Effect of Chromium Addition on the Warm Rolled Behaviour of Low Carbon Steels

by

Nadia Diane Romani

Department of Metals & Materials Engineering McGill University Montreal, Canada June 2003

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Abstract

The warm rolling of steels is becoming an increasingly common practice in industry. Nevertheless, the poor formability of warm rolled low carbon (LC) steels is problematic, as this property is important in deep drawing applications, which require uniform deformation. Compression tests were conducted to study the effect of chromium content on rate sensitivity (m), using two experimental LC-chromium alloys and an extra-low carbon (ELC) steel. Samples were tested at various temperatures and strain rates, and the flow stress was measured for each condition. A second dynamic strain aging (DSA) peak was present in both LC-Cr alloyed steels, indicating that chromium raised the DSA temperature range into that for warm rolling. At these temperatures, the chromium addition reduced m as compared to the ELC steel, increasing the shear band content of the warm rolled microstructures. This increased shear band content modified the type and distribution of the recrystallization nuclei. Thus, it is expected that LC-Cr steels will have better formability after warm rolling than unmodified LC steels.

Résumé

Le laminage ferritique à chaud des aciers devient de plus en plus commun en industrie. Malgré tout, la formabilité d'aciers à bas carbone (BC) laminés à chaud en domaine ferritique est problématique, car cette propriété est importante lors de l'emboutissage, qui requiert une déformation uniforme. Des essais en compression ont été faites pour évaluer l'effet de la concentration en chrome sur la sensibilité à la vitesse (m), en utilisant deux aciers BC alliés au chrome et un acier à faible teneur en carbone (FTC). Les échantillons ont été testés à différentes températures et vitesses de déformation et la contrainte de déformation a été mesurée pour chaque condition. Un deuxième pic de vieillissement est présent pour les deux aciers BC alliés au Cr, indiquant que le chrome agrandie la fenêtre de température de vieillissement dans le cas du laminage ferritique à chaud. À ces températures, les additions de chrome réduisent m en comparaison avec l'acier FTC, augmentant le volume des bandes de cisaillement des microstructures laminées dans ces conditions. Cette augmentation de la quantité des bandes de cisaillement modifie le type et la distribution des sites de recristallisation. Ceci étant dit, il est attendu que les aciers BC-Cr auront une meilleure formabilité après le laminage à tiède que les aciers BC non-modifiés.

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List of Symbols

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ODF	Orientation distribution function		
PLC	Portevin-LeChatelier		
Q^*	Activation energy barrier to recrystallization		
Q _{def}	Activation energy for deformation		
Q _{diff}	Activation energy for solute diffusion		
R	Gas constant		
R-value	Strain ratio		
\overline{R}	Average strain ratio		
R _{0°}	Strain ratio measured along the rolling direction		
R _{45°}	Strain ratio measured at 45° to the rolling direction		
R _{90°}	Strain ratio measured at 90° to the rolling direction		
RD	Rolling direction		
SiC	Silicon carbide		
SSA	Static strain aging		
Т	Absolute temperature		
t	Final thickness		
t ₀	Initial thickness		
t	Time		
t _i	Incubation time		
<i>t</i> _{0.50}	Time to 50% recrystallization		
w	Final width		
\mathbf{w}_0	Initial width		
$X_{ u}$	Volume fraction transformed (recrystallization)		
Z	Zener-Hollomon parameter		
α	Ferrite, constant		
γ	Austenite		
Δ	Finite change		
ΔR	Planar anisotropy		

8	Strain
ė	Strain rate
E _{adj}	Adjusted strain
ε _{CM}	Apparent strain associated with the compression machine
E _{eng.}	Engineering strain
π	Pi
σ	Stress
$\sigma_{\text{eng.}}$	Engineering stress
σ_p	Peak flow stress
$\sigma_{x_{\star}}$	Standard deviation

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Chapter 1

Introduction

The warm rolling of steels within the ferrite (α) range (650-800°C) is becoming an increasingly common practice in the steel industry, as it is very cost effective. Its major advantages are:

- Lower energy usage;
- Less scale loss;
- Increased furnace throughput;
- \triangleright Reduced roll wear.

Energy usage is reduced in warm rolling processes, since lower reheat temperatures are required as compared to hot rolling practices^{1,2,3}. Thinner gauges are also possible in warm rolled steels, in the case where heat losses would limit the gauge^{1,3}. As the rolling temperature is decreased, the thickness of the scale formed on the rolled product decreases². This is beneficial, as less scale will require less acid in the pickling process and less material loss³. Throughput is increased as a result of the lower reheat temperature, which leads to quicker reheating, and the thinner scale, which requires less pickling time^{2,3}. Due to the reduction in load associated with warm rolling and the decreased temperatures, wear on the rolls is reduced, extending their life¹. These cost savings can amount to a considerable decrease in production expenses.

Currently, interstitial free (IF) steels are being produced by warm rolling methods. The problem being addressed is the poor formability of warm rolled low carbon (LC) steels. Formability is an important property affecting the deep drawing behaviour, as uniform deformation is necessary. Research by Barnett and Jonas⁴ has indicated that this can be achieved in LC steels by alloying with chromium.

Strain ratio (R-value) is an important parameter in deep drawing. This is the ratio of the width to the thickness strain and is a measure of the resistance to thinning. A high R-value promotes uniform deformation. Work by Ray *et al.*⁵ has demonstrated that a strong {111} texture will result in a high R-value. This recrystallization texture can nucleate on shear bands in a deformed microstructure; therefore, it is important to produce shear bands during warm rolling to obtain good formability.

Rate sensitivity (m) is a measure of the change in flow stress with strain rate. This is important for shear band formation, as shear bands consist of localized flow within a grain, which occurs at a higher strain rate than in the bulk. Dynamic strain aging (DSA) has a considerable effect on rate sensitivity. DSA occurs when solute atoms pin and unpin mobile dislocations. When m is negative, intense shear bands are formed; conversely, very few shear bands are formed when m is highly positive. A moderate shear band content, as is the case with a slightly positive value of m, is desired as it will result in good formability. When excessively intense shear bands are produced, the texture becomes fragmented and the Goss texture ($\{110\}<001>$) is formed, which results in a product with poor formability.

The formability of warm rolled LC steels improves greatly when alloyed with chromium⁴. Chromium addition extends the DSA behaviour into the warm rolling temperature range, resulting in slightly positive values of m, which produce moderate amounts of shear bands. This results in good formability for LC steels containing 1.3 wt.% chromium⁴. However, a chromium addition of 1.3 wt.% is relatively expensive; thus the current work was aimed at optimizing the level of chromium addition in terms of cost and formability.

The pertinent theories governing the principal phenomena considered in this study are reviewed in Chapter 2. Strain aging will be covered, with particular emphasis on dynamic strain aging. Rate sensitivity will be discussed, as it can control the formation of in-grain shear bands during deformation. Texture and recrystallization will also be reviewed, as it is these parameters that will ultimately dictate the formability of the final product. The effect of various alloy additions will also be discussed, as it is through the addition of chromium that the current study seeks to modify the afore-mentioned phenomena to favourably affect the formability of warm rolled products.

Chapter 3 describes the various experimental techniques and materials used in this study. The experimental protocol used for compression testing is thoroughly discussed. Also, the procedures for the pilot mill trials are discussed, along with their purpose and goals. Also, the conditions for the annealing treatment and metallographic work after the warm rolling trials are explained.

Results from the various experiments are presented in Chapter 4 and the relevance of the findings is discussed in Chapter 5. Lastly, the conclusions obtained from this study are given in Chapter 6.

Chapter 2

Literature Review

2.1 WARM ROLLING

Warm rolling has aroused interest in recent years as it has the potential to diversify the product range and decrease the production cost of hot rolled products. By definition, warm rolling takes place within the ferrite temperature range (see Figure 2 - 1), for which the upper limit is approximately 900°C for LC steels. In cooling from this temperature to approximately 650°C, the steel undergoes transformation from austenite to ferrite. The FCC austenite phase is stronger than BCC ferrite; therefore, there is a reduction in the flow stress with increasing ferrite fraction, as illustrated in Figure 2 - 2. Note that in practice, warm rolling usually results in a microstructure consisting of at least 90% ferrite at the final finishing stage⁶. Thus, ferritic rolling benefits from having reduced rolling loads, as compared to rolling in the austenite (γ) range; this decreases roll wear and, therefore, extends their lives⁶.



