of this series (at lower temperatures). In addition, steel A has a higher Ar_3 compared to steel F, corresponding to a higher Si level, which not only stabilizes ferrite, but also increases the carbon activity in austenite. Both effects will elevate the Ar_3 . Finally, if the Ar_3 temperatures of steels B and C are compared, it is seen that the former has a lower value because of the higher carbon level in this grade. Nevertheless, aside from these variations in flow behaviour, the general shape of the curve is the same for all the steels in this series.

Grade	Ar ₃ (℃)		Fifth I	eviation (°C)	Ar ₁ (°C)		
	ссс	Dilatometry	ссс	Dilatometry	ссс	Dilatometry	
Steel A	755±5	745	625±5	615	590±5	570	
Steel B	750±5	-	650±5	-	630±5	-	
Steel C	775±5	765	645±5	640	635±5	615	
Steel D	725±5	720	640±5	635	615±5	585	
Steel E	730±5	-	650±5	-	625±5	-	
Steel F	720±5	-	600±5		585±5	-	
Steel G	765±5	•	660±5		640±5	-	

Table 4.3 The temperatures of the 4th, 5th and 6th deviations in Class I steels, measured by different methods

The second type of curve (Class II) [Fig. 4.23] is exhibited by steels H and I. There is a significant decrease in the slope of the true stress-true strain curve after initial It may be recalled that similar behaviour was observed in the previously vielding. mentioned series of steels, and was due to the formation of acicular ferrite. However, the temperatures at which the decreases in flow stress occur in steels H and I are at least 100° C higher than for the other series. Moreover, there is no softening behaviour, *i.e.* there is no transformation, prior to this flow stress drop. Thus, although the shape of the drops in flow stress of the two classes of curve are similar, the associated microstructural changes are different. Metallographic examination of quenched specimens before and after the flow stress drop in this second series of steels reveals that the deviation corresponds with the start of the austenite-to-polygonal ferrite transformation [Fig. 4.24]. Note that the quenched specimen (steel H) after the deviation has a very large volume fraction of ferrite. This is due to the very fast rate of transformation in this steel, which is implied by the rapid decrease in the flow stress associated with this deviation. This is confirmed by comparing this behaviour to that of steel I which shows a much smaller rate of decrease in flow stress, and a correspondingly smaller ferrite volume fraction on quenching after the deviation [Fig. 4.25].



Fig. 4.23 The true stress-true strain CCC curves of; (a) grade H (cooling rate and strain rate 0.5°C/sec and 3×10⁻⁴/sec, respectively) and (b) grade I (cooling rate and strain rate 1°C/sec and 3×10⁻⁴/sec, respectively)



Fig. 4.24 The microstructures of grade H before and after the start of austenite-to-ferrite transformation. Notice that the bainitic type morphology in the microstructure of the specimen quenched from above the Ar_3 is because of the low carbon level in this grade.

In steel H, the second deviation after this flow stress drop was found to be related to the austenite-to-ferrite transformation finish, *i.e.* the start of pearlite formation, the latter leading to a significant increase in the flow stress [Fig. 4.26], as was observed in *Class I* behaviour after pearlite formation. In the case of this steel (steel H), the start of pearlite formation begins after a short plateau [Fig. 4.23]. This plateau does not correspond to any fundamental change in microstructure. It merely illustrates a specific condition where the rate of ferrite formation, along with the degree of recovery in the previously formed ferrite, is enough to compensate for the increase in flow stress resulting from strain



Fig. 4.25 The start of austenite-to-ferrite transformation in steel I ⁽²¹⁷⁾.

hardening and the decreasing temperature. At still lower temperatures, this 'steady state' condition disappears and the slope of the true stress-true strain curve starts to rise, *i.e.* the slope of the curve becomes positive once the austenite begins to transform to the pearlitic structure [Fig. 4.23].

In both steels H and I, since this second deviation signifies the end of the austeniteto-ferrite transformation, no deviation relating to the onset of acicular ferrite is seen. Table 4.4 lists the temperatures of the deviations in this series of steels.



*CCT diagram

Grade		4r ₃ (°C)	Ar ₁ (°C)		
	CCC Other Methods		ссс	Other Methods	
Steel H	845±5	850*	775±5		
Steel I	730±5 735**		700±5	-	

**DTA testing (217)

Table 4.4The temperatures of Ar_3 and Ar_1 deviations in Class II steels, measured
by different methods

STRESS (MPa)				AL A			XX
30 -						<u>Т</u> 75µ	
	SEM		37.5um				
							•.
0 –	750	800]	850 TEMPERA	900 FURE (°C	950	1000	

Fig. 4.26 The start of pearlite formation in steel H.
4.4.4.7 Discussion

Metallographic evidence indicates that the CCC test is sensitive enough to reveal many aspects of the austenite transformation during cooling. Dilatometric tests have demonstrated that there is only a small effect of the strain applied during the CCC test on the various transformations. The CCD (continuous cooling deformation) test is thus a powerful tool to determine the transformation behaviour of microstructures generated by hot deformation.

Out of the 14 steels tested, two types of CCC curve (Class I and Class II) have been identified. In terms of chemical composition, the basic difference between these two classes is the level of alloying. Class II steels are plain C-Mn steels, whereas Class I steels are either highly alloyed with Mn and Si, and/or contain Nb and Ti as a microalloying addition. Thus, the differences in flow curve behaviour can be attributed to chemical composition effects, as will be discussed in the following sections.

1. The austenite-to-ferrite transformation start temperature

Ferrite has a lower flow stress than austenite at the same temperature because of the increased dynamic restoration process in the ferrite, which is, in turn, caused by the higher stacking fault energy and diffusion coefficient of solute atoms ⁽²¹⁸⁾. Both classes of flow curve thus respond to the formation of this softer phase in the appropriate manner, but to different extents. Class I steels exhibit a decrease in the rate of flow stress increase with decreasing temperature at the Ar_3 , whereas Class II steels reveal a large drop in the flow stress. Such a difference can be rationalised on the basis of the kinetics of the transformation, the Class I steels exhibiting the slower rate.

As is well known, the addition of solute elements can greatly decrease the nucleation and growth kinetics of polygonal ferrite (219,220). Alloying elements affect nucleation by segregating to austenite grain boundaries, decreasing their energy and, consequently, their potency as nucleation sites. In addition, by developing a solute drag force, they also decrease the mobility of the ferrite boundaries. Therefore, in all cases, the absence of strong solute drag elements in *Class II* steels appears to be the reason for these differences in behavior. The relevant compositional differences are the presence of higher levels of Si, C, and Mn in *Class I* steels. It is further anticipated that the addition of an element with strong solute drag properties, *e.g.* Nb, will lead to a steel with *Class I* type behavior, even in the absence of higher Si, C and Mn levels. As a result, it can be

concluded that the transformation rate in *Class I* steels is sluggish, because of the relatively high amounts of alloying elements.

By contrast, the behavior in *Class II* steels is dominated by the dynamic restoration of ferrite. This hypothesis is further strengthened by noting that the drop in stress level is more significant in grade H than grade I, which coincides with a higher level of Mn in the latter, leading to a larger solute drag effect. In general, for grades with the same Ar_3 (irrespective of whether they belong to *Class I* or *Class II*), the steels with the lower solute element level will exhibit a larger decrease in flow stress or decrease in the rate of flow stress increase.

2. Appearance of the acicular ferrite deviation in Class I steels

The tendency towards acicular ferrite formation increases with increasing undercooling. The appearance of acicular ferrite in *Class I* steels may then be attributed to either a delay in the austenite-to-polygonal ferrite transformation, and/or a decrease in the pearlite start temperatures. From the previous discussion, an early completion of the austenite-to-ferrite transformation is anticipated in *Class II* steels, thus preventing large undercooling, therefore leading to an absence of acicular ferrite.

3. Pearlite formation

The other, more significant difference between the aforementioned two classes is the temperature of pearlite formation, which, in *Class I*, occurs at lower temperatures. The addition of small amounts of alloying element, such as Mn, Ni, Cr, Mo, W, Ti and Nb, significantly retards the pearlite reaction, due to the change in the Ae_1 temperature and the slow diffusion of alloying elements. It is well known that austenite stabilizers, such as Mn, Ni and Nb, depress the Ae_1 temperature and, therefore, are usually expected to slow down the pearlite reaction ⁽²²¹⁾. (Note that, although Nb is a ferrite stabilizer, additions of up to 0.10 to 0.20 percent lower the Ae_3 temperature ⁽²²²⁾ and consequently expand the austenite loop.) Carbide forming elements (which are also ferrite stabilizers) such as Si, Mo, Co, W, Cr, and Ti, increase the Ae_1 temperature, but they slow down the transformation rate, and therefore behave in the same manner as the austenite stabilizers (*e.g.* compare the pearlite deviation of *Class I* steels F and G, which are without and with Ti, respectively [Table 4.3]).

4. Pearlite spheroidization

The large ferrite-cementite interfacial area in the lamellar structure of pearlite suggests, at first sight, that a structure with a smaller interfacial area, such as cementite spheres in a ferritic matrix, would be much more stable. In fact, the lamellae do spheroidize as expected, but the kinetics of pearlite spheroidization, even at temperatures close to the Ae_1 temperature, is extremely slow. The probable reason for that is as follows. The interface between cementite and ferrite in pearlite is a low energy interface, therefore the driving force to reduce the interfacial area is probably small ⁽²²³⁾.

Table 4.5 shows the deviation temperatures related to the start of pearlite spheroidization. This table shows that the corresponding deviation temperature varies

with the steel composition. It appears, however, that the variation of carbon content above a given level (e.g., 0.4%), where the other species are almost the same, has roughly no effect on the temperature of cementite spheroidization (compare steels K to M). The latter is consistent with the findings of other workers, where it was shown that the presence of any proeutectoid constituent does not affect the rate of spheroidization; that is, carbon content has no effect (223).

An increase in Si level for a given carbon content, shifts the deviation to higher temperatures, (consider steels B and D). On the other hand, a decrease in the

Table 4.5The temperatures of Ar_1 and
spheroidization deviations in Class I
steels

Grade	Ar ₁	Spheroidization (°C)		
Steel A	590±5	540±5		
Steel B	630±5	590±5		
Steel C	635±5	565±5		
Steel D	615±5	550±5		
Steel E	625±5	575±5		
Steel J	650±5	580±5		
Steel K	650±5	615±5		
Steel L	650±5	615±5		
Steel M	655 <u>±</u> 5	615±5		
Steel N	660±5	615±5		

carbon level, for a constant Si content, reduces the start temperature of pearlite spheroidization (compare steels B and C). It is worth emphasizing that the spheroidization temperature of steel A (Nb bearing TRIP steel) among the other steels is the lowest one. There is no literature available concerning the effect of Nb on the kinetics of cementite spheroidization, but this can be similar to its effects on the pearlite formation temperature (*section 4.4.4.7*). In another words, the lower the pearlite formation temperature, the lower the spheroidization start temperature.

Chapter 4 METHOD DEVELOPMENT

The time interval between the start of pearlite formation and that of spheroidization, as calculated from their related CCC curves, [Fig. 4.20], is about two minutes. Usually, spheroidization requires several hours. Clearly, the rate of pearlite spheroidization has been increased by applying concurrent straining, which is in good agreement with previous investigations (224-228). For example, Robbins et al. (225) showed that, at an effective strain rate of 2.4/sec, full spheroidization occurred in a eutectoid steel in less than 3 minutes at 700°C. The reasons for this accelerated spheroidization can be explained as follows. In general, the overall process of spheroidization involves the 'break-up' of lamellae into fragments and the rounding off and growth of these fragments by coarsening processes (224). All the stages are controlled by an effective diffusion coefficient for carbon, which arises from a volume constraint and requires concurrent diffusion of vacancies (224). Therefore, the strain induced spheroidization may be attributed to the formation of lattice vacancies and carbon vacancy complexes (224) during plastic deformation. These point defects, existing in excess of the thermal equilibrium level, will accelerate the mobility of atoms and contribute to a more rapid rate of spheroidization. Another possible contribution for accelerated spheroidization may be due to the presence of dislocations and grain boundaries created by concurrent straining; such lineal and planar imperfections could act as short circuiting paths for diffusion of carbon and iron (225).

The spheroidization or softening behaviour was not observed for *Class II* steels. In contrast, there is an increase in the rate of work hardening at far below pearlite formation temperature instead of softening, [Fig. 4.27]. The microstructural examination of steel H after the last deviation revealed the presence of a second phase (carbide) at the ferrite grain boundaries, [Fig. 4.28]. The latter can be explained as follows. It appears that the same procedure of spheroidization occurred in this case but the volume fraction of spheroidized pearlite was not enough to decrease the rate of strengthening during cooling. This early spheroidization along with concurrent straining led to the growth of carbide spheroids until a continuous second phase was formed. This subsequently grew through the ferrite grain boundaries by the concurrent straining during *CCC* testing. This growth may be attributed to the higher stress concentration in the ferrite grains than the pearlite colonies in this steel.



Fig. 4.27 The CCC curves of steels H and I in the lower range of test temperature. The superimposed linear regressions indicate the start of higher rate of work hardening.



Fig. 4.28 The back-scattered electron micrographs of steel H below the last deviation; (a) lower and (b) higher magnification.

CHAPTER 5

RESULTS

This investigation concerns the state of the retained austenite in Si-Mn bainitic TRIP steels after thermomechanical processing (TMP). As noted in the literature survey, no extensive work in this area has been performed. Thus, the effects of all the important TMP parameters should be taken into consideration, systematically. However, it is difficult to separate the effects of each parameter, since a change in one parameter frequently affects another. Nevertheless, the experimental thermal and deformation schedules were each designed to highlight the effect of specific TMP parameters.

5.1 Recrystallized Austenite

As was mentioned in *Chapter 3*, the first stage of thermomechanical processing is controlled rolling in the austenite region above the recrystallization stop temperature. The temperature in this region is high enough to enable the austenite to be quickly recovered and recrystallized. Recovery and recrystallization have usually been considered as the mechanisms of restoration through which worked metal returns, partially or completely, to its undeformed condition. These mechanisms generally operate when a metal is annealed at a high homologous temperature for a period of time. Annealing is usually carried out in

the absence of stress or strain, in which case the recovery and recrystallization that occurs is termed *static*.

Recovery and recrystallization can also take place under stress and during concurrent straining, *i.e.* during high temperature deformation. In this case, they are termed *dynamic recovery* and *dynamic recrystallization*. The main austenite features which can be affected by recrystallization processes are the austenite grain size and substructure. Therefore, each of these should be taken into consideration and examined separately.

Figure 5.1 shows the thermal and deformation schedules which were applied to generate static and dynamic recrystallization. As can be seen, after solutionizing, double hit compression at typical industrial TMP temperatures was performed. The first deformation recrystallize was to the higher temperature structure to a finer grain size (in this way obtaining the same initial condition for all the tests), and the second was to obtain a statically or dynamically recrystallized austenite. The specimen was then cooled down to a temperature below the Ar_3 , held



Fig. 5.1 Schematic diagram of heat and deformation schedule.

isothermally for different periods of time in order to form various amounts of ferrite with a given morphology (*i.e.*, polygonal). This was followed by a salt bath quench, where the specimen was held isothermally for a predetermined duration at a temperature at which the austenite would transform to bainite. The specimen was then air cooled to room temperature. Two examples of true stress-true strain behaviour due to double hit compression of steel B are shown in Fig. 5.2. The softening after the first deformation, in both cases, is characteristic of static recrystallization. The hump in the curve of the slower strain rate test is an indication of dynamic recrystallization. (No-load times between hits of the tests performed at 1050°C and 1000°C were 20 sec and 2 min., respectively.)



Fig. 5.2 Typical double-hit compression true stress-true strain curves of steel B.

5.1.1 Static Recrystallization

The austenite microstructure after static recrystallization is close to the equilibrium state. This, of course, includes the substructure, which is close to the lowest dislocation density. Therefore, the effects of austenite grain size and steel composition, can be investigated through structures generated by applying static recrystallization.

5.1.1.1 The effect of composition

In order to investigate the effect of the chemical composition variations on the state of the retained austenite, two compressive deformations ($\varepsilon_1 = \varepsilon_2 = 0.3$ and $\dot{\varepsilon}_1 = \dot{\varepsilon}_2 = 3 \times 10^{-4} \text{ sec}^{-1}$) at 1050°C were applied to the specimens, which were then subjected to the same pattern of heat treatment as shown in Fig. 5.1. Here, the temperature of isothermal holding in the two phase region was 650°C, and the isothermal bainitic treatment of 400°C for 2 min. was selected based on the results of other workers (28,34,44). The microstructure of steel A after this process, which is typical of all the steels examined, is shown in Fig. 5.3. The white area is the grain boundary allotriomorph ferrite. The rest is a combination of bainite, retained austenite and, probably, martensite. No pearlite was identified in the microstructures at the end of this process.

Figure 5.4 shows the variation of the retained austenite and its corresponding carbon concentration in the different grades of TRIP steels. The austenite grain size (40 ± 5)

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 μm) and ferrite quantity (25±2.5%) were kept approximately constant in all cases. As can be seen, the retained austenite volume fraction (V_{RA}) was highest in steel A. The V_{RA} differences between the other grades are not as significant and can be attributed to the differences in their carbon and silicon levels. Note that, although steels A and B differ in Nb only, there is an almost 30% increase in retained austenite in the Nb containing steel A. It appears that the effect of the other alloying elements, *i.e.*, Si and C, on the V_{RA} is not as important as Nb. The carbon contents of the retained austenite in all other cases are roughly the same.



Fig. 5.3 Typical microstructure of steel A after double-hit compression, and 3 min holding at 650°C, followed by quenching into the salt bath (400°C).



Fig. 5.4 Variation of (a) the retained austenite volume fraction, and (b) carbon concentration of the retained austenite in different grades.

The dispersion of retained austenite was revealed using the previously mentioned color metallography technique, [Fig. 5.5]. As can be seen, there are several sites (pools) where retained austenite is present. For example, in this case, retained austenite is enclosed by; (i) polygonal ferrite, (ii) bainite packets and (iii) upper and lower bainite platelets. Here they are mostly encompassed in upper bainite. The various retained austenite morphologies and their corresponding compositions will be discussed later in more detail.

Figure 5.6 compares the mechanical properties of steels A and B. The higher amount of retained austenite



Fig. 5.5 Color-etched microstructure of steel A.

in steel A apparently increased the total elongation. The UTS is higher in steel A than B, because of the presence of Nb (in solution or precipitated), as will be discussed in more detail later.



Fig. 5.6 Mechanical properties of steel A and B, (T-EL=total elongation, UTS=ultimate tensile strength).

5.1.1.2 The effect of austenite grain size

As is well known, the subsequent transformation kinetics of the austenite can be affected significantly by the austenite grain size. As reported by other workers ⁽²²⁹⁻²³³⁾, the effect of grain size on transformation products differs from one transformation to another. Therefore it is necessary to carefully examine the effect of austenite grain size on the state of the retained austenite.

The amount of strain is an important variable in dictating the statically recrystallized austenite grain size, with larger initial austenite grains leading to larger recrystallized grain sizes $(^{234})$. If the effect of each variable is considered to be independent, and is represented in a simple power law form, for strains less than a critical value (ϵ^*) (this critical value is a function of peak strain for dynamic recrystallization), the following relationship can be utilized to calculate the size of austenite after static recrystallization (235).

$$d_{rex} = 0.5 \times d_o^{0.67} \times \varepsilon^{-1} \qquad (\varepsilon \le \varepsilon^{\uparrow}) \qquad Eq. 5.1$$

where d_o and ε are the austenite grain size before deformation and the degree of strain, respectively. Figure 5.7 shows the austenite grain size after (a) solutionizing (without straining), (b) a single deformation ($\varepsilon = 0.3$) and (c) a double hit compression



Fig. 5.7 The austenite grain size after; (a) solutionizing (no strain), (b) a single pass deformation ($\varepsilon = 0.3$), and (c) double-hit compression ($\varepsilon_1 = \varepsilon_2 = 0.3$) at 1050°C.

 $(\varepsilon_1 = \varepsilon_2 = 0.3)$ all at 1050°C. In this figure the austenite grain boundaries were delineated by proeutectoid ferrite formed by quenching the specimen from 1050°C into a salt bath held at 650°C. In this way, the ferrite decorated the prior austenite grain boundaries. The results of the austenite grain size measurements showed good agreement with the ones calculated based on the above equation. With an initial grain size of 230µm for steel A the calculated results for a single deformation ($\varepsilon = 0.3$) and a double hit compression ($\varepsilon_1 = \varepsilon_2 = 0.3$) were 65µm and 30µm, respectively, and the measured ones were 70µm and 40µm, respectively.

