CONTINUOUS ANNEALING OF LOW-CARBON STEEL

by

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ABSTRACT

Commercial quality low-carbon steel sheet, previously cold reduced 60% was initially recrystallized at 705°C for times of 30 minutes and 24 hours to produce material of two initial grain sizes. The recrystallized strip was cold deformed at room temperature in the range 0-28% (equivalent tensile deformation) and annealed at temperatures in the range 610-715°C for 10 minutes in order to recrystallize the material for a second time. The response of the material to the amount of prior deformation as well as annealing temperature was observed by following changes in yield strength, tensile strength, uniform elongation, total elongation, and recrystallized grain size.

It was found that with prior deformations in the range 21-28% and with a high annealing temperature, 705°C, material could be produced with comparable properties to very ductile material obtained with the present annealing methods. The results suggest that by combining two recrystallization steps, the first being necessary to obtain an initial fine recrystallized grain size, with an intermediate controlled deformation step, material could be continuously produced to suit a wide range of applications. Suggestions are also made for carrying the work beyond the initial stages described in the present thesis.

RESUME

Une fenillo d'acier à basse teneur de carbone de qualité commerciale, antérieurement réduite à froid de 60%, avait été initialement recristallisée à 705°C à des temps de 30 minutes et 24 heures pour produire un matériau dont les grains sont de deux tailles différentes. La bande recristallisée a été déformée à froid à la température ambiante variant de 0-28% (déformation équivalente en tension) et recuit à des températures variant de 610-715°C pour 10 minutes pour faire recristalliser le matériau une deuxième fois. La réaction du matériau à la quantité-initiale de déformation ainsi qu'à la température de recuit a été observée par les changements de la limite d'élasticité, de résistance à la traction, d'élongation uniforme, d'élongation totale et par la taille des grains recristallisés.

Il a été constaté qu'avec des déformations initiales de l'ordre de 21 à 28%, et avec une haute température de recuit, 705°C, le matériau peut être produit avec des propriétés comparables au materiau très ductile obtenu par les methodes actuelles de recuit. Les résultats suggèrent qu'en combinant deux étapes de recristallisation, la première étant nécessaire pour obtenir une taille de grain recristallisé initialement petite, avec une étape intermédiaire de déformation contrôlée, le matériau peut être produit continuellement pour être utilisé dans un large éventail d'applications. Des suggestions sont aussi faites de manière à porter le travail au delà des limites initiales décrites dans la présente thèse.

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CHAPTER 1

1. INTRODUCTION

The importance to the steel industry of annealing practices in the manufacture of cold-reduced flat-rolled products cannot be overemphasized. Except for the very small proportion of full hard strip and sheet used in the as-cold-reduced state, some form of heat treatment is applied as a separate operation to all cold-reduced flat-rolled products in order to restore the ductility lost in cold reduction before further fabrication.

Heat treatment of cold-reduced sheet, strip and tinplate stock may be divided into batch operations, such as box annealing or open-coil annealing, and continuous operations that include continuous annealing, strand annealing and normalizing.

In the batch annealing processes, the steel is slowly raised to a temperature level at or below the lower critical temperature (approximately 720°C), soaked at that temperature for several hours and then cooled very slowly to room temperature. The complete temperature cycle takes several days but it does result in the softest possible finished product; accordingly, batch annealing is the principal heat treatment applied to cold reduced steel since the largest proportion of low-carbon steel is destined for severe fabricating operation (e.g. in the manufacture of automobiles and household appliances).

In continuous heat treatment on the other hand, the steel is heated quickly to the annealing temperature, held a short time, typically a matter of minutes, and then quickly cooled. As a consequence, the finished steel is very fine-grained and thus harder than that produced by batch annealing. This fine-grained microstructure is acceptable and even highly desirable in some applications, for example in the production of the hard tinplate desired by the can manufacturing companies. Continuous annealing can also be attractive economically, particularly if it is combined with one or more additional processes; for instance in continuous galvanizing where the combination of annealing and hot-dip galvanizing produces savings resulting from the elimination of the skin rolling, shearing, pickling, fluxing and handling steps previously carried out as separate operations.

However, the most extensive application of annealed steel sheet is in the production of formed parts, particularly for automobile production, i.e. the annealing process must yield steel sheet suitable for deep drawing. The main obstacles to the successful application of continuous annealing to drawing quality steel are metallurgical and are a direct result of the speed of the continuous annealing process. The high heating rates recrystallize the material to a very fine grain size and the short time at recrystallization temperature does not allow grain growth and equilibrium carbon precipitation from solid solution in the ferrite. Hence continuously annealed steel has a yield strength too high, and a ductility too low, for it to be suitable for forming operations and it would be susceptible to quench and strain aging, both of which are detrimental to successful formability.

Despite these drawbacks, further investigations of the continuous annealing of drawing quality steels are worthwhile due to the economic attractiveness of the process: reduction of the time spent heating tightly wound (virtually solid) coils of steel would result in significant energy savings; it would eliminate the large amount of floor space and in-process inventory of steel that batch annealing entails; it would reduce handling; and allow a smooth flow of material from hot rolling to finished material. Hence, the following section comprises a survey of literature relating to continuous annealing made with a view to investigating the possibility of a continuous process to enable the production of drawing quality sheet.

CHAPTER 2

2. LITERATURE SURVEY

2.1 Heat Treatment of Flat-Rolled Steel

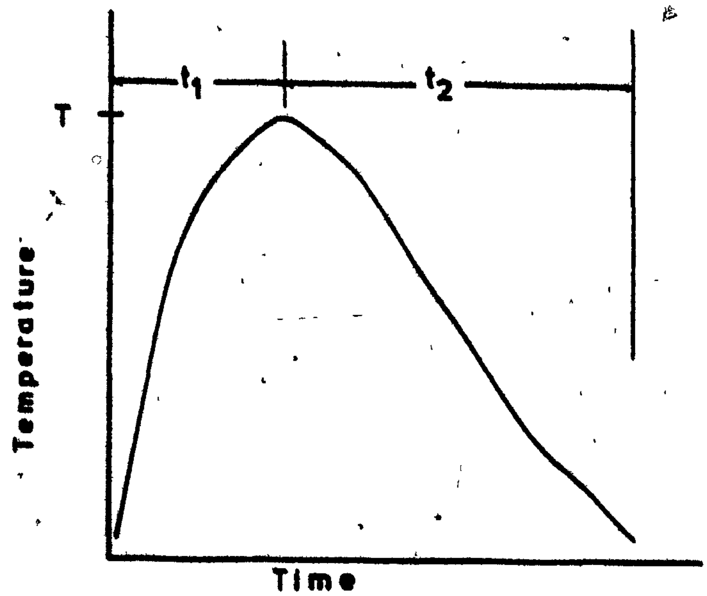
In this section both methods for the annealing of flat-rolled low-carbon steel, i.e. batch and continuous processes, are described and their relative merits assessed.

2.1.1 Batch Annealing Processes

A. Box Annealing

In box annealing a large stationary mass of steel, in the form of tight coils that can weigh up to ten tons and more, is subjected to a heat treatment cycle. The coils are stacked four or five high (i.e. eight to twelve feet high) onto shallow trays of cast iron called annealing bases. Thermocouples are inserted in standard locations in the charge and an inner cover is then lowered over each stack of coils and settled in a sand seal placed on the annealing base. A portable furnace is lowered onto the base, fuel line and thermocouple connections are made, the flow of deoxidizing gas to purge the air from the space under the inner cover is begun and the burners are ignited.[1-5]

As box annealing involves the heating and cooling of large masses of steel, processing times are necessarily long. Figure 2.1 shows a typical heating and cooling cycle; average processing times vary from six to ten days, an undesirable length of time from the standpoint of production rate. A further problem encountered in box annealing results from the use of tightly coiled steel; the outside of a coil must



T : Heating temperature (680°C)

t_1 : Heating time (2 days)
t_2 : Cooling time (4 days)

FIGURE 2.1 Box annealing heat cycle.

be heated to a higher temperature than the inside in order that the required temperature may be reached at the centre. The temperature profile across the coil results in differences in recrystallized grain size and consequently in final mechanical properties.[2]

A temperature differential also exists between the outer limits of the stack. Generally, two thermocouples are placed in any single or multiple coil stack, one in or near the top edge of the upper coil and the other near the lower edge of the bottom coil, thus giving a clear indication of the temperature differential between the outer

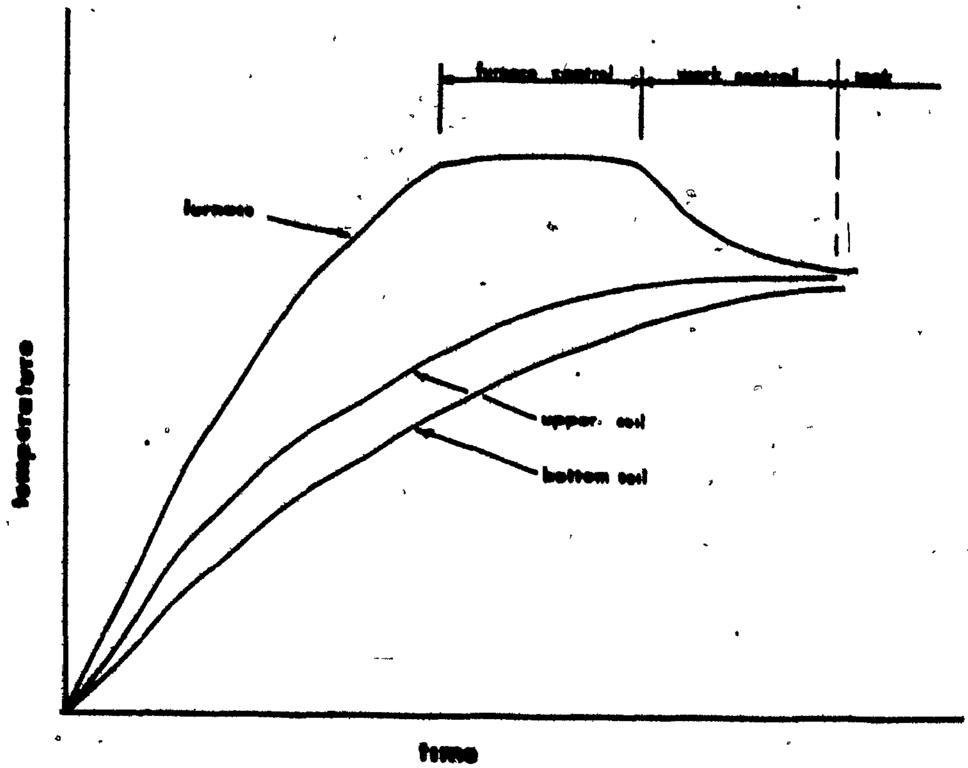


FIGURE 2.2 Typical temperature recording chart.[2]

limits of the stack as shown in Figure 2.2. This temperature differential between coils in the same stack also results in a variation of mechanical properties across the stack.

B. Open-Coil Annealing

Open-coil annealing was originally developed to shorten the long annealing times necessary in box annealing and thus increase production rates. The only difference between the two processes is that while the box annealing uses a tightly wound coil, the open-coil process uses a loose coil. Conventional tightly wrapped coils are rewound using a metal spacer between the wraps; on removing the separator, a space is left between adjacent turns. After annealing, the coil is rewound tightly for further processing or shipment.(6-10)

The chief advantage of open-coil annealing is that during annealing, the hot gases can circulate through the spaces between the coil wraps, thus improving heat transfer and resulting in reduced annealing times. An additional advantage of this free gas flow is the ability to utilize any desired annealing atmosphere, permitting such gas-metal reactions as decarburizing.

As with box annealing, furnace temperature is controlled by thermocouples spaced through the charge: usually in the furnace wall, under the bottom coil and in the mid-thickness of the top coil. Figure 2.3 illustrates a typical temperature cycle; a temperature differential does occur between coils from the same stack but it is not as large as those observed in conventional box annealing.

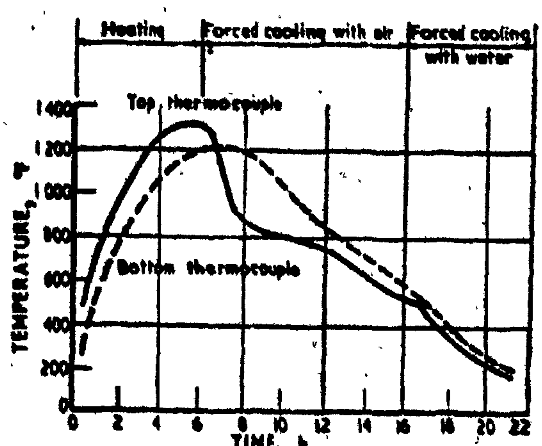


FIGURE 2.3 Cycle for annealing cold-rolled sheet by means of open-coil technique.(9)

The length of the temperature cycle in open-coil annealing, typically one or two days, represents a marked improvement over box annealing but it still represents a considerable obstacle to productivity. Other disadvantages include increased age hardening sensitivity due to the faster cooling rates, the hazard of coil breakage during rewinding and the increased operational costs of open-coiling and the rewinding of each coil as well as reduced furnace capacity caused by the larger volume of an open coil vis-a-vis a closed coil.

2.1.2 Continuous Annealing

2.1.2.1 Historical Development

When modern cold-reduction mills had become common in steel plants (i.e. since about 1930), the only unit in which there was no continuity of flow was the annealing department. This lack of flow brought about difficulties in handling the high tonnages produced by those mills, causing delays, storage problems, etc., even with the use of the newest annealing technique, open-coil annealing.

As a consequence of investigations carried out by Hague and Brace[11] and Otis[12], who explained the possibilities for the use of continuous annealing for the manufacture of tinplate stock, the first furnace for the production of continuously annealed tinplate stock was installed at the Crown Cork and Seal Company, Baltimore, U.S.A., in 1936; it was a vertical two-strip unit, with an electric radiant-heating furnace, operating at 75 ft./min.[13] Two further lines with speeds of up to 300 ft./min. were erected soon afterwards by the same company. The product was considerably stiffer and harder than the equivalent batch annealed material and was looked upon with scepticism by the tinplate industry and was not accepted by the open-top canmakers, as it was not compatible with existing equipment for forming can bodies, bottle caps, etc..

New attempts for developing continuous annealing processes were made in 1940, when a 300 ft./min. line with an electric radiant-heating furnace was installed by Dominion Foundries and Steel Company, Canada. The product of this line, although disposed of successfully, did not make a great impact on the canmaking trade and the investigations were discontinued. However, in the early 1950's canmakers realized that the high degree of uniformity in the mechanical properties of continuously annealed tinplate had a beneficial effect on canmaking speeds, once the automatic forming equipment had been adjusted to process harder sheet. Several advantages were observed for both canmakers and steel producers; the strength of the sheet prevented buckling and panelling in the finished can, and the improved flatness allowed steel producers to process larger coils with consequent beneficial influence on throughputs. Increases in throughput of between 25 and 50% were quoted.[14] With these proven advantages, the development of continuous annealing processes received greater impetus.

2.1.2.2 Continuous Annealing-Line Description

Figure 2.4[15] illustrates a typical continuous annealing line which, for convenience of description, may be divided into three sections; entry, furnace and exit. In the direction of sheet travel, the entry section consists of a double payoff reel, shears for cutting off and

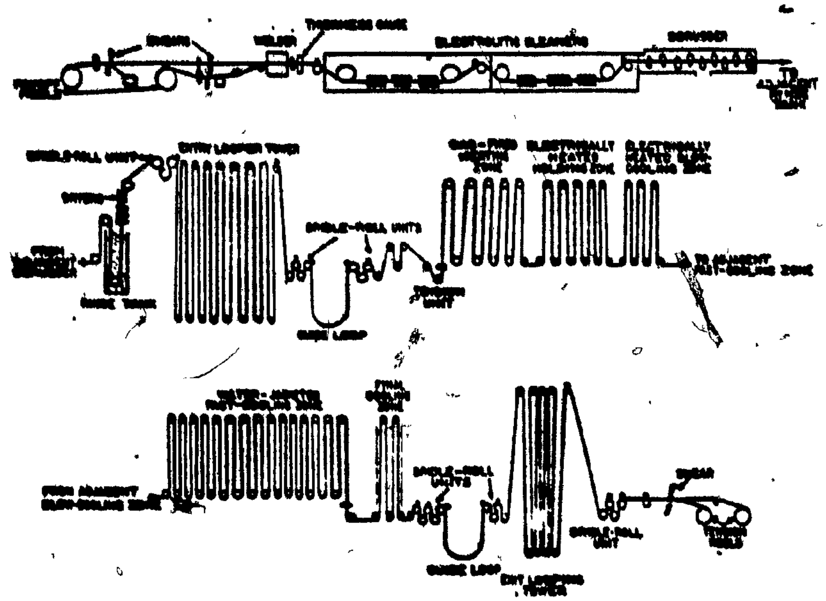
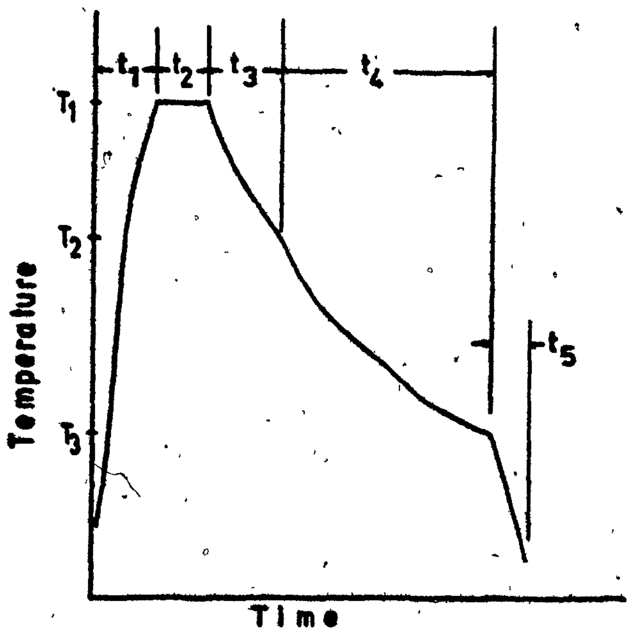


FIGURE 2.4 Schematic continuous annealing line illustrating the path of the sheet through the line(15), designed to operate at 1500 ft./min.. Not to scale.

squaring the ends of coils for welding, a mash type welding unit, a dip tank with an electrolytic cleaner to remove any contamination from the sheet, a brush scrubber unit followed by a rinse tank and drying unit, and an entry looping tower for sheet storing and from which sheet is taken to maintain constant line speed when a weld is being made. The purpose of this entry section is to provide a constant feed for the furnace section that follows. On entering the furnace section the sheet is heated in a gas-fired zone to approximately 732°C (1350°F) in less than 30 seconds; it then passes through an electrically heated holding zone, followed by an electrically heated slow-cooling zone (also referred to as retard cooling) in which the sheet is cooled to approximately 537°C (1000°F) in less than 15 seconds. Finally, the sheet is water cooled to 115°C (240°F) in one minute, then air cooled to ambient temperature. In its passage through the furnace section, the steel is protected from oxidation by a protective gas atmosphere. Leaving the furnace section, the sheet passes through the exit section where it is coiled. This section consists of a looping tower, a bridle-roll unit, a shear and two coilers. The purpose of this section is two-fold: first, to be able to stop the strip for metallurgical analysis; and second, to change over from one recoiler to the other by means of snip shearing the strip.