Figure 2 - 1: Fe-C phase diagram (Ref. 6)



Figure 2 - 2: Linear influence of ferrite fraction on flow stress at a constant deformation temperature in a LC steel (Ref. 6)

2.2 DEEP DRAWING

The strain ratio (R-value) is an important parameter in deep drawing. This is the ratio of the width to the thickness strain and is indicative of the resistance to thinning:

$$R = \frac{\ln\left(\frac{w_0}{w}\right)}{\ln\left(\frac{t_0}{t}\right)} \tag{1}$$

where w_0 and w are the initial and final widths, and t_0 and t the initial and final thicknesses, respectively. A high R-value denotes a product with high resistance to thinning in the thickness direction, which ultimately promotes uniform deformation. As most sheet metals display variations in their elastic and plastic properties with planar

orientation, the average strain ratio (\overline{R}) is a good way of expressing this value, as it accounts for the dependence of R on direction:

$$\overline{R} = \frac{R_{0^{\circ}} + 2R_{45^{\circ}} + R_{90^{\circ}}}{4}$$
(2)

Here, $R_{0^{\circ}}$ is the R-value measured along the rolling direction, $R_{45^{\circ}}$ and $R_{90^{\circ}}$ are the values measured at 45 and 90°, respectively, to the rolling direction. Another important measurement to consider is ΔR , which is a measure of the planar anisotropy:

$$\Delta R = \frac{R_{0^{\circ}} + R_{90^{\circ}} - 2R_{45^{\circ}}}{2} \tag{3}$$

 ΔR is important in the discussion of bulk deformation textures, as directionality in the mechanical properties produced during rolling has a significant impact on the fabricability of a metal⁷. For good formability, high \overline{R} and low ΔR values are required.

In order to improve the drawability of sheet metal, the crystallographic texture must be controlled⁷. The ideal texture results in slip systems oriented so that the strength in the thickness direction exceeds that within the plane of the sheet. In BCC metals, such as ferritic steels, this translates into a {111} texture. A strong {111} fibre texture will thus result in a high \overline{R} value⁵, as illustrated is Figure 2 - 3.



Figure 2 - 3: Correlation between average R and I{111}/I{001} (Ref. 5)

2.3 STRAIN AGING

2.3.1 Static Strain Aging

Strain aging can occur during or after deformation. Aging that occurs after deformation is referred to as static strain aging (SSA). Figure 2 - 4 describes the general features of SSA. A typical mild steel is strained to point X, which produces curve A that displays an upper and lower yield point. If that same specimen is immediately re-strained, the resulting stress-strain curve will be that of curve B, where the yield strength has increased and the upper and lower yield points are no longer evident. However, if this same specimen were aged at ambient temperatures or greater and then deformed, the specimen would demonstrate behaviour consistent with curve C. In this last curve, the yield point has increased once more, and the upper and lower yield points are again present.



Figure 2 - 4: Stress-strain curves for LC steel showing strain aging (Ref. 7)

The most obvious manifestation of SSA is an increase in yield stress upon aging after straining; however, other properties are also affected. This usually causes a decrease in ductility and also produces a low value of rate sensitivity⁷. SSA is a result of solute atoms migrating to dislocations and pinning them. In LC steel, the elements associated with SSA are carbon and nitrogen, as these interstitial atoms have diffusivities far greater

than the substitutional elements, and hence, can produce aging effects within short periods of time and at relatively low temperatures. As the solubility and diffusion coefficient in steel are greater for nitrogen than carbon, the effect of the former is more pronounced^{8,9,10}.

2.3.2 Dynamic Strain Aging

Strain aging that occurs *during* deformation is referred to as dynamic strain aging (DSA). This consists of the attraction of interstitial atoms towards mobile dislocations. and occurs in steels containing carbon and/or nitrogen in solution^{11,12}. DSA produces discontinuities in the stress-strain curve (as illustrated in Figure 2 - 5) and is known as the Portevin-LeChatelier (PLC) effect. DSA occurs under conditions that permit the interstitial atoms to diffuse rapidly enough to pin dislocations during deformation, i.e. at temperatures where the diffusion rate of these interstitials matches the velocity of mobile dislocations. DSA results in enhanced dislocation multiplication, which can cause high flow stresses, negative strain rate sensitivities and serrations in the stress-strain curves^{11,12}.



Figure 2 - 5: Stress-strain curves for specimens tensile tested at various temperatures at $\dot{\epsilon} = 10^{-4} \text{ s}^{-1}$ showing serrations (Ref. 14)

2.3.3 Theory of DSA

DSA occurs when solute atoms have sufficient mobility to "catch up" to mobile dislocations and form solute (Cottrell) atmospheres around them¹³. Therefore, DSA can only occur over a temperature range where the interstitial diffusivity is rapid enough to impede dislocation motion. The serrations on stress-strain curves that result from DSA occur due to alternating cycles of dislocations being pinned and breaking away from these atmospheres.

The DSA region is the temperature range within which serrated yielding is observed. Temperatures below this range do not allow sufficient mobility of the solute atoms to agglomerate around dislocations and restrict their motion during deformation. Conversely, at high temperatures, the rate of solute atom diffusion exceeds that of dislocation motion, hence the solute atoms impose only a limited drag effect.

2.3.4 Prediction of DSA at High Strain Rates

The serrations due to the PLC effect can be used to define the DSA range¹⁴. Knowledge of this range for a material measured in laboratory scale experiments at low strain rates (e.g. 10^{-2} s⁻¹), permits the prediction of DSA at the high strain rates pertaining to mill conditions¹⁵ (e.g. 10^2 s⁻¹). Note that the flow stress peak generally coincides with the upper temperature limit. This is because DSA strengthening is reduced beyond the peak; thus no pinning and unpinning of mobile dislocations occurs beyond this temperature range. The strain rate ($\hat{\epsilon}$) of a dislocation can be equated to the diffusion rate of an interstitial atom:

$$\dot{\varepsilon} = A \cdot \exp\left(\frac{-Q_{diff}}{RT}\right) \tag{4}$$

where A is a frequency factor, Q_{diff} the activation energy for solute diffusion and T the absolute temperature¹⁴ (see Figure 2 - 6). Thus, by plotting the limits of DSA behaviour at lower strain rates, the limits at higher rates can be predicted by plotting log $\dot{\epsilon}$ vs. 1/T. The gradient of these limits is related to the activation energy. Karimi-Taheri *et al.*¹⁴ used

this method to evaluate the DSA range in LC steel and measured activation energies of 84.1 and 134 kJ/mol for the minimum and maximum temperatures, respectively where the serrations occurred. The lower energy is similar to that for the interstitial diffusion of nitrogen in pure ferrite $(76.0 \text{ kJ/mol})^{16}$. The difference between the upper and lower energies is postulated to be higher due to the binding energy of interstitial atoms to the dislocation core¹⁴.



Figure 2 - 6: Temperature and strain rate range over which serrations are observed (Ref. 14)

2.3.5 DSA and Flow Stress

In order to produce a precisely dimensioned strip product, it is important to properly predict the flow stress for a given set of rolling conditions¹⁷. The flow stress as a function of strain, strain rate and temperature has been extensively modeled in the case of hot rolling^{18,19}, yet has not been accurately established for warm rolling⁶. However, it is expected that these models will also be useful in describing the flow stress at the higher ferrite temperatures. The Zener-Hollomon parameter (Z) is used to link temperature and strain rate:

$$Z = \dot{\varepsilon} \cdot \exp\left(\frac{Q_{def}}{RT}\right)$$
(5)

where $\dot{\epsilon}$ is the strain rate, Q_{def} is the activation energy for deformation, R is the gas constant, and T the absolute temperature. The Zener-Hollomon parameter allows the behaviour of different materials to be compared²⁰. In order to predict the peak flow stress (σ_p), Equation 5 can be incorporated into a hyperbolic sine law:

$$Z = A \cdot \left[\sinh\left(\alpha\sigma_{p}\right)\right]^{n'} \tag{6}$$

where A, α and n' are constants. According to Barnett *et al.*, the value of the exponent n' is much lower in LC steels than in IF ones; this can be attributed to the DSA behaviour displayed by the former materials^{12,21}. The value of n' is only 4.5 in LC steel, as compared to 9.1 in IF steel^{22,23}. Within the range of laboratory stresses and strain rates, the exponent n' has been correlated to the rate sensitivity (m) as follows²¹:

$$n' = \frac{1}{1.2\alpha\,\sigma\,m}\tag{7}$$

2.4 RATE SENSITIVITY

The rate sensitivity (m) is a measure of the rate of change in flow stress with strain rate⁷:

$$m = \frac{\Delta \log(\sigma)}{\Delta \log(\dot{\varepsilon})}$$
(8)

Rate sensitivity is important as it plays a role in the formation of in-grain shear bands during deformation. Shear bands consist of localized flow within a grain, which occurs at a higher strain rate than bulk deformation in the remaining grain. Thus negative or small positive values of m facilitate shear band formation.

2.4.1 Ideal Behaviour

As the deformation temperature is increased, the flow stress is generally expected to decrease²⁴. This phenomenon is observed in pure metals (as illustrated in Figure 2 - 7), which display this idealized behaviour. Increasing the strain rate in this case will result in a slight increase in flow stress, resulting in small positive or "normal" values of m. Thus, when DSA is absent, m is slightly positive and increases slowly with temperature. When one considers rate sensitivity within the context of metal working, strain rate and temperature are the two independent variables. In this case, the power-law viscoplastic relationship (Equation 8) can be written as follows²⁵:

$$\sigma = k(T)\dot{\varepsilon}^{m(T)} \tag{9}$$

where m(T) denotes the temperature-dependent rate sensitivity and k(T) is a temperature dependent constant.