Figure 5.8 shows the variation of retained austenite volume fraction (V_{RA}) and the corresponding retained austenite carbon content (wt%), with austenite grain size, in steels A and B. Here again, the ferrite volume fraction in all cases for each steel was kept the same (steel A: $25\pm2.5\%$, steel B: $30\pm3\%$). As can be seen in Fig. 5.8a, a decrease in the austenite grain size in steel A from $230\mu m$ to $70\mu m$ leads to a 22% reduction of the V_{RA} . The carbon concentration, however, remains more or less constant. In the case of steel B, the effect of austenite grain size on retained austenite volume fraction is opposite to that of steel A, [Fig. 5.8b]. The retained austenite in the as-solutionized specimen of steel B (*i.e.* very large grain size), has a higher carbon content compared to the smaller grain size specimens.



Fig. 5.8 Variation of the V_{RA} and the corresponding carbon concentration with prior austenite grain size for, (a) steel A and (b) steel B.

The mechanical properties of steels A and B with different grain sizes are shown in Fig. 5.9(a,b). As can be seen, the largest grain size in steel A not only resulted in the highest amount of retained austenite, but also led to the largest total elongation. In steel B, however, there appears to be an optimum retained austenite fraction which maximises the mechanical properties. In other words, the total elongation is not a function of V_{RA} only.



Fig. 5.9 Mechanical properties of (a) steel A and (b) steel B with different prior austenite grain sizes.

The dispersion of retained austenite in the samples with different grain sizes is shown in Fig. 5.10. As can be seen, the most important accommodation sites (pools) for the retained austenite (*i.e.* bainite interlayers and inter packets) did not vary with prior austenite grain size. Note, however, the change of the bainite packet sizes with prior austenite grain size. The smaller the prior austenite grain size, the smaller the bainite packet size, which is in agreement with other investigations (231-233). The other interesting effect concerns the lower bainite colony size, which is increased by increasing the upper bainite packet size.



Fig. 5.10 Color-etched microstructures of steel A with different prior austenite grain sizes; (a) 70µm and (b) 230µm.

5.1.1.3 Effect of the static recrystallization temperature

Changing the temperature of static recrystallization is equivalent to varying the finishing temperature above the T_{nr} in an industrial schedule. To investigate this, the same thermomechanical treatment, [Fig. 5.1], was carried out at a different temperature. The results are shown in Fig. 5.11. As can be seen, the change in the finishing temperature of steel A from 1050°C to 1000°C resulted in a decrease in the amount of retained austenite by 20%. On the other hand, no significant effect on the retained austenite volume fraction was found in steel B. Lowering the finishing temperature in steel A reduced the V_{RA} of steel A to a level close to that of steel B.

The mechanical properties of these specimens are illustrated in Fig. 5.12. It should be mentioned that, since the ferrite quantity differs in each steel, a comparison between the two *steels* is not valid. In both steels, total elongation decreases with the lower temperature. For steel A, this coincided with a decrease in V_{RA} . Although the level of



Fig. 5.11 Variation of the V_{RA} of steels A and B, statically recrystallized at different temperatures.

retained austenite is almost the same for both temperatures in steel B, there is a significant change in mechanical properties.



Fig. 5.12 Mechanical properties of steels A and B in specimens which were statically recrystallized at different temperatures.

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Microscopic examination revealed the presence of some colonies of pearlite in the microstructure of the specimens which were processed at 1000°C, [Fig. 5.13]. This is consistent with the work of Richman et al. (206), who believed that, with the same prior austenite grain size, a decrease in the austenitizing temperature increases the kinetics of pearlite formation. This was related to the formation of transformation sites or embryos which are stabilized by the presence of imperfections in the parent phase (austenite), and which tend to decrease reversibly in size or potency with increasing temperature. Another possibility which they noted was that a metastable clustering of carbon atoms mav occur about lattice imperfections in the parent phase, it being these imperfections that participate in the pearlite-nucleation process.



Fig. 5.13 Color-etched micrograph of steel A after recrystallization at 1000°C; The black regions are pearlite.

5.1.1.4 The effect of Nb

Nb has been found to be the most influential microalloying element in HSLA steels. It retards austenite recrystallization by the solute drag effect as well as by strain induced precipitation $(^{236})$. On the other hand, Nb in solution, in common with other alloying elements, retards ferrite and pearlite formation as well as depressing the bainite transformation to lower temperatures. Generally, the undissolved Nb in austenite (usually in the form of Nb(CN)) may have two effects which lead to the same results. Firstly, it does not have any retardation effect on the transformation kinetics as it does when it is in solution and, secondly, it actually accelerates transformation kinetics by acting as nucleation sites. The combined outcome of these effects can influence the state of the retained austenite at the end of TMP.

In order to examine these aforementioned effects, two different hot deformation schedules, in addition to the usual one, were used. One involved double hit compression

at 1000°C with a longer interpass time and longer holding period after the second pass [Fig. 5.14, path no. 2] than those applied in the previous section (path no. 1). The second schedule (path no. 3) was comprised of a deformation performed isothermally after the first pass using a slower strain rate, but to the same strain level (i.e., longer time of These schedules were deformation). considered to make at least 50% of Nb precipitate out, because dislocations increase the density of nucleation sites and the effective diffusion rate of the solute atoms, accelerating precipitation in this way (237)The effect of



for highlighting the effects of Nb.

deformation on precipitation has been systematically studied by *Weiss* and *Jonas* ⁽²³⁸⁾. Figure 5.15, taken from their work, compares the precipitation-temperature-time (*PTT*) diagrams for three conditions. A 5% prestrain accelerates the rate of static precipitation by about one order magnitude and decreases the nose temperature. The rate of dynamic precipitation is more than an order of magnitude faster than that of the static precipitation induced by a 5% prestrain.

The variations in the V_{RA} and their relative differences carbon after applying the three different conditions [Fig. 5.14] are shown in Fig. 5.16. The austenite grain size and the ferrite quantity are the same at the end of all three processes. As can be seen, the V_{RA} was not altered by the changes in hot deformation process. The



temperature) curves obtained during dynamic, strain induced (5% pre-strain) and static (no-predeformation) precipitations.

retained austenite carbon level, however, has decreased by 30% in the second and third conditions (those with longer duration at 1000°C).



Fig. 5.16 Variation of the V_{RA} and the corresponding carbon content of steel A with different conditions.

If the results illustrated in Fig. 5.16 on V_{RA} are compared with those of Fig. 5.11, it may be concluded that the decrease in the temperature of *TMP* in the case of steel A, which reduces the Nb in solid solution by promoting Nb(CN) precipitation, plays the major role in the decrease in the V_{RA} . Another interesting point is the carbon decrease in the retained austenite with increase in the length of time of the process. This appears to be associated with Nb precipitation and its subsequent coarsening, which therefore consumes carbon on the one hand, and changes the kinetics of the subsequent transformation (especially bainite formation) on the other hand. These will be discussed in more detail in the next chapter.

5.1.2 Dynamic Recrystallization

As is well known, dynamic recrystallization is observed in FCC metals and alloys (except aluminum) containing moderate to low stacking-fault energy, and is associated with relatively low rates of dynamic recovery and higher than average rates of work hardening. Dynamic recrystallization commences only when a critical strain (also called the *incubation strain*) ε_c is reached during hot deformation. This is somewhat less than the strain at the maximum stress, *i.e.* the maximum (or peak) strain ε_p ⁽²³⁹⁾.

Dynamic recrystallization gives rises to a decrease in the rate of strain hardening which leads to the peak. This can happen over several cycles (multiple peak) or continuously (single peak) ^(240,241). The former behaviour appears at low strain rates and

high temperatures. This indicates that discrete cycles of grain coarsening are occurring. The latter behaviour occurs at relatively high strain rates and low temperatures. This has been shown to be an effect of grain refinement $(^{239})$. It is important to note that dynamic recrystallization produces both a texture and grain size distribution which are different from that of static recrystallization $(^{242})$. Another noteworthy point of the dynamically recrystallized structure concerns the dislocation substructure. Here, a rather poorly recovered dislocation substructure develops in the initial stage of deformation; *i.e.* the cells have a relatively small size and more tangled walls compared to structures which have undergone a high degree of static recrystallization. This substructure, when retained to room temperature, leads to yield strengths higher than those of annealed materials $(^{239})$.

To follow the effect of the dynamically recrystallized microstructure on the state of the retained austenite, the specimens were solutionized as before and processed by double hit compression at two different temperatures. In this case, to attain dynamically recrystallized austenite, the second pass was carried out with a strain rate one to two orders of magnitude lower than that of the first pass [Fig. 5.2].

Figure 5.17 illustrates the variation of retained austenite volume fraction for different conditions of dynamic recrystallization. The decrease of the temperature (from 1050°C to 1000°C, (1)) results in a decrease in the V_{RA} by about 10%, which follows the pattern seen in static recrystallization, [Fig. 5.12]. The level of V_{RA} reduction in the former, however, is less than that of the latter. It should be mentioned that, since the differences in prior austenite grain sizes (resulting from temperature and deformation variations in this case), are in



Fig. 5.17 Variation of the V_{RA} of steel A with different conditions of dynamic recrystallization.

the range of $\pm 5\mu m$, their effects on the subsequent transformation characteristics and on the retained austenite volume fraction are negligible.

In order to see the effect of dynamic recrystallization substructure, the dynamically recrystallized austenite was cooled down rapidly to the next step of processing. In the case of condition (1) the specimen was cooled 3° C/sec. to a temperature below Ar_3 , and this was increased to 10° C/sec. in the condition (2). As can be seen [Fig. 5.17], the $V_{R,4}$ was increased again to a higher level. A comparison of this result with those of the previous section [Fig. 5.16], indicates that the dynamically recrystallized austenite substructure, when retained to transformation temperatures, is more prone to retaining austenite. The corresponding microstructural examinations did not show changes in the retained austenite size and distribution in different specimens.

The mechanical properties of the specimens dynamically recrystallized at 1000° C, followed by rapid and slow cooling, are shown in Fig. 5.18. As can be seen, the variation of total elongation follows the trend of the retained austenite volume fraction change. The *UTS* of the rapidly cooled material, even with higher amounts of retained austenite, is still higher than that of the slowly cooled structure.



Fig. 5.18 Mechanical properties of steel A for different conditions of dynamic recrystallization.

5.2 The Effect of Pancaking

In principle, a temperature (T_{nr}) can be defined in steels below which the austenite does not undergo recrystallization after high temperature deformation. This temperature depends greatly on the steel composition and the deformation conditions (strain and strain rate) ⁽²⁴³⁾. Generally, both dynamic and static recrystallization are retarded, by alloying elements in solution or when newly precipitated, to a degree that depends on the temperature and the alloying element. As was described earlier, the second stage of controlled rolling is deformation of austenite in the no recrystallization region (which results in the pancaking of austenite). The relevant microstructural features of pancaked austenite are the increase in dislocation density, formation of deformation bands, the destruction of the coherency of annealing twins with the matrix and the formation of ledge like austenite grain boundaries.

In general, both diffusional and non-diffusional transformations can be affected by the aforementioned features of pancaked austenite. This, in turn, can dictate the state of the retained austenite. Here, the effects of pancaked austenite on the retained austenite characteristics were investigated using the hot deformation schedule shown in Fig. 5.19. The specimens were deformed below the T_{nr} after applying double hit deformation at 1050°C. The rest of the process was the same as before, apart from the isothermal hold temperature in the two phase region,



Fig. 5.19 The heat and deformation schedule used to investigate the effects of pancaking.

which was increased to 700°C in order to avoid pearlite formation during isothermal holding and cooling to bainitic region, since undesired pearlite transformation can be encouraged by pancaking.

Figure 5.20a shows the variation of the retained austenite volume fraction with the amount of deformation in the no-recrystallization region for steel A. The amount of ferrite was kept the same for all cases (30 \pm 3%). As can be seen there is a maximum V_{RA} at about

20% strain. At this degree of deformation, the carbon level of the retained austenite is also maximised, [Fig. 5.20b]. Figure 5.20c displays the variation of V_{R4} with the same pancaking conditions for steel B (the amount of ferrite in these specimens was 40±4%). Again, in this case, the maximum amount of retained austenite is attained at 20% strain.



Fig. 5.20 Variation of, (a) the V_{RA} of steel A, (b) the corresponding carbon content of steel A and (c) the V_{RA} of steel B, with strain in the no-recrystallization region.

The corresponding mechanical properties of these steel A microstructures are illustrated in Fig. 5.21. The total elongation does not change systematically with retained austenite volume fraction. The ultimate strength, however, attains its maximum at about 20% deformation, coinciding with the maximum V_{RA} .

The microstructures of steel A specimens are shown in Fig. 5.22. As can be seen the increase of strain in the no-recrystallization region leads to an increase in the interlayer thickness of upper bainite platelets as the degree of pancaking increases. On the other hand the fraction of lower bainite appears to increase as the strain increases. Furthermore, the dispersion of retained austenite follows the same trend as before, *i.e.*, the main part of retained austenite volume fraction is enclosed by bainite packets and lathes.



Fig. 5.21 Mechanical properties of steel A for different amount of strain in no recrystallization region.



(a)

(b)

Fig. 5.22 Color-etched microstructures of steel A; (a) 10% and (b) 30% strain in no recrystallization region.

5.3 The Effect of Finishing Temperature in the No-Recrystallization Region

Since there is always some restoration (*i.e.*, dynamic and static recovery and recrystallization, depending on the state of strain and temperature) associated with hot deformation, the amount of strain energy accumulation (*i.e.* the density of dislocations in austenite) changes with the deformation-finish temperature. This, in turn, leads to a change in the kinetics of subsequent transformations. Figure 5.23 shows the variation of the retained austenite volume fraction with finishing temperature. The ferrite quantities for the specimens of steels A and B are 16%±2 and 32%±3, respectively. Steel A does not encounter a significant change in V_{RA} after a 30% reduction at 900°C and 800°C. This is not true of steel B where the decrease in finishing temperature resulted in a 15% increase in V_{RA} .



Fig. 5.23 Variation of the V_{RA} with finishing temperature in the no-recrystallization region; (a) steel A, (b) steel B.

The corresponding mechanical properties of the above microstructures of steel A are shown in Fig. 5.24. Clearly, the total elongation is lower in the case of the sample finished at 800°C. The ultimate strengths, however, were not changed significantly.



Fig. 5.24 Mechanical properties of steel A for different finishing temperature.

5.4 The Effect of the State of Ferrite

The austenite-to-proeutectoid ferrite reaction is the first phase transformation which occurs during equilibrium cooling of hypoeutectoid steels. As was mentioned earlier, this reaction is a diffusional nucleation and growth process. Once the reaction starts, growth of ferrite, which can only dissolve very low carbon levels, takes place by rejection of carbon into the untransformed austenite, such that the carbon content of the austenite increases with increasing ferrite volume fraction. Since the carbon requires time to diffuse uniformly through the austenite phase, a carbon gradient is established at the growing interface, and growth is controlled by long-range diffusion of carbon in austenite. The carbon gradient and, accordingly, carbon enrichment of austenite, can chemically lead to different degrees of austenite stabilization. The stability of austenite and, consequently, the state of the retained austenite may be affected, not only by the amount of ferrite (via changes in austenite carbon enrichment), but also by the morphology of ferrite (due to geometrical effects).

5.4.1 The Austenite-to-Ferrite Transformation Characteristics

The transformation of thermomechanically processed austenite to ferrite is determined by: *i*) the composition of the austenite, which controls the hardenability of the steel and thus the transformation temperature for a given cooling rate (as was shown in the *CCC* results); *ii*) the microalloying additions, and the temperature dependence of the solubility of the microalloy carbide/nitride, which will determine the solute content and the amount of undissolved carbides/nitrides; *iii*) the reheating temperature, which will dictate

the dissolved solute concentration and the austenite grain size prior to thermomechanical processing; iv) the morphology and the grain size of austenite after *TMP*, which will control the ferrite nucleation rate through grain boundary surface area, serrations on grain boundaries, deformation bands, recovered substructures, etc.; v) the presence of undissolved or strain induced microalloy carbides, which will decrease the solute concentration and may also act as nuclei for ferrite formation; vi) the rate of cooling after *TMP*, which, for a constant microstructure and composition of the austenite, will greatly affect the transformation temperature and, thus, the nucleation and growth rates of the ferrite ⁽²⁴⁴⁾. This in turn will determine the ferrite grain size and morphology.

The significance of the above factors can be evaluated by their effects on the Ar_3 temperature. In fact the control of ferrite characteristics by these factors is through their effects on this critical temperature. For instance, the effect of austenite grain size on the Ar_3 temperature of steels A and B, obtained by CCC testing, is tabulated in Table 5.1. As

can be seen, the bigger the austenite grain size, the lower the Ar_3 temperature. This effect of the austenite grain size on its subsequent transformation characteristics is analogous with cooling rate and strain accumulation. In this way, both the kinetics of the transformation start

 Table 5.1
 Variations of ferrite formation characteristics with prior austenite grain size

Steel	Grain Size (µ)	n	Ln b	Ar ₃ (°C)
A	230	1.359	-9.674	755±5
A	70	1.804	-11.100	775±5
A	40	0.870	-5.250	810±5
B	350	1.225	-8.301	750±5
B	80	1.971	-11.114	780±5
B	45	1.117	-5.678	815±5

and the progress of the transformation are affected. Figure 5.25 shows the results of isothermal (650° C) holding on the progression of ferrite formation for steels A and B. As can be seen, the larger the austenite grain size, the slower is the rate of ferrite transformation.

The isothermal transformation rate of austenite to proeutectoid ferrite at temperatures in the two phase region can be described by the *Avrami* equation ⁽²⁴⁵⁾.

$$X = 1 - \exp(-bt^n) \qquad \qquad Eq. \ 5.2$$

where X is the fraction transformed and t is time. The parameters n and b in this equation can be obtained from a least-squares fit (246) of the kinetic data to a plot of Ln Ln[1/(1-X)] vs Ln t. It should be mentioned that the Avrami equation is based on a total fraction of unity. Here, since the total fraction of ferrite formed at a given temperature is less than unity, the transformation data have to be normalized with respect to the total volume fraction transformed at the end of the transformation (247) before the least-square fit is made. The total ferrite fraction was determined by holding a specimen for a very long time at the desired isothermal temperature, and measuring the resulting ferrite fraction. The results of the calculation for different grain sizes and the identical isothermal holding temperature are given in Table 5.1. As can be seen, in all cases the kinetics of ferrite formation in steel B are higher than in steel A. This, as was mentioned before, can be related to the solute drag like effect of Nb on the austenite-to-ferrite transformation.



Fig. 5.25 Variation of the ferrite volume fraction with isothermal holding duration for different prior austenite grain sizes; (a) steel A and (b) steel B.

A final effect of austenite grain size concerns the variation of the ferrite morphology at the end of the CCC test, this being brought about by changing the Ar_3 temperature, which in turn is due to the change of the austenite grain size. This can be observed in the CCC curves of different austenite grain sizes [Fig. 5.26]. It can be seen that the deviation which was related to acicular ferrite formation, has disappeared on changing the austenite grain size from $230\mu m$ to $40\mu m$. It should be noted that, the higher flow stresses in the coarser austenite grain ($230\mu m$) can be related to grain boundary sliding, operating at high temperatures and low strain rates.



Fig. 5.26 The CCC true stress-temperature curves of steel A for two different prior austenite grain sizes.

5.4.2 The Effect of Ferrite Quantity

To determine the effect of ferrite quantity on the amount of retained austenite, the specimen, after performing the basic *TMP* at higher temperatures, was held at a temperature below the Ar_3 (650 °C) for various times, followed by quenching into the salt bath. Figure 5.27 shows typical microstructures of steel A after 1 and 5 minutes holding at 650 °C. The increase in the ferrite quantity after the longer holding time is quite clear. The variation of retained austenite quantity with the amount of ferrite for steels A and B, for prior austenite grain sizes of $40\mu m$ and $45\mu m$, respectively, is shown in Fig. 5.28. It seems that, by increasing the amount of ferrite, the retained austenite quantity, after a small maximum, decreases. Furthermore, it appears that there is an optimum amount of ferrite which maximises the amount of retained austenite.

Figure 5.29 shows the effect of austenite grain size and ferrite volume fraction of steel A on the V_{RA} . As can be seen, the existence of an optimum ferrite fraction which leads to a maximum amount of retained austenite appears to be repeated for all austenite grain sizes. Furthermore, it was observed that [Fig. 5.30], for steels A, B and C, having an intermediate prior austenite grain size, the V_{RA} increased beyond a certain ferrite fraction (in this case ~50%).



Fig. 5.27 Optical microstructure of steel A after, (a) 1 min. and (b) 5 min. holding at 650°C.



Fig. 5.28 Variation of the retained austenite volume fraction with the amount of ferrite; (a) steel A and (b) steel B.



Fig. 5.29 Variation of the retained austenite volume fraction with the ferrite quantity of steel A for different prior austenite grain sizes; (a) $40\mu m$, (b) $70\mu m$ and (c) $230\mu m$.