A typical continuous annealing heating cycle is shown in Figure 2.5. Since its initial acceptance for the annealing of tinplate stock, this basic heating cycle has undergone numerous modifications, some of which are illustrated in Figure 2.6.[16] Each modification is designed to increase the production rate from a continuous annealing installation without increasing furnace size. In cycle B, the heating rate and soaking are the same as those employed in the conventional method (cycle A) but the cooling time has been reduced by water quenching the material from 300°C (572°F) to room temperature. Cycle C involves fast heating and eliminates the soaking period and the sheet is again water quenched from 300°C, thus achieving a time-saving in both heating and cooling stages; the most notable application of this annealing method was in four units installed by the Bethlehem Steel Company in



T_1 : Heating temp. (732°C)
T_2 : Temp. at the end of slow cooling zone (537°C)
T_3 : Temp. at the end of fast cooling zone (115°C)

t_1 : Heating time (20 sec)
t_2 : Soaking time (20 sec)
t_3 : Slow cooling time (12 sec)
t_4 : Fast cooling time (60 sec)
t_5 : Final cooling time (8 sec)

FIGURE 2.5 Typical continuous annealing heating cycle.

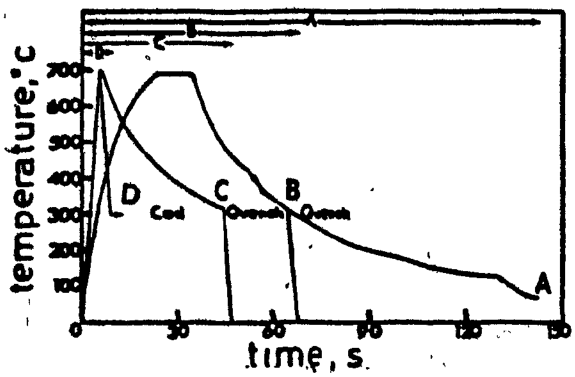


FIGURE 2.6 Deviations from the conventional continuous annealing cycle.(16)

1957. In cycle D the steel is quenched from the annealing temperature to 300°C, coiled and then held for at least 1 hour at 300°C. However, these modifications, and others made solely with the aim of increasing production rate, often had a detrimental effect on sheet quality and thus were not entirely successful.

2.1.3 Properties of Annealed Steel

The batch annealing methods with their long processing times yield sheets with very different characteristics from those produced by continuous annealing (Table I).

Continuously annealed steels have substantially higher yield and tensile strengths than comparable batch annealed steels and consequently exhibit lower ductility. Thus continuously annealed steels are unsuitable when a high degree of formability is required, e.g. the press-forming of automobile components. This serious limitation to the use of continuously annealed steels is discussed further in the next section.

TABLE I

Typical Mechanical Properties of Annealed Low-Carbon Steel

Mechanical Properties	Batch Annealed	Continuously Annealed
Yield strength (Psi)	$28x10^3$	$58x10^3$
Tensile strength (Psi)	$47x10^3$	$61x10^3$
Elongation in 2 in. (%)	44	20

2.2 The Production of Deep Drawing Quality Sheet by Continuous Annealing

2.2.1 Properties and Selection Criteria for Formability in Low-Carbon Sheet

During drawing operations, sheet steel undergoes severe plastic deformation; a number of test procedures have been found useful in the selection of steel for deep drawing, the tension test being one of the most useful since it relates mechanical properties to formability. For optimal formability, tensile strength should be high and yield strength low*; the greater the spread between the yield and tensile

* But low yield strength resulting from an exceptionally large grain size is undesirable due to "orange peel" (roughness) formation during forming; a very large grain size will also result in low values for elongation and tensile strength.

strengths, the more suitable the steel is for severe forming operation.[17,18] One criterion for formability is the yield-tensile ratio: the lower the yield-tensile ratio, the higher the formability. The conventional percent total elongation, being the measure of the ability of the material to undergo plastic deformation, is another important formability criterion but even more important is uniform elongation since it corresponds to the amount of plastic deformation possible before localized necking occurs and beyond which forming operations are undesirable.

Cold-rolled sheet for drawing applications is manufactured[19,20] in three main qualities: (i) commercial quality, produced from rimmed or capped steel with 0.15% maximum carbon content; (ii) drawing quality, produced from rimmed steel of SAE grade 1006 or ASTM A-619 for cold rolled sheet; and (iii) drawing quality special killed steel used to make parts involving difficult draws or where minimal aging characteristics are required. Typical mechanical properties are shown in Table II.[20] The variation in properties is greater for commercial quality than for drawing quality and is at a minimum for special killed steel.

Other criteria may also be used to assess the formability of steel sheet, e.g. plastic strain ratio "r", work hardening exponent, as well as values obtained from various cup-drawing tests, such as the Olsen and Erichsen. However, these tests are less commonly applied and specified than the ones quoted earlier. Hardness and Olsen cup test are formability tests usually made on incoming steel. These tests are not always an exact measure of formability but they are commonly used because they require a minimum of specimens for testing and are made quickly on relatively inexpensive machines.

TABLE II

Typical Mechanical Properties of Cold-Rolled Low-Carbon Sheet Steel for Forming

Steel Quality	Hardness Rockwell B	Yield Strength 1000 Psi	Tensile Strength 1000 Psi	Total Elongation in 2" (%)
CQ	40/60	28/36	40/48	36/41
DQ	38/48	25/32	40/46	38/43
DQSK	36/45	23/28	40/46	40/45

CQ: commercial quality
DQ: drawing quality
DQSK: drawing quality, special killed

The Olsen cup test is made on a sheet specimen 3 3/4 in. wide held between flat ring dies of 1 in. inside diameter. A ball of 7/8 in. diameter is pushed progressively against the sheet to form a cup while the punch load and height of cup are indicated continuously. The Olsen cup value recorded is the height of the cup in thousandths of an inch at the instant when the punch load starts to drop. Sheet thickness for the standard Olsen cup test is limited to 0.062 in., because of the clearance between the ball and the ring.

2.2.2 Metallurgy of Continuous Annealing

Once its composition has been fixed, the tensile and other properties of a steel are determined by its microstructure: grain size and shape, carbide morphology and inclusion content. The inclusion count is controlled by the steelmaking practice but the other factors are determined during final recrystallization annealing. There are two metallurgical phenomena in annealing[21] that are of importance in the production of drawing quality sheet: recrystallization and grain growth, and aging reactions. Each is discussed briefly in the following section.

2.2.2.1 Recrystallization and Grain Growth

In continuous annealing, the sheet is rapidly heated and held for a short time at the recrystallization temperature. Studies[22-24] of the continuous annealing of low-carbon steel have shown that with heating rates in excess of 125°C/sec. up to an annealing temperature of 700°C, recrystallization occurs instantaneously[16], i.e. within a matter of seconds. However, it recrystallizes to a fine grain size that does not then coarsen appreciably even after a 20-hour soak at this temperature, (Figure 2.7). This contrasts with the distinctly coarser initial grain size obtained with the slow heating rates characteristic of batch annealing also illustrated in Figure 2.7 (and presumably due to differences in the nucleation and growth rates obtained with slow heating rates).

It is generally held that sheet steel of very fine grain size does not have the good ductility that is characteristic of batch-annealed sheet steel; the finer the grain size, the higher the tensile strength and consequently the lower the ductility. As the grain size becomes finer the yield elongation increases and this also results in a loss of ductility. The yield elongation must be eliminated by temper rolling after annealing, and hence as the grain size becomes finer, more

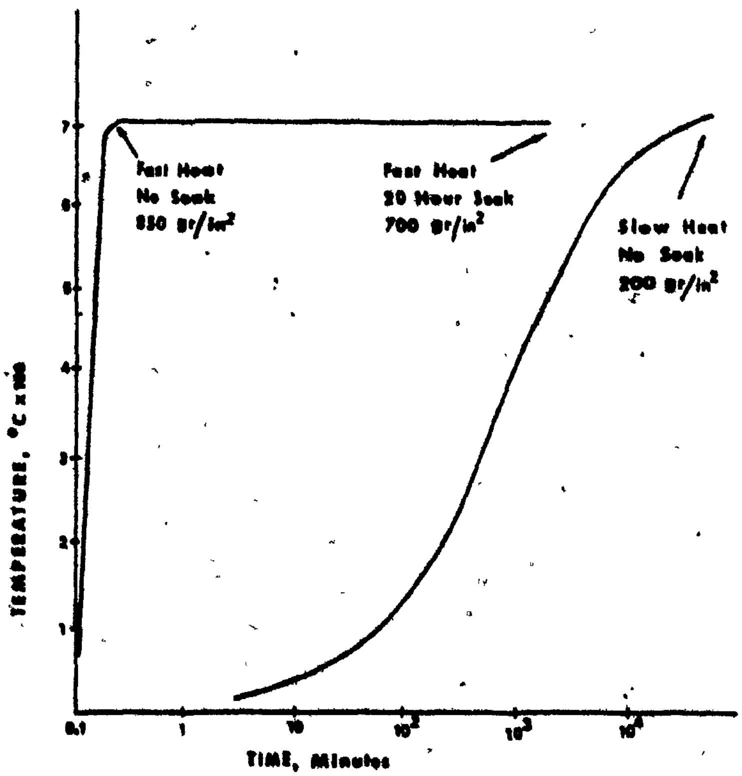


FIGURE 2.7 Illustration of how heating rates, rather than soaking time, affect annealed grain size of killed steel. This figure has been adapted from the original.[21]

temper rolling is needed to eliminate yield elongation resulting in a further loss of ductility. Thus, to make continuously annealed material successful for forming and drawing operations, a way to achieve a grain size equivalent to that obtained in batch methods must be found. Two ways in which this objective might be reached are discussed below.

(a) One of the characteristics of recrystallization is that the coarser the grain size before cold working, the coarser will be the grain size after annealing. Thus, in order to produce large-grained material by continuous annealing, the starting material for cold rolling operations should be coarse-grained hot rolled stock obtainable by finishing "cold" and coiling "hot" (e.g. finishing at 816°C and coiling at 704°C).* Experiments have shown[21] that by following this practice continuously annealed sheet can be produced having a grain size and softness close to that of batch annealed sheet. However, the carbide distribution is poor in such material: the coarse carbides formed as a result of hot coiling are strung out during cold rolling and their distribution remains unaffected by subsequent annealing. These carbide stringers detract from the transverse ductility of the sheet, and hence from its formability.

(b) Another characteristic of the recrystallization process is that the recrystallized grain size is strongly dependent on prior cold reduction, being coarser with decreasing prior reduction, Figure 2.8. Hence it is possible that an appropriate cold reduction (less than that used in current practices, i.e. 60% approximately) prior to a final annealing would give a desirable grain

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* Rather than the usually quoted temperatures of 900°C and 550°C, approximately.

size; furthermore, control of grain size by prior reduction would not result in the undesirable carbide structures associated with method (a) above.

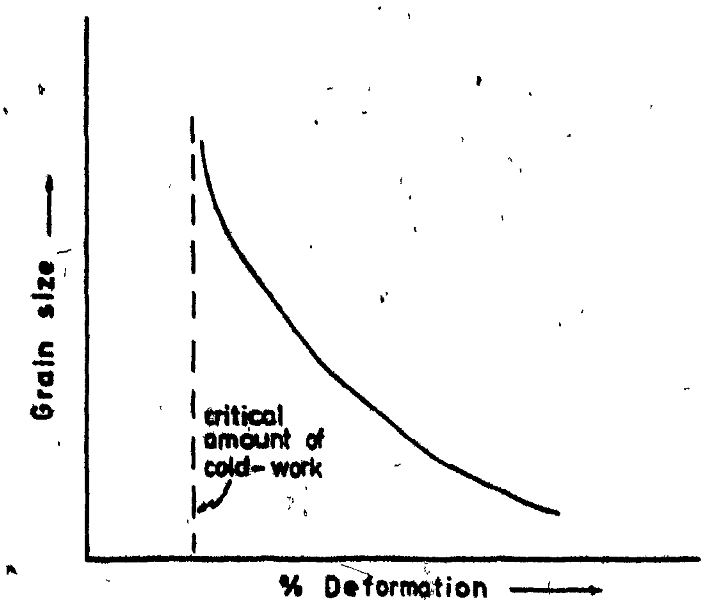


FIGURE 2.8 Effect of prior reduction on the recrystallized grain size.(25)

2.2.2.2 Aging

Aging, which is a time-dependent change in the properties of a material[26], takes place when solute elements are retained in solution after quenching or fast cooling. Due to the fast cooling rates usually employed in continuous annealing, steels annealed by this method are subject to two common aging phenomena[27]: quench aging and strain aging.

A. Quench Aging

Quench aging in steels is the term applied to the precipitation of carbon and/or nitrogen as carbides, nitrides, or carbo-nitrides from supersaturated ferrite. Due to the predominance of carbide precipitation in carbon steels over that of nitrides, carbon is the principal agent in quench aging. As shown in Figure 2.9, the solubility of carbon, which occupies interstitial positions in the ferrite lattice, decreases sharply with decreasing temperature, to vanishingly small values at room temperature. If carbon is retained in solid solution by rapid cooling, as in continuous annealing, carbon can precipitate subsequently at slightly above room temperature as finely dispersed particles of iron carbide. The consequences of such precipitation are manifested in increased hardness, yield and tensile strength and decreased ductility.

In batch annealing on the other hand, cooling from the annealing temperature is slow enough to allow for the precipitation of virtually all excess carbon thus ensuring that quench-aging effects will be kept at a low level at room temperature. Slow cooling is most important[28] in the range from the recrystallization temperature down to about 300°C

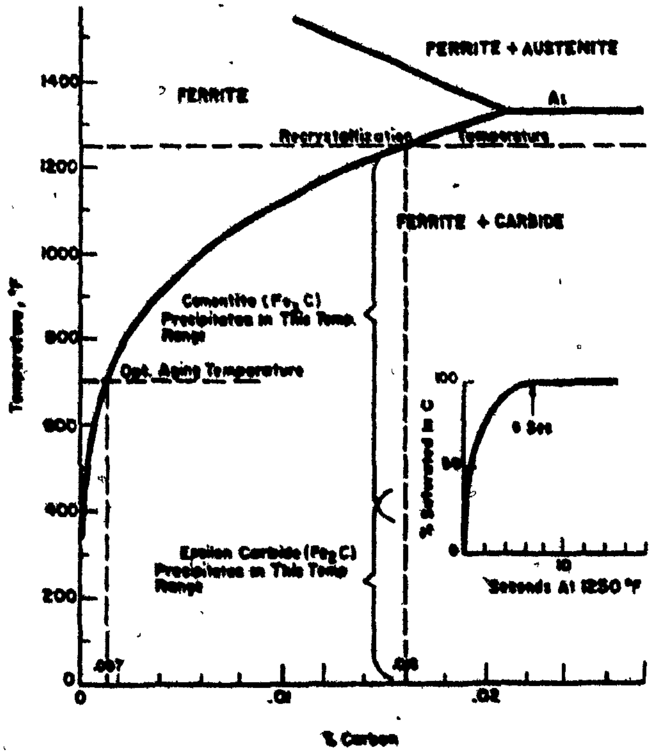


FIGURE 2.9 Solubility of carbon in ferrite.(21)

(572°F) and the optimum time in this critical temperature range is 25 seconds, i.e. rapid cooling (quenching) can be used below 300°C as in cycles B and C in Figure 2.6.

Another method suggested(16) to alleviate the quench aging problem involves quenching the steel from the annealing temperature to 300°C at an unrestricted rate, coiling and then holding the steel, in coil form, for at least one hour at 300°C. The holding period at this temperature would allow carbon to precipitate as large stable carbides. But coiling has to be done in an inert atmosphere which would have to be maintained until the coil reaches 200°C (392°F) or less before the coil could be exposed to the air.

B. Strain Aging

Strain aging is aging induced by cold working and results from the excessive amount of carbon that remains in solution in the ferrite of continuously annealed steels, due to too rapid cooling and retarded carbide nucleation associated with a low carbon content.[27] Most of the manifestations of strain aging are similar to those of quench aging, but strain aging has another characteristic. Following annealing, low-carbon sheet steel is lightly rolled ("temper rolled") to eliminate the abrupt yield elongation characteristic of this material. If aging takes place after temper rolling, the yield elongation returns and the sheet is then susceptible to discontinuous yielding, "fluting" and "stretcher strains" on subsequent deformation. Hence the solution to the problem of strain aging, as with quench aging, lies in the removal of all non-equilibrium carbon from solid solution in the ferrite.

2.3 Developments in Continuous Annealing Practice

Since its adoption as an industrial process, continuous annealing has been modified many times. Three important modifications are discussed in this section, two of which are relevant to the production of low-carbon deep drawing steels.