Deformation Temperature

Figure 2 - 7: Variation in the stress with deformation temperature under ideal conditions (Ref. 24)

2.4.2 DSA and Rate Sensitivity

DSA has a considerable effect on m. When Cottrell atmospheres form around dislocations, solute carbon atoms lead to an increase in the work hardening rate that

results in a distinct peak in the flow stress vs. temperature curve¹⁵. This peak can be responsible for negative values of m^{26} . This phenomenon is present in LC grades, but is absent in IF steels, as they lack the solute carbon required for DSA behaviour¹². Figure 2 - 8 illustrates the peak in the flow stress vs. temperature curve that is responsible for the negative m values caused by DSA. As a result of these negative rate sensitivities, the flow stress decreases with increasing strain rate, a trend that ultimately leads to unstable deformation and flow localization¹⁴. This is because the material deforms more rapidly in these regions than in the bulk.



Figure 2 - 8: Schematic representation of stress vs. deformation temperature for two strain rates illustrating the effect of DSA

LC steels typically display strong DSA peaks, the temperature of which increases with strain rate¹⁵. At temperatures corresponding to this peak, an increase in strain rate results in negative m values in the DSA region. Conversely, the resulting steep slope at temperatures above the peak results in regions of highly positive m¹². The shape of the stress vs. temperature curve is not dependent upon strain rate, and hence does not change with increasing strain rate; however, the curve is shifted to higher temperatures as the strain rate is increased¹⁵.

2.5 SHEAR BANDS

Shear bands are banded features that are often apparent in deformed ferrite microstructures (see Figure 2 - 9). These lie at angles of \pm 20-35° to the rolling direction and are contained within individual deformed grains⁶. At high rolling loads in Fe-N alloys, it has been found that the maximum occurrence of shear bands coincides with the maximum temperature limit of the DSA range¹². The behaviour of shear bands is consistent with that of other flow localization phenomena, as they too are sensitive to deformation temperature, rate sensitivity and work hardening rate²⁷.



Figure 2 - 9: Shear bands in a warm rolled IF steel (Ref. 28)

2.5.1 Shear Bands and Rate Sensitivity

The frequency of shear banding depends on the value of m as bands are a form of localized high rate flow¹⁵. Negative values of m will promote this formation, as it is under these conditions that a portion of the material (i.e. the shear bands) will deform at a strain rate greater than that of the bulk with a reduction in flow stress. In regions of highly positive m, the bulk deforms at a lower stress than the shear bands, and hence little or no banding is present. Thus as a general trend, warm rolled LC steels display a decreased occurrence of shear banding with increasing rate sensitivity⁶.

It is, however, possible for shear bands to form with small positive values of m. Other phenomena that are relevant to the formation of shear bands include deformation heating, hard grains and strain path change. Since shear bands consist of intense local deformation, this will result in localized heating within the shear band, which is referred to as deformation heating²⁷. Supporting the relationship between the frequency of shear bands and rate sensitivity, it has been found that alloying a LC steel with 1.3 wt.% chromium can increase the shear band fraction from almost 0 to 30% (for a rolling temperature of 600°C. This phenomenon occurs due to the decrease in rate sensitivity, which is a result of the chromium addition²⁹ (see 2.8 Effect of Alloy Additions).

As predicted from theory, there is a decrease in flow stress that results from an increase in temperature; thus deformation heating causes shear bands to deform more easily. Hard grains are grains with an unfavourable orientation, which makes it difficult to produce slip within the material. In order to compensate, the material can deform by other means, such as through the formation of shear bands. It should also be noted that the bulk of the material deforms in the pure shear type of plane strain; by contrast, shear bands undergo simple shear²⁷. This change in strain path, from pure to simple shear, also results in a decrease in the flow stress¹², as simple shear essentially involves deformation in a single plane, while pure shear involves a combination of two types of simple shear, as illustrated in Figure 2 - 10^7 .



Figure 2 - 10: Strain path of (a) pure shear (b) simple shear (Ref. 7)

2.5.2 Shear Bands and Texture

Shear bands are effective nucleation sites for recrystallization as they consist of highly strained regions. Shear bands are desirable in warm rolled products, as they can provide nucleation sites for the desired {111} texture after annealing, which leads to greater R-values in the final product¹⁵. Duggan et al. showed that the new grains produced were of the same orientation as the shear bands³⁰. However, intense shear bands can be detrimental, since they provoke nucleation of the Goss {110}<001> component and hence weaken the desired {111} texture²⁸. Excessive shear banding can also cause fragmentation of the warm rolling texture, as they can become a significant volume fraction of the bulk⁶. Therefore, it is important to produce moderate (but not intense) shear bands during warm rolling to obtain good formability.

2.6 RECRYSTALLIZATION

Warm rolled materials must be annealed in order to produce the desired formability properties of the final product. Annealing, where a deformed material is returned to its strain-free condition by holding at an elevated temperature, consists of three stages: recovery, recrystallization and grain growth. Recrystallization is characterized by a decrease in strength accompanied by an increase in ductility, due to the decrease in dislocation density⁷.

Recrystallization is driven by the stored energy of cold work. A strain-free grain nucleates and the high angle boundary migrates and annihilates the dislocations it encounters. Recrystallization is affected by the following parameters:

- > Amount of prior deformation, which is the driving force;
- > Temperature, which relates to the kinetics of the process;
- > Time, which is also related to the kinetics;
- The initial grain size, which is also a driving force, as it affects the degree of strain hardening and also the potential density of nucleation sites;

- The composition of the material being recrystallized, which is responsible for solute drag, which decreases the net driving force;
- The amount of recovery prior to the start of recrystallization, which decreases the retained strain, which also decreases the net driving force.

2.6.1 Kinetics of Recrystallization

Various attempts have been made to model the kinetics of recrystallization. For isothermal transformations, the volume fraction transformed (X_v) with time (*t*) can be expressed by the Johnson-Mehl-Avrami (JMA) equation^{31,32,33}:

$$X_{v} = 1 - \exp(-Kt^{n}) \tag{10}$$

where *n* is the kinetic exponent, which depends on the growth geometry and the mechanism of transformation, and K is a parameter that takes the numerical and growth constants into account. This model is illustrated in Figure 2 - 11^{34} .



Figure 2 - 11: Recrystallized fraction (X_{ν}) as a function of the annealing time (*t*) for Al after 5.1% tensile deformation (Ref. 34)

An extension of this model for primary recrystallization is the Johnson-Mehl-Avrami-Kolmogorov (JMAK) equation³⁵. The JMAK model incorporates modifications to simple first-order reaction rate kinetics to include the linear dependence of transformation rate on volume fraction of the remaining parent phase $(1 - X_{\nu})^{36}$. These modifications encompass: the time dependence of the nucleation rate, the dimensionality of the growing particle, whether growth is linear or parabolic, and the eventual impingement of growing particles at long times. The general form of the JMAK equation is:

$$X_{v} = 1 - \exp\left(-\left(k(t - t_{i})\right)^{n}\right)$$
(11)

where t_i is the incubation time, and k is a thermal activation rate constant. The value of the kinetic exponent (n) typically varies between 1 and 4³⁶. The rate constant is defined as follows:

$$k = k_0 \exp\left(\frac{-Q^*}{k_B T}\right) \tag{12}$$

where Q^* is the activation energy barrier for recrystallization, k_0 and k_B are constants, and T is the absolute temperature. The value of k can be calculated by measuring X_v at various temperatures, whilst Q^* can be inferred from Kissinger plots³⁷ or an Arrhenius-type equation (Equation 12)³⁶.

The JMAK model assumes that the nucleation sites are randomly distributed, which is an essential feature of this approach³⁸. The JMAK diagram is constructed by plotting log $[\ln(1/(1-X_v))]$ against the log of the annealing time. The use of a log-log scale results in a straight line, the slope of which is equal to the exponent *n*. However, it is unusual for experimental data to demonstrate good agreement with JMAK kinetics; this is due to the complex mechanisms involved in recrystallization³⁸. One such example involves the work of Vandermeer and Gordon³⁹ on 40% cold rolled aluminum containing copper, which is shown in Figure 2 - 12. Specimens subjected to lower annealing temperatures and long annealing times tended to recrystallize more slowly than predicted by the model.



Figure 2 - 12: JMAK plot of the recrystallization kinetics of aluminum containing 0.0068 at.% Cu, deformed 40% by rolling (Ref. 39)

2.6.2 Recrystallization in Warm Rolling

Recrystallization is one of the processes that impacts the mechanical properties of warm rolled materials. The most significant features of recrystallization to be considered are: the stored energy/dislocation density, which is the driving force; the inhomogeneity of the dislocation density, which affects the characteristics of nucleation and grain growth; and the crystallographic texture, which influences the orientations of the recrystallized grains¹².

In-situ recrystallization can occur during warm rolling. Previously, dynamic recovery (DRC) was considered the prevalent softening mechanism during the high temperature deformation of ferrite (warm rolling conditions)^{7,40,41}. However, other studies have shown that dynamic recrystallization (DRX) can take place under exceptional warm rolling conditions^{40,42,43,44}. The studies by Glover and Sellars⁴⁰ and Matsubara *et al.*⁴² both show evidence of a clear transition from DRC to DRX. A stress state was identified by these workers below which the scale of the structure changed drastically after deformation. This evidence implies a change of softening mechanism.