Fig. 5.30 Variation of the retained austenite volume fraction with the ferrite quantity of different steels for intermediate austenite grain size; (a) steel A, (b) steel B and (c) steel C.

5.4.3 The Effect of Ferrite Formation Temperature

Since the temperature of the isothermal austenite-to-ferrite transformation can affect the size, distribution, morphology and composition (carbon level) of ferrite and the remaining austenite (untransformed austenite), it will therefore influence the state of the retained austenite. The effect of the isothermal holding temperature on the retained austenite volume fraction is given in Table 5.2. It should be noted that, since the Ar_3 temperature was the same, in order to obtain a given amount of ferrite by isothermal

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holding, higher isothermal temperatures required longer holding durations. By comparing these data with the values in section 5.4.2, it can be concluded that, in steel A, increasing the temperature leads to reduction of V_{RA} . In the case of steel B, however, there is no significant effect of temperature.

Steel	Temp. (°C)	V _F (%)	V _{RA} (%)
A	650	22.5	9.5
A	700	28.0	7.5
A	750	14.5	8.0
A	750	37.0	5.9
B	650	36.0	7.1
B	700	46.5	4.9
B	750	29.5	7.2
B	750	34.0	6.5

Table 5.2Variation of the V_{RA} with the temperature of
isothermal holding in the two phase region

5.4.4 The Effect of Ferrite Grain Size

Austenite pancaking is one of the most common methods used to refine the ferrite grain size. This is related to the additional ferrite nucleation sites such as austenite grain boundary serrations, deformation bands and twins. In the case of the steels used in this work, the $T_{nr'}$ s for grades A and B were estimated to be about 930°C and 910°C, respectively. The effect of 20% and 30% strains in the no recrystallization range on the microstructure of steel A is shown in Fig. 5.31. As can be seen, the increase of the strain from 20% to 30% resulted in ferrite grain refinement. Table 5.3 is an alternative presentation of the previously reported pancaking data for steel A. The results show that a decrease in the ferrite grain size by pancaking does not increase the retained austenite quantity, contrary to the conclusions of Kawano et al (29,248).

Strain in the No Rex. Region	20%	30%	
Strain during 7-to-a Transformation	-	-	20%
Ferrite Volume Fraction	27.5%	30.0%	25.0%
Ferrite Grain Size	19 µm	13 µm	10 µm
Retained Austenite Volume Fraction	9.11%	7.92%	9.60%

Table 5.3Variation of the V_{RA} with polygonal ferrite grain size obtained by different
TMP schedules

Another way of refining the ferrite grain size, which is well established, is straining the specimen during transformation (dynamic transformation of austenite-to-ferrite). In this way, not only the ferrite grains can be refined, but a higher dislocation density (finer subgrain size) is established in the ferrite. To carry out the dynamic transformation, the

Fig. 5.32

The heat and deformation schedule used to apply

dynamic transformation of

austenite-to-ferrite.

specimen was deformed isothermally at a temperature below the Ar_3 (*i.e.* in the two phase region), [Fig. 5.32]. The effect of this process on the final microstructural components is tabulated in Table 5.3. As can be seen, the deformation during transformation not only has more effect on the ferrite grain refinement, [Fig. 5.33], but also has more influence on the retained austenite volume fraction compared to pancaking.



Fig. 5.31 Typical microstructures in steel A after, (a) 20% and (b) 30% strain in the no recrystallization region.





Fig. 5.33 Typical microstructures in steel A after, (a) 20% strain in no recrystallization region and (b) 20% strain during austenite-to-ferrite transformation.

5.4.5 The Effect of Ferrite Morphology

From the different morphologies of ferrite, two different categories can be distinguished. The first is characterised by a transformation without any surface relief effects and, consequently, leads to lower dislocation densities at the end of transformation, *e.g.* polygonal ferrite. The other type exhibits surface relief and higher densities of dislocations, *e.g.* acicular and widmanstatten ferrite. To generate acicular ferrite in addition to polygonal ferrite, the thermal and deformation schedule shown in Fig. 5.34 was applied, (this schedule was based on the *CCC* results). Here, the isothermal hold in the two phase region and the quench in the salt bath stages were substituted by continuous cooling in the furnace (in the two phase region) and with compressed air and argon (from the two phase region to the bainite hold temperature), respectively. The presence of the acicular ferrite in the microstructure after this treatment for steels A and B is seen in Fig. 5.35.



TIME

Fig. 5.34 The heat and deformation schedule used to generate acicular ferrite.



Fig. 5.35 Typical microstructures of (a) steel A and (b) steel B, revealing acicular ferrite.

Figure 5.36 shows the microstructural components in samples with and without acicular ferrite for steels A and B. As can be seen, the presence of acicular ferrite in steel

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A has resulted in a significant increase in the quantity of retained austenite. The opposite behaviour is, however, observed in steel B. Further, in Fig. 5.37, in both steels, the white components (*i.e.*, the martensite/retained austenite fraction) are mostly enclosed by fine acicular ferrite or coarse granular bainite. Essentially, the prior austenite grains have been fragmented by these phases, and the distributions of the white components are dictated by this grain fragmentation.

Since the condition of the prior austenite can affect the acicular ferrite characteristics, the prior austenite structure was varied. This was done by generating dynamically recrystallized and pancaked austenite prior to transforming to acicular ferrite.



Fig. 5.36 The microstructural components of, $(a_1 \text{ and } a_2)$ steel A with and without acicular ferrite, respectively, and $(b_1 \text{ and } b_2)$ steel B with and without acicular ferrite, respectively.

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Fig. 5.37 The retained austenite distributions in (a) steel A and (b) steel B; left and right microstructures with and without acicular ferrite, respectively.
5.4.5.1 Dynamically recrystallized austenite

The austenite was dynamically recrystallized using the same schedule as section (5.1.2), and this was transformed to acicular plus polygonal ferrite by applying the aforementioned continuous cooling treatment [Fig. 5.34]. The amount of ferrite (acicular plus polygonal) was kept the same in all the cases (~35%). In this series of experiments, fast cooling between the temperature of *TMP* finish (1050°C) and the Ar_3 was again

practiced. As can be seen in Fig 5.38, the slow cooling resulted in almost the same results as before (i.e. Fig. 5.28 with only polygonal ferrite present in microstructure), although the the retained austenite volume fraction is still higher than for specimens without acicular ferrite, if the results are normalized by the amount of ferrite. The faster cooling from the finishing temperature to the start of ferrite formation has led to higher amounts of retained austenite at the end of process.



Fig. 5.38 Variation of the V_{RA} with different conditions of dynamic recrystallization in specimens with acicular plus polygonal ferrite.

5.4.5.2 Pancaked austenite

The variation of the retained austenite volume fraction with the degree of strain in

no-recrystallization region, for the microstructures consisting of acicular plus polygonal ferrite, is shown in Fig. 5.39. As can be seen, the V_{RA} increases with the degree of deformation in the no-recrystallization region up to 20% strain; thereafter it drastically decreases. In any case, for a given amount of ferrite, the volume fraction of retained austenite of the specimen with acicular ferrite is much higher than structures with polygonal ferrite only. Note, however that the



Fig. 5.39 Variation of the V_{RA} with strain in no recrystallization region in specimens with acicular plus polygonal ferrite.

mechanical tests on the sample with 14% retained austenite volume fraction did not show extraordinary mechanical properties, (30% total elongation and 1100 MPa UTS).

5.5 The Effect of Bainite Characteristics

In all the previous experiments, the last part of the thermomechanical treatment (before air cooling to room temperature) was isothermal holding at a given temperature (400°C) in the bainite region. According to the literature, this temperature is low in the upper bainite range for the particular steels used in this work. Both upper and lower bainite consist of aggregates of platelets or laths of ferrite separated by regions of what *Christian* ⁽²⁴⁹⁾ terms *residual* phases. Depending on the steel chemical composition, the temperature and the duration of the isothermal hold in the bainite region, these latter phases can be made up of untransformed austenite, or phases such as martensite or carbide, which form subsequent to the growth of bainitic ferrite.

5.5.1 The Effect of Isothermal Holding Duration

For the purpose of assessing the effect of hold time in the bainite region, the specimen was treated as per section 5.1. The only differences were the isothermal holding temperature in the two phase region (700°C instead of 650°C), and holding time at 400°C. The effect of hold time on the amount of retained austenite for the steels A and B is shown in Fig. 5.40. It is apparent that, for both steels, increasing the time leads to a maximum V_{RA} . The mechanical properties of the specimens of steel A are illustrated in Fig. 5.41.



Fig. 5.40 Variation of the V_{RA} with holding time at 400°C; (a) steel A and (b) steel B.

The total elongation appears to coincide with the changes in the V_{RA} , but the tensile strength increases continuously with holding time at 400°C. The color etching of the samples shows less lower bainite as the hold duration increases, [Fig. 5.42].



Fig. 5.41 Mechanical properties of steel A for different holding duration at 400°C.



Fig. 5.42 Color-etched microstructures of steel A for different holding duration at 400°C; (a) 2 min. and (b) 5 min.

5.5.2 The Temperature of Isothermal Holding

In this series of tests, the specimens of steels A and B were treated as above, but two other temperatures were also examined. Figure 5.43 shows the variation of V_{RA} with the temperature of the bainite hold. This appears to follow the results of previous workers, who demonstrated a maximum at 400°C for V_{RA} . The same maximum can also be seen in the total elongation [Fig. 5.44]. The tensile strength, however, continually decreases with increasing bainite hold temperature. The corresponding color micrographs of Fig. 5.45, clearly reveal the difference in the bainite morphology. As can be seen, an increase in the bainite hold temperature resulted in the increase of lath (platelet) size and interlayer thickness.



Fig. 5.43 Variation of the V_{RA} with the temperature of bainite formation; (a) steel A and (b) steel B.



Fig. 5.44 Mechanical properties of steel A for different temperatures of bainite formation.



Fig. 5.45 Color-etched microstructures of steel A for different temperatures of bainite formation; (a) 300°C and (b) 500°C.

5.6 The Retained Austenite Morphology

As is apparent from the name, retained austenite is retained from the prior austenite which was present at higher temperatures. The prior austenite may undergo different stages of transformation during cooling to room temperature. The application of *TMP* on the parent austenite, depending on the mode of transformation, stimulates or retards the kinetics of its subsequent transformations. The change in the kinetics of transformation, in turn, alters the morphology of the phase being formed. All of these parameters are interlinked, and identifying the individual effects on the morphology of the retained austenite is very difficult.

Based on the results of the present work, the morphology of retained austenite can be classified into five different categories:

1) blocky shape enclosed by polygonal ferrite, [Fig. 5.46]

2) blocky and/or plate shape enclosed by acicular ferrite, [Fig. 5.47]

3) blocky shape enclosed by bainite packets, [Fig. 5.48]

4) interlayer residual phase between:

a) upper bainite platelets, [Fig. 5.49a]

b) intermediate temperature bainite platelets, [Fig. 5.49b]

c) lower bainite laths, [Fig. 5.49c]

5) interlayer film enclosed by martensite laths.

5.6.1 Blocky Shape Enclosed by Polygonal Ferrite

The first category appears to be the result of ferrite impingement during growth. Therefore, this morphology is common to the specimens with finer austenite grain size and/or longer duration of isothermal holding in the two phase region. The fact that this type of retained austenite is trapped by ferrite leads to two important conclusions; firstly, because of the ferrite matrix, this type of retained austenite does not lead to TRIP strengthening during initial stages of straining up to higher levels of deformation; secondly, since there is no constraint from the surrounding matrix (lower dislocation density), its stability is due to carbon content and size. In addition, the distribution of this type of retained austenite is not homogenous. A typical *TEM* and compositional variation of this morphology is shown in Fig. 5.46. As can be seen, there are higher levels of Mn and lower amounts of Si in comparison with the ferrite matrix. It should be mentioned that the *EDX* analyses were carried out using a beam with energy of 200 kV and spot size of maximum 50 nm and the semiquantitative microanalysis was performed based on the simplification that Fe%wt+Mn%wt+Si%wt=1.

5.6.2 Blocky and/or Plate Shape Enclosed by Acicular Ferrite

Due to the size and distribution of acicular ferrite, which is a function of its formation temperature, the shape and size of the entrapped retained austenite can be changed. As is well known, the mechanism of acicular ferrite formation is shear, thereby imposing a shape change (invariant plane strain) to the surrounding matrix. Accordingly, this type of retained austenite morphology has a higher dislocation density than that of the first category. A typical *TEM* and compositional profile of this class of retained austenite is shown in Fig. 5.47. The Si and Mn variation almost follows the pattern of the first morphology.



(b)



Fig. 5.46 TEM of blocky shape retained austenite enclosed by polygonal ferrite; (a) bright field and (b) dark field.

5.6.3 Blocky Shape Enclosed by Bainite Packets

The third class of the retained austenite morphology can be seen in almost all the specimens of this work (except those which were treated in the lower bainite region). As was briefly discussed earlier, the size and the distribution of the bainite packets change with the prior austenite grain size and the bainite hold temperature. Such changes lead to variations in the size and distribution of the retained austenite enclosed between the sheaves. Moreover, the dislocation density depends on the temperature of entrapment. The higher the bainite hold temperature, the lower the dislocation density. Figure 5.48 shows a typical *TEM* of this morphology along with its compositional profile. The trend of Si and Mn variations once again seems to be similar to the previous ones.

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(a)

(b)





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Fig. 5.48 TEM of blocky shape retained austenite enclosed by bainite packets; (a) bright field and (b) dark field.

5.6.4 Interlayers or Films

The final classes of the retained austenite morphologies are the films. Since the interlayer space between platelets (or laths in lower bainite and martensite) reduces with decreasing bainite hold temperature, the thicknesses of the retained austenite films are correspondingly diminished. On the other hand, the plastic constraints emanating from the shear transformation and the accommodation of plastic deformation are also increased by the reduction of temperature. This, in turn, leads to a higher dislocation density in the retained austenite in comparison with the previous morphologies. Typical *TEM*s of these

types of retained austenite morphology are shown in Fig. 5.49(a,b,c). As can be seen in these figures, the decrease in the temperature of isothermal holding in the bainite region results in a finer interlayer retained austenite film. The variations of Si and Mn, however, appear to follow the same trend as before, *i.e.* a higher Mn and lower Si level in the retained austenite with respect to the matrix.

Figure 5.50 shows the morphology and compositional variations of the retained austenite formed at 400°C, but after a longer hold time (5 min.). The only difference is the





(a)





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trend of the Si and Mn profile in the retained austenite. This one shows lower Mn and higher Si compared to the ferrite matrix. This can probably be related to the participation of some Mn in the carbides which form during the longer holding time.



(a)

(b)



Fig. 5.49b TEM of interlayer retained austenite trapped between intermediate temperature bainite platelets; (a) bright field and (b) dark field.



(a)

(b)



Fig. 5.49c TEM of interlayer retained austenite trapped between lower bainite platelets; (a) bright field and (b) dark field.

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(a)

(b)



Fig. 5.50 TEM of retained austenite in specimen held for longer duration (5 min.) at 400° C; (a) bright field and (b) dark field.

CHAPTER 6

DISCUSSION

In the following, the results of the previous chapter will be discussed in detail and, wherever possible, the present results will be compared with the literature. As was emphasized in *Chapter 2*, almost all the previous investigations on the state of the retained austenite concerned the effect of intercritical annealing, as opposed to thermomechanical processing. For this reason, it is worth beginning this chapter with a short comparison between the parent austenite obtained by intercritical annealing (IA) and thermomechanical processing (TMP).

6.1 The Characteristics of Parent Austenite after IA and TMP

The formation of austenite during intercritical annealing of low carbon and dual phase steels has been the subject of a number of investigations (250-255). In all cases, the mechanism and kinetics of austenite formation and its final morphology were shown to be mainly dependent on the prior microstructures (52,256-259). For example it has been shown that, on transforming from a pearlitic microstructure, there are three favorable nucleation sites for the formation of austenite [Fig. 6.1]: (a) the ferrite/cementite interface, (b) the line of intersection between the cementite platelets and surfaces of the pearlite colony, and (c) the points of intersection between the cementite platelets and the edges of the pearlite

colony (256). It was also confirmed that the growth of austenite into pearlite is a carbon diffusion-controlled process.

Furthermore, in a low-carbon steel with a ferritic microstructure, the nucleation of austenite takes place mainly at ferrite grain boundaries ⁽²⁶⁰⁾ and usually develops allotriomorphic austenite. Like allotriomorphic ferrite, an allotriomorphic austenite exhibits a *Kurdjumov-Sachs* orientation relationship with one ferrite grain and grows predominantly into the adjacent grain only by the migration of



Fig. 6.1 Three nucleation sites for austenite formation during intercritical annealing in a plane section of three pearlite colonies.

incoherent interfaces. In ferrite plus spheroidized cementite microstructures, austenite nucleates at the junctions between the cementite particles and ferrite grain boundaries. The austenite film then grows and completely envelopes the carbide, and further growth of austenite occurs by the continuous diffusion of carbon, from the dissolution of the carbide, toward the growing austenite envelope ⁽²⁶¹⁾.

In the above cases, the austenite, on cooling, transforms to one or more of the austenite decomposition products, depending on the hardenability and cooling rate. Since the austenite has formed on the ferrite grain boundaries, the various austenite transformation products of intercritically annealed hot rolled or normalized steels are also located on ferrite grain boundaries. Significant partitioning of Mn to austenite (250,252,254,255) and, to some extent, Si to ferrite (262,263) occurs during intercritical annealing.

Finally, starting from bainitic and martensitic microstructures of low-carbon-lowalloy steels, austenite forms in two morphologies: *acicular* and *globular*. The acicular austenite is promoted by an initial acicular microstructure, low isothermal austenitizing temperatures, and slow heating rates. The nucleation sites in this case are primarily lath boundaries and prior austenite grain boundaries. If the duration of holding is long enough, these transitional acicular nuclei, grow rapidly and eventually coalesce to form a globular morphology. Globular austenite can form directly with conditions opposite to those mentioned above. The mechanism of austenite formation from these structures tends also to be diffusion-controlled and is linked with the fine dispersion of carbide particles. One other very important factor controlling the austenite formation characteristics is cold rolling of the steel prior to annealing. Here, the austenite transformation products are located, not only on the ferrite grain boundaries, but also within ferrite grains (264).

The austenite which forms after applying a solutionizing heat treatment followed by IMP: (i) is chemically more homogeneous, (ii) may have a different grain size, (iii) may contain different levels of strain accumulation (dislocation density) within the grains and at grain boundaries (due to pancaking and dynamic recrystallization substructure), and (iv) its morphology is not a function of prior microstructure.

Even when this austenite is cooled down into the intercritical region in order to promote isothermal ferrite formation, its morphology is always equiaxed and its chemical composition differs from that produced by intercritical annealing. The reason for the latter is rooted in the observation of two basically different modes of growth of proeutectoid ferrite in austenite in Fe-C-X alloys: (i) growth with partitioning of the alloying element X between α and γ under local equilibrium conditions, and (ii) growth with no bulk partitioning of X between α and γ . It has been demonstrated that the latter mode is much more common, and, depending on whether X is an austenite or ferrite stabilizer, involves a narrow zone of enrichment or depletion, which moves ahead of the α/γ interface. It has been shown that this occurs for alloys in which X = Si, Mo, Co, Al, Cr and Cu. The strong austenite stabilizers Ni, Mn and Pt are the only elements to show partitioning to the austenite phase. This suggests that the austenite particles formed by TMP, are more enriched in Mn than those produced by IA. Two other reasons for this are (i) during IA, austenite forms from ferrite and carbides, which can be considered as extra barriers against bulk and interface diffusion of Mn from the parent phases to the austenite being formed, because the diffusion coefficients of Mn in both of these phases are smaller than for austenite; (ii) the temperature of IA is not high enough to ensure extensive dissolution of MnS and (Fe, Mn)₂₃C₆, thus reducing the solid solution concentration of Mn.

As an overall summary, the austenite which forms during intercritical annealing, depending on the temperature and the time of treatment, can be different in morphology, possesses a finer particle size, is more dispersed and chemically heterogeneous. All of these may promote the stabilization and, consequently, the retention of austenite at the end of process. The thermomechanically processed austenite, however, has some other features which cannot be obtained by intercritical annealing. These are the subjects of this chapter.

6.2 The Effect of Alloying Elements

All substitutional alloying elements, except cobalt, decrease the M_s (martensite start) temperature in proportion to the level in solid solution. It has been generally accepted that the M_s temperature can be considered to be a measure of austenite stability. A low M_s is indicative of high austenite stability, *i.e.* high resistance to martensite formation, thereby leading to higher amounts of retained austenite at the end of the

process. As shown in Fig. 6.2, the quantity of retained austenite in a carbon steel is markedly dependent upon the *dissolved* carbon content. Apart from carbon, the other main alloying elements which dictate the retained austenite characteristics in the TRIP steels used in this study are Si, Mn and Nb. The major role of Si, as was mentioned in *Chapter* 2, is to hinder carbide precipitation, therefore keeping the carbon in solution.