2.3.1 BISRA Process

Methods for reducing the size and capital cost of conventional continuous annealing units have been studied and developed by the British Iron and Steel Research Association. Gibbon[29] has analysed the first pilot BISRA compact annealing process in which electrical resistance heating of the steel was combined with preheating and subsequent quenching in a liquid lead-bismuth alloy. The first compact annealing line was designed to process 127 mm wide steel strip and consisted of two mild steel tanks filled with lead-bismuth eutectic, the strip being preheated on passing through the first tank, then electrically heated to the annealing temperature before being quenched in the second tank (Figure 2.10). This pilot plant was later redesigned so that the preheat and quench tanks were in the form of ducts (Figure 2.11), thus reducing the amount of lead-bismuth eutectic required. Other investigations[30,31] indicated that bismuth could replace lead-bismuth as the heat-transfer medium, thus eliminating a possible health hazard, and also that the annealing cycle could be appreciably shortened without affecting product quality. The use of liquid sodium as a heat-transfer medium has also been studied[32]; the advantages are claimed to be more rapid heating and cooling rates, the possibility of almost complete heat recovery, and reduction in space requirements over conventional annealing lines.

In essence, the BISRA annealing process involves heating the cold-reduced strip as rapidly as possible to the annealing temperature (700°C), quenching immediately to an intermediate temperature (150-260°C) and holding at this temperature to overage the material. This process produces material comparable in properties with conventional continuously annealed material of temper universal specification, i.e. material suitable for tinplate but not for forming operations.

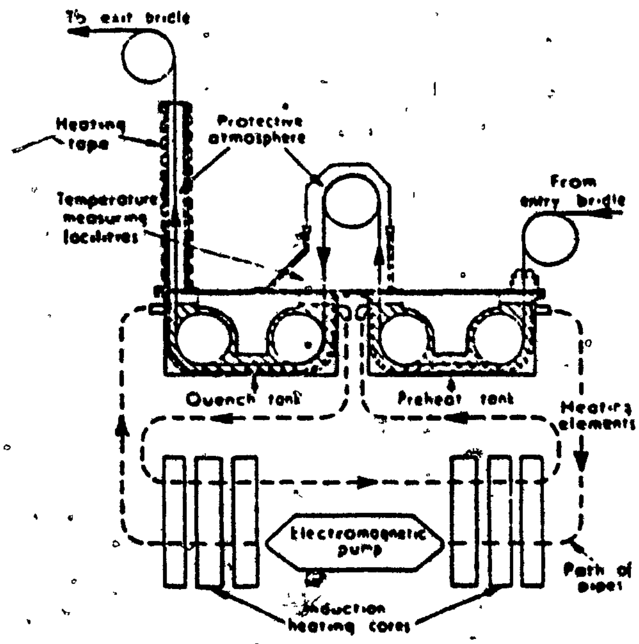


FIGURE 2.10 Schematic layout of first BISRA compact annealing pilot plant.(29)

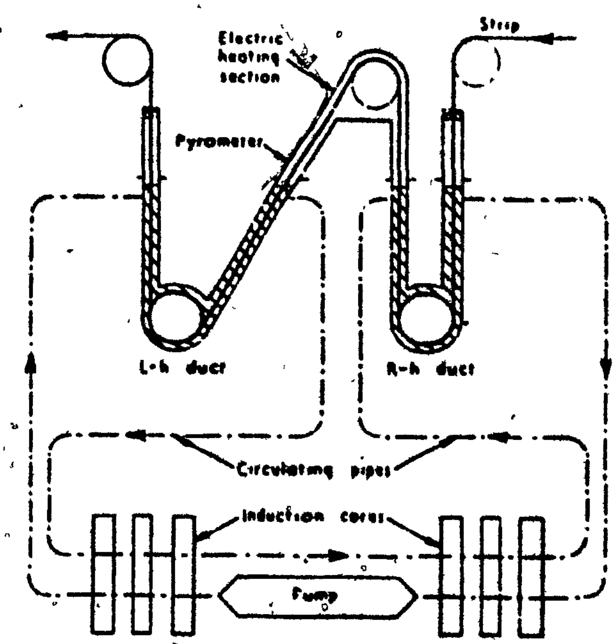


FIGURE 2.11 Schematic layout of second BISRA compact annealing pilot plant.(29)

2.3.2 CAPL Process

CAPL technology (Continuous Annealing and Processing Line) was developed by Nippon Steel Corporation and has been in operation since 1972.[33-37] It was developed in an attempt to produce continuously annealed low-carbon cold-rolled steel sheet with forming characteristics equal to those obtainable by batch annealing. CAPL is a continuous annealing line which incorporates electrolytic cleaning, continuous annealing and overaging, temper rolling, finishing and inspection. Line specifications are given in Table III.

TABLE III

Principal Specifications of Continuous Annealing and Processing Line (CAPL).[36]

Production capacity		34,000 tons/month
Line speed		200 m/min
Strip size:	thickness	0.4-1.2 mm
	width	750-1240 mm
Maximum coil weight		45 tons
Total line length		291 metres

A typical CAPL Line arrangement is illustrated in Figure 2.12 and consists of four sections: electrolytic cleaning, furnace, temper rolling and inspection facilities. After cleaning, the strip enters the furnace section through an entry looping tower with a strip storage capacity of 300 m (985 ft) and after annealing is temper rolled and then levelled prior to inspection.

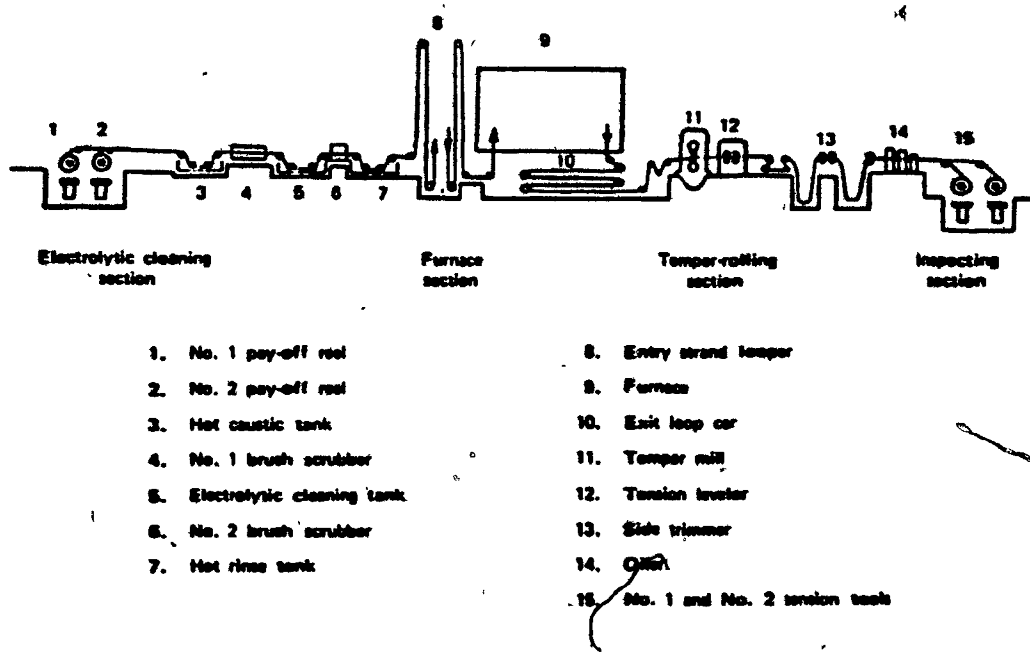
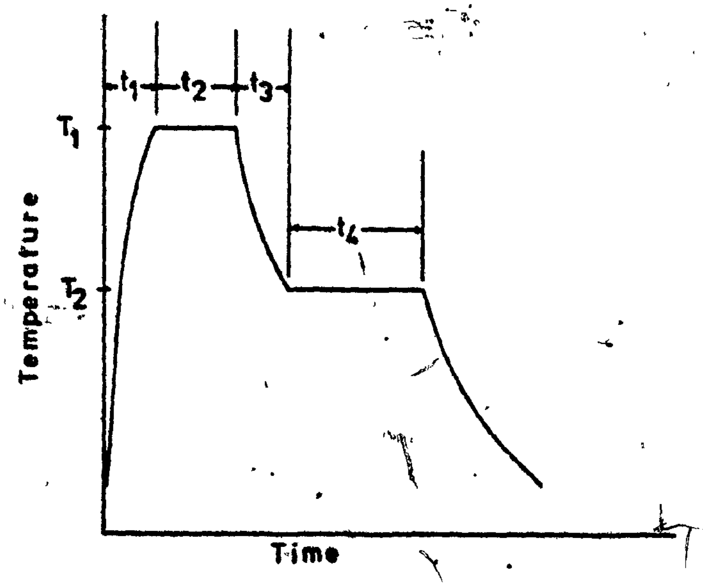


FIGURE 2.12 Schematic line arrangement of CAPL technology.[33]

The continuous annealing furnace consists of the following chambers connected in series: a heating chamber with gas heating from a radiant tube system, an electrically heated soaking chamber, a primary cooling chamber with a jet cooling system in which water-cooled air effects forced cooling, an electrically heated overaging chamber and finally, a secondary cooling chamber again of the jet-cooling type where the air flow is water cooled initially and then refrigeration cooled to provide a final cooling of the strip before exit. If a heating cycle were drawn for the strip as it passed through this furnace system, it would resemble that shown in Figure 2.13. On introduction to the heating



T_1 : Heating temperature (700°C - 900°C)
T_2 : Overaging temperature (300°C - 450°C)

t_1 : Heating time (2 min)
t_2 : Soaking time (2 min)
t_3 : Primary cooling time (~ 15 sec)
t_4 : Overaging time (5 min)

FIGURE 2.13 Heat cycle of CAPL process.

chamber strip is heated rapidly to temperatures between 700°C and 900°C, this heating being effected within two minutes. The steel then passes through the soaking chamber in which it is maintained at the annealing temperature for up to two minutes before being led to the primary cooling chamber where the steel is rapidly cooled to the overaging temperature at cooling rates of approximately 5-30°C/sec. The overaging time is 5-8 minutes at 300-450°C. The steel thus overaged is cooled below 50°C within two minutes in the secondary cooling chamber.

One important restriction on material destined for CAPL processing is that it must have a low manganese content since excess manganese was found to be detrimental to the production of drawing quality sheet by continuous annealing.[34] Thus, manganese must only be present in stoichiometric amounts for combination with sulphur and oxygen. It was established that the following relationship must be adhered to:

$$0 \leq \{Mn\ \%\} - \frac{\text{Atomic weight of Mn}}{\text{Atomic weight of O}} \times \{O\ \%\} - \frac{\text{Atomic weight of Mn}}{\text{Atomic weight of S}} \times \{S\ \%\} \leq 0.15$$

or: $0 \leq K \leq 0.15$

It was found that this "K-value" could be calculated using the following empirical equations:

For rimmed and capped steels:

$$K = \{Mn\ \%\} - \frac{55}{16}\{O\ \%\} - \frac{55}{32}\{S\ \%\}$$

For Al-killed steels:

$$K = \{Mn\ \%\} - \frac{55}{32}\{S\ \%\}$$

A further restriction placed on steel for CAPL processing is that following hot rolling it must be coiled at temperatures in the range 675-800°C. As explained in Section 2.2, hot coiling from the hot mill gives a large hot-rolled grain size and, consequently, a large annealed grain size after cold-rolling.

2.3.3 NKK Process

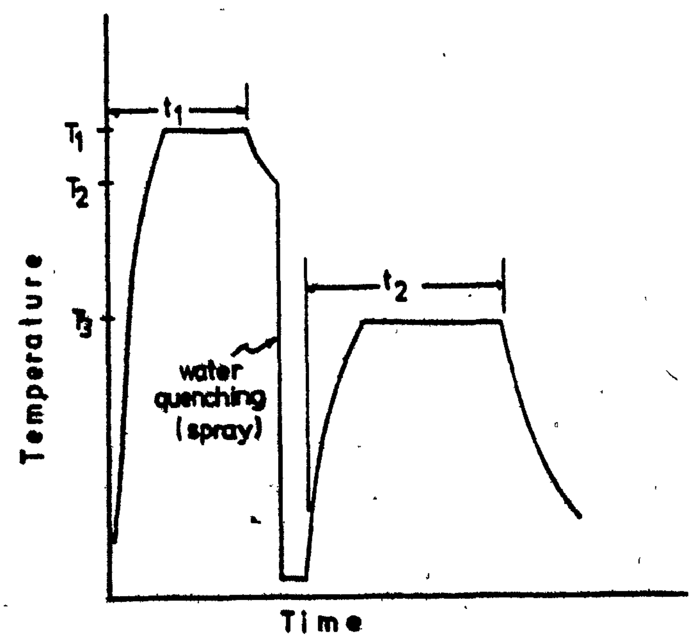
The continuous annealing line modifications developed by Nippon KoKan K.K. of Japan[38-43] also aim to produce drawing quality sheet by close control of both steel grade and its processing. One advantage of the NKK line is that it is so arranged that by a simple switching of strip feed patterns, either tinplate can be produced by a conventional annealing cycle or drawing quality sheet by the NKK modified cycle. The principal line specifications are given in Table IV.

Strip feed into an NKK modified furnace passes first through a normal entry zone incorporating cleaning facilities. The annealing furnace itself consists of three regions: heating, accelerated aging and cooling. The distinctive feature is that the overaging is preceded by water quenching; this is done in a water spray cooling system located beneath the furnace. The heat cycle pattern of this process is shown in Figure 2.14.

TABLE IV

Outline of the Continuous Annealing Equipment at NKK's Fukuyama Works.(43)

	No.1 CAL	No.2 CAL
Nominal capacity	32,000 tons/month	43,000 tons/month
Line speed	572 m/min	250 m/min
Strip size		
thickness	0.15-0.6 mm	0.4-1.2 mm
width	457-1067 mm	610-1300 mm
Maximum coil weight	32 tons	32 tons
Total line length	131.56 m	156.5 m



T_1 : Heating temperature (680°C - 760°C)
T_2 : Quenching temperature (450°C - 700°C)
T_3 : Overaging temperature (300°C - 500°C)

t_1 : Heating and soaking time (10 - 300 sec)
t_2 : Overaging time (0 - 150 sec)

FIGURE 2.14 Pattern of the heat cycle. NKK's continuous annealing.(41)

As with the CAPL furnace, the NKK process has been designed to give optimum results with sheet steel of closely controlled chemical composition. Thus a special degassing method was developed[42] which enabled steels with less than 0.01% C to be produced. These ultra-low carbon steels are then hot rolled with the further requirement that coiling after hot rolling must be done at temperatures over 650°C. It was also found that best results were obtained with steels that had been aluminum killed. With these prerequisites, elements in solid solution, such as C, N, Mn, Al, are minimized so that grain growth after hot coiling is enhanced and the aging susceptibility of the steel is minimized. Thus, the NKK could be described as a method for manufacturing cold drawing quality steel which combines a specific degassing method, hot rolling conditions and continuous annealing.

2.3.4 Comparison of CAPL and NKK Modifications with Conventionally Processed Steels

Proponents of the CAPL and NKK processes claim that low-carbon sheet steel produced by their methods has properties comparable to that produced by batch annealing: these claims are supported by the data presented in Table V.

TABLE V

Mechanical Properties Comparison of CAPL and NKK Processes

Mechanical Properties	Batch[1] Annealing	Continuous[2] Annealing	Continuous Annealing	
			CAPL[3]	NKK[4]
Yield strength (Psi)	$28.2x10^3$	$58.2x10^3$*	$27.9x10^3$	$31.3x10^3$
Tensile strength (Psi)	$46.9x10^3$	$60.5x10^3$*	$46.6x10^3$	$46.8x10^3$
Elongation in 2 in. (%)	44	20	44.2	45

1 Nippon Steel Corp.(33)

2 Steel Company of Wales (Tinplate Div.)(13)

3 Nippon Steel Corp.(33)

4 Nippon Kokan Kabushiki Kaisha(43)

* Originally expressed in tons/in^2 (assuming: 1 ton = 2240 lb)

2.4 Summary and Conclusions

Batch operations (i.e. box annealing and open-coil annealing) involve many consecutive processes such as coiling after cold-rolling, uncoiling for electrolytic cleaning, coiling for heat treatment, uncoiling for temper rolling and final coiling for shipment. Each separate process requires heavy machinery for coil handling and floor space for coil storage. The necessity for these separate operations and the long times involved in the batch annealing process itself, often up to a week, makes for long processing times from steelmaking to shipment so that batch annealing is a low-productivity operation.

In continuous processing, although essentially the same operations are involved, i.e. electrolytic cleaning, temper rolling, etc., all coiling and recoiling have been eliminated and with them problems of handling, storage, etc.. As a result, continuous operations involve short processing times. Continuous annealing cycles are of the order of two minutes so that continuous annealing is a high productivity operation. Also, other problems such as ridges, buckles, stickers, sand spots, localized overheating, poor flatness, etc. to which batch annealed materials are prone, are eliminated or reduced in continuous annealing operations.

As a result of the large masses of steel involved in batch annealing, the heating rates are very slow allowing the material to recrystallize to a large grain size and the slow cooling rates employed give long times at high temperatures favoring carbide precipitation. These characteristics of batch annealing allow the production of steel with excellent formability such as that required by the automobile industry. In contrast, the heating rates in continuous annealing are extremely high causing the steel to recrystallize to a fine grain size, which results in a steel that is stiffer and harder than its batch annealed equivalent. Due to the rapid cooling rates in continuous annealing, sufficient carbon always remains in solid solution to give a product with a high aging susceptibility. Thus, the use of continuous annealing has been limited to the production of tinplate stock, and sheet for galvanizing and aluminizing.

Table VI summarizes the effect of the process characteristics on the material properties and hence on their suitability for forming operations. From this Table it may be seen that there are two main obstacles to the successful application of continuous annealing to the production of drawing quality steel:

1) The problem of obtaining a coarse enough grain size; and
2) the problem of precipitating enough carbon from solution in the ferrite to eliminate or reduce aging effects.

TABLE VI

Characteristics of Conventional Batch and Continuous Annealing

Batch Annealing	Continuous Annealing
Slow heating: grain size - large yield strength - low tensile strength - low elongation - large Slow cooling: aging susceptibility - low	Fast heating: grain size - fine yield strength - high tensile strength - high elongation - small Fast cooling: aging susceptibility - high
FORMABILITY: GOOD	FORMABILITY: POOR

Continuing efforts to improve continuous annealing lines have resulted in the development of several patented processes. The BISRA method attempts only to improve continuous annealing for the production of tinplate stock but the CAPL and NKK processes represent attempts to produce drawing quality steel by continuous annealing practices. However, both the latter methods rely on close control of chemical composition and hot-rolling practice. Hot coiling after hot-rolling has the undesirable effect of producing coarse carbides that cannot be evenly dispersed by subsequent cold rolling and the close controls necessary of chemical composition, particularly in NKK process, are definite drawbacks.