Both studies reported the presence of dislocations within newly formed grains, high angle grain boundaries and distinctive flow curves^{40,42}.

The dislocation density is dependent upon grain orientation during deformation; therefore, deformed grains with different orientations have different driving forces for recrystallization²⁸. As discussed by Urabe and Jonas, Taylor factors, which represent the stored energy of deformation for grains containing shear bands, have been used to study recrystallization behaviour^{45,46}. It was determined that, within warm rolled steels, the high Taylor factor orientations were those of the ND fibre. These ND fibres tend to contain a significant amount of shear bands.

Hutchinson noted that recrystallization nucleated preferentially in grains containing shear bands⁴⁷. It has been shown by Barnett that a {111} recrystallization texture can be nucleated by the presence of in-grain shear bands^{29,48}. Duggan *et al.* have reported similar results for a cold rolled ultra-low carbon steel⁴⁹. However, Ushioda *et al.* have shown that *intense* in-grain shear bands, produced by warm rolling at temperatures corresponding to the DSA flow stress peak, resulted in a reduction of the {111} texture intensity after recrystallization⁵⁰. Thus, the presence of moderate numbers of shear bands are thought to be required to nucleate {111} orientated recrystallized grains. The latter type of texture is expected to result in an annealed product with good formability.

2.7 TEXTURE

The annealing (recrystallization) texture is of vital importance in attempting to produce warm rolled steel with good formability²⁸. As illustrated in Figure 2 - 3, R-value increases with increasing intensity of the $\{111\}$ texture; therefore, steel with a strong $\{111\}$ texture will have a high R-value and will deform uniformly, allowing deep drawing to very thin gauges.

2.7.1 Effect of Deformation Temperature

Deformation temperature has a considerable impact on the texture in LC steels due to the variation in m, and hence on shear band formation. It was noted by Barnett and Jonas¹² that there is an increase in the {111} and {001} texture intensities with rolling temperature. Two phenomena were used to attempt to explain this correlation: slip configuration and grain fragmentation. The former was deemed to have a more significant impact in warm rolling. Grain fragmentation has a randomizing effect on the deformation texture (i.e. it weakens the texture) and prolific amounts of shear bands are known to further weaken the texture, as they transfer flow away from the matrix.

2.7.2 Bulk Deformation Textures

Warm rolled materials display bulk textures similar to those of cold rolled products^{12,51}. The predominant orientations can be grouped according to two families: the rolling direction (RD) fibre, which is parallel to the <110> axis, and the normal direction (ND) fibre, which is parallel to the <111> axis⁶. Figure 2 - 13 illustrates this texture notation. It is within this context that ΔR is important, as it measures the distribution of texture along any one fibre. Though a strong texture is desirable, it is important that it be evenly distributed along the different directions to promote uniformity in the properties.



Figure 2 - 13: $\phi_2 = 45^{\circ}$ ODF plot showing the locations of the main rolling and recrystallization textures (Ref. 6)

2.7.3 Recrystallization Textures

The ideal {111} texture, which is required for good formability, nucleates on shear bands, as long as they are of moderate intensity. Currently, warm rolled LC materials have poor formability due to the absence of a distinct {111} recrystallization texture, which can be attributed to the lack of in-grain shear bands produced within the warm rolling temperature range⁶. In order to increase the fraction of shear bands present, the rate sensitivity must be decreased, which can be achieved by modifying the DSA behaviour of the LC steel. It would be ideal to remove all the soluble carbon from solution (as in an IF steel), using techniques such as vacuum degassing; however, this is not cost effective. Current research indicates that adding alloying elements, such as chromium, can reduce m, and hence, increase the amount of shear bands that will be produced. This should result in a strong {111} texture, which should ultimately produce a material with a high R-value, leading to good formability.

2.8 EFFECT OF ALLOY ADDITIONS

The addition of alloying elements has a significant impact on the final properties of a finished steel. In the current context of warm rolling, the alloying element being investigated is chromium. It has been deemed responsible for modifying the behaviour of LC steels regarding flow stress, DSA effects and shear band formation⁵².

2.8.1 Impact on Flow Stress and DSA

As is the case with carbon, chromium also has a significant impact on the flow stress of LC steels as a function of deformation temperature. Firstly, as previously shown^{15,52} the presence of chromium in LC steels results in decreasing the height of the peak in the flow stress vs. temperature curve, due to a reduction in the solute carbon content. It also creates a second peak in the flow stress vs. temperature curve, as shown in Figure 2 - $14^{6,15,52}$. This second peak occurs at temperatures higher than those associated with the carbon/nitrogen DSA peak⁶; this temperature is high enough to allow
chromium diffusion⁵². This broadens the flow stress peak, which then extends into the warm rolling region. In this way, the rate sensitivity is lowered across the entire temperature range^{6,15}. It is believed that the solute chromium forms complexes with solute carbon within the warm rolling range. These interact with the mobile dislocations during deformation at the higher temperatures, as they are less mobile than solute carbon atoms alone^{15,52}. Note that chromium will also strengthen the material through solid-solution hardening, due to the introduction of substitutional chromium atoms into the matrix.⁷ This difference between the sizes of the atoms in the lattice renders the material more difficult to deform and, hence, stronger.

The second peak in the flow stress vs. temperature curve was observed in LC steels with additions of 1.3 wt.% Cr and greater^{15,52}, but not in materials with additions of 0.5 wt.% Cr¹⁵. Though ideal levels of rate sensitivity were not reached, the addition of 1.3 wt.% chromium resulted in its decrease to significantly lower levels within the warm rolling temperature range, as compared to the LC samples¹⁵.



Figure 2 - 14: Temperature vs. flow stress curves for tensile test data showing the effect of an addition of 1.3 wt.% Cr (Ref. 6)

2.8.2 Shear Bands

The decrease in m resulting from chromium additions thus impacts on the formation of shear bands, a decrease in rate sensitivity increasing the occurrence of shear bands. It was found that a decrease in rate sensitivity in the warm rolling temperature range (approximately 350-500°C under laboratory conditions), resulted in an increase in the occurrence of shear bands in a LC-0.5 wt% Cr steel as compared to a LC base grade^{6,14,28}.

Chapter 3

Experimental Details

Compression tests were conducted to study the effect of chromium content on rate sensitivity. Two experimental alloys, as well as an ELC steel, were tested at temperatures ranging from room temperature to 800°C, at different strain rates. Stress vs. strain plots were calculated for every experiment in order to determine the flow stress at 10% strain and hence calculate the rate sensitivity.

In addition to the compression tests, warm rolling trials were conducted using the pilot mill at CANMET in Ottawa. Analysis of the shear band content in the warm rolled samples allowed the rate sensitivity calculations from the compression tests to be compared with the metallographic observations. Lastly, some annealing experiments were carried out using the warm rolled samples to study the recrystallization behaviour of the materials.

3.1 MATERIALS

Test samples were manufactured from three different materials: an ELC steel and two experimental alloys. The chromium content was varied between the two alloys: a high (0.80 wt.%) and a low (0.49 wt.%) chromium LC steel. The experimental alloys contained boron additions of 39 and 45 ppm, respectively. The detailed compositions of the various materials are listed in Table 3 - I. Cylindrical samples (see Figure 3 - 1) were machined in order to carry out the compression tests; these were 11.4 mm in height and 7.6 mm in diameter.

	С	Mn	Cr	В	Ti	Al	Ν
ELC	0.020	0.120	0.071	=	-	0.048	0.0067
LC-low Cr	0.040	0.144	0.490	0.0039	0.0150	0.032	0.0032
LC-high Cr	0.040	0.151	0.800	0.0045	0.0150	0.037	0.0025

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Figure 3 - 1: Schematic drawing of compression specimens

3.2 COMPRESSION TESTS

Compression tests were carried out using a servo-hydraulic axial compression machine, which is equipped with a water-cooled furnace (see Figure 3 - 2). A temperature controller was used to control the heating cycle of the furnace, with a K-type thermocouple arranged to remain in constant contact with the surface of the specimen. A quartz tube, which encompassed the specimen and the anvils within the furnace, was vented with both compressed air and argon gas. The compressed air was used to rapidly cool the specimen, whilst the argon gas provided an inert atmosphere during testing in order to reduce oxidation of the specimen and tools. The compression machine was operated by means of a computer equipped with MTS TestStar[™] software, which recorded force, temperature and displacement at regular time intervals during deformation.





Figure 3 - 2: Experimental set-up used for compression testing

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In order to minimize the frictional forces between the sample and the anvils during the test, the sample was placed between two sheets of Pelco mica, which had been lubricated with boron nitride. The boron nitride was in the form of a fine powder (~1 micron) and was combined with ethanol in order to produce a slurry.