Generally, the way the alloying species influence the retained austenite characteristics, is through their effect on the subsequent austenite transformation



Fig. 6.2 Retained austenite in steel as a function of carbon content.

kinetics. The changes in the CCT (continuous-cooling-transformation) diagrams ⁽³⁵⁾ as functions of Si and Mn content are shown in Fig. 6.3. As can be seen, an increase in the silicon level from 1% to 2% results in a retardation of the austenite to pearlite transformation, which is in good agreement with the CCC results, Table 4.3. This can be related to the higher carbon activity in ferrite and the inhibition of carbide precipitation from austenite due to the higher levels of Si.

Another important effect is the expansion of the bainite field towards higher temperatures as the Si content increases. The start of martensite transformation on cooling appears to be unchanged. The increase of the Mn level from 1.5% to 2%, however, moves the austenite-to-ferrite and pearlite transformation start to longer times. On the other hand, the start of the bainite transformation appears to be unchanged. Thus, crossing the bainite transformation line on cooling can be achieved with a lower cooling

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rate (*i.e.* it is easier to obtain bainite during cooling). In addition, the stability of austenite, as indicated by the M_s temperature, increases with the Mn level.

From the above, for a given cooling rate, in order to have ferrite in addition to bainite and retained austenite in the microstructure, a compromise between Si, Mn and carbon concentrations is necessary. A word of caution should be added here. Although, the trends in transformation behaviour in Si-Mn TRIP steels follow the criteria. the transformation above temperatures presented in Fig. 6.3 do not correspond to the steels and procedures used in this work, especially under isothermal conditions.

Furthermore, it should be noted that, although the higher carbon level leads to more retained austenite, the increase of carbon, as the *CCC* results revealed, Table 4.5, leads to a higher chance of pearlite formation in the microstructure. Since pearlite consumes the carbon by carbide precipitation, the stability of the retained austenite is decreased.

As the C and Si levels in the steels in this work vary by less than 35%, an



Fig. 6.3 Changes in the CCT diagrams as a function of Si and Mn content.

appreciable difference in the retained austenite volume fraction due to the changes in the levels of these elements cannot be distinguished (265). However, C and Si do affect the *CCT* curves, and in the same way, *i.e.* an increase should increase the bainite start temperature. This can be seen by comparing steels C and D in Fig. 6.4, where the lower carbon content leads to thicker bainite platelets (laths). For the same isothermal holding

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temperature in the bainite region, the latter can be an indication of a transformation that is closer to the upper bound temperature of upper bainite.



Fig. 6.4 Color-etched microstructures of, (a) steel C and (b) steel D.

6.2.1 The Effect of Nb

As was shown in the results sections, Nb in solution in austenite had a strong influence on the retention of austenite. For example, as shown in Fig. 5.4a, steel A, exhibited at least 27% higher retained austenite volume fraction than steel B after the same TMP, (it should be recalled that the only compositional difference between these steels is Nb). Furthermore, it was shown that, by taking the Nb out of solution, (by means of the change in processing temperature, [Fig. 5.11], or accelerating the Nb(CN) precipitation by concurrent straining [Fig. 5.16]), the retained austenite volume fraction in steel A decreased and almost approached that of steel B. The effect of Nb on the austenite transformations of different steels has been extensively investigated (102,222,266,267). The effect on the austenite-to-ferrite transformation was discussed in *Chapter2*, but Nb can also affect the hardenability and the start temperature of martensite transformation (M_s) . It was demonstrated that the presence of small levels of dissolved Nb in Fe-22.5%Ni lowered the M_s temperature (266),

as shown in Fig. 6.5. The presence of low concentrations of Nb was also reported to act as an austenite stabilizer (222) in HSLA steels. In addition, it has been well established that the hardenability increases with increasing amount of microalloys, such as Nb, Ti and V, dissolved in austenite (244,268).

The mechanism of increased hardenability by microalloys is not well understood. *Pickering et al.* ⁽²⁴⁴⁾ suggested that dissolved Nb would decrease both the nucleation rate and the growth rate of ferrite. It is possible that both segregation of Nb to austenite grain boundaries (in a manner similar to boron) and decreasing carbon diffusion (because



Fig. 6.5 The effect of solute Nb on the martensite start (M_s) temperature of Fe-22.5%Ni alloys.

of its strong affinity for carbon) reduce the nucleation rate. Also, both the solute drag like effect and the decreasing carbon diffusion rate decrease the growth rate of ferrite (267). It is interesting to note that, on a weight percent basis, Nb is about 10 times more effective than Mn in lowering the ferrite start temperature and, accordingly, increasing the hardenability (222).

On the other hand, it has been shown that the volume fraction of bainite increases with an increase in the level of dissolved microalloying elements in steels at the solution temperature. Figure 6.6 shows a quantitative analysis of microstructures in microalloyed direct-quenched steels ⁽²⁶⁷⁾. As can be seen, Nb has a significant effect on increasing the volume fraction of bainite. However, any thermomechanical treatment applied to the parent austenite before direct quenching into the bainitic region may change the situation. As was mentioned earlier, the effect of dissolved Nb on the austenite-to-ferrite and bainite

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Fig. 6.6 Variation of the bainite volume fraction with the amount of soluble microalloying elements at reheating temperature in direct quenched ferrite+bainite steels.

Fig. 6.7 Schematic diagram showing the effect of boron on the isothermal transformation diagram of low carbon-0.5%Mo steel.

transformation can be considered to be analogous to boron. It can be seen, in Fig. 6.7, that boron retards the heterogeneous nucleation of allotriomorphic ferrite to a greater degree than that of bainite (there is little effect on the rate of the bainite reaction). In fact, this permits boron containing steels to be cooled continuously to fully bainitic microstructures. This mostly arises from the segregation of boron at the austenite grain boundaries, thereby reducing their interface energy per unit area (269). (It was also reported that boron decreases the self diffusivity of iron at austenite grain boundaries, and possibly the carbon diffusivity, by blocking interstitial sites or by increasing the modulus of the boundary (270).) Thus, for a given cooling rate during quenching and a given isothermal holding temperature and duration, the bainite volume fraction should not be increased significantly by Nb additions, unless another factor accelerates the bainite transformation. The latter is possible by TMP, as will be shown later.

In summary, solute Nb increases hardenability and decreases the M_s temperature by solid solution strengthening of parent austenite. Therefore, for a given *TMP* schedule, austenite in a microalloyed Si-Mn TRIP steels has a higher propensity to be retained or stabilized. This is firstly due to the inhibiting effect on carbide precipitation (higher hardenability), as seen in the *CCC* results in *Chapter 4*, and secondly through the presence of dissolved microalloying elements leading to solid solution strengthening in the austenite, therefore reducing the M_s temperature. Basically, strengthening of the austenite by solid

solution hardening (266), and, accordingly, the increase of elastic constants of the austenite (271-273), hinders both the nucleation as well as the growth of martensite.

6.3 The Austenite Grain Size

As was shown in section 5.1.1.2, the austenite grain size had two opposing effects on the retention of austenite at the end of processing. In the case of steel B, [Fig. 5.8b], decreasing the prior austenite grain size resulted in a higher volume fraction of retained austenite. By contrast, the retained austenite volume fraction decreased with a smaller prior austenite grain size of steel A, [Fig. 5.8a].

In order to discuss the effects of austenite grain size on the retained austenite characteristics, the changes in the austenite transformation characteristics due to the variations of the austenite grain size must be well understood, because the state of the parent austenite is influenced by its transformation products. Moreover, it is not actually the *prior* austenite which is retained at room temperature, but it is the metastable austenite which has been stabilized by a variety of mechanisms. Thus, before discussing the effects of austenite grain size on the transformation products, the different mechanisms of austenite stabilization will be summarized.

6.3.1 Parameters Affecting the Austenite Stability

It was emphasized in the results chapter that, during the processing of TRIP steels, the characteristics of the parent austenite are changed. Examples of this are as follows: repeated static recrystallization [Fig. 5.7] resulted in finer prior austenite grain size; dynamic recrystallization (especially when followed by a faster cooling rate), section 5.1.2, and pancaking of parent austenite, [Fig. 4.10] and section 5.2, produces different densities and configurations of deformation substructure; ferrite formation characteristics (volume fraction, size and morphology) during intercritical processing, section 5.4, may change the chemistry and morphology [Fig. 5.35] of the prior austenite. As will be discussed in the following, all of these can affect the stability of the parent austenite.

Generally, austenite stability has been shown to depend on austenite composition (274), grain size of the *remaining* austenite (particle size) (275), austenitizing parameters (139,149-151), cooling rates (138,169), specimen thickness (274), applied stress (133,138,143,145), pressure (276), austenite-to-martensite-to-austenite reverse cycling (277), and thermal arrest (thermal stabilization) whilst cooling (278). Austenite

stability, in fact, can be considered as a part of the kinetics of the martensitic transformation, in that it can be considered to retard the nucleation and propagation events of the latter.

The fundamentals of the mechanisms of austenite stabilization are, thus, based on how the aforementioned parameters may influence the nucleation and growth of martensite. Since the martensitic transformation progresses by shear, the rate of martensite formation is lower when the austenite resistance to shear is higher. Microscopic features, such as grain size and austenite fragmentation, and submicroscopic characteristics, such as the nature and distribution of defects, have been invoked to qualitatively explain austenite stability^(139,149-151,275). These mechanisms will be covered more deeply at the appropriate points in the discussion.

6.3.2 The Effect of Austenite Grain Size on Subsequent Transformations

It is well known that the transformation kinetics (*i.e.*, hardenability) of steels can be affected by the austenite grain size (279). Essentially, transformation to ferrite or pearlite is nucleated at grain boundaries. Therefore, the greater the grain boundary area per unit volume (*i.e.* the finer the austenite grain size), the greater will be the density of nucleation sites and the lower the hardenability.

As was shown in Fig. 5.26, the refinement of the prior austenite grain size from $230\mu m$ to $40\mu m$ led to completely different *CCC* behaviour in steel A. For example, the finer austenite grain exhibited a lower flow strength during *CCC* testing. In addition, it was emphasized that the Ar_3 was increased by the decrease in the prior austenite grain size. The latter is consistent with the results of *Ouchi et al.* (280). The austenite grain size influences the morphology of the transformation products, as well. For example, a coarser austenite grain size retards ferrite formation to lower temperature, leading to widmanstätten side plates, or even intragranular widmanstätten plates, instead of grain boundary allotriomorphs. This is indicated in Fig. 5.26 by the deviation due to the transition in ferrite morphology from polygonal to polygonal+acicular. The latter was not encountered in finer austenite. In the case of pearlite, while its rate of growth is independent of the austenite grain size, the nucleation rate increases with increase in the grain boundary area ⁽²⁸¹⁾. The formation of pearlite in fine-grained austenite is, therefore, more rapid. For example, the pearlite formation temperature in steel A, [Fig. 5.26], was increased about 30°C by decreasing the prior austenite grain size from 230 μm to 40 μm .

Note that austenite with a finer grain size usually transforms to fine grained ferrite, pearlite, bainite, or martensite (282).

Increasing the austenite grain size decreases the rate of bainite formation (231-233). It is believed that the effect of austenite grain size on the rate of bainite transformation is generally smaller than that of the transformation to pearlite (232). Furthermore, it was shown that the bainite packet size decreases with austenite grain size refinement. Figure 6.8 shows the bainitic structures of steel B with different prior austenite grain sizes. As can be seen, the average packet size in the finer austenite grain structure, is smaller than that of the coarser one. The following empirical relationship (230) between the average size of the bainite packet \overline{d}_B and the corresponding average size of austenite grain \overline{d}_A has been evaluated:

$$\vec{d}_{B} = 1.4 \vec{d}_{A}^{0.55}$$
 Eq. 6.1



Fig. 6.8 The SEM of bainite packets of steel B with different prior austenite grain sizes; (a) $350\mu m$ and (b) $45\mu m$.

The austenite grain size influences the martensitic transformation in different ways. Since martensite growth relies on maintaining a certain coherency with the surrounding austenite, a high-angle grain boundary is an effective barrier to plate growth. Thus, while the austenite grain size does not affect the number of martensite nuclei in a given volume, the final martensite plate size is a function of the grain size. Another important feature of the austenite grain size is its effect on the residual stress after the transformation is completed. In large grain sized microstructures the dilatational strain associated with the transformation causes large residual stresses to be built up between adjacent grains. This can even lead to grain-boundary rupture and substantially increase the dislocation density in the martensite and its surrounding matrix. Finer austenite grain sizes, however, tend to be more self-accommodating ⁽²⁸³⁻²⁸⁴⁾.

Finally, the effect of austenite grain size on the martensitic transformation start temperature (M_s) has remained controversial. Some workers believed that the M_s temperature increases (in the range of 10 to 20°C) with increasing austenite grain size ⁽²⁸⁵⁾. Their explanations were centered around the observation that grain boundaries act as barriers to martensite propagation, and that the size of the initial plates is controlled by the grain diameter. They postulated that the amount of martensite formed is the product of the number of nuclei that propagate and the size of each martensite plate ⁽²⁰⁸⁾. The grain size effect has also been discussed in terms of the magnitude of the surface energy associated with plates of various sizes; this approach also explains a decrease in M_s with decreasing size ⁽²⁸⁶⁾. On the other hand, there are other workers who suggest that the existence of pre-embryos and dislocation substructure are the main factors controlling the M_s temperature ⁽²⁸³⁾. The effect of dislocation substructure will be discussed later in more detail.

From the above discussion, the coarser the austenite grain size, the higher the hardenability of austenite, but the effect on the martensitic transformation start temperature is very small. It was understood that coarser austenite led to higher amounts of retained austenite at the end of the process, as is true of water quenched steels (287). This may not be the case when the austenite is directly quenched from the two phase (austenite+ferrite) region into the bainitic region, as was done in the present work. For the same cooling rate and isothermal holding temperatures, coarser austenite grain sizes led to bainite morphologies closer to the lower temperature morphologies, *i.e.* thinner bainitic platelets and hence denser packets. On the other hand, as was mentioned above, the

coarser the austenite grain size, the bigger the size of bainite packets [Fig. 6.8]. This, in turn, results in coarser austenite particles trapped in between the bainitic packets, which are more prone to transform to lower bainite or martensite on subsequent cooling to room temperature. Thus, the lower amounts of retained austenite in the coarse-grain austenite observed in steel B [Fig. 5.8b] occur for the following reasons: (i) the larger and denser bainitic packets reduce the total interpacket and interlayer material; (ii) the coarser austenite particles are more likely to transform to martensite, as will be explained in the following section. This rationale, however, does not follow in the case of steel A because of the presence of Nb. Instead, coarser austenite grain sizes lead to higher levels of dissolved Nb because of the effect of deformation, which was used in prior austenite grain refining, on promoting the kinetics of Nb(CN) precipitation.

Although a decrease in the prior austenite grain size increased the retained austenite in steel B, the total elongation did not follow this trend. This is an indication of a higher stability of retained austenite in the coarser austenite grain size, because of a higher carbon concentration and probably because of enclosure in a more compact bainite morphology.

6.3.3 Effect of Austenite Particle Size on the Austenite Stabilization

One of the most important objectives in thermomechanically processing TRIP steels was to refine and morphologically change the prior austenite grains. This austenite is then changed by subsequent transformations of austenite during cooling to room temperature. For example, the size of prior austenite is changed (refined) by polygonal ferrite formation and growth at the austenite grain boundaries [Fig. 5.27], or by acicular ferrite [Fig. 5.37] and bainite formation [Fig. 5.45] within the austenite grains. The latter results in austenite fragmentation, *i.e.* the production of austenite particles with smaller sizes. Furthermore, bainite was the last desired transformation in all the *TMP* schedules used in this work, and it too produces small austenite particles. Thus, a fundamental explanation of the stability of austenite in the form of small particles is necessary.

It has been generally observed that austenite stability increases as the austenite particle size decreases. This was not rigorously understood in early papers, but it was postulated that the reason would likely involve both martensite nucleation and growth. Subsequently, it was suggested ^(275,288) that, when a fixed volume of austenite is divided into discrete regions, it is possible to isolate regions which do not contain an effective martensite nucleation site. It is also possible that, even when an effective martensite

1.1

nucleus is present (e.g., the stacking faults in the austenite particle), the austenite particle can be stabilized by the matrix constraints resulting from the partially coherent nature of the ferrite-austenite interfaces.

In subsequent work (98,289,290), it was shown that the austenite-to-martensite transition in small particles (below 5 µm) does not occur because the particles do not contain the crystal defects necessary to nucleate the martensite transformation. Even when the transformation nucleated in a particle, it did not always proceed to completion because the elastic constraints of the surrounding matrix increase too rapidly as the martensite lath thickens (291) These ideas were supported by the observation that the martensitic transformation could be nucleated wherever dislocations, introduced by plastic deformation, intersected the particles^(290,292). This was consistent with the early work of Wayman⁽²⁹³⁾, who showed that deformation of the microstructure would introduce deformation structure into the retained austenite particles, which could serve as a martensite nucleus. Deformation of the material would also produce strain gradients in the ferrite at the ferrite-austenite interface. These strain gradients could effectively reduce the constraints of the partially coherent interface, by reducing the amount of ferrite deformation required to maintain compatibility across the interface during the shape change accompanying the transformation of an austenite particle to martensite (293).

Rigsbee explained the increase in the stability with decreasing particle size by the probable higher level of chemical partitioning (Mn to remaining austenite and Si to ferrite), and by the increased effectiveness of the interface (relative to the volume of the particle) in constraining the particle against the required particle shape changes accompanying a martensitic transformation (50).

As a summary, the higher stability of smaller austenite particle can be related to the chemical stabilization by Mn, insufficient defects for nucleation and inhibition of growth by plastic constraint.

6.4 The Influence of Dynamic Recrystallization

As was mentioned in section 5.1.2, in the metals which undergo dynamic recrystallization, a poorly recovered dislocation substructure develops in the initial stages of deformation, in which the substructure cells have a relatively small size and more tangled walls, compared to structures which result from a high degree of recovery (239). Broadly speaking, the defect density in dynamically recrystallized austenite is higher, and the dislocation substructure is more heterogeneous than in the statically recrystallized one (241). In order to evaluate the effects of these differences in the substructure on the retained austenite characteristics, static and dynamic recrystallization schedules, Figs. 5.1 and 5.2, were applied to the specimens.

As a brief review of the results, Figs. 5.11 and 5.17, for steel A, can be compared. The change in the recrystallization mechanism from static to dynamic at 1050°C did not affect the retained austenite volume fraction. This can be explained by the presence of a substantial driving force for recovery of the substructure at 1050°C. The decrease in the recrystallization temperature, however, resulted in the observed higher volume fraction of retained austenite after dynamic recrystallization.

Since the prior austenite grain size was the same for both dynamically and statically recrystallized cases at 1000°C, the two major differences between these austenite structures, are: (i) the level of solute Nb in steel A (the effect of Nb was discussed already, *section 6.2*), and (ii) the substructures. With regard to Nb levels, during dynamic recrystallization, austenite undergoes continuous straining and, as was shown previously in Fig. 5.15, this will likely take the Nb out of solution. Dynamically recrystallized austenite should therefore exhibit less retained austenite. To more thoroughly specify the effect of dynamic recrystallization substructure, some specimens were cooled faster to the next *IMP* stage (isothermal holding in the two phase region). This allowed a substantial part of the substructure of the dynamically recrystallized austenite to be retained to the two phase region. The results, as were illustrated in Fig. 5.17, revealed that the faster cooled specimen retained a higher volume fraction of austenite. To rationalize these results, the different features of dynamic recrystallization substructure and their effects on the parent austenite stabilization (changes in the M_s temperature) will be discussed below.

Figure 6.9 shows the three types of dislocation density distribution developed in a dynamically recrystallized microstructure (195). It has been shown that these can be

retained to room temperature ⁽²³⁹⁾. Thus, if the specimen is cooled quickly enough after dynamic recrystallization, the higher temperature substructures and defects will be included in the subsequent structure. This would be encouraged in the presence of Nb, with regard to pinning by precipitates and solute drag effects. The effect of substructures on the austenite stabilization (reducing M_s temperature) was discussed in *Chapter 2 (section* 2.8.3). Referring to Eqs. 2.6 and 2.8, an increase in dislocation density, *i.e.*, the conditions after dynamic recrystallization especially after the faster cooling rate, decreases the strain energy per unit length of dislocation, Γ_s , which would reduce the embryo sizes. As was explained, the decrease of embryo size in this way leads to a higher non-chemical free energy (the term $\Delta G_{Non-Chem}^{\gamma \to \alpha'}$ in Eq. 2.4), and this leads to a lower M_s temperature.