All the suggested modifications, with the exception of the reduction of carbon to ultra low level in NKK, are aimed at achieving a large grain size despite the fast heating rates used in continuous annealing. However, there is yet another method for achieving a large grain size on annealing and one which is independent of both chemical composition and hot coiling temperature, viz. the degree of strain prior to annealing. Although this strong influence of prior deformation on finished grain size is well known, attempts have not been made seriously to investigate the integration and regulation of prior strain into the continuous annealing cycle. One objection that can be made is that it would be costly to anneal a sheet, roll it to a controlled degree and then reanneal but this objection must be weighed against the cost of close chemical control of manganese, oxygen and sulphur in CAPL process, or the cost of installing complex degassing equipment, as in the NKK process. Hence, the following experimental work represents an initial step in the formulation of a continuous annealing cycle in which prior reduction will be the critical factor determining final grain size and thus final mechanical properties.

CHAPTER 3

3. EXPERIMENTAL PROCEDURE

3.1 Introduction

As a result of the survey of literature relating to continuous annealing outlined in Section 2, it was concluded that there was a good possibility of producing steel sheet with good formability by introducing an intermediate deformation step prior to final annealing. The experimental procedure used to investigate this possibility is outlined below.

The starting material for the investigation was mild steel sheet cold rolled 60%. As a first step, this material was annealed so that two sets of fully recrystallized samples, one with a grain size approximately twice that of the other, were produced. Specimens of these two kinds of material were then deformed in tension at incremental strains up to the onset of plastic instability. Tensile deformation was employed to produce this intermediate strain since it was fouhd to be more controllable (at least for small strains) than rolling to a given specimen thickness. The deformed specimens were then given a final annealing treatment in order to recrystallize them completely. Following the final heat treatment, the samples were examined metallographically and their mechanical properties determined using standard procedures.

An appraisal of these results showed that higher levels of pre-strain than had been initially planned would be necessary to obtain a wide range of recrystallized grain sizes. Since the higher strains were greater than those which it would be possible to produce using simple tension, deformation by rolling had to be resorted to. Although, as pointed out earlier, rolling reduction was less controllable than straining in tension, rolling only became necessary at the higher strain levels where precision is less important (viz. at reductions greater than 22%, approximately). Rolled specimens were annealed, tested and examined metallographically in the same way as specimens deformed in tension.

3.2 Material

The starting material for the experiments was rimmed steel sheet* supplied in coil form from the normal production lines of Dominion Foundries and Steel Co., (DOFASCO), Hamilton, Ontario. The steel was in the cold-rolled condition, having been cold-reduced 60% to a thickness of 0.035 in. after cold rolling and was typical of material intended for the manufacture of Commercial Quality Drawing Steel. This steel is covered by SAE 1008 specification.(44)

Chemical analysis of the steel was performed by Dofasco. The results are given in Table VII, which also lists the chemical analysis for SAE 1008 steel.

* Hot rolled finishing temperature, 1600°F (871°C).
Coiling temperature, 1200°F (650°C).

TABLE VII

Chemical Composition of Starting Material

Dofasco		SAE 1008
wt. %	Element	wt. %
0.07	C	0.1 max
0.25	Mn	0.3-0.5
0.005	P	0.040 max
0.011	S	0.050 max
0.002	Si	-
0.02	Cu	-
0.01	Ni	-
0.03	Cr	-
0.11	Sn	-
Balance	Fe	Balance

3.3 Material Preparation

3.3.1 Preparation of Specimens for Recrystallization I Annealing

Rectangular specimens (2 in. x 1 in.) were cut from the coil, the long dimension of the specimens being parallel to the rolling direction. These samples were used for hardness tests and metallographic studies of annealing behaviour.

Specimens for tensile testing were also cut from the sheet in such a way that the rolling direction was parallel to the length of the sample*, the finished tensile specimens being shaped on a "Tensilkut"

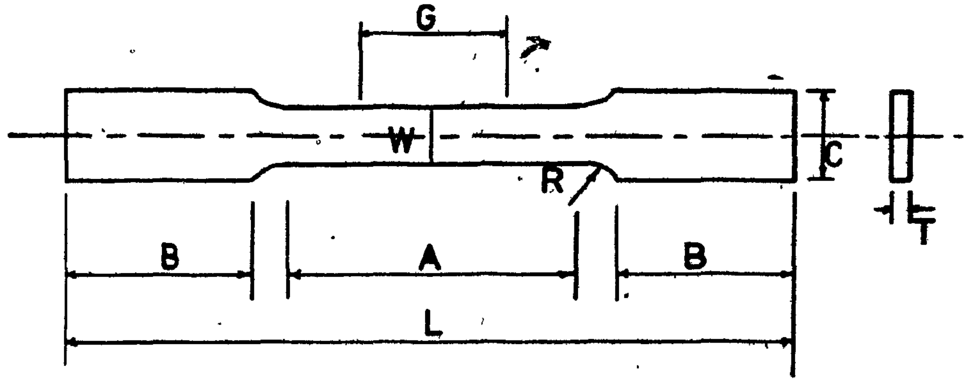
* The ductility of sheet steel as measured by total elongation, or uniform elongation, is generally slightly greater in the direction of rolling than in the transverse direction.(17)

shaping mill in accordance with ASTM specification E8-61T.[45] Figure 3.1 illustrates the standard tension test specimen and lists the dimensions specified for the 2 in. gauge length test pieces which were used in this investigation. In calculating specimen cross sectional area at least three width and thickness measurements were taken from the reduced section of the sample (Section A in Figure 3.1) and each value was the average of two readings.

3.4 Material Processing

3.4.1 Recrystallization I

The aim of the first recrystallization treatment (Recrystallization I) was to produce fully recrystallized material of two particular standard grain sizes. Initially, it was necessary to establish the range of temperatures over which the cold-rolled steel would be completely recrystallized so that a suitable recrystallization temperature could be chosen. Annealing experiments were therefore carried out using rectangular specimens in a lead bath. A steel jig was constructed which allowed the simultaneous annealing of up to five samples; samples were hung vertically from the jig at a spacing of 0.8 cm so that the lead could circulate freely thus ensuring a maximum heat transfer rate. When the jig, loaded with specimens, was lowered into the lead bath, there was an initial temperature drop of 3-5°C due to the introduced mass of cold metal but the set temperature was quickly regained. Specimens were held for periods of time ranging from 10 to 60 minutes at temperatures in the range 450-700°C. At the end of the desired time interval the jig was removed from the lead bath and cooled in air. After this annealing



G: Gage length	2.000 ± 0.005 in
W: Width	0.500 ± 0.010 in
T: Thickness	thickness of material
R: Radius of fillet	0.5 in., min
L: Overall length	8.0 in., min
A: Length of reduced section	2.25 in., min
B: Length of grip section	2.0 in., min
C: Width of grip section	0.75 in., min

FIGURE 3.1 Standard rectangular tension test specimen with 2 in. gage length.(45)

treatment, specimens were tested for hardness (see Section 3.5.1) and the values obtained from three specimens after each recrystallization annealing treatment averaged for an overall hardness value for each annealing temperature. The recrystallization temperatures were then established by plotting the hardness measurements against the annealing temperatures.

Having determined the annealing temperature range over which a recrystallized material could be produced it was necessary to establish the minimum time to produce a recrystallized material. A series of annealing treatments were carried out in the range 600-700^{o}C and for annealing times of 2 to 8 minutes. As before, annealing was followed using hardness measurements.

It was observed following these recrystallization-annealing treatments that some lead always remained on the specimens hindering hardness measurements. Thus, in order to obtain clean specimens it was decided that they would be enclosed in steel envelopes for all subsequent heat treatments. An important advantage of using steel envelopes was that they not only provided a protective sheath for the samples but also, when properly sealed, neutralized the entrapped atmosphere and hence minimized specimen-surface oxidation. To ascertain the optimum time for recrystallization at 700^{o}C using these envelopes, a series of isothermal heat treatments was carried out. Steel envelopes were loaded with rectangular samples, sealed by folding the open end at least three times to ensure air-tightness and then placed into the Lindberg Hevy-Duty electric resistance furnace. The envelopes and enclosed samples reached furnace temperature after two minutes from which point annealing times of 60 seconds to 24 hours were measured. On removal from the furnace the steel envelopes were allowed to air cool, and the samples were tested for hardness (Section 3.5.1).

3.4.2 Pre-Strain - Recrystallization II

The aim of the recrystallization I treatment was the production of specimens with a particular recrystallized grain size. Samples were then given a pre-strain prior to the final anneal, Recrystallization II. In order to investigate the effect of different amounts of pre-strain, tensile specimens were strained by amounts ranging from 1 to 24%, using an Instron TT-D Universal Testing Machine at a crosshead speed of 0.05 in./minute. The strain was measured using a 2 in. strain gauge extensometer and the values were read directly from the autographic chart recording. Once the desired value of pre-strain was attained the Instron was stopped and the specimens removed.

The pre-strained specimens were sealed in steel envelopes and placed in the Lindberg Hevy-Duty electric resistance furnace for the final annealing treatment. This process, recrystallization II, was carried out at temperatures in the range 610-715^{o}C for a constant annealing time (10 min.), after which time specimens were air cooled and their mechanical properties determined.

3.5 Material Testing

3.5.1 Hardness Testing

Hardness was measured with a Rockwell Superficial Hardness Testing Machine on the R30-T scale. An average of ten readings per specimen was recorded as the hardness of the specimen.

3.5.2 Tensile Testing

All tensile tests were carried out at room temperature on an Instron TT-D Universal Testing Machine, using wedge-action grips and a crosshead speed of 0.5 in./min.. The loads were measured with a standard Instron GR 20,000 lb. load cell, while the extensions were measured with a strain gauge extensometer designed for 1 or 2 in. gauge length. The Instron crosshead movement, which was equipped with a control dial that could be adjusted and read to ± 0.002 inches, was used to calibrate the extensometer. This gave an extensometer error of ± 0.2% (in the case of 1 in. gauge specimen) and ± 0.1% (in the case of 2 in. gauge specimen) to all calculations of percentage elongation, i.e. elongations quoted as 1.0% or 10.0% should be read as (1.0 ± 0.2 or 1.0 ± 0.1)% or (10.0 ± 0.2 or 10.0 ± 0.1)% respectively, in order to include extensometer error. An autographic chart recording of load versus extension was obtained from each test, using an X-Y chart drive system at the greatest sensitivity consistent with chart size limitation. The extensometer recorded the entire test, up to and including fracture. The total and uniform elongations were read directly from the chart recording. The yield load (taken at 0.2% offset), and the maximum load, were each divided by the original cross-sectional area of the specimen to obtain yield strength and tensile strength. The load cell had been calibrated with a standard 500 lb. load. Since the loads involved in testing were generally of the order of 1,000 lb., the load values read off the chart had a maximum error of ± 1.0 lb., or ± 60 Psi.

The yield strengths and tensile strengths, total and uniform elongations of two tensile specimens from each pre-strain and annealing temperature combination were averaged to yield the final results for each condition.

3.5.3 Metallography

Longitudinal cross-sections of selected specimens were mounted in Quickmount (a cold setting resin) and then successively ground on 220, 320, 400 and 600 grit silicon carbide papers. This was followed by polishing on Microcloth, initially with 5 micron diamond paste and finally with 1 micron diamond paste. The specimens were etched for microexamination with 2% nitric acid in alcohol and photomicrographed using standard procedures.

3.5.4 Quantitative Metallography

To estimate the grain size of the final annealed material, a quantitative metallographic examination was carried out on metallographic samples prepared from the tensile specimens. The lineal intercept method was employed for this purpose. The test line length was 100 mm and the magnification for each sample was selected so that a single test line length would yield at least 10 intercepts. For each sample, five fields were randomly selected and five test lines were used in each field employing the same line test arrangement in order to obtain an average value for each specimen.

No determination of grain size can be an exact measurement. Thus, no estimation is complete without a determination of the precision with which the determined grain size represents the actual average grain size of the specimen examined. Thus, in accordance with common engineering practice, 95% confidence limits were established for each grain size measurement.

3.6 Pre-Straining by Rolling

Analysis of the preceding results revealed that larger deformations, beyond the plastic instability limit, needed to be investigated. Consequently, one set of recrystallized samples with GS-I as starting grain size was reduced in a rolling mill. These specimens (initially 8.5 in. x 0.5 in.) were passed through a Stanat 4-high mill until thickness reductions in the range 24-32% were achieved. This 4-high mill had 0.75 in. diameter work rolls and 4 in. diameter back-up rolls.

Following cold reduction, the specimens were recrystallized at 705°C for 10 minutes and then allowed to air-cool. Specimens were then tension tested and examined metallographically according to the procedures outlined in Section 3.5.

CHAPTER 4

4. EXPERIMENTAL RESULTS

4.1 Introduction

The experimental results have been ordered in a manner analogous to that used to describe the experimental procedure. Firstly, the results obtained from the Recrystallization I treatments that were used to establish the annealing parameters are presented. These are followed by the results obtained from the pre-strain + Recrystallization II treatments. The tensile test results that show the effect of pre-strain, annealing temperature and starting grain size on the mechanical properties of the material after Recrystallization II are presented in sets of graphs, each set corresponding to one specific mechanical property. The effect of pre-strain and starting grain size on the final recrystallized grain size is then plotted. Then follows a series of photomicrographs that has a two-fold aim: to document the recrystallized microstructures and to relate the final grain size to the observed mechanical properties. Finally, the results of the rolling mill experiments are presented with relevant graphs and photomicrographs.

4.2 Recrystallization I

Hardness results from the rolled rectangular samples annealed for 10 minutes in the lead bath are presented in Figure 4.1 where they are plotted against annealing temperature.

From Figure 4.1 it may be seen that the hardness of the steel in cold rolled condition was about 78 R30-T and this hardness was retained

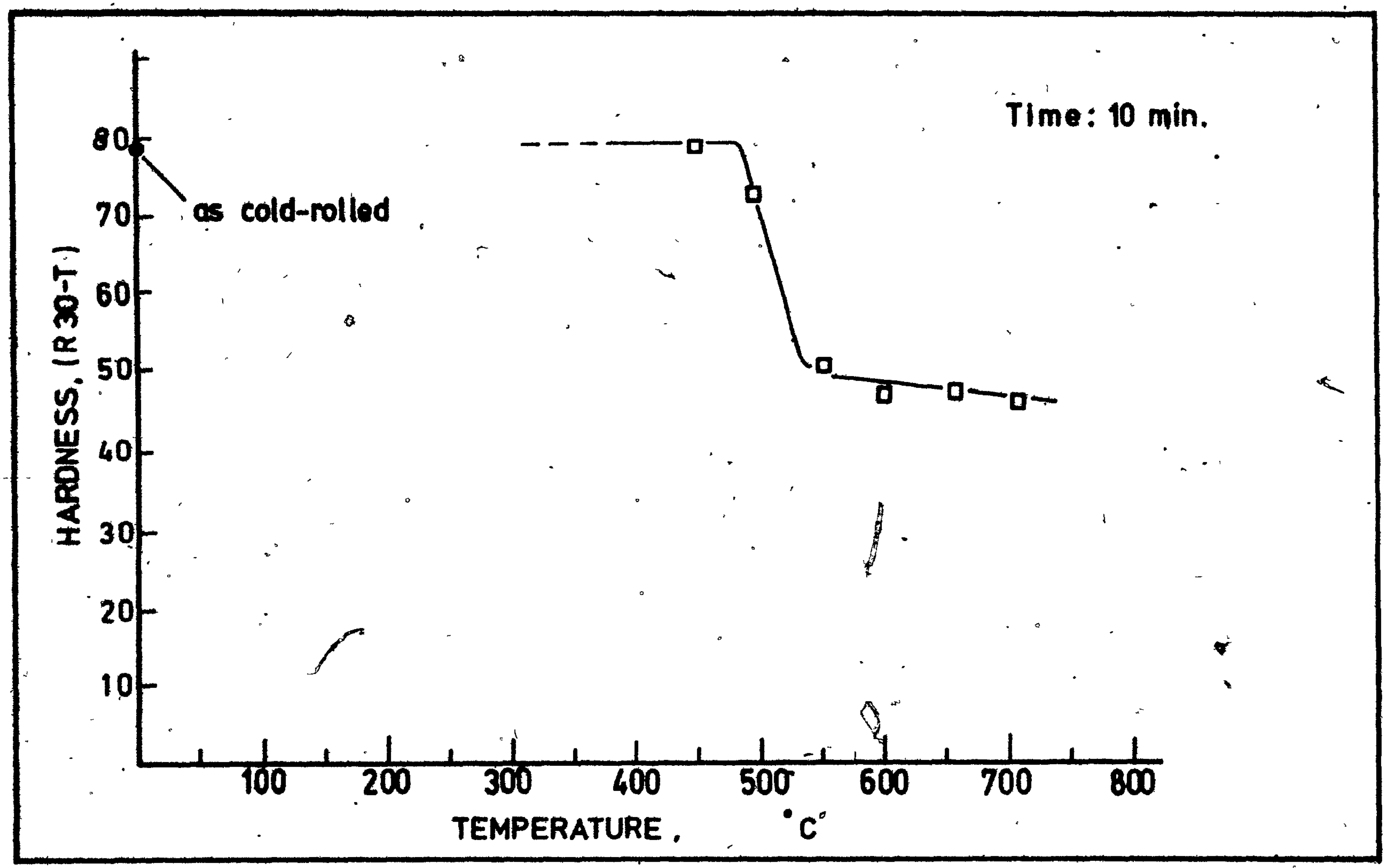


FIGURE 4.1 Effect of annealing temperature on superficial hardness of 60% cold-rolled rimmed steel sheet.

with no appreciable change after annealing at 450°C; on increasing the annealing temperature by 100°C (i.e. up to 550°C) the hardness dropped about 30 hardness points. Further increments in temperature had no dramatic influence on the hardness which continued to decrease smoothly and gradually. Increasing annealing times up to 60 minutes had no significant effect on the hardness results.

The drastic decrease in hardness that took place between 450 and 550°C was directly related to the change in microstructure where the highly distorted cold-worked grains were replaced by new deformation-free grains. The slight decrease of hardness at higher temperatures was due to the growth of these new grains. Therefore, it may be said that recrystallization took place between 450 and 550°C, so that recrystallized material was definitely complete when the cold rolled material was heated above 550°C for ten minutes.