The heating schedule used is illustrated in Figure 3 - 3. The temperature was first raised to 900°C over a span of 10 minutes (1), and then maintained at that temperature for 15 minutes (2). This annealing treatment was carried out to dissolve all the carbides present, so as to obtain the maximum amount of carbon in solution possible. Tests were also conducted where the holding period was 30 minutes; however, there were no notable differences associated with this extended annealing time. The samples were then rapidly cooled to the test temperature by blasting compressed air through the tube (3). This was done to minimize carbide precipitation. The test temperature was held (4) for 5 minutes, which allowed sufficient time for the furnace temperature to stabilize before the compression was carried out (5). The cooling rate was approximately 100°C/minute. Appendix contains the various heating schedules for the different test temperatures, as well as a plot of the heating profile during testing.



Figure 3 - 3: Heating schedule (schematic)

Once the heating schedule was completed, the samples were compressed at a constant strain rate up to a true strain of approximately 20%. Tests were carried out at

three different strain rates: 10^{-1} , 10^{-2} , and 10^{-3} s⁻¹. Using MTS TestStarTM software, force, temperature and displacement values were recorded at regular intervals throughout the deformation, with approximately 2000 points per experiment. The appendix contains the test schedules used for each strain rate.

3.3 ROLLING TRIALS

Warm rolling trials were carried out at a pilot mill to validate the results obtained from the laboratory experiments. Prior to warm rolling, each material was hot rolled to a thickness of 1.17 cm at a temperature exceeding 950° in order to avoid pancaking the austenite. Test slabs of each material were used, with dimensions of 16.51x9.52x1.17cm. All billets were solution treated at 950°C for 30 to 60 minutes prior to rolling in order to dissolve all the carbides present, then air cooled down to the rolling temperature on a ceramic block and covered with a ceramic wool blanket to facilitate slow cooling. An embedded thermocouple was used to monitor this temperature. Rolling was conducted using a single pass in order to eliminate interpass static recrystallization. A roll gap of 1.52 mm (60/1000 in.) and a rolling speed of 45 rpm were used, resulting in a strain rate of 30s⁻¹. The rolls were lubricated with a soluble oil after every 5 passes to minimize friction effects, which have been known to adversely affect the deformation textures⁵³. The rolled billets were immediately water quenched in order to prevent the occurrence of static recrystallization.

Rolling trials were carried out at 440, 640, 710, and 780°C. The three higher temperatures were chosen to correspond to the warm rolling region, which is approximately between 600 and 800°C for pilot mills operating at a strain rate of 30 s⁻¹. Earlier trials had included tests at 850°C; however, it was determined that this temperature was too high as static recrystallization had occurred prior to quenching. The trials at 440°C, which is well below the warm rolling temperature range, were conducted to obtain data within the region of negative rate sensitivity. The warm rolling region typically exhibits positive rate sensitivity.

3.4 ANNEALING

Annealing trials were conducted, where samples of the warm rolled materials were immersed in a molten salt bath for various time intervals then immediately water quenched. Salt was used for the annealing trials, as its high heat transfer rate allows for very short annealing times, as compared with air conduction in a muffle furnace. Annealing samples 15mm long by 5 mm wide (see Figure 3 - 4) were cut from the 710°C warm rolled specimens of each material. Samples were annealed at 750°C for 10, 20, 30, 45 and 100 seconds to observe the nuclei of the recrystallized grains.

A high temperature was required for annealing since the diffusion of chromium in steel is sluggish. 750°C was selected as it is a high temperature that still lies below the phase transformation temperature (A_{C_1}) within the single phase region. Annealing above the transformation temperature will result in a transformation texture rather than the desired recrystallization texture.



Figure 3 - 4: Schematic illustration of samples used for the annealing trials

3.5 METALLOGRAPHY

Samples from the warm rolling trials and the annealing tests were (hot) mounted in Bakelite to facilitate further examination. All samples were mounted so as to expose the microstructure in the RD-ND plane (i.e. perpendicular to the transverse direction). Figure 3 - 5 is schematic illustration showing the warm rolled section examined. Mounted samples were ground, polished and etched prior to microscopic analysis. The samples were all ground and polished in the same manner; however; different etching times were required. For grinding, silicon carbide (SiC) paper was used with water as the lubricant. Each sample was ground on strips, which were consecutively finer, as follows: 60, 120, 240, 320, 400, 600 and 1200 grit. Polishing was carried out with cloths using diamond abrasive liquid, on 3 and 1 μ m wheels.

The etchant used for this study was 2%. Nital, which was made using 2 vol.% nitric acid (HNO₃) with ethanol making up the remainder. The warm rolled samples were etched by immersion for a total of 17 seconds, while the annealed samples required only 13 seconds to properly expose their microstructures.



Figure 3 - 5: Schematic illustration of the warm rolled samples studied

3.6 MICROSCOPY

Once polished, samples were examined using an optical microscope with a digital camera attached. This arrangement enabled digital micrographs, at a magnification of up to 1000x, to be captured for the various samples.

3.6.1 Shear Band Counting

In order to calculate the fraction of grains that contained in-grain shear bands, a simple point counting technique was used. Several digital micrographs were taken of each sample at magnifications ranging from 200-500x, depending on the grain size of the sample produced by the rolling temperature. Next, an 8 point by 11 point grid was superimposed on the images. The shear band fraction was determined by taking the ratio of the number of points within grains containing shear bands to the total number of points. Figure 3 - 6 is an illustration of this technique; the grains containing shear bands have been highlighted (in white) to facilitate distinction from those grains without. This procedure was repeated approximately 5 times per specimen, in order to obtain a representative value of the shear band fraction.



Figure 3 - 6: Microstructure of the LC-Low Cr sample warm rolled at 440°C with the grains containing shear bands highlighted in white, Mag. 200x

3.6.2 Recrystallized Fraction

The fraction of the material that had recrystallized was determined using a point counting method similar to that employed for the shear bands. Digital micrographs were taken for each sample at the various temperatures and a 10 point by 9 point grid was superimposed over this image. The recrystallized fraction was then determined by calculating the ratio of the points that lie within recrystallized grains to the total number of points. This procedure was repeated 4 times per temperature in order to obtain an accurate estimate of the fraction of material that had recrystallized. Figure 3 - 7 is an example of this method, where the recrystallized grains have been highlighted (in white) for emphasis. For the sake of simplicity, only recrystallized grains that coincided with grid points were redrawn.



Figure 3 - 7: Microstructure of the ELC sample annealed at 750°C for 10 s showing the recrystallized grains highlighted in white, Mag. 500x

Chapter 4

Results

4.1 COMPRESSION TESTS

4.1.1 Flow Stress

From the data collected for each compression test, the engineering stress (σ_{eng}) was calculated according to the following equation:

$$\sigma_{eng} = \frac{4F}{\pi d_0^2} \tag{13}$$

where F is the force developed and d_0 is the initial diameter of the specimen. Figure 4 - 1 is a typical plot of engineering stress as a function of engineering strain (ε_{eng}), described in Equation 14, where h_0 is the initial height of the specimen and Δh is the change in height of the specimen:

$$\varepsilon_{eng} = \frac{\Delta h}{h_0} \tag{14}$$

The engineering stress at 10% strain (10% $\sigma_{eng.}$ or 10% flow stress) was established from the plot, as indicated in Figure 4 - 1. This value was chosen because the effect of barrelling is minimal at 10% strain and yet there is sufficient work hardening for the effect of DSA to be detectable at this point on the stress-strain curve. Engineering stress and engineering strain were used instead of true stress and true strain in order to compare the results directly with those of other researchers^{4,6,12,15}. The 10% flow stress was plotted as a function of temperature for the ELC sample, as well as for the LC low and high chromium alloys, as represented in Figures 4 - 2, 4 - 3 and 4 - 4, respectively. As a means of comparing the general behaviour of the three different steels, Figure 4 - 5 was constructed using data for a strain rate of 10^{-3} s⁻¹ for each material. The ELC material exhibited a single, sharp peak in the flow stress, while the chromium alloys both contained a second minor peak, resulting in a broadening of the overall DSA region. It was also apparent that the peak flow stresses for the chromium alloys were lower than for the ELC steel.



Figure 4 - 1: $\sigma_{eng.}$ vs. $\epsilon_{eng.}$ Strain, LC-High Cr, 700°C, $\dot{\epsilon} = 10^{-1} s^{-1}$



Figure 4 - 2: 10% Flow Stress vs. Temperature, ELC sample



Figure 4 - 3: 10% Flow Stress vs. Temperature, LC-Low Cr sample



Figure 4 - 4: 10% Flow Stress vs. Temperature, LC-High Cr sample



Figure 4 - 5: 10% flow stress vs. deformation temperature for all materials, strain rate = 10^{-3} s⁻¹

4.1.2 Serrations

The DSA range for the materials in question was verified by studying the presence of serrations on the stress-strain curves. The equation describing this boundary was derived by taking the logarithm of Equation 4 as follows:

$$\dot{\varepsilon} = A \cdot \exp\left(\frac{-Q_{diff}}{RT}\right)$$
$$\ln \dot{\varepsilon} = \left(\frac{-Q_{diff}}{R}\right) \frac{1}{T} + \ln A \tag{15}$$

Thus, ln \dot{e} was plotted against inverse absolute temperature, as shown in Figures 4 – 6 to 4 – 8. Two straight lines were drawn on each graph to indicate the limits of the region

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where serrations were observed. According to Equation 15, the slopes of these lines equal $-Q_{diff}/R$; this permits determination of the activation energies associated with the diffusion of the DSA solutes. These values, along with results obtained by others^{14,41-43}, are listed in Table 4 - I. There were no serrations observed on the stress-strain plots at temperatures above the flow stress peaks.