Fig. 6.9 Three stages of dislocation density distribution developed in DRX grain structures; (a) DRX nucleus, (b) Growing DFX grain and (c) DRX grain undergoing strain hardening. d_o initial (annealed) dislocation density, d_c critical value required for dynamic nucleation, D current DRX grain size, present state presented by full lines, one or more earlier states by broken lines ⁽¹⁹⁵⁾.

As was illustrated in the corresponding results section (section 5.1.2), the present results support the ideas of the effects of substructure on austenite stabilization. By having some part of the substructure of dynamically recrystallized specimen retained, the yield strength of the remaining austenite is increased, and the retained austenite volume fraction is higher. Furthermore, the parent austenite substructure, which can be inherited to some degree by other transformation products (e.g. bainite), also increased the subsequent UTS. This can be seen in Fig. 5.18, where the UTS in the specimen with more retained austenite is higher than that of the one with less retained austenite.

6.5 The Effect of Pancaking

The effects of austenite deformation in the no recrystallization (pancaking) region on the retention of austenite and its characteristics were shown in *sections* 5.2, 5.3 and 5.4.5.2. The variation of retained austenite volume fraction with strain in the no recrystallization region always exhibited a maximum, [Figs. 5.20 and 5.39]. The presence of this maximum results from two opposing responses of the retained austenite volume fraction to changes in the strain in the no recrystallization region. They in turn can be divided into direct and indirect effects, the latter via the subsequent transformation characteristics of the prior austenite. As was mentioned earlier, it is the latter that mainly dictates the state of the retained austenite at the end of the process. In the following, the important microstructural features of prior austenite after pancaking will be outlined. This will then be followed by a consideration of the effect of pancaking on the *subsequent* austenite.

6.5.1 Microstructural Characteristics of the Pancaked Austenite

The deformation of austenite in the no-recrystallization region leads to the formation of pancaked austenite grains. Among the austenite features (e.g., grain size, composition, etc.), the substructure is of vital importance, since it determines the mechanism and magnitude of the effects on the subsequent transformations. These can be qualitatively observed in Figs. 4.9 and 4.10. The former shows an example of the effects on the extent of transformation to ferrite (section I.2 of Chapter 4). The latter is an example of the influence of pancaking substructure on the preferred nucleation sites of the subsequent transformation (to ferrite).

Apart from the overall increased dislocation density and serrated grain boundaries *(section 5.2)*, the deformation structures, *i.e.*, the local heterogeneities introduced by deformation, in hot deformed austenite, can be generally classified into three types, based on their morphological appearance. These are *deformation bands* (or *annealing twins* which have lost their coherency with the matrix during pancaking), *microbands* and heterogeneities near the grain boundary regions (181,201,294).

The morphology, density and distribution of deformation structures are expected to change according to the deformation conditions. It is clear that the density of these defects increases with increasing amounts of deformation and decreasing finishing temperature. The latter can be observed in Fig. 5.23b, which illustrates the effect of finishing temperature in the no recrystallization region on the retained austenite volume fraction for steel B. As can be seen, a decrease in the finishing temperature resulted in a higher retained austenite volume fraction. Since every other relevant parameter was kept constant, this effect can be qualitatively linked to the higher density of defects in the deformation structure. The prior austenite grain size has been shown to influence the characteristics of these features. Since the grain boundary area per unit volume of a specimen increases with decreasing prior austenite grain size, it was found that strains are accommodated (concentrated) mainly at, or close to, the grain boundaries in fine grained austenite ⁽¹⁸¹⁾. The deformation structures therefore appeared not to develop appreciably in fine grained austenites compared to coarse grained ones.

In addition to the above, a sharp rolling texture is present in the pancaked austenite. Accordingly, the stored energy of deformation depends upon the orientation of austenite. It is well known that the components of the austenite rolling texture transform at different rates ⁽²⁹⁵⁾. Thus, transformation of the pancaked austenite would be expected to proceed inhomogeneously.

Having evaluated the deformation structure in pancaked austenite, the next step, in relation to the processing steps, is to understand the effects of these local heterogeneities on ferrite and pearlite formation.

6.5.2 Transformation of Pancaked Austenite to Ferrite and Pearlite

Since the lattice adjacent to the deformation structure is highly strained and severely distorted, these become preferred sites for ferrite and pearlite nucleation. They influence not only the kinetics of formation, but also the morphology and size of these transformation products. This can be better appreciated by referring to Figs. 4.10 and 4.19, where the presence of deformation structures led to different amounts (*i.e.* kinetics), [Fig. 4.10], and size of ferrite, [Fig. 4.19], formed from pancaked austenite. For a given composition, the effective nucleation area per unit volume (S_{ν} , which includes both grain boundaries and the boundaries of severely distorted local inhomogeneities, mostly deformation bands) increases as the finishing temperature is decreased and total reduction (ψ) below the no recrystallization temperature is increased. Therefore, for a given undercooling, the nucleation frequency increases with increasing ψ . Consequently, a transformation during cooling should start earlier as the degree of prior austenite reduction is raised. This effect is clearly documented in previous work (202,217,296). Two examples of the variation of the Ar_3 with the degree of austenite deformation below the no

recrystallization temperature are also shown in Fig. 6.10. As can be seen, even small

deformations of austenite in the norecrystallization region evidently cause a marked increase in ferrite nucleation frequency.

It has also been shown (85) that pearlite nucleation can be affected by deformation in the no recrystallization region, occurring much earlier than in strain free austenite. In the latter, the preferred nucleation sites for pearlite nodules are triple junctions of grain boundaries, while in pancaked austenite the preferred nucleation sites are at grain boundaries as well as at triple junctions. This trend of acceleration of transformation was also followed by the





steels which were used in this study. For example, the variations of the Ar_3 and Ar_1 with the amount of strain above 800°C for steel C, obtained by CCC testing, are tabulated in Table 6.1. As can be seen, the increase in prestrain led to higher values for both the Ar_3 and Ar_1 temperatures. It is worth mentioning that the relative importance of nucleation sites for transformations is considered to depend on the amount of deformation. The austenite grain boundaries are the most preferred sites for relatively low levels of deformation, whereas annealing twin boundaries and deformation bands become more important with increasing deformation in the no recrystallization region.

 Table 6.1
 Variations of the ferrite and pearlite formation temperatures with strain above 800°C for steel C

Prior Austenite Grain Size	Strain above 800°C	Ar ₃	Ar ₁
350µ	15%	775±5	635±5
350µ	25%	785±5	650±5

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In the series of tests which were performed to investigate the effects of pancaking on the retained austenite, the above effects on the ferrite and pearlite formation characteristics were taken into consideration through knowledge of the Ar_2 and Ar_1 temperatures measured by CCC testing. In the hot deformation schedule, the isothermal holding temperature in the two phase region was increased to 700°C to avoid undesired pearlite formation (during isothermal holding and subsequent quenching into the salt bath), and the holding time was decreased when the strain in the no recrystallization region was increased to compensate for the increase of the Ar_3 . As was mentioned in the corresponding results section (section 5.2), this led to almost the same amount of ferrite in the pancaked specimens. No pearlite was seen in the microstructure by direct quenching the sample from isothermal holding in the two phase region into the salt bath. The only difference between the specimens deformed to different pancaking strains was the ferrite grain size, which decreased with increasing strain. As was shown in section 5.4.4, the effect of ferrite grain size on the retained austenite, compared to the other parameters, is not as significant. This will be discussed in more detail in section 6.6.3.

6.5.3 The Transformation of Deformed Austenite-to-Bainite

The effect of the level of pancaking on the microstructure of steel A was shown in Fig. 5.22. As was pointed out *(section 5.2)*, the increase in the amount of strain in no recrystallization region resulted in an increase in the interlayer thickness of upper bainite and also an increase in the fraction of lower bainite. Also, as was illustrated in Figs. 5.20a and 5.21, the corresponding mechanical properties, in particular total elongation, did not follow the same trend of retained austenite volume fraction. These results can be rationalized, partly by the effect of deformation substructure on the bainite transformation characteristics and, therefore, the austenite stabilization, and partly by its effects on the martensitic transformation. The latter is the subject of the next section but the former will be discussed below.

It has been shown that the deformation of austenite in the no recrystallization region, or prior to transformation, retards the decomposition of austenite-tobainite (147,297). This was related to the mechanical stabilization of austenite due to the increase of defect density by pancaking or ausforming (the deformation of austenite in the metastable region between the ferrite and bainite curves of the *TTT* diagram). In the case of the latter, it was shown that samples of deformed austenite resulted in lower maximum volume fractions of bainitic ferrite, in comparison with those transformed from undeformed austenite. This is in spite of the fact that the deformed austenite transformed initially at a higher rate (298) to bainite. This effect was not reported to appear during transformation at higher temperatures, presumably because the total volume fraction of bainite that can form is then reduced. Stabilization, therefore, is only manifested when the regions of austenite which are easily transformed are exhausted, these being regions left unchanged by the inhomogeneous deformation (*e.g.*, pancaking). The non-uniformity of stabilization is reflected in the microstructure that develops. In this case, the sheaves of the bainite which do form were found to be aligned along specific directions within individual austenite grains, in contrast with the usual bainitic transformation where sheaves grew in many different orientations (299).

In Si(>1%)-Mn steels. Bhadeshia and Edmonds ⁽²⁷⁾ noted that separate bainite C-curves can be distinguished. Figure 6.11 represents the TTT curve of upper and lower Fe-0.43%C-3.00%i. -bainite for 2.12%Si. Since the carbon, silicon and manganese content of the steels used in this work are lower, the boundary temperatures of upper and lower bainite and also M_s temperature are shifted to higher values. The existence of separate regions for upper and lower bainite in the steels used in this work is





supported by referring to the microstructures of specimens held at 500°C and 300°C in the salt bath, [Fig 5.45], which shows two completely different morphologies of bainite related to upper and lower bainite.

By comparing the microstructures of the pancaked specimens together with one which was not pancaked, it can be seen that the increase of strain in the no-recrystallization region resulted in upper bainite with thicker platelets and a higher volume fraction of lower bainite, [Figs. 5.5 and 5.22]. These are indicative of a higher temperature morphology of upper bainite, and a higher amount of remaining austenite after isothermal holding in the salt bath (which is subsequently transformed to the lower bainite), respectively. This effect of strain can be easily rationalized by referring to Fig. 6.11. As can be seen, by assuming that the upper bainite C-curve is moved to lower temperatures by

pancaking, for the same isothermal transformation temperature in the bainite region, the aforementioned changes in bainite morphology can be obtained.

By referring to Figs. 5.20, it appears that the best bainite characteristics, to achieve more retained austenite and higher concentrations of carbon in the retained austenite, were attained with 20% pancaking. However, the total elongation values did not follow this trend, [Fig. 5.21], but continuously decreased with increasing pancaking strain. This strongly supports the above discussion of the shift in the bainite C-curves by pancaking, because the higher the pancaking strain, the greater the lower bainite volume fraction, the latter decreasing the formability⁽³⁰⁰⁾. The decrease of carbon concentration in the retained austenite of the highly pancaked specimen (Fig. 5.20c) can be related to the increase of carbide precipitation, probably by the increase of the lower bainite volume fraction in the microstructure. This postulate is based on the *TEM* observation of the upper bainites formed after 2 mins. holding at 400°C. No carbide precipitation was observed in the latter, but the presence of carbide in lower bainite was always seen (this will be demonstrated in more detail in *section 6.7*).

Once the bainite transformation ceases, the microstructure possesses some austenite with the stability necessary for retention at the end of processing. As was mentioned already, one of the commonly accepted criteria for austenite stability is the magnitude of M_s temperature. The lower the M_s temperature, the higher the stability of austenite and therefore the higher the amount of retained austenite. In the present case, in which pancaked austenite has undergone the bainitic transformation, two separate categories of conditions affect the stability of austenite (i.e. the M_s temperature) at the The first emanates from bainite transformation characteristics, time of retention. (section 5.6). The formation of bainite not only divides the parent austenite into the smaller particles with different morphology (section 5.6), size and carbon concentration which have their own mechanisms of stability (section 6.3.3), but introduces some dislocations (substructure) in to the remaining austenite in its vicinity (because of the shear mechanism of formation). The second is due to the deformation substructure which has survived in the austenite remaining from the pancaking period. This and the resultant substructure from the bainite transformation have their own particular effects on the stability of austenite. The latter can be examined by measuring the M_s temperature of the remaining austenite or by measuring the volume fraction of retained austenite after various amounts of tensile deformation on the sample at room temperature. Neither of these was in the scope of the present work.

Based on the discussion of section 2.8.3, pancaking increases the strength of austenite by work hardening, and can therefore be considered to decrease the M_s temperature. However, as the stability of the retained austenite was not in the scope of the present work, there is no evidence of the effect of pancaking on the M_s temperature of the austenite at the time of retention. By referring to the effects of austenite strengthening on the martensitic transformation, the following may be postulated. Although the deformation structures of pancaked austenite can be partly inherited by bainite laths (301), the dislocation density of the remaining austenite remains the same. In addition, the shear transformation of austenite-to-bainite, and its required accommodation by plastic deformation, introduces more substructure to the remaining austenite. On the other hand, the higher temperature substructure of pancaked austenite will be more 'passive' than the lower temperature ausformed one, with respect to inhibiting the martensitic transformation. This may be concluded by assuming higher levels of recovery and segregation at higher temperatures, generally decreasing the free energy of the pancaked austenite. In addition, recovery could lead to subgrains with relatively high angle boundaries. This, in turn, can lead to martensitic transformation suppression, analogous to the austenite small particle effect (section 6.3.3). At higher temperatures, the higher diffusion rate and segregation of alloying elements to the grain and deformation structure boundaries can lead to the poisoning of martensite embryo formation sites via localized strengthening of the austenite.

The deformation temperature in the no recrystallization region is important, since the lower the temperature, the lower the degree of restoration of deformed structure. However, changing the pancaking temperature in steel A, Fig. 5.23, did not affect the retained austenite volume fraction significantly. This can be related to the presence of Nb(CN) precipitation in the microstructure. The decrease in carbon concentration of the retained austenite in steel A is due to the increase in lower bainite volume fraction and therefore higher driving force for carbide precipitation. The latter effects influence the corresponding mechanical properties by decreasing the total elongation and increasing the UTS, (Fig. 5.24). The change of pancaking temperature in steel B, however, reduced the extent of restoration, thereby leading to a higher amount of retained austenite.
6.6 The Effect of Ferrite

The effects of the condition of ferrite on the retained austenite characteristics were mentioned in *section 5.4*. It was shown that there was an optimum amount of ferrite which resulted in the observed higher volume fraction of retained austenite, [Fig. 5.28]. On the other hand, it was pointed out that, although this optimum ferrite quantity appeared to be independent of prior austenite grain size [Fig. 5.30], the trend of the variation in retained austenite volume fraction with ferrite quantity was changed for an intermediate prior austenite grain size [Fig. 5.30]. Furthermore, the volume fraction of retained austenite was found to be significantly affected by a change in ferrite morphology, [Fig. 5.36].

Generally, the presence of ferrite in the microstructure is a factor which imparts ductility and elongation to the structure. For example, as was previously mentioned (*Chapter 2*), for dual phase steels, the presence of a highly ductile ferrite matrix is imperative for superior elongation $^{(67)}$. The necessary volume fraction of ferrite in the microstructure of these steels, in order to attain more than 25% ductility, is about 80% $^{(302)}$. In the case of *Si-Mn* TRIP steels, since formability is not the only required mechanical property, these guidelines concerning ferrite are only of limited application. On the other hand, in TRIP steels, the proeutectoid ferrite has an important role in dictating the subsequent transformations. The different aspects of thermomechanically formed proeutectoid ferrite which affect the retained austenite characteristics will be discussed below.

6.6.1 Ferrite Quantity

The changes in the retained austenite characteristics (mainly volume fraction) with ferrite quantity, (section 5.4.2), and with the combined effects of prior austenite grain size and ferrite quantity [Figs. 5.29 and 5.30], can be rationalized as follows. Generally, ferrite, which is the first transformation product of hot deformed austenite in this work, changes the composition and morphology of the remaining austenite (*i.e.* the austenite which remains after completing the transformation of the parent austenite-to-ferrite). Since the first and predominant ferrite morphology (at this stage) is the grain boundary allotriomorph (polygonal), the way in which it affects the characteristics of the remaining austenite is through its compositional and geometrical configuration. These, in turn, are affected by the factors dictating the ferrite nucleation and growth progression (isothermal temperature and holding duration, solute drag like effect, etc.).

From the point of view of geometrical configurational, the only way to stabilize the remaining austenite (apart from chemical stabilization, which will be discussed below) is by trapping the austenite through the impingement mechanism during growth of polygonal ferrite. The remaining austenite has a smaller particle size, [Fig. 5.46], thereby assisting in its stabilization, *(section 6.3)*.

In the following, the compositional variations during ferrite formation, which result in chemical stabilization, will be considered, and used to explain the effect of ferrite quantity on the retained austenite volume fraction.

During the formation of ferrite, carbon diffuses in the austenite ahead of the α/γ interface. Figure 6.12a shows a portion of the *Fe-Fe₃C* diagram, and Fig. 6.12b shows a plot of carbon concentration normal to the α/γ boundaries. In this case, since, equilibrium is assumed to be attained locally at the α/γ interface, the carbon concentrations in α and γ at this interface, denoted by $C_{\alpha}^{\alpha\gamma}$ and $C_{\gamma}^{\gamma\alpha}$, respectively, correspond to those of the equilibrium phase diagram. As can be seen, [Fig. 6.12c], by increasing the holding time or ferrite volume fraction at a given temperature the remaining austenite becomes more homogeneous with respect to carbon. Note also that the average carbon concentration of the untransformed austenite is increased, as was confirmed by the neutron diffraction results [Fig. 4.14].



Fig. 6.12 (a) A portion of the Fe-Fe₃C phase diagram, (b) Carbon concentration profile along a direction perpendicular to the austenite/ferrite interface, (c) Carbon concentration profile in front of growing ferrite at different time intervals or different volume fractions of ferrite.

Generally, there are three models concerning the growth process in Fe-C-X alloys, where X can be Mn, Si, Ni, Cr, etc. These are differentiated through the following assumptions, (i) the establishment of local equilibrium at α/γ boundaries with bulk partitioning of both X and C between γ and α , (ii) local equilibrium at α/γ interfaces with localized "pile-up" of X in front of the advancing α/γ boundaries and (iii) paraequilibrium (the situation in which the substitutional alloying elements are unable to partition during the isothermal holding period) at α/γ boundaries, with partitioning of carbon only. The best overall agreement between experimental and calculated growth kinetics and compositional profile was reported with the paraequilibrium concept (303). It should be noted that the operation of paraequilibrium growth conditions implies that the concentration of X in ferrite is indistinguishable from that in austenite. If there is significant size misfit between X atoms and Fe atoms, particularly in austenite but also in ferrite, X atoms will accumulate in disordered areas of austenite-ferrite boundaries during growth (this is known as solute drag-like effect, SDLE). The "sweeping up" by-a-movingboundary mechanism of X segregation in the SDLE theory is one basic difference with respect to the familiar solute drag effect operative during grain growth and recrystallization, wherein X atoms diffuse to a stationary boundary with volume diffusioncontrolled kinetics.

Assuming the existence of paraequilibrium growth conditions, because of the short isothermal holding times used in this investigation, it is the carbon (at this stage) which is mostly dictating the stability of remaining austenite not the other alloying elements such as Mn. The carbon enrichment of austenite associated with the increase of ferrite volume fraction, via rejection of carbon into the remaining austenite, results in an increase of austenite stability against the subsequent transformations. This, in turn, would help to retain higher levels of the austenite at room temperature. Increasing the ferrite volume fraction beyond a certain level, however, appears to decrease the retained austenite volume fraction. From metallographical examinations, such as shown in Fig. 6.13, this can be related to either the shift of pearlite C-curve to the left or approaching the pearlite C-curve during the isothermal holding period in the corresponding *TTT* diagram (an increase in the chance of pearlite formation). Since pearlite consumes the carbon, the stability of the remaining austenite is reduced, lowering the volume fraction of retained austenite.

As was illustrated in Figs. 5.29 and 5.30, a different behaviour of the retained austenite volume fraction with the amount of ferrite was found for an intermediate prior austenite grain size. The increase of retained austenite volume fraction beyond a given



Fig. 6.13 (a) The typical microstructures of steel A after long isothermal holding duration at 650°C; (b) Color-etched micrograph corresponds to the same specimen.

amount of ferrite (*i.e.* beyond a certain holding time) in the specimens of intermediate austenite grain size (Fig. 5.30) can be rationalized as follows.