Figure 4.2 shows the hardness results obtained from rolled samples which were annealed at the temperatures indicated for periods of time ranging from 2 to 8 minutes. From this figure it may be seen that for all the temperatures investigated the annealed hardness values were about 45-50 R30-T which corresponds to a recrystallized material (see Figure 4.1).

Therefore, it may be stated that using the lead bath, the annealing temperature range for producing recrystallized material for annealing times as little as two minutes was 600°C to above 700°C (but less than 723°C, at which temperature the ferrite-austenite transformation takes place).

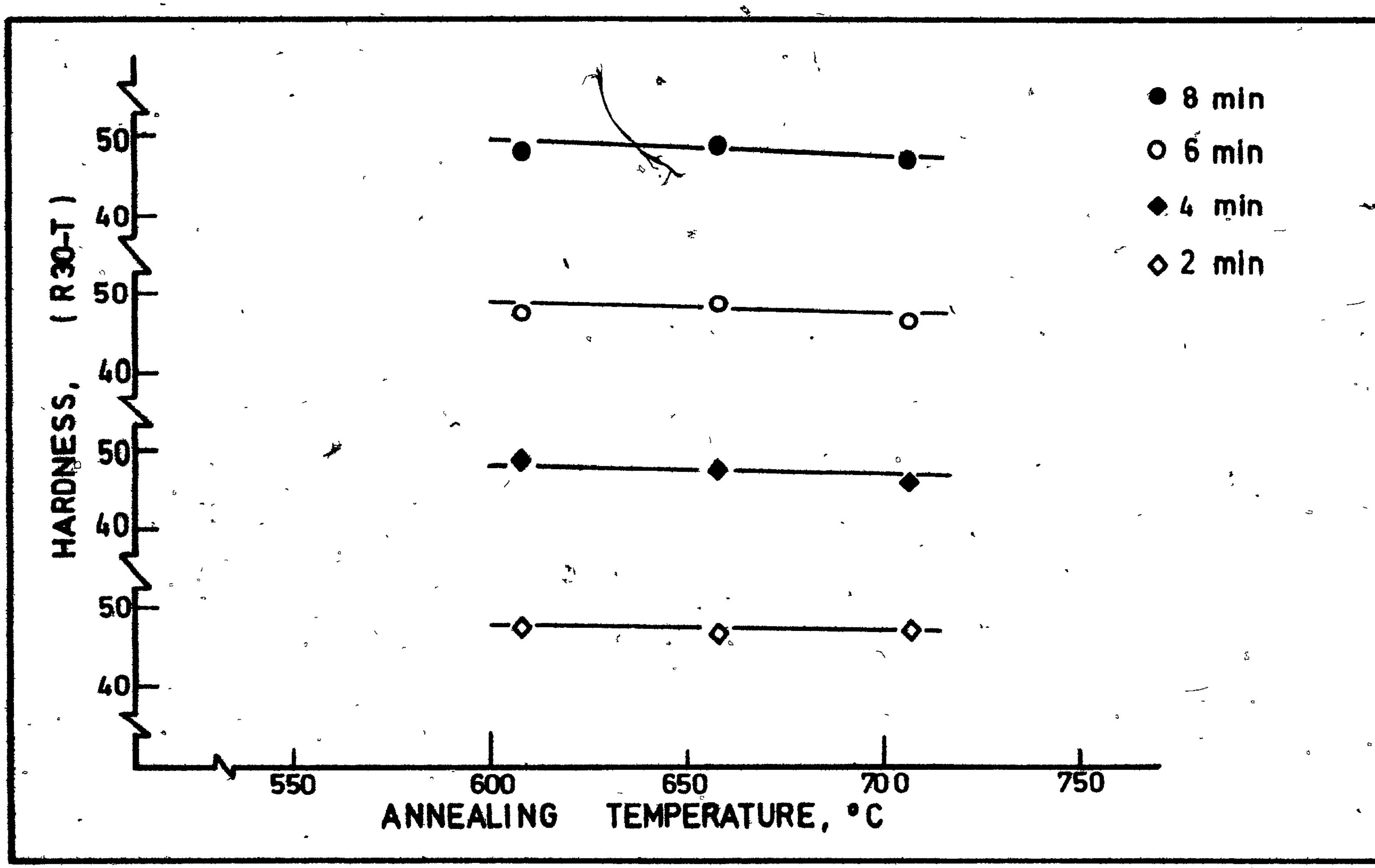


FIGURE 4.2 Comparative graph showing the effect of annealing temperature and time on superficial hardness of cold-rolled material. Hardness of cold-rolled material: 78 R30-T.

As these experimental results were determined with bare specimens treated in a lead bath, the experiments were repeated using the steel envelopes that were to be used in all further experiments. Figure 4.3 shows the hardness results for samples isothermally annealed at 700°C for various times, each plotted point being marked with its equivalent in either minutes or hours to facilitate interpretation. From this figure it may be seen that annealing at 700°C for short periods of time, i.e. less than 2 minutes, had no effect on hardness, when a metal foil container was employed. With increasing annealing times up to 3.5 minutes a drop of about 30 hardness points with respect to the cold rolled hardness took place; longer times further decreased the hardness due to grain growth. Thus, fully recrystallized material was produced using steel envelopes when holding times at 700°C were 4 minutes or more. As expected, the times required to produce a recrystallized material were longer when steel envelopes were employed (see Figure 4.2 which shows that at a temperature of 700°C, recrystallization is complete in 2 minutes).

These preliminary experiments were necessary in order to become familiar with the annealing characteristics of the cold rolled material. As a result of these tests, it was decided to recrystallize cold rolled samples in steel envelopes, using a temperature of 705°C. Two times were chosen: (a) 30 minutes and (b) 24 hours. These two treatments were necessary to provide two different starting grain sizes, a fine one (GS-I) and a coarser one (GS-II), in order to investigate the effect of this variable on subsequent behaviour.

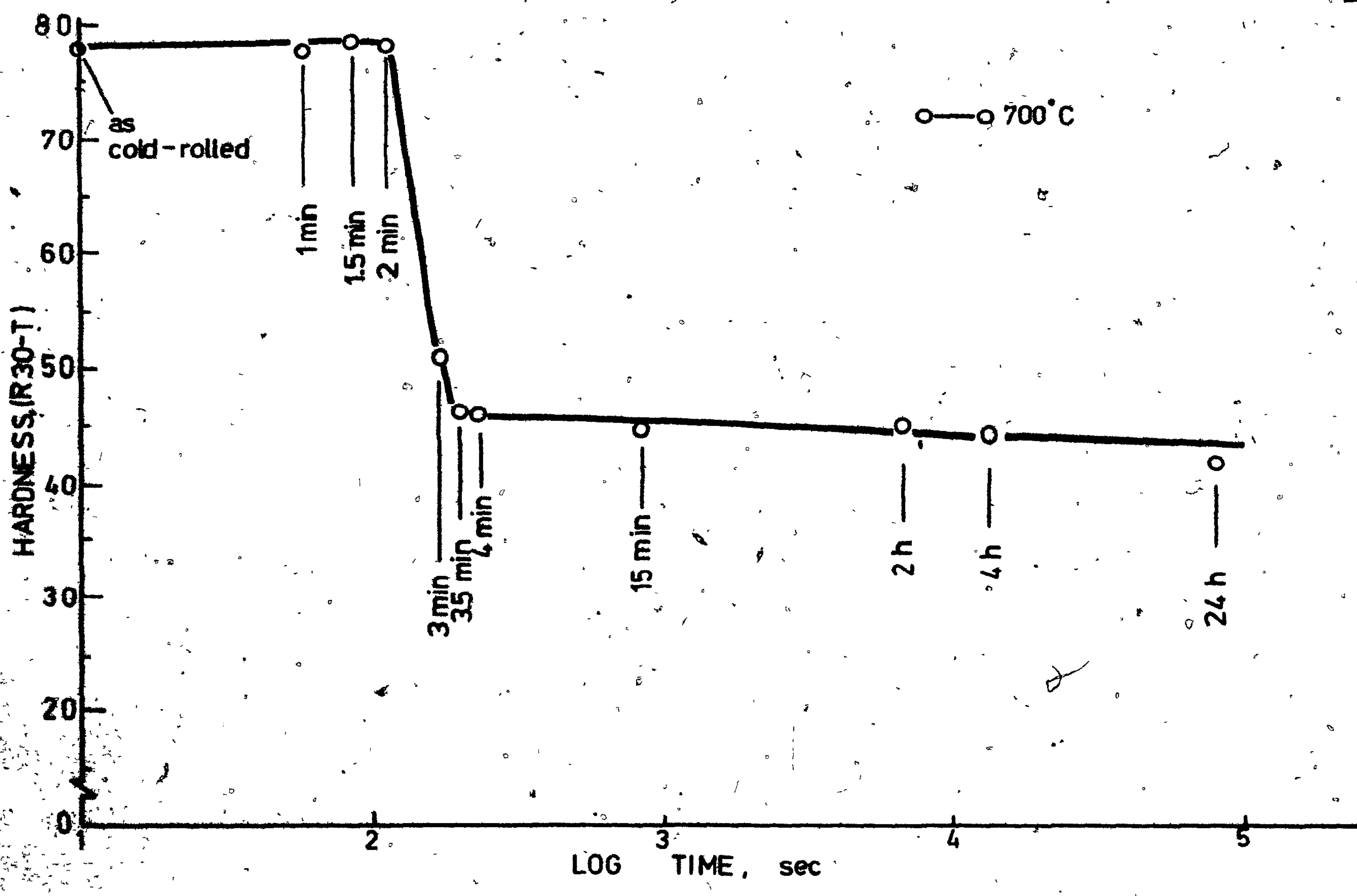


FIGURE 4.3 Effect of isothermal annealing treatment on hardness of cold-rolled steel using metal foil envelopes.

GS-I was measured to be 10.1 ± 0.21 ASTM grain size number (= 0.0096 mm); GS-II was measured as 8.28 ± 0.215 ASTM grain size number (= 0.0185 mm); i.e. grain size ratio was approximately 2:1.

4.3 Pre-Strain - Recrystallization II

The steel samples, in the form of tensile specimens, and having either GS-I or GS-II as a result of the recrystallization I treatment, were then given a pre-strain followed by a final recrystallization anneal. The annealing temperatures used were in the range 600-723°C, the annealing time being kept constant at 10 minutes.

At the completion of final recrystallization, the samples were allowed to air cool in the steel envelope. The cooling rate was estimated to be 35°C/min. and it was established (see Appendix A) that this allowed sufficient time for equilibrium carbon precipitation.

The tensile properties of the specimens were then determined. The results are presented in the following sections.

4.3.1 Yield Strength

From the tensile test results the yield strength of specimens annealed at a particular temperature has been plotted against the degree of pre-strain and is shown in Figures 4.4 and 4.5 for GS-I and GS-II starting grain sizes, respectively. Each plotted point represents the mean of two yield strength determinations.

In both figures the yield stress value plotted for zero pre-strain was the yield strength of the material immediately after recrystallization I. As expected, the material with the larger grain size, GS-II, had a lower stress value.

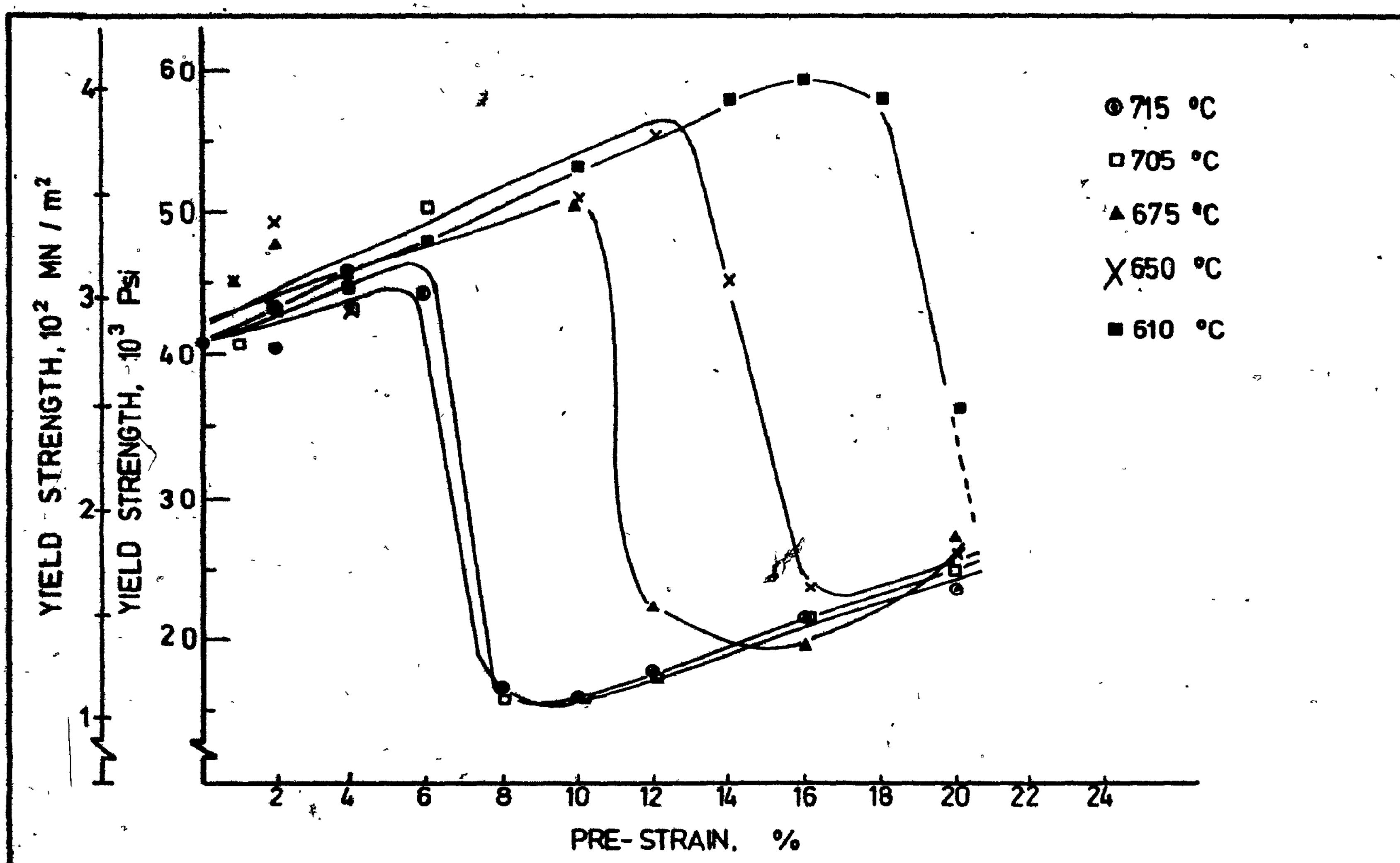


FIGURE 4.4 Effect of pre-strain and annealing temperature on yield strength. Starting grain size, GS-I. (Annealing time, 10 minutes).

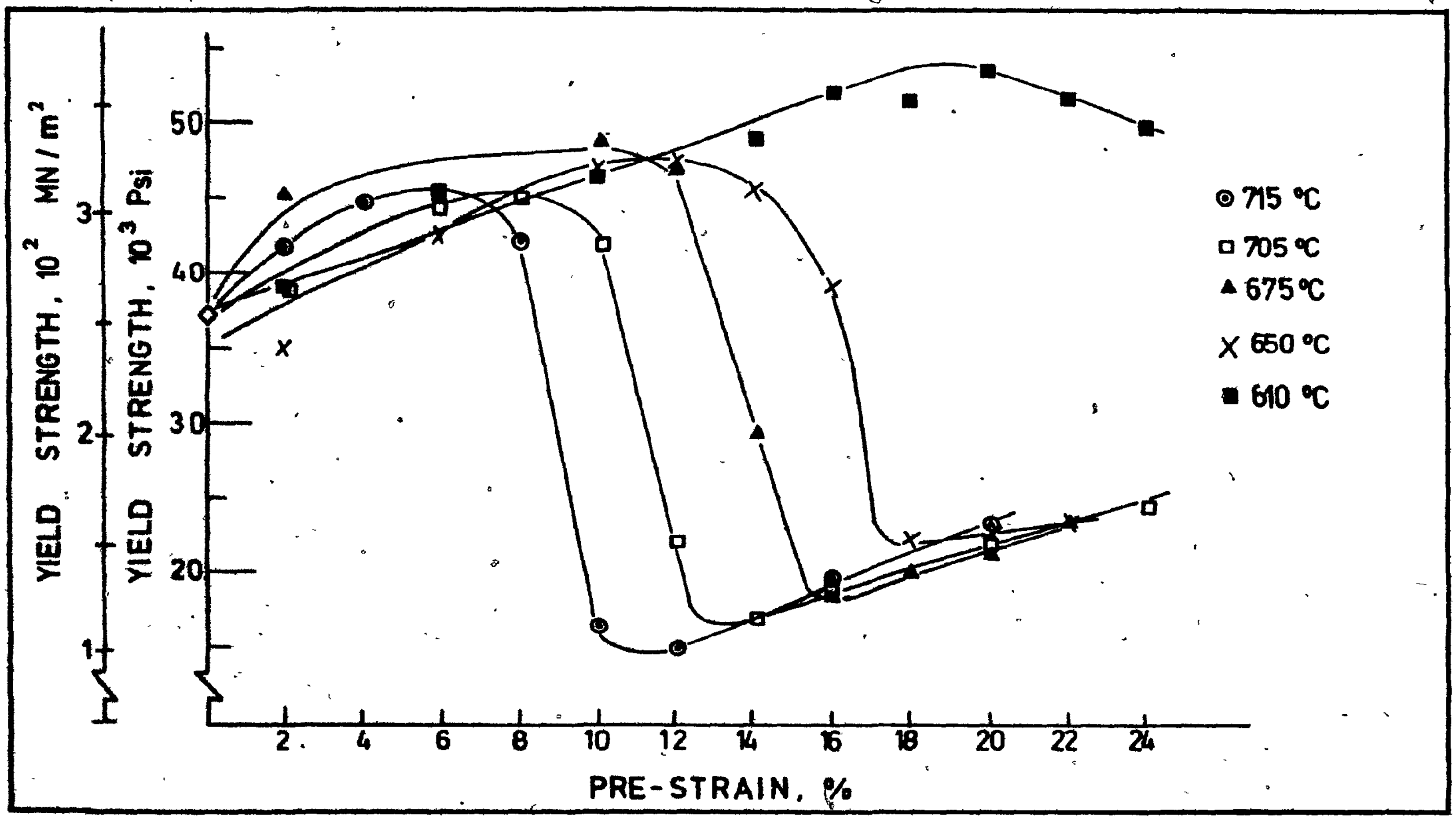


FIGURE 4.5 Effect of pre-strain and annealing temperature on yield strength. Starting grain size, GS-II. (Annealing time, 10 minutes.)