 Table 4 - I: Activation energies for solute diffusion measured from the boundaries
 of the serrations

	Q _{diff} - Onset	Q_{diff} - Termination
ELC	71.2 kJ/mol	123 kJ/mol
LC-Low Cr	62.3 kJ/mol	95.6 kJ/mol
LC-High Cr	64.8 kJ/mol	95.3 kJ/mol
Karimi-Taheri et al. ¹⁴	84.1 kJ/mol	134 kJ/mol
Kinoshita et al.54	79.9 kJ/mol	-
Lean ⁵⁵ , Keh <i>et al</i> . ⁵⁶	79.5 - 83.7 kJ/mol	128 kJ/mol



Figure 4 - 6: Arrhenius plot for ELC steel







Figure 4 - 8: Arrhenius plot for LC-High Cr alloy

4.1.3 Rate Sensitivity

Once the 10% flow stress was plotted against temperature for all strain rates, the rate sensitivities for the various materials were calculated. Flow stress was plotted against strain rate on a log-log scale for each temperature, and a straight line was fitted to each data series, as shown in Figure 4 - 9. Log scales were used since the slope of a straight line on this graph is the rate sensitivity at the given temperature, as indicated by Equation 8:

$$m = \frac{\Delta \log \sigma}{\Delta \log \dot{\varepsilon}}$$

Rate sensitivity was then plotted against temperature (see Figure 4 - 10). The values for the non-DSA "ideal" behaviour were calculated from previous tests using an IF steel^{29,57}, which is known from theory to display ideal behaviour, as it lacks the interstitial atoms in solution necessary for DSA effects.



Figure 4 - 9: Log(Flow Stress) vs. Log(Strain Rate), LC-High Cr sample



Figure 4 - 10: Rate Sensitivity vs. Temperature

4.2 WARM ROLLING MICROSTRUCTURES

The data from the warm rolling trials conducted at the CANMET pilot mill are listed in Table 4 - II. The samples were water quenched within 3 seconds of rolling. Specimens from these warm rolled materials were prepared as described in section 3.5 and the RD-ND cross-sections were viewed using an optical microscope to determine the shear band content. The microstructures all contained elongated grains parallel to the rolling direction, typical for ferritic-rolled microstructures. In-grain shear bands were also present in a portion of these grains and their fractions and intensities varied between specimens. Using a simple point counting technique and a standard grid, the fraction of

Samle	PI	reheat	Rolling	Starting		-
	Time	Temperature	Temperature	Thickness	rinal Lnickness	Keduction
	min.	Э°	J°	um	um	%
ELC	30	1050	780	10.3	3.15	69.4
I	30	1050	710	10.3	3.28	68.1
ł	30	1050	640	10.3	3.45	66.4
I	30	1050	440	10.3	4.22	59.0
LC-Low Cr	30	950	780	11.7	3.01	73.7
I	30	950	710	11.7	3.73	68.0
I	30	950	640	11.7	4.44	62.0
I	30	950	440	11.7	5.13	56.1
LC-High Cr	30	950	780	11.7	3.20	72.6
I	30	950	710	11.7	3.84	67.2
I	30	950	640	11.7	4.42	62.2
I	30	950	440	11.7	5.13	56.1

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material containing in-grain shear bands (shear band fraction) was determined for the various materials and deformation temperatures. The shear band fraction vs. temperature for the three steels is displayed in Figure 4 - 11. It is apparent that the fraction of grains that contained shear bands decreased with increasing rolling temperature for all materials. However; this decrease was significantly reduced in the chromium-modified steels. Typical microstructures of the LC-High Cr sample warm rolled at the various temperatures are shown in Figure 4 - 12. At a rolling temperature of 780°C (Figure 4 - 12d), the presence of equiaxed grains indicates that static recrystallization had occurred prior to quenching.



Figure 4 - 11: Shear band content of the various materials



Figure 4 - 12: Microstructures of the LC-High Cr sample warm rolled at (a) 440, (b) 640, (c) 710 and (d) 780°C, Mag. 50x

4.3 ANNEALING EXPERIMENTS

Samples of the warm-rolled materials were annealed in a salt bath, as described in section 3.4, and then studied using an optical microscope. These experiments were carried out in order to gain some insight into the recrystallization behaviour of the materials. The annealing behaviour is important as it affects the formability of the final product, which is critical for deep drawing applications. A concurrent project focusing on

the recrystallization textures of the materials after annealing is being carried out⁵⁸. The fraction of material that had recrystallized for each sample was calculated using a simple point counting technique and this fraction is plotted as a function of annealing time in Figure 4 - 13. The error bars and confidence limits associated with this figure will be discussed in section 5.3. It is apparent from this graph that all three materials exhibited behaviour consistent with JMA kinetics, as each curve is "S" shaped. The time to 50% recrystallization ($t_{0.50}$) was estimated from this plot and is listed in Table 4 - III. The ELC sample recrystallized most rapidly, followed by the LC-High Cr alloy.



Figure 4 - 13: Fraction Recrystallized vs. Annealing Time

Sample	t _{0.50}
ELC	14 s.
LC-Low Cr	75 s.
LC-High Cr	39 s.

Table 4 - III: Time to 50% recrystallization

The microstructures at various annealing times for the LC-High Cr alloy rolled at 710° C are shown in Figure 4 - 14. After 10 seconds, little or no recrystallization had occurred. The grains were elongated along the rolling direction, a substantial portion of which contained shear bands. As the annealing time was increased, so did the number of recrystallization nuclei. These new grains contained no shear bands and possessed an equiaxed morphology. The nuclei of the recrystallized grains were located primarily on the in-grain shear bands in the chromium alloys (Figure 4 - 15), but along grain boundaries for the ELC steel (Figure 4 - 16).







(d) 45 s

(e) 100s

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Figure 4 - 15: Microstructure of LC-High Cr alloy showing recrystallized

grains on shear bands, Mag. 1000x



Figure 4 - 16: Microstructure of ELC steel showing recrystallized grains on grain boundaries, Mag. 1000x

Chapter 5

Discussion

5.1 DSA BEHAVIOUR

5.1.1 Flow Stress

The variation of flow stress with temperature was plotted for each material, as seen in Figures 4 -2 to 4 -4 above. All three materials behaved as expected from previous results^{4,15,52}. Strong peaks were noted on the ELC curves (see Figure 4 -2), due to the presence of carbon and nitrogen in solution, which is consistent with DSA theory. The curves for each of the three strain rates were similar in shape. It was also noted that the curves shifted to higher temperatures as the strain rate was increased. Since dislocations move more rapidly at higher strain rates, solute carbon and nitrogen atoms must diffuse at greater rates in order to pin the mobile dislocations. Thus an increased temperature is necessary in order to provide sufficient mobility to the solute atoms for DSA hardening conditions to be satisfied.

The only discrepancy found was in the 10^{-3} s⁻¹ data; they did not display the decrease in flow stress in the lower temperature range (~ 100° C) associated with the curves for the other two strain rates. This behaviour was attributed to the low temperature, which certainly impeded the necessary diffusion rate. Since it was the warm rolling temperature range that was of interest in this study, this abnormality was not of great concern, as it was not within the temperature range of interest.

The flow stress vs. temperature plots for the LC-Low Cr and LC-High Cr alloys were shown above in Figures 4 -3 and 4 - 4, respectively. Both materials exhibited similar DSA behaviours. The most obvious difference between these curves and those of the ELC steel is that the latter contained a single sharp DSA peak, as compared to the broader, double peaks present in the two chromium alloy curves. As with the ELC steel, the curves for the three different strain rates were similar in shape and shifted to higher temperatures as the strain rate was increased. The lack of a dip in the 10^{-3} s⁻¹ curve was also noted in the LC-High Cr alloy, due to the sluggish diffusion rates associated with the low temperature. This was absent in the LC-Low Cr sample. The item of greatest interest was the appearance of the second DSA peak, which was present in both the high and low chromium LC alloys. This was attributed to the addition of chromium^{4,15,52}. It has been postulated that the formation of an interstitial carbon-chromium type complex is responsible for this behaviour; however, this mechanism is not clearly understood⁵⁹. This complex removes carbon from the matrix and thus alters the DSA behaviour.