Since no microstructural changes (apart from ferrite quantity) were observed in the corresponding specimens during the intercritical treatment, another chemical stabilization factor (in addition to carbon) may be influencing the austenite retention. As already noted, it has previously been understood $(^{304})$ that no partitioning of X between austenite and ferrite occurred at any temperature from the Ae_3 to the Ms temperature, when X was Si, Mo, Co, Al, Cr and Cu. This was a consequence of the paraequilibrium $\gamma/(\alpha + \gamma)$ boundary lying very close to the orthoequilibrium or actual equilibrium $\gamma/(\alpha + \gamma)$ boundary. Thus, the temperature range between these two boundaries was too small to permit sufficient undercooling to be achieved to allow formation of partitioned ferrite at detectable rates. In the case of Mn, Ni and Pt alloying additions, however, partitioning was observed above a characteristic critical temperature, [Fig. 6.14] (³⁰⁵). As is well known, the partitioning of any alloying element depends on the diffusion characteristics.

Chapter 6 DISCUSSION

The latter is a function of temperature, time, concentration gradients and diffusion path and diffusion extent. From the above discussion, it appears that, in the steels which were used in this work, the latter parameters led to partitioning of Mn to the remaining austenite in the case of intermediate prior austenite grain Since Mn is an austenite size. stabilizer, the diffusional (e.g., pearlite) and martensitic transformations are both retarded.



Fig. 6.14 Ratio of atom fraction Mn in ferrite to that in austenite for ferrite allotriomorphs.

leading to a higher amount of the retained austenite for intermediate prior austenite grain size at the end of process. This means that the enrichment and homogenization of the remaining austenite with respect to Mn, compensated for the effect of excess carbon of the remaining austenite, avoiding pearlite formation in the specimens with higher amounts of ferrite (as was observed for the other prior austenite grain sizes).

6.6.2 Ferrite Formation Temperature

As was pointed out in section 5.4.3, a comparison between the data in Table 5.2 and Fig. 5.28 revealed that an increase in the isothermal holding temperature in steel A led to a reduction of retained austenite volume fraction. In the case of steel B, however, there was no indication of significant change of retained austenite volume fraction with change in ferrite formation temperature. These findings will be explained in the following paragraphs.

It was stressed in *Chapter 2* that the temperature at which ferrite forms is important during the intercritical annealing of dual phase steels. As was shown in Fig. 2.3, the volume fraction of the retained austenite in dual phase steels initially increased with increasing annealing temperature and, after reaching a maximum, decreased at higher annealing temperatures. Since, in those tests [Fig. 2.3], only the temperature was varied without changing the holding time, the results can be rationalized as follows. As the temperature increases (for a constant isothermal holding duration), the amount of austenite increases, thus providing more initial austenite to be retained. The factors which dictate the stability of austenite in this stage were considered to be increasing enrichment of austenite with respect to C and Mn, and the austenite particle size. Beyond the maximum, [Fig. 2.3], with increasing isothermal temperature the amount of austenite increased along with the austenite particle size, the latter destabilizing the austenite.

The above rationale is not immediately applicable to this work. Since the ferrite volume fraction is the parameter which most influences the magnitude of C and Mn rejection to the remaining austenite, the change in isothermal holding temperature in the two phase region, for a constant ferrite quantity, should not change the retained austenite volume fraction. This is consistent with the results of steel B for medium amounts of ferrite (<35%).

On the other hand, since ferrite growth is a parabolic function of time, a much longer isothermal hold would be required (especially at higher temperatures), for a given ferrite volume fraction. (For example, compare the time duration needed for 30% and 40% ferrite in steel A or B, [Fig. 5.25].) Longer times can result in a more enriched and homogeneous austenite with respect to C and Mn, thereby leading to an austenite with higher stability. This occurred in the case of steel B with more than 40% ferrite after an isothermal treatment at 700°C. By comparing the results of Table 5.2 with Fig. 5.28, for the same amount of ferrite (~45%), the retained austenite volume fraction is higher with increasing isothermal hold temperature. These results are consistent with those mentioned in the previous section and those of Sakuma et al. (34). This means that the temperature of isothermal treatment in the two phase region becomes a dictating factor when a higher amount of ferrite (~35%, in the case of steel B) is required.

The inverse behaviour in steel A, (*i.e.*, a decreasing retained austenite volume fraction with increasing temperature), can be related to the presence of Nb in this steel. As already mentioned, an increase of the temperature leads to an increase of holding time to attain the same amount of ferrite. On the other hand, the higher the isothermal holding temperature, the higher the amount of Nb(CN) precipitation (closer to the nose of *PTT* (precipitation-temperature-time) C-curve) [Fig. 5.15]. Accordingly, by decreasing the solute Nb in austenite, as was shown earlier, the stability of austenite decreases via a decrease in austenite flow strength.

6.6.3 Ferrite Grain Size

As was shown in *section 5.4.4*, ferrite refinement by means of pancaking resulted in a decrease in the retained austenite volume fraction. However, this was somewhat increased by applying dynamic transformation. The latter even more effectively refined ferrite. Theoretically, the smaller the ferrite grain size, the greater the total area of the austenite/ferrite interface. This, in turn, increases the rate of carbon and Mn diffusion during the ferrite growth, thereby leading to faster partitioning of alloying elements. Based on this assumption, *Kawano et al.* (29,248) related the amount of retained austenite obtained at the end of process to the ferrite grain size. In this important work, the effects of pancaking on the subsequent transformations of austenite and its stability were not taken into consideration. Therefore, an unresolved question was the actual magnitude of the ferrite grain size effect on the retention of austenite. This became clear from the results of present work.

Since the polygonal ferrites formed from pancaked austenite do not inherit the pancaking substructures, dislocation pipe diffusion is not significantly involved in the overall diffusion rate in the ferrite. On the other hand, the pancaked austenite can itself affect subsequent transformations and, therefore, retention of austenite, as discussed in section 6.5. It was pointed out earlier, and also tabulated in Table 5.3, that the decrease in the ferrite grain size, for a given ferrite volume fraction, did not lead to higher amount of retained austenite. However, there is some increase in the retained austenite volume fraction by straining in the two phase region (during transformation). Lowering the finishing temperature to the temperature of austenite-to-ferrite transformation (i.e. leading to 'dynamic' transformation, where the transformation occurs due to undercooling in the presence of plastic deformation), increases the rate of transformation, thereby decreasing the ferrite grain size more significantly than pancaking. It also increases the strength of ferrite by work hardening. The latter is a very important factor in increasing the rate of diffusion and thus accelerating C and Mn partitioning into the remaining austenite. Since the deformation during the austenite-to-ferrite transformation is distributed between ferrite and austenite (although it is mostly concentrated in ferrite), the remaining austenite has a less dense substructure for the same amount of straining in the no recrystallization region. The austenite remaining after dynamic transformation is therefore less favorable (with respect to pancaked austenite) for accelerating the pearlite transformation. It is worth mentioning that, although the effects of dynamic transformation and the ferrite grain size

refinement on the retained austenite volume fraction are consistent with the results of *Kawano et al.*, the mechanisms, as explained above, differ.

6.6.4 Ferrite Morphology

In section 5.4.5, it was shown that the change in ferrite morphology from polygonal to acicular resulted in austenite fragmentation, [Fig. 5.37]. Figure 6.15 shows another view of austenite fragmentation by

acicular ferrite in large austenite fragmentation by acicular ferrite in large austenite grains. As can be seen, the fragmentation effectively refines the austenite particle size, assisting in the retention of a higher volume fraction of austenite, as was illustrated in Fig. 5.36. It was pointed out that the presence of acicular ferrite in the microstructure of steel A resulted in a significant increase in the retained austenite volume fraction. However, this trend was not followed by steel B. An explanation for these results is given below.

Referring back to the thermodynamic condition for martensitic transformation (section 2.8.3, Eq. 2.3), for the Fe-Cr-C system, it was shown that ΔG_{Strain} is only 5% of $G_{Chem}^{\alpha} - G_{Chem}^{\gamma}$ and hence is not an important factor in controlling the M_s temperature ⁽¹⁵²⁾. The initial value of ΔG_r (increase in interfacial



Fig. 6.15 Color-etched microstructure of steel B in which prior austenite grain (350µm in size) has been fragmented by acicular ferrite.

energy per mole of martensite) is referred to as $\Delta G_{r.s.}$, and depends on the austenite grain size. The smaller the austenite grain size, the greater the ΔG_r . The temperature at which the driving force $G_{Chem}^{\alpha} - G_{Chem}^{\gamma}$ exceeds $\Delta G_{r.s.} + K.E.$ determines the M_s temperature. However, in the presence of a large amount of acicular ferrite, the remaining austenite is blocked off into small volumes by the existing acicular ferrite. Thus, the average size of the plates formed in these small particles must be smaller than those formed before fragmentation. Thus, ΔG_r now will be larger than the initial $\Delta G_{r.s.}$. The reaction will proceed only when the temperature is lowered to a point where $G_{Chem}^{\alpha} - G_{Chem}^{\gamma}$ exceeds the new value of $\Delta G_{r.s.} + K.E.$. This process proceeds as more acicular ferrite forms and the austenite regions become reduced further in size. This indicates the important role of austenite fragmentation in stabilizing.

The response of steel A, *i.e.* the increase of the retained austenite volume fraction, to the parent austenite fragmentation by acicular ferrite, therefore, follows the above rationale. The results of steel B are contradictory because of the presence of pearlite in its microstructure. Since the pearlite transformation start temperature of steel B is higher than that of steel A, (Table 4.5), the same procedure (cooling rate, Fig. 5.34) to obtain acicular ferrite resulted in pearlite formation in steel B. This decreased the carbon in the remaining austenite, leading to a reduction in the retained austenite volume fraction.

The last important point is the effect of acicular ferrite on the fragmentation of *pancaked* austenite. As was shown in Fig. 5.39, pancaking and acicular ferrite were combined and led to a significantly high volume fraction of the retained austenite. But the latter did not result in any extraordinary improvements in mechanical properties. This is firstly because of the presence of more acicular ferrite instead of polygonal ferrite and, secondly, the special pools for retained austenite (mostly enclosed by acicular ferrite, Fig. 5.37) in the corresponding microstructure. The former resulted in a relatively lower formability, since the formability of acicular ferrite is lower than that of polygonal ferrite. The latter, as will be discussed in more detail later in *section 6.8*, led to decreased effectiveness of TRIPing.

6.7 Bainite Characteristics

The effects of bainite characteristics were evaluated in section 5.5. It was demonstrated that the change in the isothermal holding period at 400°C resulted in a variation in the retained austenite volume fraction at the end of process, [Fig. 5.40]. The results showed an optimum holding time for retained austenite volume fraction in both steels A and B. The corresponding total elongation values, [Fig. 5.41], followed the same trend exhibited by the retained austenite volume fraction. A change in the bainite formation temperature led to a small change in the retained austenite volume fraction when the temperature was decreased from 500°C to 400°C [Fig. 5.43]. However, a significant decrease was seen for the specimens treated at 300°C. These results will be discussed in this section.

6.7.1 The Effects of Isothermal Hold Duration

Increasing the holding duration from 2 to 5 min. at 400°C, as was shown in Fig. 5.42, resulted in less lower bainite in the microstructure. This means that more transformation took place at the holding temperature, decreasing the austenite available to transform to lower bainite during subsequent air cooling. The drastic increase in the retained austenite volume fraction with increasing holding time can be related to the increase of carbon concentration in the remaining austenite due to the carbon rejection from bainitic ferrite. This, in turn, leads to a higher volume fraction of the retained austenite at the end of the process.

An increase in the holding duration (e.g., beyond 5 mins. in the present case), however, resulted in a decrease in the retained austenite volume fraction. The *TEM* of the corresponding specimen revealed carbide precipitation at the boundaries of bainitic ferrite platelets, [Fig. 6.16]. The carbide phase associated with upper bainite precipitates from the carbon enriched austenite and is almost always cementite. As a consequence of the transformation to bainite, the austenite which is trapped between platelets of upper bainite, as was emphasized above, is enriched in carbon. If the carbon concentration of the remaining austenite exceeds the value given by the extrapolated $\gamma/(\gamma + \theta)$ phase boundary [Fig. 6.17] then cementite precipitation from the enriched austenite lying adjacent to the platelets of bainitic ferrite, becomes thermodynamically possible ⁽²³³⁾. The shaded area in this figure represents austenite which is unstable with respect to the precipitation of cementite. If it is additionally assumed that bainite growth stops when the



Fig. 6.16 TEM of upper bainite carbide precipitation (dark field).

Schematic illustration of the thermodynamic condition which has to be satisfied before cementite may precipitate from the remaining austenite.

carbon concentration of the remaining austenite exceeds the T'_0 phase boundary ¹, then, subject to kinetics, carbide precipitation is expected to accompany the growth of upper bainite, if the transformation temperature is below T_c , [Fig. 6.17]. The latter is expected to coincide with the conditions (holding duration and temperature) of specimens held for a prolonged (10 min.) time at 400°C. Consequently, the sequence of the aforementioned reactions can be summarized as follows:

$$\gamma \rightarrow \gamma + \alpha_{b,supersaturated} \rightarrow \alpha_{b,unsaturated} + \gamma_{enriched} \rightarrow \alpha_{b,unsaturated} + \alpha + \theta$$

where the α refers to the secondary ferrite which forms as the carbides precipitate from the remaining austenite. In other words, prolonged holding leads to carbide precipitation, which destabilizes the austenite. It should be noted that the morphology of the cementite formed at the lath boundaries [Fig. 6.16] is dependent on the carbon content of the steel. In low carbon steels (*e.g.*, steels used in this work), the carbide will be present as

and the T_o curve modified to account for this stored energy is called the T'_o curve.

¹The T_0 curve describes the locus of all points in the phase diagram where austenite and ferrite of the same chemical composition have identical free energies. For bainite, whose growth is accompanied by an invariant-plane strain shape deformation, the strain energy of transformation is about 400 J mol⁻¹ (233),

discontinuous stringers and isolated particles along the lath boundaries, as is visible in Fig. 6.16, while at higher carbon levels the stringers may become continuous (306).

The corresponding mechanical properties are in good agreement with the aforementioned phenomena. As was seen in Fig. 5.41, the maximum retained austenite volume fraction and its carbon concentration coincide with the maximum total elongation. On the other hand, the *UTS* increases with increasing holding duration, at first because of the increase in the retained austenite volume fraction and, in the later stages, due to the presence of very fine carbides in the microstructure.

Following on from this discussion, it is worth comparing the effects of holding duration in the two phase region, Figs. 5.25 and 5.28, and in the bainite formation range, Fig. 5.40. The variation in the retained austenite volume fraction showed a maximum in both cases. The increase in the retained austenite volume fraction with hold time before reaching the maximum, in both cases, was related to the rejection of carbon to the remaining austenite. Furthermore, the subsequent decrease was explained by the start of carbide precipitation, *i.e.* pearlitic carbide in the former case and bainitic carbide in the latter. Finally, it should be mentioned that the carbon content is much lower in the bainitic ferrite. Therefore, the remaining austenite enclosed by bainitic ferrite is more enriched with carbon, and thus more stable, than that of proeutectoid ferrite.

6.7.2 The Temperature of Isothermal Holding

It is well known that the variation of isothermal holding temperature in the bainitic region leads to a change of bainite morphology. The presence of two separate C-curves in *TTT* diagrams of *Si-Mn* bainitic steels, as was reported by *Bhadeshia* and *Edmonds* (27), indicates two distinct morphologies for upper and lower bainite. These morphologies are clearly differentiated in this work. As is visible in Fig. 5.45, the morphology of the upper bainite bears a close resemblance to widmanstätten ferrite, since it is composed of long ferrite platelets free from internal precipitation.

A decrease in isothermal temperature, as can be seen in the same figure, resulted in a decrease in the apparent thickness of the bainite platelets, again in good agreement with the literature (307,308). The lower bainite (formed at 300°C) appears more acicular, with finer platelets than the upper bainite. It is worth mentioning that, in general, isothermal transformation in both the lower and upper bainite temperature ranges results in an aggregate of bainitic ferrite and retained austenite. The retained austenite volume fraction, however, was much lower in the case of the lower bainite. This can firstly be related to the higher austenite-to-bainite transformation rate at lower temperature and secondly to the ease of carbide precipitation at lower temperature. This is consistent with *Hehemann's* (106) work.

Figure 6.18 shows the *TEM* of precipitated carbides in the bainitic ferrite platelets. This illustrates a striking microstructural characteristic of lower bainite, *i.e.* the growth of carbide rods within the bainitic ferrite plates. These carbides have a given growth direction and an orientation relationship with the ferrite which is identical to that found in tempered martensite. However, in contrast to tempered martensite, the cementite particles in lower bainite exhibit only one variant of the orientation relationship, such that they form parallel arrays at about 60° to the axis of the bainite plate (233,306).

As was shown in Fig. 5.44, the best mechanical properties were found in specimens treated at 400°C. Although, in steel A, the retained austenite volume fraction was not significantly changed and the retained austenite carbon concentration was reduced by decreasing the hold temperature from 500°C to 400°C, the total elongation is higher for



Fig. 6.18 TEM of lower bainite carbide precipitations; (a) bright field and (b) dark field.

the latter temperature. This emphasizes the effect of retained austenite particle size. As has been well established, the bainitic packet size and the width of laths for a given austenite grain size decrease with decreasing transformation temperature (300,309). These, in turn, can lead to finer blocky retained austenite trapped in between bainitic packets and also result in thinner retained austenite films enclosed by bainite platelets. The reduction of total elongation on passing from upper to lower bainite can be related to the decrease of the retained austenite volume fraction on the one hand and, on the other hand, to the presence of carbides in the microstructure. The latter contributed to the relatively large increase in the UTS, [Fig. 5.44].

One more point worth mentioning is the almost identical elongation behaviour of the higher (500°C) and lower (300°C) temperature specimens. Although the retained austenite volume fraction is lower in the latter case, it possesses a finer retained austenite particle size along with a much higher carbon concentration, [Fig. 6.19]. Thus the two structures have comparable ductilities.



Fig. 6.19 Variation of the retained austenite carbon content of steel A with hold temperature in the bainite region.

6.8 Retained Austenite Morphology and Mechanical Properties

Throughout this thesis, it has been emphasized that the volume fraction of retained austenite is not the only parameter that affects the final mechanical properties. The morphology, distribution, size, dislocation substructure and chemical composition of the retained austenite also play important roles. The most important factor, however, is morphology, since the others are directly (e.g., size) or indirectly (e.g., distribution) influenced by, or predicted from, the morphology.

Most of the retained austenite reported in this work was finally formed under the influence of bainite transformation characteristics. Accordingly, the prevailing retained austenite morphologies were types 3 and 4 (*i.e.*, blocky and interlayer). The type 3 or blocky regions of austenite trapped between sheaves of bainite has been recognized to be relatively unstable (310-315) (for a given particle size), and undesirable. It appears that the blocks of retained austenite tend to transform to high carbon untempered martensite under the influence of small stresses and, consequently, can impart an embrittling effect. The films of the retained austenite that are trapped between the platelets of ferrite in a sheaf, however, are much more stable. The latter is partly because of their higher carbon concentration and also because of the physical constraint against transformation due to the close proximity of plates in all directions.

By assuming that a fraction, ϕ , of a packet is comprised of films of retained austenite, it was demonstrated that the ratio of the volume fractions of film and blocky austenite (prior to any martensitic transformation) can be given by ⁽³¹⁶⁾:

$$(V_{R,A})_F/(V_{R,A})_B = (\phi V_{\alpha})/(V_{R,A} - \phi V_{\alpha})$$

where $(V_{R,A})_F$ and $(V_{R,A})_B$ are the volume fractions of film and blocky type retained austenite respectively, and V_{α} and $V_{R,A}$ are total volume fractions of bainitic ferrite and retained austenite, respectively. When the microstructure is composed of just bainite and retained austenite, it was found, experimentally, that high strength and good toughness could be obtained by maintaining the above ratio to a value greater than 0.9 (310,311). The question then arises as to the factors which control this ratio for the steels used in this work. Based on an overview of the present results, the way to minimize the volume fraction of blocky austenite is to lower the transformation temperature to permit the bainite reaction to proceed. This should be limited to a temperature at which the rate of carbide precipitation is not so high.

As was shown, the greatest elongation was observed when the specimen was held at an intermediate temperature (400°C) during bainite transformation. This may be due to the fact that, at this temperature, the retained austenite is present mainly in the form of films between platelets of bainite, rather than as blocky regions between sheaves of bainite. This greater ability of interlayer retained austenite to increase the elongation has also been demonstrated by *Sandvik et al.* (308).

The variations of Mn in the different retained austenite morphologies, found in this work, are consistent with the results of *Bangaru* ⁽¹⁷⁾. Since partitioning is thus observed, the paraequilibrium condition appears not to be completely fulfilled. This can be related to the prior austenite history, the low cooling rate from the bainite formation temperature to room temperature and the presence of proeutectoid ferrite in the microstructure. The latter can change the balance of alloying elements in the austenite before transformation.