The variation of the yield stress with the amount of pre-strain was similar regardless of the annealing temperature or the initial grain size. As the pre-strain increased, the yield stress initially increased until, over a very narrow pre-strain increment, the yield stress dropped to a low value. Once this had occurred, the yield stress resumed its steady increase with increasing pre-strain.

The major effect of increasing annealing temperature from 610^{o}C to 715^{o}C was to lower the pre-strain value at which the drop in yield stress occurred; additionally, as the annealing temperature increased the maximum value of the yield stress before the drop in yield stress occurred also decreased as did the minimum value after the yield stress drop. The behaviour of the yield stress was similar for both GS-I and GS-II starting grain sizes, except that for the larger grain size starting material, the yield strength maxima were smaller and the pre-strain at which the drop in yield stress occurred was displaced to higher pre-strain values.

Yield strength results are given in Table B-I and B-II (Appendix B) for both GS-I and GS-II starting grain sizes, respectively.

4.3.2 Critical Strain

It is clear from Figures 4.4 and 4.5 that the yield drops occurred at fairly sharply defined levels of pre-strain, depending on the annealing temperature and the initial grain size. In Figure 4.6 these strain levels, or critical strains, have been correlated with annealing temperature and initial grain size. As would be expected,

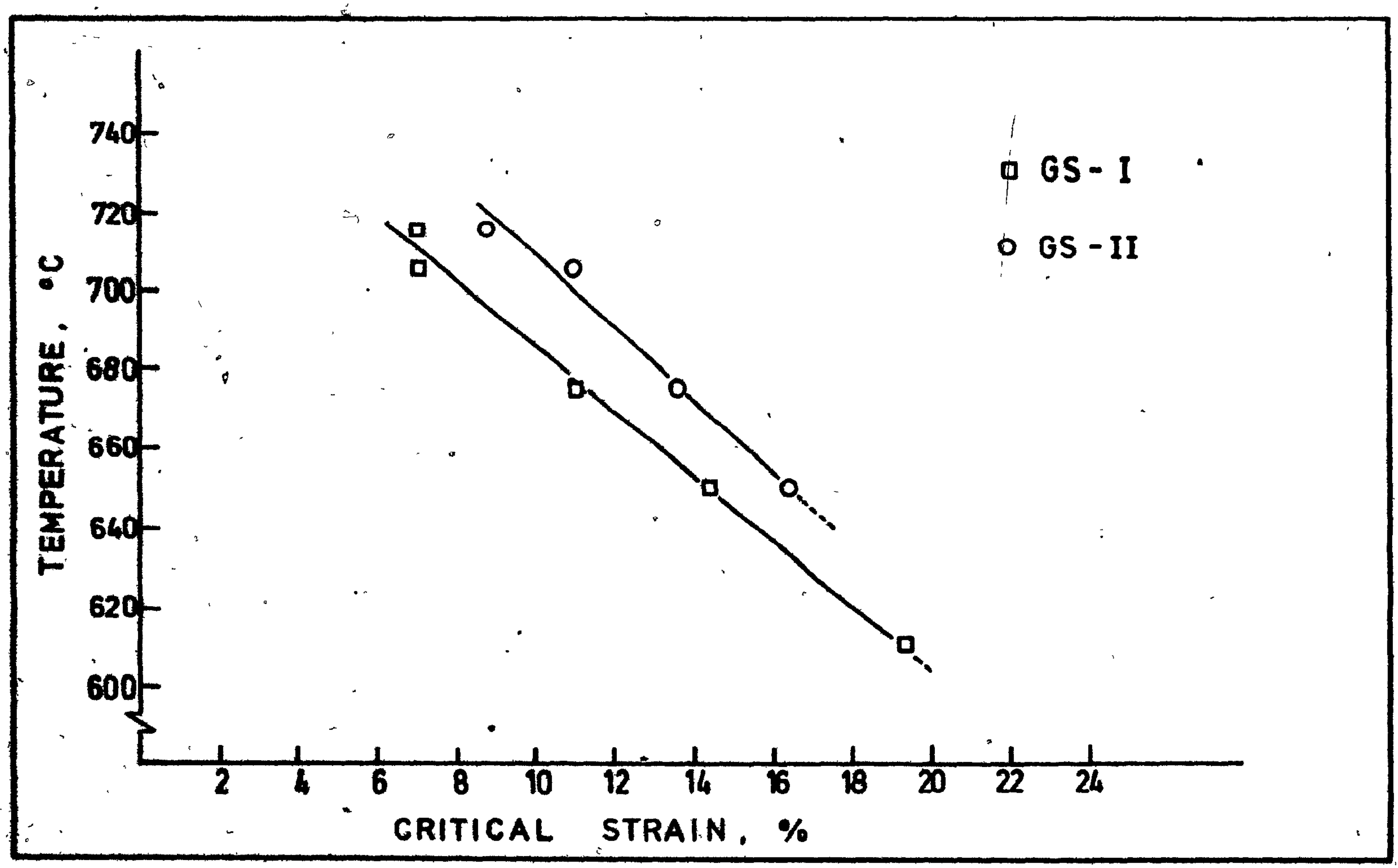


FIGURE 4.6 Effect of annealing temperature and starting grain size on critical strain. (Annealing time, 10 minutes.)

the critical strain decreased as the annealing temperature was raised. Additionally, at the same annealing temperature, the material with the largest grain size required a higher degree of pre-strain to reach the critical level.

4.3.3 Tensile Strength

Figure 4.7 and 4.8 show the effect of pre-strain and annealing temperature on the tensile strength. These figures summarize the results for the five annealing temperatures employed in the experiments and the two starting grain sizes, GS-I and GS-II, respectively. Each plotted point is the average of two tensile strength determinations (see Tables B-III and B-IV, Appendix B).

From Figure 4.7 and 4.8 it may be seen that for both starting grain sizes, tensile strength behaviour was similar to that observed for the yield strength. The position of the curves was dependent on the starting grain size of the material; for the smaller starting grain size, the tensile strength values were higher than those of the larger grain size. As before, there was a monotonic increase of tensile strength with increasing pre-strain until a critical value was reached. Further pre-straining beyond this value led to a drop in the tensile strength of the annealed specimens. The pre-strain at which this drop took place was again temperature dependent, so that as the annealing temperature was lowered the drop in tensile strength occurred at higher percentages of pre-strain.

As before, the tensile strength continued to increase with increasing pre-strain after this critical region was passed.

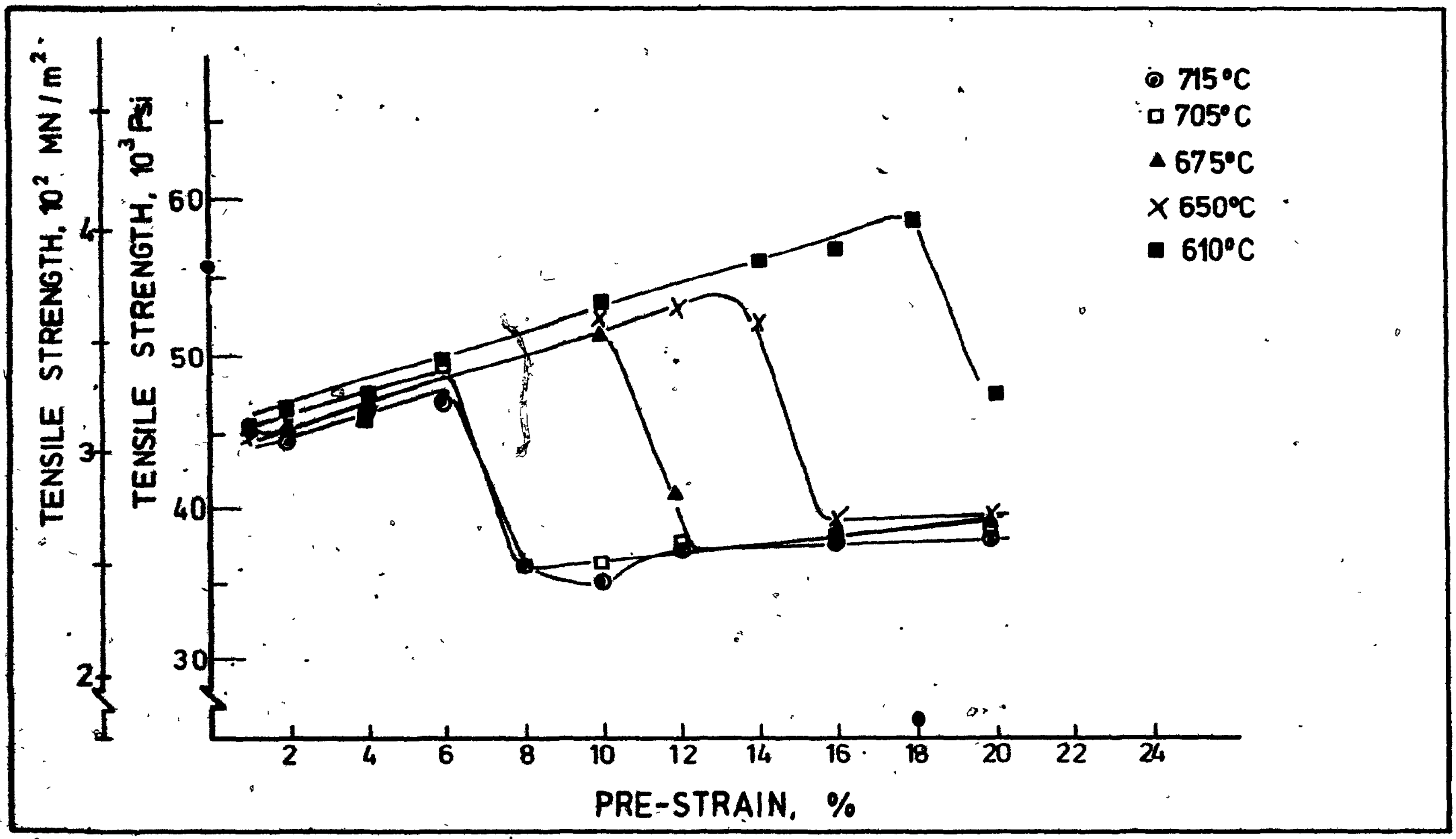


FIGURE 4.7 Effect of pre-strain and annealing temperature on tensile strength. Initial grain size: GS-I. (Annealing time: 10 minutes.)

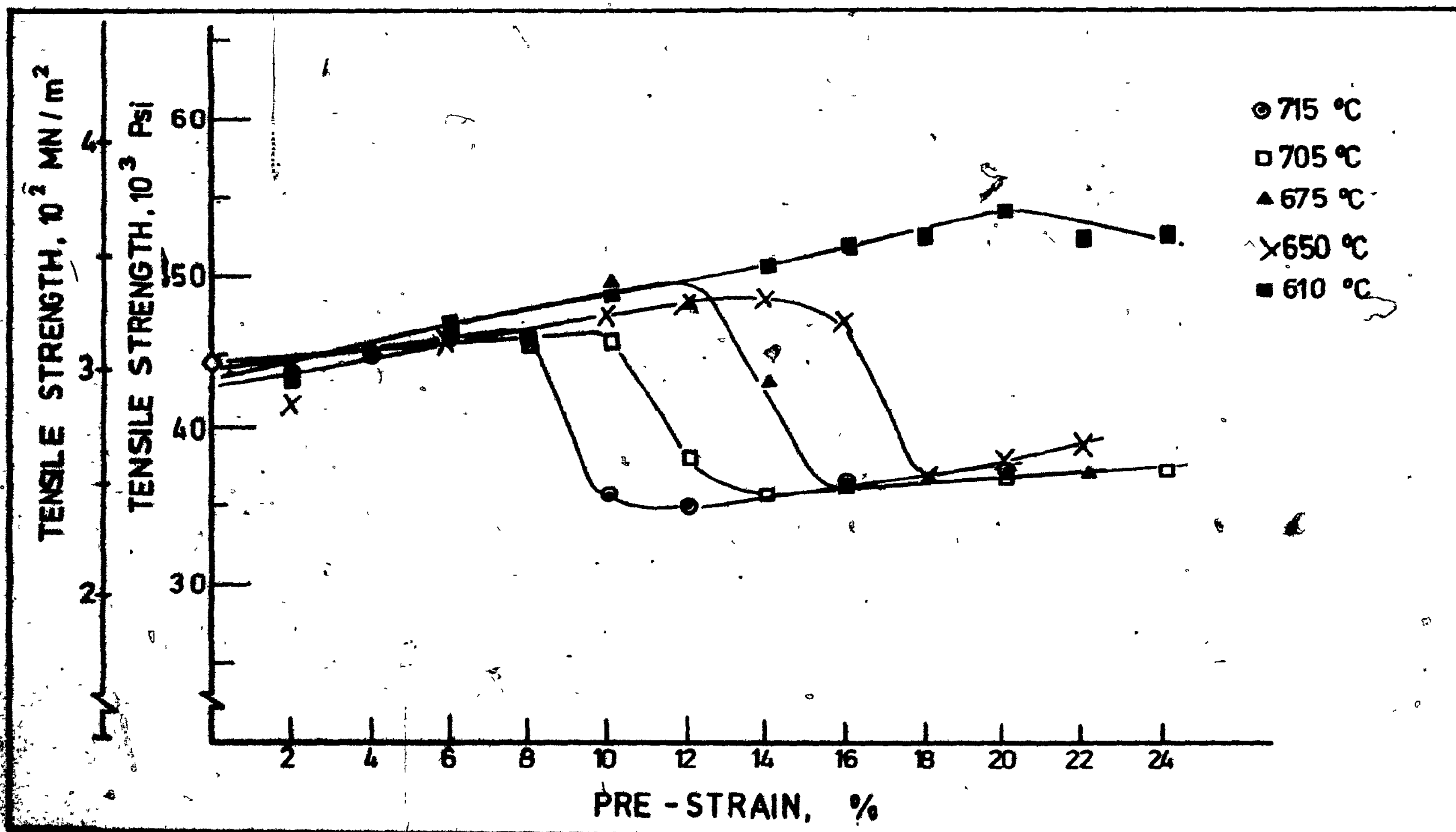


FIGURE 4.8 Effect of pre-strain and annealing temperature on tensile strength. Initial grain size: GS-II. (Annealing time: 10 minutes.)

In contrast with the yield strength results the magnitude of the drop of tensile strength was of the order of $\sim 10 \times 10^3$ psi compared with $\sim 30 \times 10^3$ psi drop in yield strength. Thus the effect of annealing temperature and pre-strain on tensile strength was much less marked.

4.3.4 Uniform Elongation

As the uniform elongation can be correlated directly with the amount of deformation that can occur before the onset of plastic instability, beyond which forming operations are impracticable, it was decided to present the ductility results in terms of the uniform elongation only. (However, total elongation results are presented in Appendix B, graphically in Figures B-IV(a) and B-IV(b) and in tabulated form in Tables B-IX and B-X).

Figures 4.9 and 4.10 show the effect of annealing temperature and pre-strain on the uniform elongation of specimens with GS-I and GS-II as starting grain sizes, respectively. Each plotted point is the average of two uniform elongation results (Appendix B, Tables B-V and B-VI).

From Figures 4.9 and 4.10, it may be seen that the dependence of uniform elongation on the degree of pre-strain was different from that observed for the yield and tensile strength. At the highest annealing temperature, 715°C, the uniform elongation remained constant until a critical region of strain was reached; in this region the uniform elongation dropped to a minimum level before returning abruptly to just above its previous level. When lower annealing temperatures, 650-705°C,

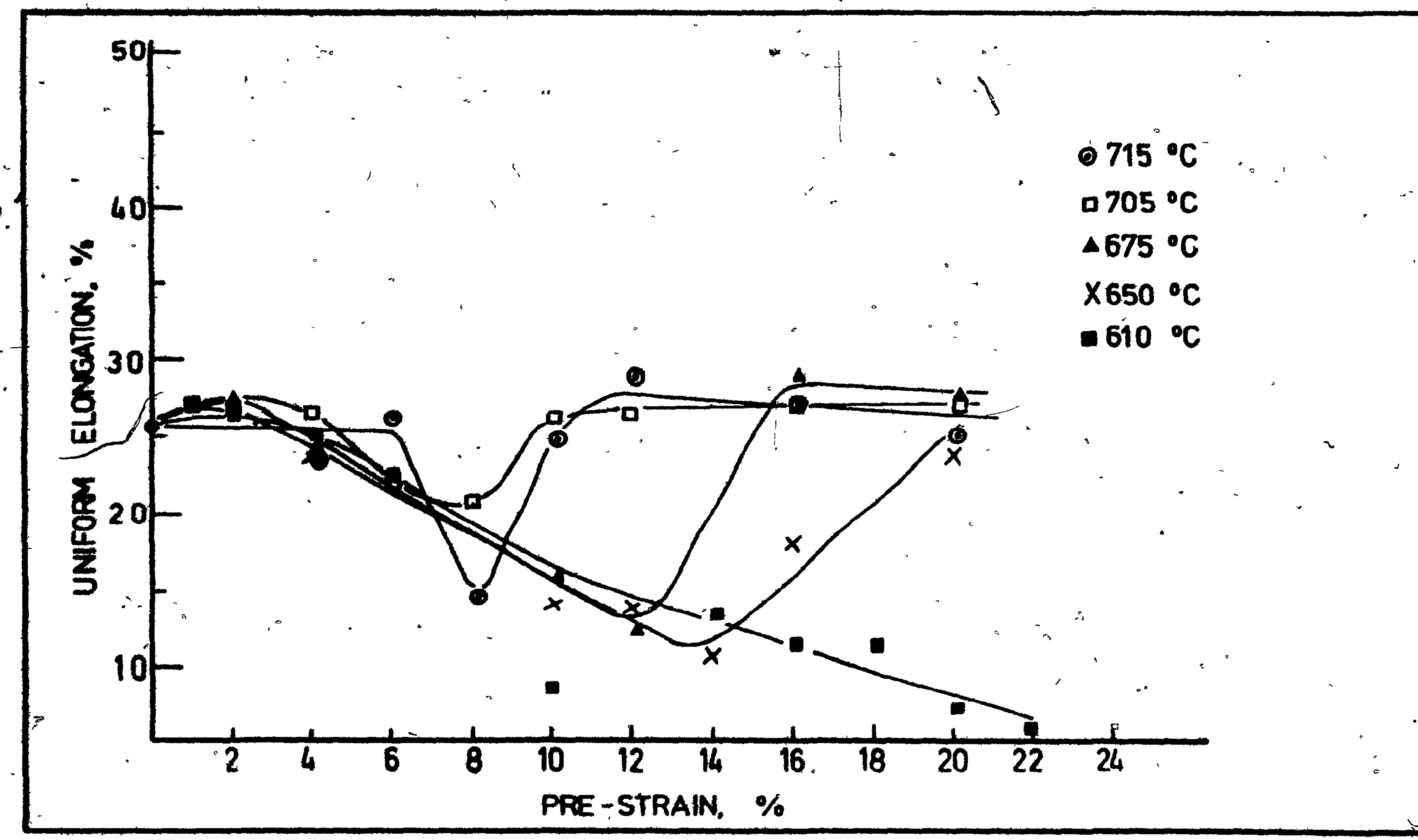


FIGURE 4.9 Effect of pre-strain and annealing temperature on uniform elongation. Starting grain size: GS-I. (Annealing time: 10 minutes.)