To examine the effect of chromium addition more closely, a graph comparing the flow stress at 10% strain of each of the three materials was constructed for a common strain rate, as shown in Figure 4 - 5. There was a notable decrease in the flow stress peak, which resulted from the chromium addition. This was probably due to the formation of chromium carbide precipitates, which removed carbon from solution, therefore decreasing the DSA effect due to carbon. Despite this decrease in the flow stress peak, solute hardening occurred as a result of the chromium addition⁷. At temperatures above the DSA region, there was an increase in the flow stress of the chromium-modified alloys as compared to the ELC steel. The second peak present in the alloys containing chromium was due to the DSA effect of the carbon-chromium complex. This is located at a higher temperature than the carbon peak, as the larger chromium molecule requires higher temperatures to obtain sufficient mobility to pin the mobile dislocations. This broadening of the overall curve due to the second peak affected the DSA behaviour in the warm rolling temperature range (approximately 350-500°C under laboratory conditions) altering the rate sensitivity within this range.

5.1.2 Serrations

The presence of serrations was observed on the stress-strain curves of all three steels and an Arrhenius plot of ln \dot{e} vs. inverse temperature was constructed for each (see Figures 4 – 6 through 4 – 8). The activation energies associated with the onset and termination of DSA behaviour (serrations) were determined and were listed in Table 4 - I. The activation energies associated with the onset and termination of serrated behaviour in the ELC steel, which were 71.2 and 123 kJ/mol respectively, were consistent with those obtained by other researchers^{14,54-56}. Values for this lower and upper limit for the chromium alloys were similar to each other, approximately 63 and 95 kJ/mol, respectively, but these are much lower than the other values. This difference is attributed to the chromium additions, as all the other samples were LC steels. The effect of chromium addition on these transitions is complex and remains to be explained.

The boundaries associated with the onset and termination of serrated behaviour are vital in establishing the extrapolated warm rolling temperature range. Actual warm rolling is carried out at strain rates of the order of 10^2 s⁻¹, while the laboratory scale experiments were conducted at about 10^{-2} s⁻¹. The serrated behaviour was present at all strain rates; this observation permits determination of the temperature range over which DSA behaviour is expected to be present. Once this range is established, laboratory scale experiments can be conducted to predict the behaviour expected under mill operations. Typical warm rolling operations are conducted between 650 and 850°C, while this temperature range, once extrapolated to lower strain rates, is roughly equivalent to 350 to 600°C for laboratory experiments, as indicated in Figure 2 - 6 and Figures 4 - 6 to 4 - 8.

5.1.3 Rate Sensitivity

The rate sensitivities of the three materials were calculated from the compression test results. Figure 4 - 10 above showed the rate sensitivities of the test materials as a function of deformation temperature. As illustrated in Figure 2 - 8, the presence of interstitial elements, such as carbon and nitrogen, produces a strong peak on the flow stress vs. temperature plot due to the DSA behaviour associated with these elements. The

introduction of this DSA behaviour results in negative as well as highly positive values of m. The DSA peak leads to negative rate sensitivities up to approximately 250°C; the value of m then steadily increases up to 800°C. The addition of chromium, and the resulting broadening of the DSA peak, altered the rate sensitivity within the extrapolated warm rolling temperature range as compared to the behaviour of the ELC steel. The rate sensitivity of the ELC sample was negative at temperatures below 250°C and then rapidly increased to highly positive values at more elevated temperatures. The alloys containing chromium exhibited similar behaviour without such extremes.

Since the ELC steel had a higher flow stress peak and a sharper peak (Figure 4 - 5), it also exhibited a more negative minimum rate sensitivity as compared to the two chromium alloys. The addition of chromium resulted in considerable peak broadening, leading to a slight reduction in the rate of change of flow stress with temperature, and hence a reduced rate sensitivity. Within the warm rolling temperature range, the rate sensitivities of both the low and high chromium LC alloys were significantly lower than that of the ELC steel. The behaviour of the LC-High Cr alloy was of particular interest. Within the extrapolated warm rolling range, its behaviour was similar to ideal behaviour, yielding a slightly positive m. Work by Barnett *et al.*^{29,57} revealed similar behaviour when 1.3 wt.% chromium had been added to warm rolled steels. A strong {111} texture resulted from this alloy addition, which is similar to that obtained for warm rolled IF steels that have good formability. From these results, it was expected that the LC-High Cr alloy would produce a moderate amount of shear bands during warm rolling and ultimately produce a product with good formability.

It should be pointed out that the two chromium modified steels investigated here both contained about 40 ppm boron. Such boron additions tend to segregate to grain boundaries (and dislocations) and can have significant effects on recrystallization behaviour. These can in turn affect the textures formed during annealing. There are some indications⁵⁸ that the present boron additions are in fact modifying the annealing textures in an undesirable way. However, definitive results regarding these effects were not available at the time of submission of this thesis.

5.2 SHEAR BAND FORMATION

The samples warm rolled at 440, 640, 710 and 780°C were studied and their shear band content determined. Microstructures of the LC-High Cr alloy at the various rolling temperatures were displayed in Figure 4 - 12 above and the fraction of material containing in-grain shear bands was plotted as a function of rolling temperature in Figure 4 - 11. The shear band fraction decreased with increasing rolling temperature in all three materials; however, this decrease was most drastic in the ELC steel. As shown in Figure 4 - 10, the rate sensitivity of the ELC steel increased rapidly with temperature within the extrapolated warm rolling range. In a region of highly positive m, little or no shear bands will form as flow localization is not favoured. The greatest fraction of shear bands present was in the 440°C specimens in all cases, which corresponded to a region of negative m, as this lies within the temperature range of the DSA peak on the flow stress vs. temperature plot. In regions of m below zero, this material will deform at a flow stress below that of the bulk. Therefore, these regions experience flow localization as the material seeks to deform in the regions requiring the least work. The significant decrease in shear band fraction at 780°C in both the low and high chromium LC alloys occurred since the temperature was sufficient for recrystallization to occur. These recrystallized grains are apparent in Figure 4 - 12d.

The data illustrated in Figure 4 - 11 confirm the results obtained from the compression tests. The warm rolled samples (640 and 710°C) were characterized by a moderate amount of shear bands when chromium was added. The measured rate sensitivities predicted this outcome. As the LC-High Cr alloy contained a slightly greater shear band fraction than the low Cr alloy in the warm rolling temperature range, it is expected that this material will yield a stronger {111} texture, as this texture is thought to nucleate on shear bands. Grains with higher dislocation densities will recrystallize preferentially during annealing⁴⁷. Shear bands contain dislocations that can provide preferential sites for recrystallization nuclei with a {111} orientation²⁸. The orientations of the nuclei depend on the orientations of the material within the shear bands. Since the LC-High Cr alloy contained the greatest amount of shear bands, but not excessively so, it

is predicted to ultimately have better formability than both the LC-Low Cr and ELC samples as it is expected to possess a stronger {111} texture.

5.3 RECRYSTALLIZATION BEHAVIOUR

Specimens cut from samples warm rolled at 710°C were annealed to produce partial recrystallization microstructures and these specimens were shown in Figure 4 - 14. The fraction of material recrystallized was plotted against annealing time in Figure 4 - 13. All three materials displayed recrystallization kinetics consistent with JMA behaviour in that the recrystallized fraction vs. annealing time plots were "S" shaped. In the ELC samples, recrystallization was very rapid; over 90% recrystallization was achieved in 20 s. This was believed to have occurred largely due to the presence of solute carbon⁴, as increased solute carbon leads to stronger DSA behaviour, increasing the dislocation density and, therefore, the driving force for recrystallization. The LC-Low Cr alloy recrystallized at a much slower rate than the ELC steel, due to the increased grain boundary pinning associated with the presence of chromium carbide precipitates. The LC-High Cr alloy recrystallized at an intermediate rate. The LC-High Cr alloy recrystallized more rapidly than the Low Cr alloy due to its greater shear band content and hence increased the dislocation density⁶.

In order to examine the recrystallization kinetics, the experimental data were compared to the JMAK model by plotting $\ln[1/(1-X_{\nu})]$ against annealing time, where X_{ν} is the fraction of recrystallized material, on a log-log scale³⁸ as shown in Figure 5 - 1. Next, straight lines were fitted to the data, the slopes of which are equal to the exponent *n* of Equation 10^{38} :

$$X_{v} = 1 - \exp(-Kt^{n})$$

This method provides an equation to fit the data on the JMA plot (see Figure 4 - 13). For this purpose, the temperature-dependent constant K was estimated using Solver (Excel). The error bars on Figure 4 - 13 were calculated using the expression proposed by Gladman and Woodhead⁶⁰:

$$\left(\frac{\sigma_{X_v}}{X_v}\right)^2 = \frac{1 - X_v}{n_r} \tag{16}$$

where σ_{Xv} is the standard deviation and n_r is the number of points measured in the recrystallized grains. In the current study, the *total* number of points counted was used for n_r , as suggested by Orsetti Rossi and Sellars⁶¹. This substitution yields more reasonable values in the vicinity of 50% volume fraction recrystallized, allowing $t_{0.5}$, the time to 50% recrystallization, to be determined with acceptable accuracy. This method is valid within the range $0.1 \le X_v \ge 0.9$, indicated by the confidence limits on Figure 4 - 13. A fraction of recrystallized material below 0.1 would require too many measurements to obtain accurate results and above 0.9, it becomes difficult to differentiate the recrystallized grains from the untransformed regions^{38,61}. Note that several values were outside the range of accuracy: $0.1 \le X_v \ge 0.9$, which is delimited by the dashed horizontal lines in Figure 4 - 13 (confidence limits).