6.9 Control of the Microstructure

In the previous sections, the various microstructures, which were produced by different TMP, and the related mechanical properties, were discussed. In each case, knowing the steel composition and from the information generated by CCC testing, a thermomechanical processing schedule was designed to obtain particular microstructures. Generally, the microstructure at the end of processing was composed of ferrite, bainite, retained austenite and martensite. In some cases, the presence of pearlite in the microstructure was also observed, Fig. 6.14. Based on the results of this work, the optimization of microstructural constituents in TRIP steels can be directed in two ways. The first concerns the overall effects of micro-constituent characteristics (type, quantity, size and morphology) on dictating the volume fraction and the stability of retained austenite (*i.e.* the state of the retained austenite). The second one is the mechanical properties of the final microstructure and their dependence on the variation of microstructural component characteristics.

6.9.1 Retained Austenite Characteristics

The relevant retained austenite characteristics are volume fraction, chemical composition, morphology, size and distribution. As was shown in *Chapter 5*, each of these could be influenced by changes in the condition of the other microstructural components. For example, an increase of polygonal ferrite quantity resulted in a higher volume fraction of retained austenite, Figs. 5.28 and 5.29. The latter decreased beyond an optimum ferrite volume fraction, (~35%). In addition, increasing the amount of polygonal ferrite beyond an optimum led to retained austenite of the more undesirable type 1 morphology, Fig. 5.46. The change in the morphology of ferrite from polygonal to acicular, however, resulted in different characteristics (volume fraction and morphology) of the retained austenite. Therefore, the acicular structure is the optimum morphology of ferrite to obtain a higher volume fraction of retained austenite, although there is a chance of pearlite formation. The latter can be overcome by a proper processing schedule and/or alloying (*e.g.*, Nb, B, Mo, etc.).

Bainite, which is the main micro-component in the reported microstructures, also exhibited an optimum morphology, Fig. 5.43, and quantity, Fig. 5.40. As was explained in *section 5.5*, the optimum morphology of bainite to achieve the desired characteristics of retained austenite was the one obtained at 400°C, with an intermediate hold time (~5 min.). It should be noted that these conditions are based on a microstructure containing $30\pm3\%$ polygonal ferrite before the bainite transformation. These conditions would be modified if the balance of the other microstructural components changes.

6.9.2 Optimization of Mechanical Properties

Generally, a modification of microstructure is performed to optimize mechanical properties, and these are, in turn defined by the subsequent application of the product. Si-Mn bainitic TRIP steels in the form of sheet have been used mostly in the automotive industries, for automatic structural press forming parts, bumpers, reinforcement parts, doorguard bars, *etc.* The steels for these applications must possess high strengths and good formability. For some forming operations, *e.g.* press forming, formability in TRIP steels can be evaluated by the product of tensile strength and total elongation, $(TS \times T. EL)$. The higher the value of this parameter, the better the formability. If the volume fraction of microstructural components (*e.g.*, bainite, ferrite and retained austenite) is kept the same, the highest value of $TS \times T$. EL is obtained when the retained austenite stability is optimised. The production of Si-Mn TRIP steels in the form of bar and rod and using them to make for example high strength fasteners, (bolts, nuts, studs, steering links, tie rods, *etc.*), also demands similar mechanical properties. (In fact, the results of this work will be used to develop an appropriate composition and *TMP* schedule for fasteners.)

The mechanical properties, reported in *Chapter 5*, were obtained mainly by changing the *TMP* parameters, keeping the balance of microstructural components constant. For example, for steel A with about 30% polygonal ferrite and 9.5% retained austenite, Fig. 5.9, the value of $TS \times T$. *EL* is 46,250 MPa.%, (37%×1250 MPa). Changing the ferrite morphology from polygonal to acicular altered this to 33,000 MPa.% (30%×1100 MPa), section 5.4. Thus, although acicular ferrite and pancaking, [Fig. 5.39], increased the retained austenite volume fraction by about 50% (from 9.5% to 14.1%), the overall mechanical properties decreased by 40%. This was due to the lower stability of retained austenite and the lower formability of acicular ferrite in comparison to polygonal ferrite (233).

Bainite characteristics could always be altered to optimize mechanical properties [Figs. 5.41 and 5.44]. For example, for a structure with ~30% polygonal ferrite, the optimum combination of elongation and strength (61180 MPa.%) was obtained at 400°C with 5 min. hold time. The optimum bainite transformation is a function of the microstructure of interest (e.g. quantity of polygonal ferrite). The important point, however, is that the properties arising from TRIPing are dictated by the bainite formation characteristics. The latter rely on the microstructure from which the bainite forms and the *TMP* schedule. If other mechanical properties, not dependent on TRIP properties, are required, (e.g., higher strength or formability), the characteristics of the other microconstituents must be changed, and these will in turn alter TRIP properties, or will require changes in *TMP* to maintain TRIP properties.

CHAPTER 7

CONCLUSIONS

In the present work a new test technique, based on continuous cooling deformation testing, was developed for investigating microstructural changes during hot deformation. Using this method, the austenite microstructural, mechanical and transformation characteristics, which evolve during continuous cooling, can be followed by applying constant strain rate deformation during cooling, and analyzing the subsequent flow stress behaviour. The analysis, which is based on changes in the flow behaviour due to the microstructural variations that may or may not accompany the stages of transformation, revealed the following points:

-The occurrence of dynamic recrystallization in the austenite recrystallization region (above the T_{nr}) is revealed (as a hump) during CCC testing. Since dynamic recrystallization is very sensitive to the conditions of TMP (e.g., cooling rate, strain rate, starting microstructure and the steel composition), the position of the hump in the CCC curves depends on the steel composition and CCC conditions. Accordingly, this deviation would not necessarily appear in all cases.

-The start of the austenite no-recrystallization region (revealed by a significant decrease in the rate of dynamic restoration) coincides with a sudden increase in the slope of the CCC

curves (second deviation). This is not observed in the steels designated as *Class II* because of the lower level of alloying elements and hence the lack of solute drag and precipitation effects. The rate of work hardening (strain accumulation) below this deviation is more pronounced in steels containing Nb than the others. The latter shows that Nb exerts both solute drag and precipitation effects on the restoration stop processes.

-For deformation bands and twins to become preferential sites for ferrite nucleation, strain accumulation in the vicinity of these boundaries in the austenite no-recrystallization region must take place.

-CCC testing reliably defines the changes in the mechanical properties of the austenite in the metastable state. The resulting analysis of the flow behaviour was confirmed by metallography and neutron diffractometry.

-Metastable austenite appears to be associated with a higher flow stress or a higher strain hardening component with respect to the stable state.

-Neutron diffractometry at high temperatures indicated a faster rate of contraction of the austenite lattice as the temperature decreases through the metastable state, compared with that observed through the stable austenite region.

-The concept of solute carbon clustering can be utilized to explain the changes in the austenite lattice parameter and strengthening as the temperature passes through the equilibrium temperature of the austenite-to-ferrite transformation.

-The increase in the carbon content retards the Ae_3' to lower temperatures and also leads to a retardation of the lattice parameter contraction below the Ae_3' .

-The austenite lattice parameter reduction during cooling reveals a leveling off of the austenite lattice parameter after formation of a given amount of ferrite, which is a confirmation of carbon rejection to the remaining austenite.

-The austenite to ferrite transformation in Class II steels occurs at a faster rate than that of Class I steels because of the solute element additions in the latter. This leads to a drop in the flow stress at the Ar_3 in Class II steels, whereas the Class I steels only exhibit a decrease in the rate of flow stress increase with temperature. However, the Ar_3

temperatures in the *Class II* steels are not always higher than those of the *Class I* steels, suggesting that the solute elements affect growth rates more than nucleation rates.

-The larger the austenite grain size, the lower the Ar_3 temperature.

-If the strain accumulation in the vicinity of deformation bands and twins is not sufficient to make them equivalent to grain boundaries (with respect to ferrite nucleation) at the Ar_3 , these will eventually become preferential sites for ferrite nucleation below the Ar_3 .

-The formation of acicular ferrite in Class I steels is due to the postponement of all stages of the austenite transformation to lower temperatures. The deviation which is related to such acicular ferrite formation disappears on changing the austenite grain size from $230\mu m$ to $40\mu m$.

-Changes in the ferrite morphology can affect the CCC curve because of variations in the kinetics of ferrite formation.

-The end of the austenite-to-ferrite transformation (start of pearlite formation) results in a very rapid increase in the rate of strengthening below the Ar_3 . The increase in alloying element concentration, in particular Nb, decreases the start temperature of pearlite formation in both *Classes* of steels.

-Continuous deformation below the pearlite formation temperature accelerates the rate of cementite spheroidization. The start of pearlite spheroidization coincides with a decrease in the rate of strengthening. The latter is not observed in *Class II* steels, since the pearlite fraction in the microstructure of these group of steels is not as significant. Further continuous deformation in *Class II* steels leads to coalescence of the cementite spheroids and eventually results in cementite penetration through the ferrite grain boundaries. This is accompanied by an increase in the rate of work hardening far below the pearlite formation temperature, instead of softening.

The present investigation on the transformation characteristics of Si-Mn TRIP steels after thermomechanical processing resulted in the following conclusions:

-In the different grades of TRIP steels used in this work, when the prior austenite grain size and polygonal ferrite quantity are kept constant, the V_{RA} differences between the

other grades are not so significant (less than 10%) and can be attributed to the differences in their carbon and silicon levels. The higher the Si and C concentrations, the higher the retained austenite volume fraction. The addition of Nb to the same composition, however, resulted in a 30% increase in V_{RA} and an increased total elongation. The UTS was higher in steel A, the Nb bearing steel, than steel B (Nb free grade).

-A decrease in the austenite grain size in steel A from $230\mu m$ to $70\mu m$ led to a 22% reduct on of the V_{RA} . The carbon concentration of the retained austenite, however, remains more or less constant. In the case of steel B, the effect of austenite grain size on the retained austenite volume fraction is opposite to that of steel A. The largest grain size in steel A not only resulted in the highest amount of retained austenite, but also led to the largest total elongation. In steel B, however, an optimum retained austenite volume fraction (obtained for intermediate prior austenite grain size) was observed which maximises the mechanical properties. The most important accommodation sites (pools) for the retained austenite (*i.e.* bainite interlayer and inter packets) did not vary with prior austenite grain size. However, the bainite packet sizes changed with prior austenite grain size. The smaller the prior austenite grain size, the smaller the bainite packet size.

-The change in the finishing temperature of steel A from 1050°C to 1000°C (above the T_{nr}) resulted in a decrease in the amount of retained austenite by 20%. On the other hand, no significant effect on the retained austenite volume fraction was found in steel B. Lowering the finishing temperature in steel A reduced the V_{RA} of steel A to a level close to that of steel B. In both steels, total elongation decreased with the lower finishing temperature. For steel A, this coincided with a decrease in V_{RA} . Although the level of retained austenite was almost the same for both temperatures in steel B, there was a significant change in the mechanical properties.

-Promoting Nb(CN) precipitation by the change in *TMP* conditions resulted in a decrease in the V_{RA} . This was attributed to a reduction of Nb in solid solution and therefore a decrease in parent austenite stability.

-The dynamically recrystallized austenite substructure, when retained to transformation temperatures, is more prone to retaining austenite (more V_{RA}). The variation of total elongation correlates with that of the retained austenite volume fraction change. The UTS in the rapidly cooled material, which retained the dynamically recrystallized austenite

substructure, is still higher than that of the slowly cooled structure, even though the amount of retained austenite is higher in the former.

-The variation of the retained austenite volume fraction with strain in the norecrystallization region always exhibited a maximum. For both steels A and B, although the ferrite quantity was not the same, the maximum V_{R4} was observed at about 20% strain. The total elongation, in the case of steel A, does not change systematically with the retained austenite volume fraction. The ultimate strength, however, attains its maximum at about 20% deformation, coinciding with the maximum V_{R4} . The increase of the strain in the no-recrystallization region leads to an increase in the interlayer thickness of upper bainite platelets as the degree of pancaking increases. On the other hand, the fraction of lower bainite appears to increase as the strain increases.

-In both steels A and B, the n value in the *Avrami* equation exhibits a maximum for intermediate prior austenite grain size, and is higher for steel B.

-By increasing the amount of ferrite, the retained austenite volume fraction, after a slight plateau, decreases. Furthermore, it appears that there is an optimum amount of ferrite which maximises the amount of retained austenite. The existence of an optimum ferrite fraction which leads to a maximum amount of retained austenite, appears to be repeated for all austenite grain sizes. An increase of the V_{RA} beyond a certain ferrite fraction (in the case of steel A, ~50%) occurs for an intermediate austenite grain size. This behaviour also takes place in the other steels.

-Increasing the temperature of ferrite formation in steel A leads to a reduction of V_{RA} . In the case of steel B, however, there is no significant effect of temperature.

-The results of this work show that a decrease in the ferrite grain size by pancaking does not increase the retained austenite quantity, contrary to the conclusions of *Kawano et al.* (29,248). On the other hand, deformation during dynamic transformation not only has more effect on ferrite grain refinement, but also has more influence on the retained austenite volume fraction compared to pancaking.

-The presence of acicular ferrite in steel A resulted in a significant increase in the quantity of retained austenite. Further, in both steels, the martensite/retained austenite phases are mostly enclosed by fine acicular ferrite or coarse granular bainite. Essentially, the prior austenite grains have been fragmented by these phases, and the distributions of these phases are dictated by this grain fragmentation. In any case, by retaining the dynamically recrystallized substructure or applying pancaking, for a given amount of ferrite, the volume fraction of retained austenite of the specimen with acicular ferrite is much higher than structures with polygonal ferrite only. Note, however, that the mechanical tests on the sample with 14% retained austenite volume fraction did not show extraordinary mechanical properties.

-The increase in the isothermal holding time during bainite formation leads to a maximum V_{RA} . The total elongation appears to coincide with the changes in the V_{RA} , but the tensile strength increases continuously with holding time at 400°C.

-The variation of V_{RA} with the temperature of the bainite hold appears to follow the results of previous workers, who demonstrated a maximum at 400°C for V_{RA} . The same maximum can also be seen in the total elongation. The tensile strength, however, continually decreases with increasing bainite hold temperature. In addition, an increase in the bainite hold temperature resulted in the increase of lath (platelet) size and interlayer thickness.

-For the Si-Mn steels investigated in this work, the morphology of retained austenite can be classified into five different categories: (1) blocky shape enclosed by polygonal ferrite, (2) blocky and/or plate shape enclosed by acicular ferrite, (3) blocky shape enclosed by bainite packets, (4) interlayer residual phase between: (a) upper bainite platelets, (b) intermediate temperature bainite platelets, (c) lower bainite laths, (5) interlayer film enclosed by martensite laths.

Statement of Originality and Contribution to Knowledge

The present work includes the following original contributions:

A novel mechanical method, based on continuous cooling deformation testing, was developed for investigating microstructural changes during hot deformation. A major attraction of this test technique is that the specific microstructure, for which the transformation characteristics are required, can be generated by hot deformation, and then immediately evaluated by continuous cooling deformation.

For the first time, it was shown that the mechanical properties of the austenite change while passing from the stable to the metastable state. This was indirectly confirmed by metallography and neutron diffractometry. It appeared that metastable austenite was associated with a higher flow stress or a higher strain hardening component with respect to the stable state.

Neutron diffractometry at high temperatures was utilized to monitor the austenite lattice parameter variation with temperature. This indicated a faster rate of contraction of the austenite lattice as the temperature decreases through the metastable state, compared with that observed through the stable austenite region. To explain the changes in the austenite lattice parameter and strengthening as the temperature passes through the equilibrium temperature of the austenite-to-ferrite transformation, the concept of solute carbon clustering was applied. Although the latter is an old concept, illustrating the agreement between theory and experiment, in particular, the employment of neutron diffractometry at high temperatures is an original approach.

A Si-Mn and Nb bearing TRIP steel was used for the first time and therefore all the results pertaining to this steel are original.

The prior austenite grain size obtained by different *TMP's* (*RCR* or *DRCR*) has opposing effects on the retained austenite characteristics when a microalloying element Nb is added. When Nb is kept in solution, it strongly increases the retained austenite volume fraction.

It was shown that, if the dynamically recrystallized austenite substructure is retained to transformation temperatures, a higher volume fraction of retained austenite is obtained.

The variation of retained austenite volume fraction with strain in the norecrystallization region always exhibited a maximum. An increase of strain in the norecrystallization region leads to an increase in the interlayer thickness of the upper bainite platelets as the degree of pancaking increases. On the other hand, the fraction of lower bainite appears to increase as the strain increases.

The dynamic transformation of ferrite was shown to be more effective than pancaking on ferrite grain refinement and increasing the amount of retained austenite.

It was shown that the prior austenite grains can be fragmented by acicular ferrite, and the distributions of the retained austenite are dictated by this grain fragmentation. In any case (regardless of the *TMP* conditions) for a given amount of ferrite, the volume fraction of retained austenite of the specimen with acicular ferrite is much higher than structures with polygonal ferrite only.

Recommendations for Future Work

As was frequently emphasized throughout this thesis, the deformation characteristics and the stability of retained austenite during forming processes were not in the scope of this work. These can be reasonably deduced from a consideration of the present results. However, a systematic investigation of the stability of retained austenite in as-thermomechanically processed microstructures is highly recommended. This may be done by applying deformation (*e.g.*, uniaxial tension) at room temperature and then measuring the variation of retained austenite volume fraction with the amount of strain. These data can be then used to model the stability of retained austenite.

Since retained austenite measurement demands a special specimen preparation procedure (if x-ray diffraction is used), the geometry of the as-hot deformed and the cold worked material is important. On the other hand, for a closer approach to industrial processing conditions, the best simulation of such processes should be used to generate representative structures. Hot torsion and laboratory scale rolling mills are the two available tools which can be utilized to simulate industrial conditions. If the former is used, the gauge length portion of the torsion specimen can be used for uniaxial tension. In this case, because of inhomogenous deformation, neutron diffraction is recommended for retained austenite measurement. Since the latter is based on bulk penetration, no specific surface preparation (electropolishing) is necessary. On the other hand, if laboratory scale rolling is utilized, flat tensile specimens should be tested to allow easy sample preparation for x-ray diffraction.

After optimizing the thermomechanical processing schedule to obtain given desired properties, the chemical compositions of Si-Mn TRIP steels should be defined by considering the industrial demands. For example, a decrease of carbon content from 0.2% to 0.15% results in a substantial increase in the ferrite quantity and decrease in the retained austenite volume fraction. This gives higher formability at the expense of strength and TRIP properties. The results of this work on Si-Mn and Nb bearing TRIP steel shows that the presence of Nb while it is in solution plays a crucial role in austenite retention. The precipitation of Nb (which is not always completed) is not detrimental to austenite retention, but increases the strength of the material. Therefore, in a situation that a HSLA steel along with higher formability is required, the addition of Nb is recommended.

As was shown in some cases (e.g., processing for acicular ferrite formation), some amount of pearlite was observed in the Nb free steels. If the presence of a higher retained austenite volume fraction along with acicular ferrite is needed, or if there is some practical limitation for cooling between the run out table and the coiler (which is almost always in the range of pearlite formation), the addition of small amounts of boron or Mo is recommended. These retard pearlite formation during cooling by poisoning (*i.e.* by segregation leading to a lowering of the grain boundary energy of) the prior austenite grain boundaries.

There is almost no literature specifically directed to the effect of pancaking on the kinetics of bainite and martensite formation (in spite of the effects of ausforming, which is somewhat analogous to pancaking). Systematically resolving these effects will help to better understand *TMP* and, consequently, to better design the optimum *TMP* schedule.