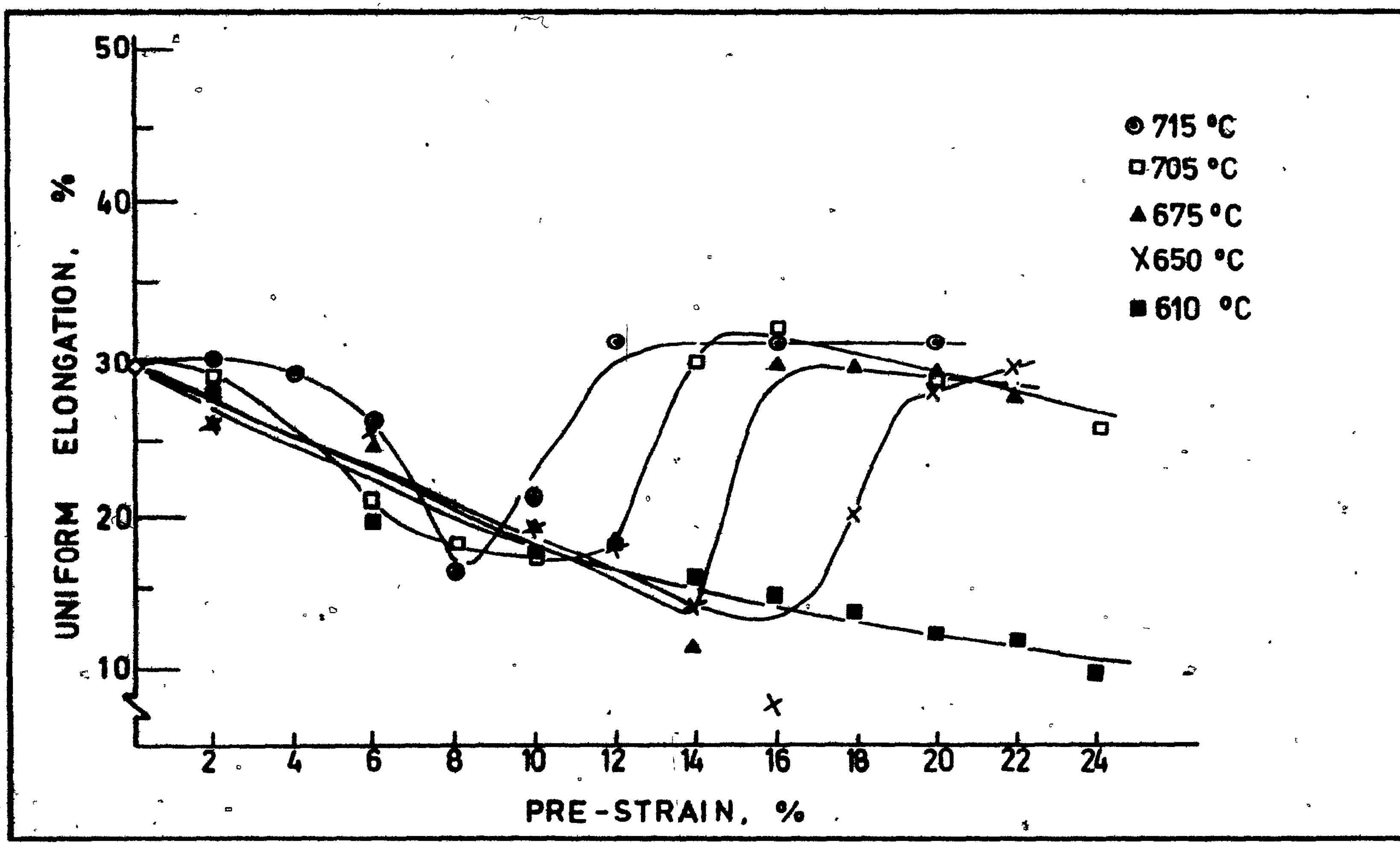


FIGURE 4.10 Effect of pre-strain and annealing temperature on uniform elongation. Starting grain size: GS-II. (Annealing time: 10 minutes.)

were used, the uniform elongation decreased slowly to a minimum value with increasing pre-strain, the minima occurring at higher pre-strains with lower annealing temperatures, and abruptly returned almost to their initial values when a critical value of strain was reached. With the lowest annealing temperature, 610°C, the uniform elongation decreased steadily up to a maximum pre-strain, 22%-24%. This relationship was observed for both GS-I and GS-II samples. As might be expected, the minima are closely related to the amount of pre-strain at which a drastic drop in yield strength occurred, i.e. to the critical strain.

4.3.5 Yield-Tensile Ratio

The yield-tensile ratio is an important criterion for the selection of material intended for severe forming operations. The lower this value, the greater is the spread between yield strength and tensile strength, and consequently, the more suitable the material is for the severe deformations encountered, for example, in pressing operations.

Figure 4.11 and 4.12 show the yield-tensile ratio for the two grain sizes, respectively, at the five annealing temperatures employed (the yield-tensile ratio results are fully tabulated in Tables B-VII and B-VIII, Appendix B).

4.4 Grain Size Variation

The effect of pre-strain on the final recrystallized grain size is shown in Figures 4.13-17 for both starting grain sizes. Each graph corresponds to one annealing temperature and an annealing time of 10 minutes.

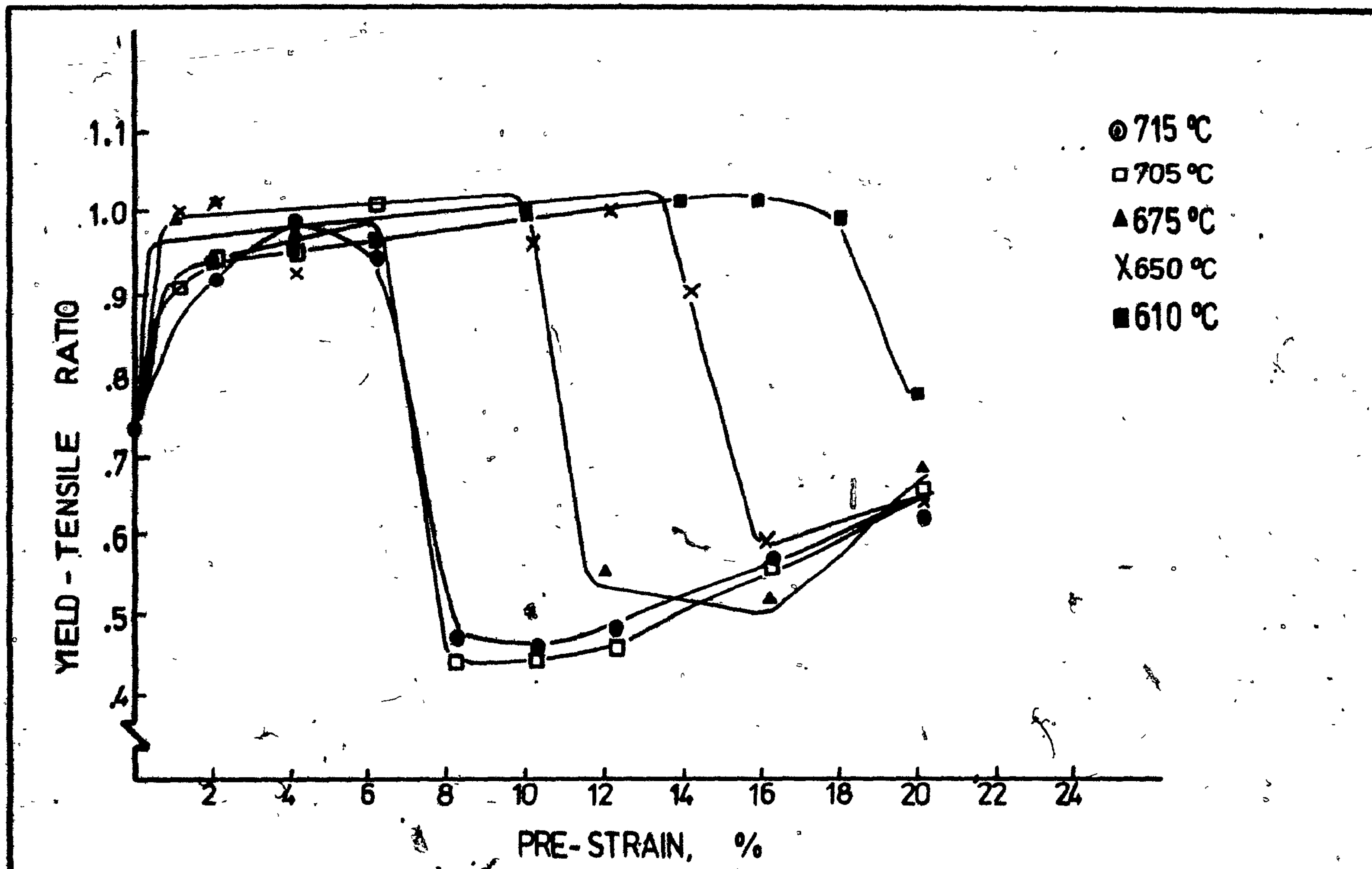


FIGURE 4.11 Effect of annealing temperature and intermediate strain on yield-tensile ratio. Starting grain size, GS-I. (Annealing time: 10 minutes.)

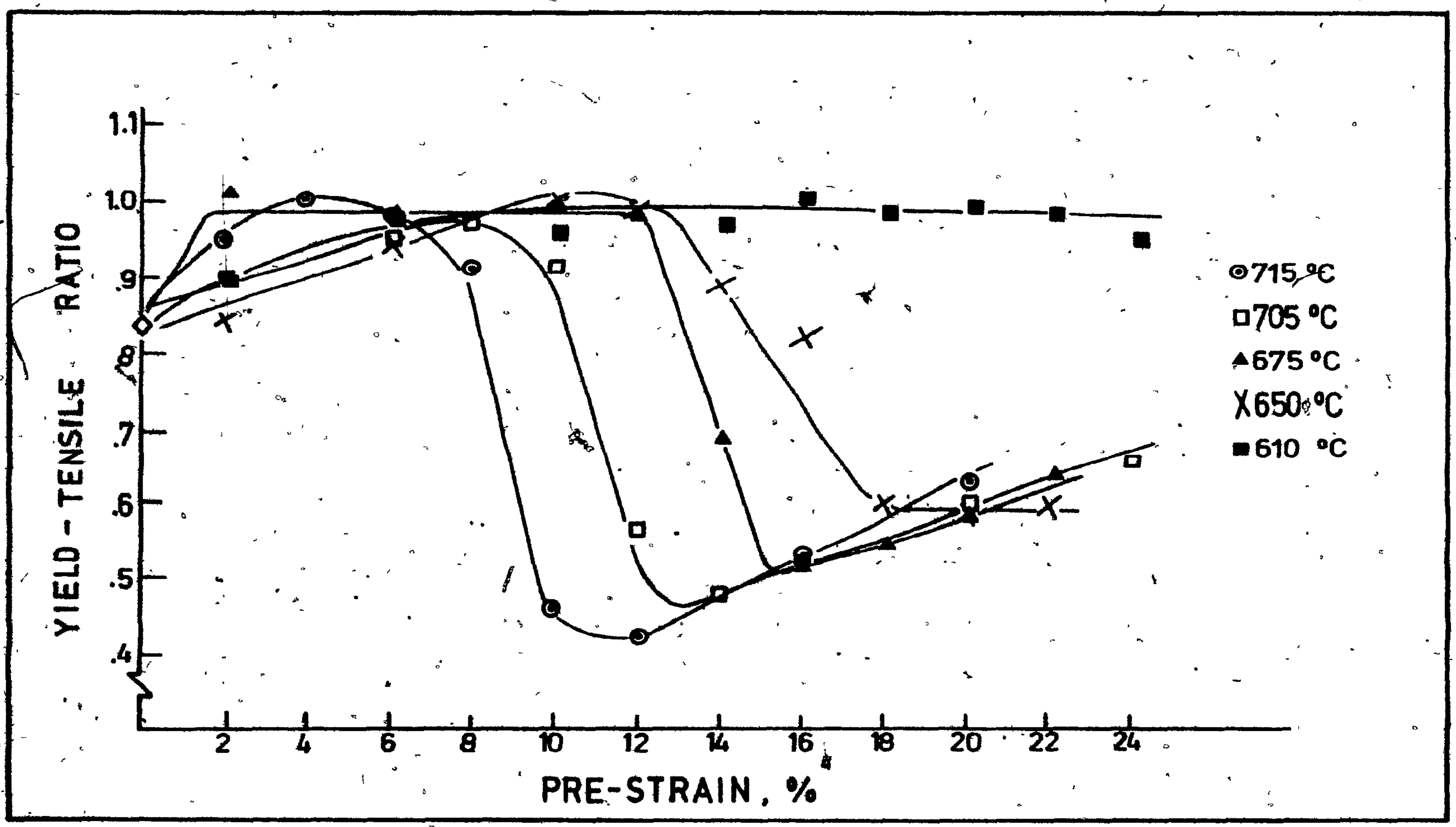


FIGURE 4.12 Effect of annealing temperature and intermediate strain on yield-tensile ratio. Starting grain size, GS-II. (Annealing time: 10 minutes.)

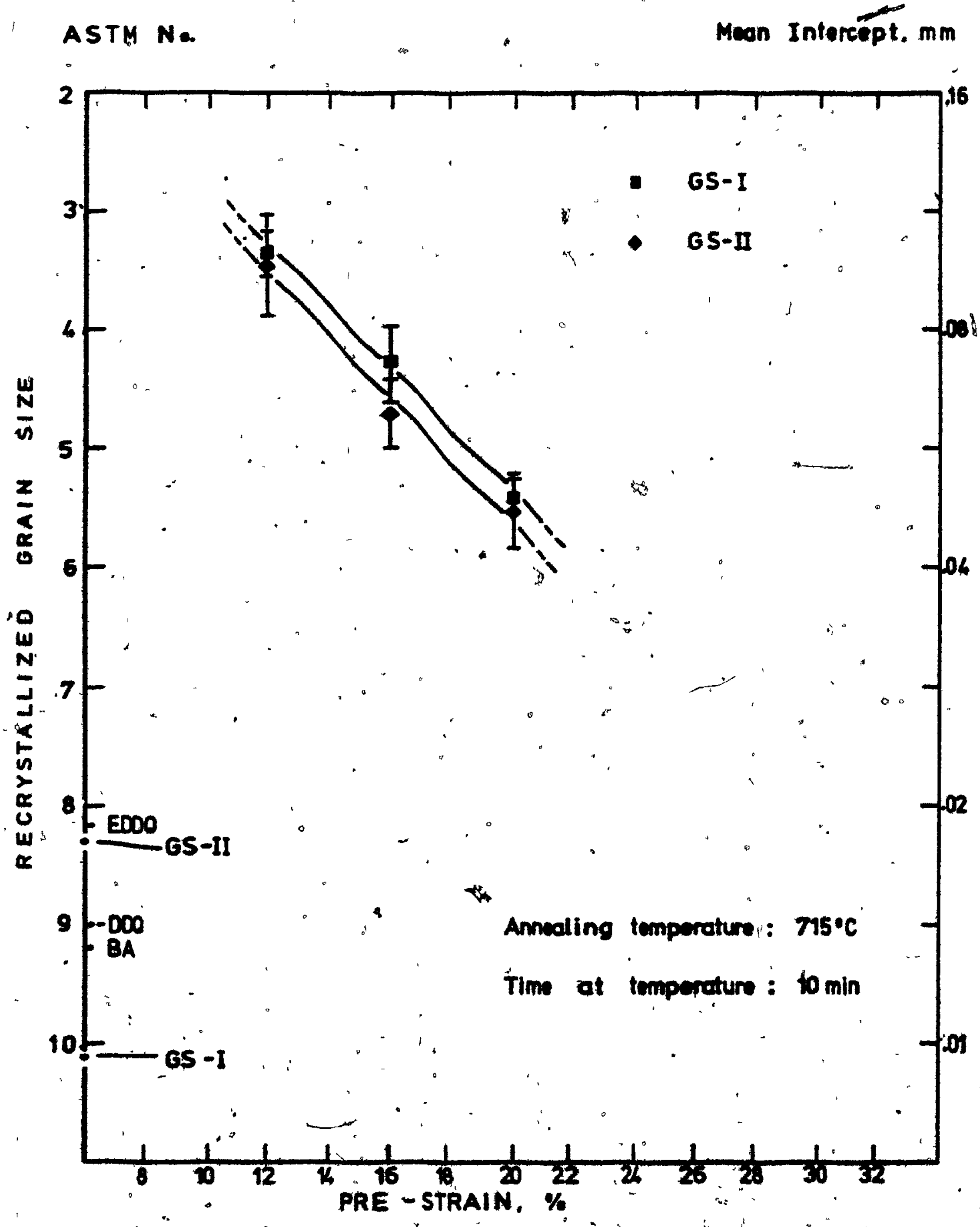


FIGURE 4.13 Effect of pre-strain and starting grain size on the final recrystallized grain size. Annealing temperature: 715°C.