Figure 5 - 1: Experimental JMAK plots for the three test materials

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The JMAK model was applied to the annealing data from each material, with the results illustrated in Figure 5 - 1. A straight line was fitted to each series in order to measure the slope, which corresponds to the exponential factor in the JMA equation (n). The measured values of n are listed in Table 5 - I below. However, the use of these values resulted in a poor fit to the recrystallization data once inserted into Equation 10. For this reason, values for n were established empirically instead and these values were employed to plot the recrystallization behaviour that was presented in Figure 4 - 13 above.

Sample	n (JMAK)	n (Experimental)
ELC	1.7	3.7
LC-Low Cr	2.4	3.3
LC-High Cr	2.2	3.5

Table 5 - I: Values of *n* for the various materials

5.3.1 Formability and Texture

In a concurrent project by Toroghinezhad *et al.*⁵⁸, samples of these warm rolled materials were annealed and the textures were measured. It was noted that the addition of chromium decreased the effect of rolling temperature on the texture intensity. In both chromium alloys, the {111} texture intensity was significantly increased at warm rolling temperatures compared to the ELC material. Shear bands were deemed to be vital in providing intragranular nucleation sites for {111} oriented grains. However, a {111} texture sufficiently strong to provide good formability was not produced. It is postulated that this was due to the disruption of the annealing texture by the presence of solute boron⁵⁸.

Chapter 6

Conclusions

- All three steels exhibited DSA behaviour during compression testing. The ELC material showed a sharp, single peak in the flow stress vs. temperature curve, whilst the Cr modified alloys both displayed a decrease in flow stress, as well as the presence of a second DSA peak at a higher temperature than the carbon peak. It is postulated that the formation of an interstitial carbon-chromium type complex is responsible for this behaviour. This second peak extended the DSA behaviour into the warm rolling range.
- 2. Serrations were present on the stress-strain curves, at certain temperatures, for all three steels. Arrhenius plots detailing the temperatures of onset and termination of these serrations suggest that the activation energy for diffusion of the DSA solute in the LC-Cr alloys is less than in the ELC steel. There is no current explanation or interpretation of this observation.
- 3. All three materials exhibited negative rate sensitivities at temperatures below 250°C and the ELC material displayed high positive values above 400°C. The addition of chromium reduced the rate sensitivity in the extrapolated warm rolling temperature range, as it resulted in considerable broadening of the DSA peak and a reduction in the flow stress within this range. The rate sensitivity of the LC-High Cr alloy (0.8 wt.% Cr) was lower than that of the LC-Low Cr material

(0.5 wt.%). Within the warm rolling temperature range, its behaviour was similar to that of materials that do not display DSA behaviour; thus a slightly positive m was observed.

- 4. The warm rolled microstructures all contained in-grain shear bands, the fraction of which decreased with increasing temperature in each case. More shear bands were present in the Cr-modified alloys than the ELC material at warm rolling temperatures, due to their decreased rate sensitivities.
- 5. All the warm rolled and annealed materials displayed recrystallization kinetics consistent with the JMA model. The ELC steel recrystallized the most rapidly, followed by the LC-High Cr alloy. The LC-High Cr sample was thought to recrystallize more rapidly than the low Cr alloy, since it contained a greater density of shear bands and hence possessed a higher dislocation density. The latter provides a higher driving force for recrystallization.
- 6. It was noted that recrystallization was nucleated primarily along shear bands in the case of the Cr-modified alloys, but along grain boundaries in the ELC steel, as the ELC sample contained few or no shear bands within this temperature range.
- 7. Although the compression tests and warm rolling trials led to predictions that the LC-High Cr alloy would produce a warm rolled product with good formability, texture measurements resulting from a concurrent project do not support this analysis. The {111} texture was strengthened as compared to the ELC steel, but not sufficiently to provide good formability. More work must therefore be done to further strengthen this {111} texture. It appears advisable that no boron additions be made to future experimental LC-Cr alloys, as the segregation of solute boron to grain boundaries may be responsible for disrupting the annealing texture.
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Appendix

HEATING PROFILE

The following heating schedule was used for all compression tests:

- 1) Heat to 900°C in 10 minutes.
- 2) Hold at 900°C for 15 minutes.
- 3) Cool quickly to A
- 4) Allow to settle at B
- 5) Hold at B for 5 minutes
- 6) Test

Temperature B is the test temperature and A is a temperature below the test temperature. Because the sample was cooled using compressed air, once the air was turned off, the temperature of the specimen rose significantly. Therefore, it was decided that the specimen should be overcooled so that this natural increase in temperature would bring the sample to the desired test temperature (see Figure A - 1). It was apparent that the cooling rate was not constant. The cooling rate was in the order of 300°C/min down to 500°C, but then slowed to approximately 50°C/min. The average cooling rate across the entire range was estimated at 100°C/min. Table A - i contains the temperature values of A and B used for the different test temperatures.



Figure A - 1: Temperature profile, LC-High Cr, 300°C, strain rate = 10^{-1} s⁻¹.

Test Temperature	А	В
800°C	750°C	800°C
700°C	650°C	700°C
600°C	550°C	600°C
500°C	450°C	500°C
400°C	370°C	400°C
300°C	270°C	300°C
250°C	220°C	250°C
200°C	170°C	200°C
150°C	125°C	150°C
100°C	85°C	100°C
25°C	85°C	25°C

Table A - i: Values of A and B for all Test Temperatures

TEST SCHEDULE

The sample was placed in the furnace and the upper anvil was lowered to just a few millimetres short of contact with the sample. The general schedule, which was programmed into the MTS TestStarTM software, was almost identical for the three strain rates used $(10^{-1} \text{ s}^{-1}, 10^{-2} \text{ s}^{-1} \text{ and } 10^{-3} \text{ s}^{-1})$ except for the time between points and the deformation rate. Table A - ii contains the particulars for each strain rate and the general program is located in Table A - iii.

Table A - ii: Test Particulars

Strain Rate	Time Between Points (Δ)	Deformation Rate (R)
10^{-1} s^{-1}	0.00100 s	1.14 mm/s
10^{-2} s^{-1}	0.0100 s	0.114 mm/s
10^{-3} s^{-1}	0.100 s	0.0114 mm/s

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Table A - iii: General Test Schedule

STEP	START	FINISH		DETAILS
Specimen Contact	<procedure>start</procedure>	Contact Detect done	 Shape 	e: RAMP
			 Rate: 	0.100 mm/s
			 Relati 	ive End Level: -10.00 mm
Contact Detect	<procedure>start</procedure>		 Signa 	ul: Channel 1 Force
			• Uppe	r Limit: +32.500 kN
			 Lowe 	rr Limit: -50.000 kN
Heating Hold	Contact Detect done	Start Test. Trigger	• Time:	: 60 min
			 Contr 	rol: Channel 1 Force
Start Test	Contact Detect done		Butto	n 1
			 Trigg 	er 1 (manual)
Data Acquire	Start Test. Trigger	Deformation done	• Time	Between Points: Δ
			 Buffe 	r Size: 10 000
			 Buffe 	r Type: LINEAR
			 Speci 	imen Data File
			 Includ 	de: time, Channel 1 Force, displacement,
			tempe	erature
Deformation	Start Test. Trigger		 Shape 	e: RAMP
			 Rate: 	R
-			 Relati 	ive End Level: -2.200 mm
Unload	Deformation done		 Shape 	e: RAMP
			 Rate: 	5.00 mm/s
			 Relati 	ive End Level: 5.000 mm

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CORRECTION OF STRESS-STRAIN DATA

Once the data from the compression tests were collected and analyzed, a plot of engineering stress vs. engineering strain was produced for each test. A typical plot of such data is illustrated in Figure A - 2. It is noted, however, that there is a linear portion at the beginning of the stress-strain curve; this corresponds to the elasticity of the compression machine and tools. The data were corrected in order to account for this apparent strain. A line was drawn through this straight segment and the equation that fitted it was calculated (y = mx + b). The apparent strain associated with the compression machine (ϵ_{CM}) was then determined using the equation of this line as follows:

$$\varepsilon_{CM} = \frac{\sigma_{eng.} - b}{m}$$
(i)

where σ_{eng} is the engineering stress, b is the y-intercept of the straight line and m is its slope. The adjusted strain (ε_{adj}) was then calculated by subtracting the strain of the compression machine from the engineering strain (Equation ii), which represented the actual strain in the sample. It was this value that was then plotted against engineering stress (Figure A - 3) and used for further calculations.

$$\boldsymbol{\varepsilon}_{adj_{\perp}} = \boldsymbol{\varepsilon}_{eng_{\perp}} - \boldsymbol{\varepsilon}_{CM} \tag{11}$$



Figure A - 2: Uncorrected Stress-Strain plot for the LC-Low Cr specimen, 800°C, strain rate = 10^{-1} s⁻¹



Figure A - 3: Corrected Stress-Strain plot for the LC-Low Cr specimen, 800°C, strain rate = 10^{-1} s⁻¹