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Appendix I

Computer Program

10	REM						
20	REM	CONSTANT TRUE STRAIN RATE INTERRUPTED COMPRESSION TEST					
30	REM						
40	REM	*******PROGRAMMED BY AB	BASS ZAREI HANZAKI*******				
50	REM						
60	REM						
70	ERASE	ERASE					
80	DIM X(50,1), Y(50,1)						
90	COMMON A(450,2),C(450,2),D(450,2),Z(450)						
95	COMMON F(15)						
100	COMMON H0, A0, D1, D2, D5, B, S0, I1, I2						
110	COMMON C1,S1,S2,S3,L0,A,J,N\$.M\$,T\$						
120	PRINT "S	PRINT "SAMPLE IDENTIFICATION"; \ INPUT N\$					
130	PRINT \PRINT "MATERIAL"; \ INPUT M\$						
140	PRINT \PRINT "TEST TEMPERATURE"; \ INPUT T\$						
150	PRINT \ PRINT "INITIAL HEIGHT (mm)"; \ INPUT H0						
160	PRINT \PRINT "INITIAL DIAMETER (mm)"; \INPUT D0 \ A0=PI*(D0)^2/4						
170	PRINT \PRINT "NUMBER OF DEFORMATIONS:"; \INPUT D5						
180	PRINT \PRINT "PRE-DEFORMATION"; \INPUT DI						
190	IF D5=1 THEN GO TO 210						
200	PRINT \PRINT "SECOND DEFORMATION"; \INPUT D2						
210	PRINT \PRINT "FIRST STRAIN RATE (1/S)"; \ INPUT SI						
215	PRINT \PRINT "SECOND STRAIN RATE (1/S)"; \INPUT S3						
220	PRINT \ PRINT "TIME INTERPASS (s)"; \ INPUT T						
230	PRINT \PRINT "LOAD RANGE (kip)"; \INPUT L \L0=L*4.4482*1000						
240	PRINT \PRINT "STROKE RANGE (mm)"; \ INPUT SO						
245	PRINT \PRINT "HOW MANY DATA DO YOU NEED ?"; \ INPUT D						
200	ERASE						
200							
200	PRINT "DATE: "; \ PRINT DAT5; \ PRINT "TIME: "; \ PRINT H;":"M						
203							
270							
275	PRINT						
200	PRINT "	FII F NAMF	·"· \ PRINT NS				
300	PRINT "	MATERIAI.	· · · · PRINT MS				
320	PRINT "	TEST TEMPERATURE (C)	."·\PRINT T\$				
325	PRINT		,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,				
330	PRINT "	NITIAL HEIGHT (MM)	;":\PRINT HO				
340	PRINT "	NITIAL DIAMETER (MM)	:":\PRINT D0				
350	PRINT		, , , , , , , , , ,				
360	PRINT "/	NUMBER OF DEFORMATIONS	:":\PRINT D5				
365	PRINT "/	NUMBER OF DATA	:":\PRINT D				
370	PRINT	· ·					
380	PRINT "/	FIRST DEFORMATION	:"; \ PRINT D1				
390	PRINT "	SECOND DEFORMATION	:"; \ PRINT D2				
400	PRINT ".	FIRST STRAIN RATE	:"; \ PRINT S1; \ PRINT "1/SEC."				

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405	PRINT "SECOND STRAIN RATE	"; \PRINT S3; \PRINT "I/SEC."				
410	PRINT "TIME INTERPASS	PRINT T; PRINT "SEC."				
420	PRINT \ PRINT					
425	PRINT "LOAD RANGE	:"; \ PRINT L; \ PRINT "KIP"				
430	PRINT "STROKE RANGE	<pre>:":\PRINT S0;\PRINT "MM"</pre>				
440	W=SYS(4)					
450	PRINT \ PRINT					
460	PRINT "PRESS RETURN TO START THE"	TEST"; \ INPUT G\$				
470	PRINT \ PRINT					
480	PRINT "STARTING THE AUTOMATIC POSITIONING OF THE PISTON"					
490	FGARB(1,"R", TIME 100,4)					
500	ADIMMED(1,A)					
510	FGGO					
520	IF A<-3.00000E-03 THEN GO TO 540					
530	GO TO 500					
540	FGSTOP					
550	S2=A*L0/A0					
560	PRINT \ PRINT "THE STRESS ON THE SAMPLE IS: ": \ PRINT S2: \ PRINT "MPa"					
570	ETIME \ SLEEP(1)					
580	ADIMMED(3,B)					
590	T1=D1/S1/50 \ T2=D2/S3/50					
595	PRINT T1.T2					
600	REM					
610	FOR I=1 TO 50					
620	H1=H0/EXP(D1*I/50)					
630	X(1.0)=(H1-H0)/S0+B					
640	NEXT I					
650	REM					
660	H2=H0/EXP(D1)+.254					
670	H=(H0-H1)/S0					
680	FOR $I=1$ TO 50					
690	$H_3 = H_2/EXP(D_2*I/50)$					
700	$Y(I_0) = (H_3 - H_2)/S_0 + 02 + (B - H)$					
710	NEXT I					
720	RFM					
722	$T_3=D1/S1 \setminus V_3=T_3/05 \setminus T_8=V_3/D \setminus T_8T_8<1$	ፐዝFN ፕՋ= 1				
776	13-D131 \ V3-13/.03 \ 16-V3/D \ IF 16-1 ITEN 18=1 TA-D2/22 \ \/A-T4/.05 \ T0-\/A/D \ IF T0-1 TIEN 18=1					
728	ער גאר אריער איין איין איין איין איין איין איין איי					
730	CKTIME(1.05)					
740	$\Delta DTIMED(1 \land 3 TS 1)$					
750	$\Delta DTMED(2 A 3 10000 1)$					
760	$\Delta DTMED(3 A 3 T0 1)$					
700	DEM CET ID THE CVCTEM					
780	ECADE UND TIME TI ADDAVY					
700	$\mathbf{FCARD}(\mathbf{I}, \mathbf{K}, \mathbf{IIIVIE} \mathbf{I}, \mathbf{ARRAI} \mathbf{A})$ $\mathbf{FCARD}(\mathbf{I}, \mathbf{R}, \mathbf{TR} \mathbf{E} \mathbf{I} 0 0 0 0 \mathbf{E} 0 2 0 1 \mathbf{I} \mathbf{R}$	עת				
900	POARD(I, K, IIIVIE I.00000E-03,02+(D-F.					
810	ADDIT ADDOUN	RI THE DEFORMATION", \ INPUT G5				
010						
02U 020	$FG51A105(1,W) \setminus IF W \le 20 \text{ THEN GO 10 820}$					
040	ADSIOP(I)					
84U	KEM					
820	IF DS=1 THEN GO TO 910					
860	ETIME / SLEEP(I)					
870						
890	FGARB(1,"R", TIME T2, ARRAY Y)					

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895 FGGO \ ADGO(3) 900 FGSTATUS(1,S) \ IF S<>0 THEN GO TO 900 910 ADSTOP \ FGSTOP \ CKSTOP 920 REM 930 FGIMMED(1,"R", TIME .5,0) 940 PRINT \ PRINT \ PRINT 950 PRINT "****** THE EXPERIMENT IS FINISHED ******"; \ INPUT G\$ 960 FOR I=1 TO A 970 REM C1 IS THE MTS COMPLIANCE 980 C2=-ELEVEL(A(I,2))*L0 990 C1=.198769*(1-EXP(-1.15596E-07*C2^2))+2.00063E-05*C2 1000 Z(I)=H0+(ELEVEL(A(I,1))-B)*S0 1010 PRINT LLOG(H0/Z(I)) 1020 NEXT I 1030 PRINT \PRINT 1031 X0=1.00000E-05 1032 FOR I=10 TO A 1033 X1=LOG(H0/Z(I)) 1034 IF X1<X0 THEN 1037 1035 X0=X1 1036 NEXT I 1037 PRINT "II=";I-1 \ II=I-1 1040 G2=-ELEVEL(A(11,2))*L0*Z(11)/A0/H0 1041 G3=.1*G2 1042 FOR J=I1 TO A 1043 X2=-ELEVEL(A(J,2))*L0*Z(J)/A0/H0 1044 IF X2>G3 THEN 1046 1045 NEXT J 1046 PRINT "12=";J \ 12=J 1049 J=1 1070 FOR I=1 TO A 1080 IF I>I1 THEN IF I<I2 GO TO 1110 1090 $D(J,1)=A(I,1) \setminus D(J,2)=A(I,2)$ 1100 J=J+1 1110 NEXT I 1120 REM 1130 ERASE 1140 PRINT \PRINT 1150 PRINT "RESULTS FROM THE EXPERIMENT:" 1160 PRINT \PRINT 1170 PRINT "INITIAL POSITION:": \ PRINT B 1180 PRINT "TOTAL NUMBER OF DATA POINTS:"; \ PRINT J 1190 PRINT "NUMBER OF POINTS FOR THE FIRST DEFORMATION:"; \ PRINT II 1195 $F(0)=B \setminus F(1)=J \setminus F(2)=I1$ 1196 $F(3)=I2 \setminus F(4)=H0 \setminus F(5)=D0 \setminus F(6)=D5 \setminus F(7)=D1 \setminus F(8)=S1$ 1197 $F(9)=T \setminus F(10)=D2 \setminus F(11)=L \setminus F(12)=S0$ 1198 F(13)=B 1200 PRINT \PRINT \PRINT 1210 W=SYS(4) 1220 PRINT "DO YOU WANT TO SAVE THE DATA"; \ INPUT G\$ 1230 PRINT \ PRINT 1240 IF G\$="N" THEN GO TO 1280 1250 OPEN "DUI:T"&N\$ FOR OUTPUT AS FILE #1

1255 OPEN "DU1:K"&N\$ FOR OUTPUT AS FILE #2

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- 1260 FOUT(1,D(1,1),,0,F)
- 1265 FOUT(2,F(1),,0,F) \ CLOSE #2
- 1270 CLOSE #1
- 1280 PRINT \ PRINT "DO YOU WANT TO SEE THE DATA"; \ INPUT G\$
- 1290 IF G\$="N" THEN GO TO 1360
- 1300 OPEN "DU1:T"&N\$ FOR INPUT AS FILE #1
- 1305 OPEN "DU1:K"&N\$ FOR INPUT AS FILE #2
- 1310 FINP(1,C(1,1),,0,F) \ CLOSE #1
- 1315 FINP(2,F(1),,0,F) \ CLOSE #2
- 1320 FOR I=1 TO J
- 1325 PRINT I
- 1330 PRINT ELEVEL(C(1,2))*L0/A0*H0/Z(1),
- 1340 PRINT Z(I),LOG(H0/Z(I))
- 1350 NEXT I
- 1355 PRINT "B=";F(0), "J=";F(1), "I1=";F(2)
- 1360 PRINT \PRINT "DO YOU WANT TO SEE THE GRAPHICS"; \ INPUT F\$
- 1370 IF F\$="Y" THEN CHAIN "GRAF5.BAS" LINE 270
- 1380 END

Appendix II

Quantitative Analysis (Multiphase) by X-Ray Diffraction

Quantitative analysis by diffraction is based on the fact that the intensity of the diffraction pattern of a particular phase in a mixture of phases depends on the concentration of that phase in the mixture⁽⁸⁶⁾. The exact expression for the intensity diffracted by a single phase specimen in a diffractometer is:

 B. D. Cullity; 'Elements of X-Ray Diffraction', Second Edition, Addison-Wesley Publishing Company, Inc., 1978, 407.

$$I = \left(\frac{I_0 A \lambda^3}{32 \pi r}\right) \left[\left(\frac{\mu_0}{4 \pi}\right)^2 \left(\frac{e^4}{m^2}\right) \right] \left(\frac{1}{V^2}\right) \left[\left|F\right|^2 P\left(\frac{1 + \cos^2 2\theta}{\sin^2 \theta \cos \theta}\right) \right] \left(\frac{e^{-2M}}{2\mu}\right)$$

- *I* : integrated intensity per unit length of diffraction line (joul/sec.m)
- I_0 : intensity of incident beam (joul/sec.m²); true intensity

A : cross-sectional area of incident beam (m^2)

- λ : wavelength of incident beam (m)
- *r* : radius of diffractometer circle (m)

 μ_0 : 4 π ×10⁻⁷(m Kg C⁻²)

- e : Charge on electron (C)
- m : mass of electron (Kg)
- V : volume of unit cell (m^3)
- F : structure factor
- P : multiplicity factor

 θ : Bragg angle

- e^{-2M} : temperature factor
- μ : linear absorption coefficient (m⁻¹)

The quantity I_0 in the above equation is a true intensity, *i.e.* energy per unit area per unit time (joule/sec.m²), and I_0A is the power of the incident beam. This means that I is the power per unit length of diffraction line in the diffracted beam. If this beam is then incident on a film or counter for a certain time, t, then the response of the film or counter is a measure of the energy I.t per unit length of the diffraction line. This is the quantity

Appendix II

commonly called the integrated intensity. A more descriptive term is the "total diffracted energy".

Direct Comparison Method

In this method we suppose that:

$$k_{2} = \left(\frac{I_{0}A\lambda^{3}}{32\pi r}\right) \left[\left(\frac{\mu_{0}}{4\pi}\right)^{2} \left(\frac{e^{4}}{m^{2}}\right) \right]$$

and

$$R = \left(\frac{1}{V^2}\right) \left[|F|^2 P\left(\frac{1 + \cos^2 2\theta}{\sin^2 \theta \cos \theta}\right) \right] (e^{-2M})$$

Then:

$$I = \frac{k_2 R}{2\mu}$$

where k_2 is a constant, independent of the type and amount of the diffracting material, and R depends on θ , hkl, and the material.

The value of C_{γ}/C_{α} can therefore be obtained from a measurement of I_{γ}/I_{α} and a calculation of R_{γ} and R_{α} . (Note that the calculation of R values requires a knowledge of the crystal structures and lattice parameters of both phases). Once C_{γ}/C_{α} is found, the value of C_{γ} can be obtained from the additional relationship:

$$C_{\gamma} + C_{\alpha} = 1$$

In choosing the diffraction lines to measure, we must be sure to avoid overlapping of closely adjacent lines from different phases. In this work, the (111) and (200) planes for austenite and (110) plane for ferrite were used. In order to precisely calculate R_{γ} and R_{α} the following computer program was utilized.

10	REM	
20	REM	**********************CALCULATION OF " R " VALUE*****************
30	REM	
40	REM	*******PROGRAMMED BY ABBASS ZAREI HANZAKI*******
50	REM	
60	REM	

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70	INPUT " TEST IDENTIFICATION	: " : M\$				
80	INPUT " MATERIAL	: " : N\$				
90	INPUT " POINT OF MAX. INTENSITY	: " ; A0				
100	INPUT " LATTICE PARAMETERS a AND b	:";A,B				
110	INPUT " MULTIPLICITY FACTOR	:";P				
120	REM " WL IS COPPER WAVE .	LENGTH X-RAY IRRADIATION "				
130	A1=(A0/2)*(3.141592654/180)					
140	WL=1.54439					
150	B1=\$IN(A1)					
160	B2=B1/WL					
170	V=A*(B^2)					
180	REM "F1 IS ATOMIC SCATTERING FACTOR "					
190	IF 0!<=B2 AND B2<=.1 THEN Z0=26-23.1; Z1=Z0*10;					
		Z2=Z1*B2 : F1=26-Z2				
200	IF .1<=B2 AND B2<=.2 THEN Z? 23.1-18.9; Z1=Z	0*10:				
		Z2=Z1*(B21) : F1=23.1-Z2				
210	IF .2<=B2 AND B2<=.3 THEN Z0=18.9-15.6: Z1=Z	0*10:				
		Z2≕Z1*(B22) : F1=18.9-Z2				
220	IF .3<=B2 AND B2<=.4 THEN Z0=15.6-13.3: Z1=Z	0*10:				
		Z2=Z1*(B23) : F1=15.6-Z2				
230	IF .4<=B2 AND B2<=.5 THEN Z0=13.3-11.6 : Z1=2	20*10:				
		Z2=Z1*(B24) : F1=13.3-Z2				
240	INPUT "SELECT 1 FOR AUSTENITE, 2 FOR FERI	RITE AND 3 FOR MARTENSITE"; K				
250	REM "F2 IS STRUCTURE FA	CTOR "				
260	IF K=1 THEN F2=16*(F1^2)					
270	IF K=2 THEN F2=4*(F1^2)					
280	IF K=3 THEN F2=4*(F1^2)					
290	REM " LPF IS LORENTZ-POL	ARIZATION FACTOR "				
300	LPF=(1+(COS(A1*2))^2)/((SIN(A1)^2)*COS(A1))					
310	REM "TFM IS TEMPERATURE FACTOR "					
320	FM=(.350810735#)*(B2^2)					
330	TFM=EXP((-2)*FM)					
340	R=(1/(V^2))*((F2^2)*P*LPF)*TFM					
350	PRINT "R-VALUE=";R					
355	PRINT B2, V, F1, F2, LPF, TFM					
356	PRINT BI					
360	M\$=M\$+".PRN"					
370	OPEN M\$ FOR OUTPUT AS#1					
380	PRINT #1 , M\$					
390	PRINT #1 ,A0,R					
400	CLOSE #1					
410	STOP					

410 STOP 420 END

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Appendix III

III.1 Shear Punch Test

Basically, the shear punch test was developed (179) to complement the ball microhardness test and to improve on some of the disadvantages of the microhardness test, in particular the sensitivity to microstructure because of small sampling volumes. The test is based on a blanking operation common to sheet metal forming. Consequently, much of the understanding of the processes involved in the shear punch test was obtained from this literature.

III.2 The Approach

The apparatus is shown schematically in Fig. III.1. It consists of an upper and lower housing. The upper housing contains a punch, and the lower one holds a die. The

central axes of the punch and die are exactly aligned in the upper and lower housings. The housings are fitted to the upper and lower rams of an MTS load frame, [Fig. 3.1]. The sample is placed at the center between the die and a washer. The latter is built in to clamp and prevent the sample from moving during punching. A flat tip punch is forced through the sample, and the action of the punch against the die shears a circular disk from the sample, completing the test. Although the punch apparatus can be constructed for either a 6.25, 3, or 1 mm-diameter punch, the 3 mm punch was chosen since TEM disks can be produced from the specimen of interest at the same time. Bv performing the punch operation in the MTS load frame, which was equipped with a load cell and a displacement transducer, the force on the punch can be monitored as



Fig. III.1 Schematic illustration of the shear punch apparatus.

a function of lower housing travel.

III.3 Specimen Behavior

An example of a load-displacement curve produced in this way is given in Fig. III.2. As can be seen, it has all the features of a load-displacement curve produced in a uniaxial tension test; that is, an initial linear region, a deviation from linearity, a nonlinear increase in load with displacement, a load maximum, and a decrease in load with displacement to the point of failure. The specimen deformations associated with these various features were analyzed, and a schematic example of this analysis is also shown in Fig. III.2. The latter was originally done by sectioning a brass sample at various points in the load-displacement curve and etching to reveal the grain structure. This in turn serves as a crude grid to demonstrate deformation. As can be seen, the point of deviation from linearity corresponds to permanent penetration of the punch into the specimen. Further deformation proceeds in a process zone approximately coincident with the annulus between the punch and the die hole. The specimen thins considerably in this region under both rising and falling load conditions, and finally the remaining ligament fails catastrophically.



Displacement

Fig. III.2 Typical load-displacement curve of a shear punch test and schematic illustrations of a specimen cross section, depicting the extent of deformation at several points of displacement.

III.4 Test and Specimen Considerations

To achieve reproducible load-displacement curves for analysis, alignment of the apparatus is critical. For instance, the point of deviation from linearity is of major interest. To obtain a distinguishable linear region from which deviation is recognizable, it is essential that the initial punch penetration of the specimens be uniform around the punch circumference. This requires that the punch be exactly perpendicular to the specimen and hence have no misalignment during shearing. This can be achieved by minimizing the tolerance between the punch and the die hole. However, punch-hole tolerance is fixed by the necessity of minimizing the contribution of punch-die friction to the load-displacement data. It was found that a 25 μm tolerance between the punch and die-hole provides adequate alignment without sacrificing load accuracy (179).

The nature of the load displacement data is, of course, a function of the punch-die geometry, particularly the punch profile and the punch-die tolerance. Attempts were made to maintain these constant and to verify this by periodically punching a set of reference materials and comparing results with standards.

III.5 Data Correlations

In order to calibrate shear-punch data with the standards, a series of shear punch and uniaxial tension tests were performed on a wide range of carbon and TRIP steels. In most cases at least six shear punch tests and three to six tensile tests were performed for each material. All tests were performed at room temperature. It was found (179) that the load at deviation from linearity, P_y , correlates well with the uniaxial yield stress, and that the maximum load, P_m , correlates with the ultimate tensile strength. The following empirical equation (179) was utilized to calculate yield stress and ultimate tensile strength:

$$\sigma_{eff} = \frac{P - F}{2\pi rt} = C\sigma$$

where

P = either the yield load or maximum load,

 $\sigma = corresponding uniaxial stress,$

F =friction load,

- r = punch radius,
- t = specimen thickness, and

C = constant

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F can be determined from the load displacement trace after the disk is punched through, or it can be determined from a plot of P versus σ as the offset at $\sigma=0$. By applying the above correlation, it was found that for the yield data, C = 0.50, and for the maximum load data, C = 0.62. The 95% confidence interval on predicted σ for a given P is $\pm 10\%$ of σ_y for yield and $\pm 5\%$ of σ_{UTS} for ultimate tensile strength. The uncertainty is somewhat larger for the yield than the maximum loads, largely because of the greater difficulty associated with determining P_y . The following empirical equation ⁽¹⁷⁷⁾ was used to calculate the total elongation:

$$El_{TOTAL} = \frac{d_f}{t}$$

where $El_{TOT,U}$ is total elongation and d_f is displacement at failure. It should be mentioned that the above empirical equation was originally found for area reduction calculation. But the results of this work on the calibration and correlation of punch data with uniaxial tension ones manifested that the (d_f/t) ratio corresponded to the total elongation, (T. El± 5%).