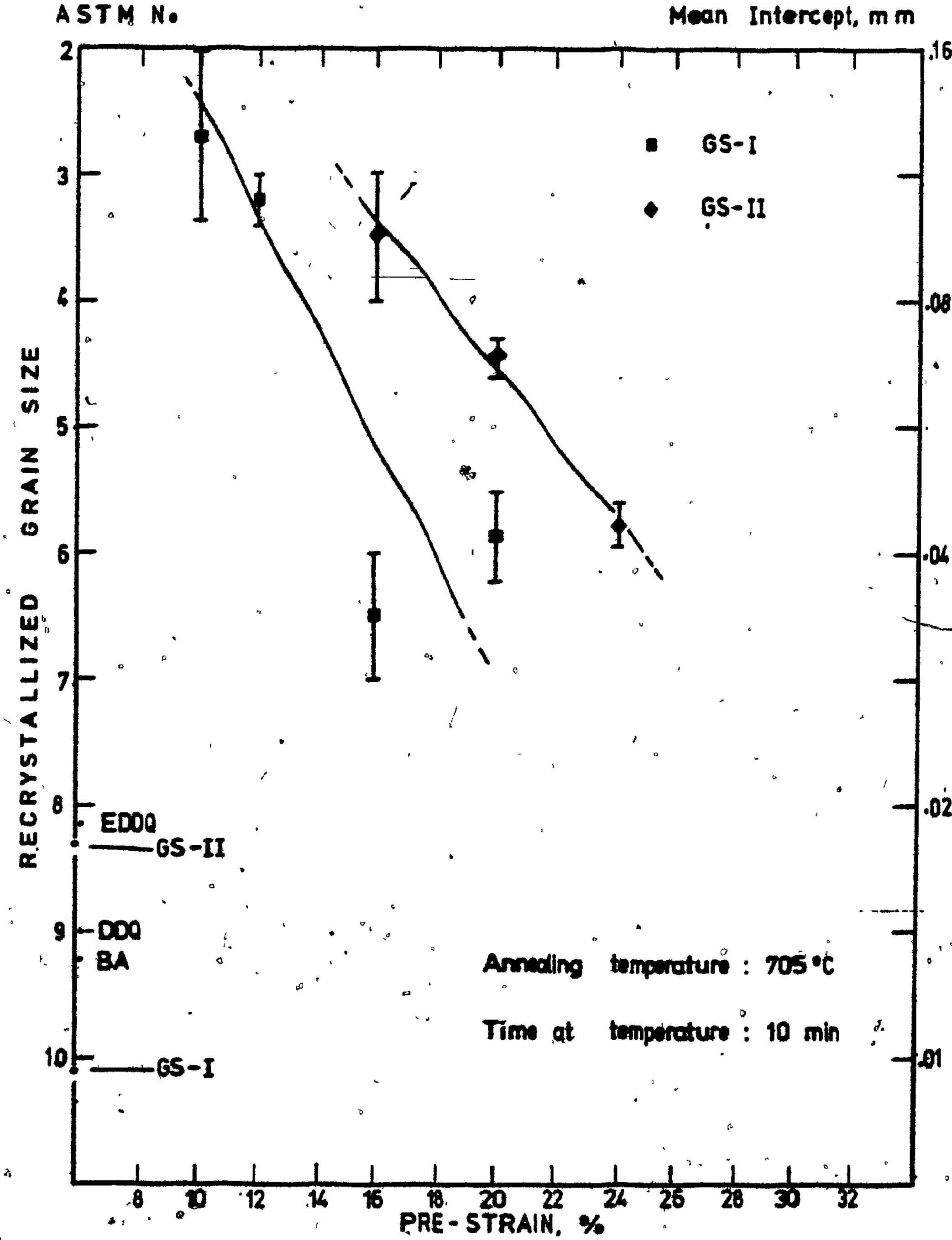


FIGURE 4.14 Effect of pre-strain and starting grain size on the final recrystallized grain size. Annealing temperature: 705°C.

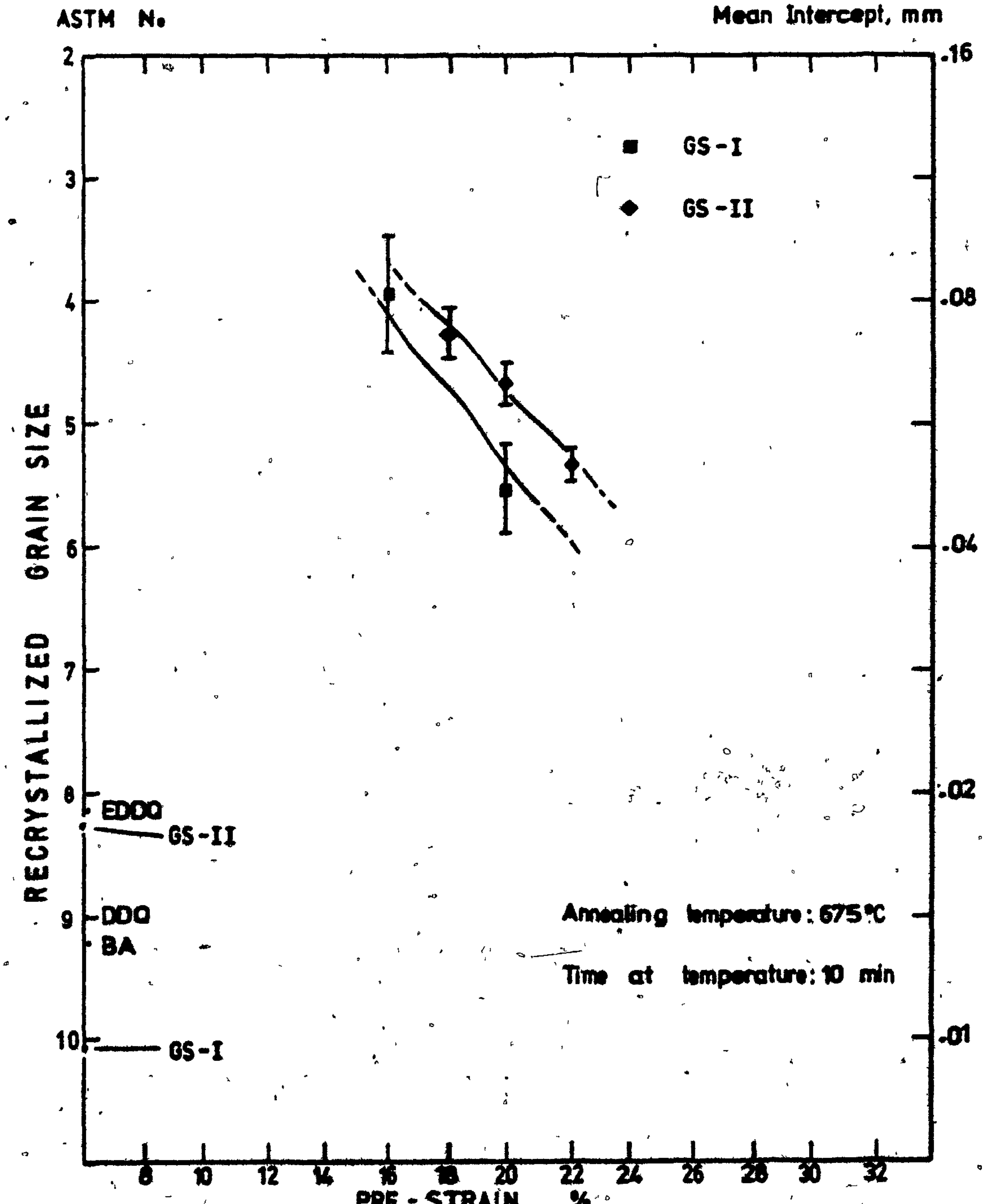


FIGURE 4.15 Effect of pre-strain and starting grain size on the final recrystallized grain size. Annealing temperature: 675°C.

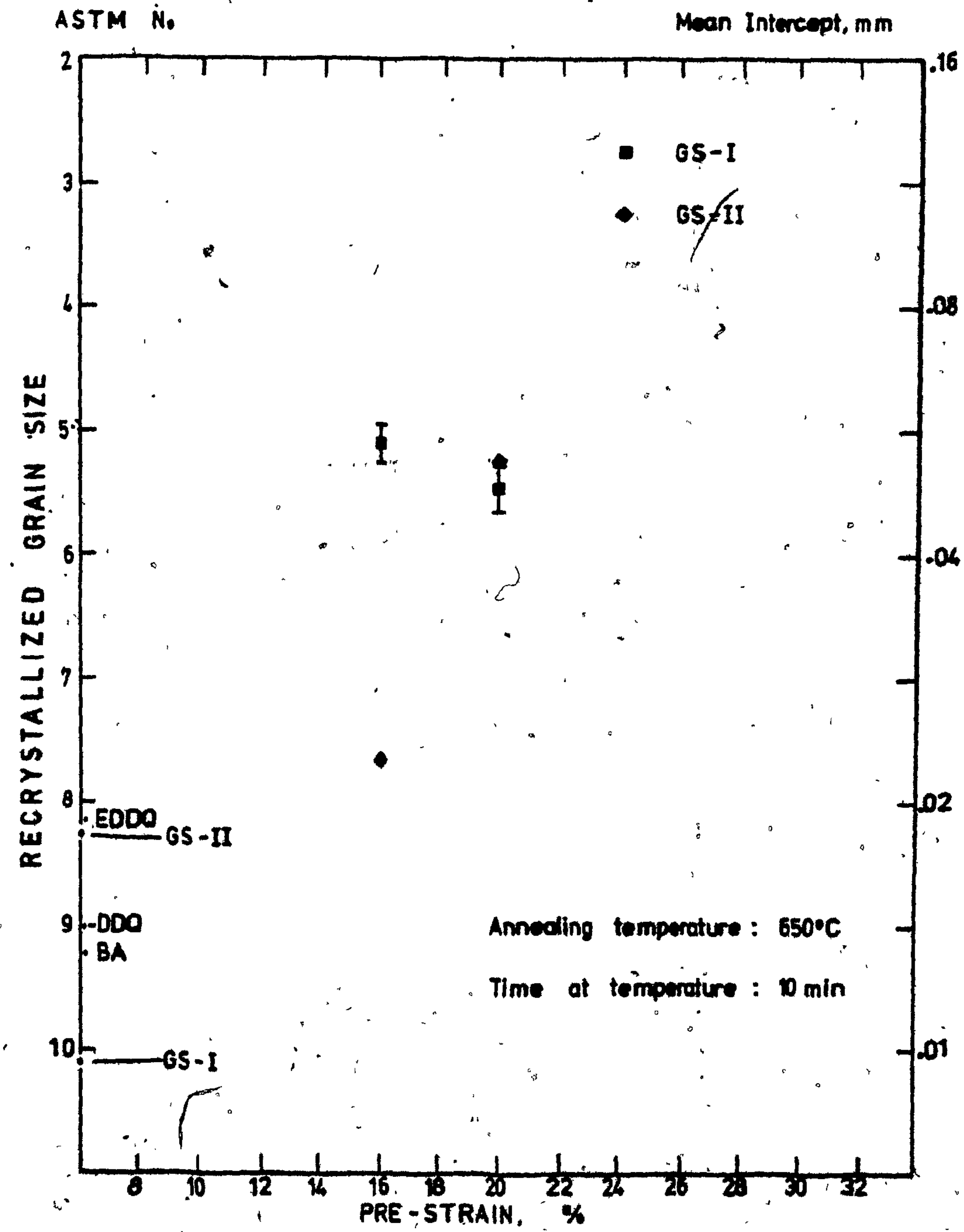


FIGURE 4.16 Effect of pre-strain and starting grain size on the final recrystallized grain size. Annealing temperature: 650°C.

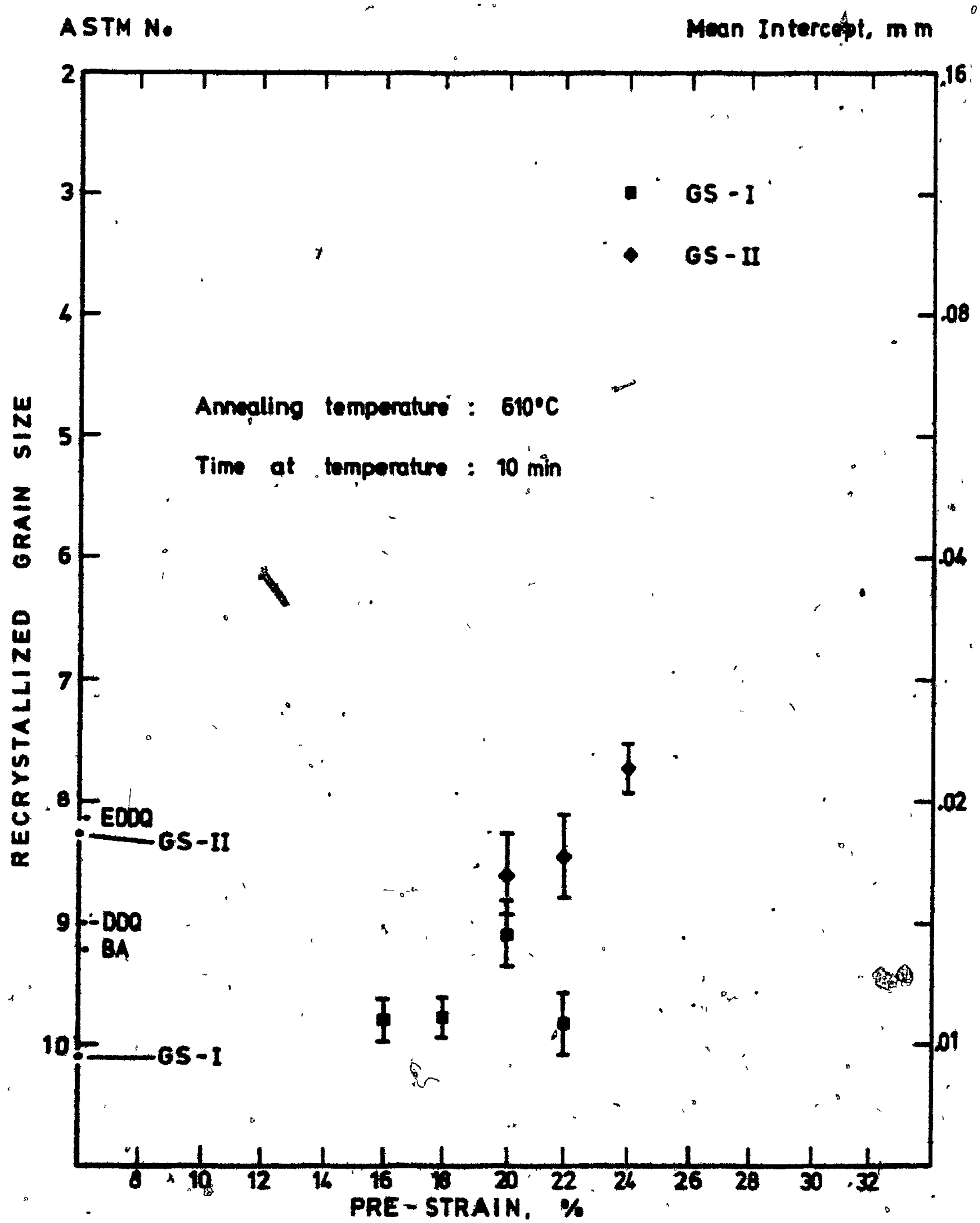


FIGURE 4.17 **Effect of pre-strain and starting grain size on the final recrystallized grain size. Annealing temperature: 610°C.**

Grain size measurements were made only on fully recrystallized samples, i.e. those which had been pre-strained by more than the critical strain. All samples, in any particular series, which had been pre-strained beyond the critical value were used for such grain size estimations.

The grain size is expressed as the mean intercept distance ($\bar{l}$) in millimeters calculated to 95% confidence limits (see Section 3.5.4) and it is also plotted in terms of ASTM grain size numbers. Limit bars on each plotted point indicate the spread of grain size measurements. The two starting grain sizes (GS-I and GS-II) employed in the experiments are shown on the graphs, as well as the grain sizes corresponding to batch annealed (BA) material, and to two Japanese steels (DDQ and EDDQ).* This additional information was included on the graphs to enable comparisons to be made between the final recrystallized grain sizes obtained by strain-annealing and those normally produced in the industry.

As expected, material that had been pre-strained just beyond the critical strain had a coarse recrystallized grain size and as the amount of pre-strain increased, the grain size decreased. This behaviour was observed for the annealing temperatures 715, 705 and 675°C and for materials of both starting grain sizes. The coarser grained starting material yielded the largest final grain sizes, and in all cases the final grain size was considerably coarser than that of commercial materials.

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* DDQ = deep drawing quality
EDDQ = extra deep drawing quality

When an annealing temperature of 650°C was employed (Figure 4.16), material with GS-I, the small initial grain size, exhibited the grain size - pre-strain relationship observed at the higher annealing temperatures. For the larger-grained GS-II material previous graphs (e.g. 4.5, 4.6) have shown that critical strain at 650°C was approximately 17% so that the grain size value plotted at 16% pre-strain was an unrecrystallized grain size.

It has been shown previously (Figure 4.4 and 4.5) that for the annealing temperature 610°C, the critical amount of strain was never exceeded but fell outside the limitations of the experimental method. Recrystallized grains were not obtained at this annealing temperature and measurement of the pre-recrystallization grain size showed it to vary little from the starting grain sizes.

4.5 Metallography

4.5.1 Photomicrography: Criterion for the Selection of Specimens

Because of the very large number of samples that would be required, it was impractical to produce a photographic record of all samples, so the following selection criterion was adopted. It has been stated (Section 2.2.1) that two important characteristics in formability selection are high uniform elongation and low yield-tensile ratio. Thus for each initial grain size and for each annealing temperature used, uniform elongation and yield-tensile ratio were plotted as a function of pre-strain. Using each of these graphs, Figures 4.18-27, a specimen was selected as indicated by the dashed line joining optimum uniform elongation and yield-tensile ratio values at a particular level of pre-strain.

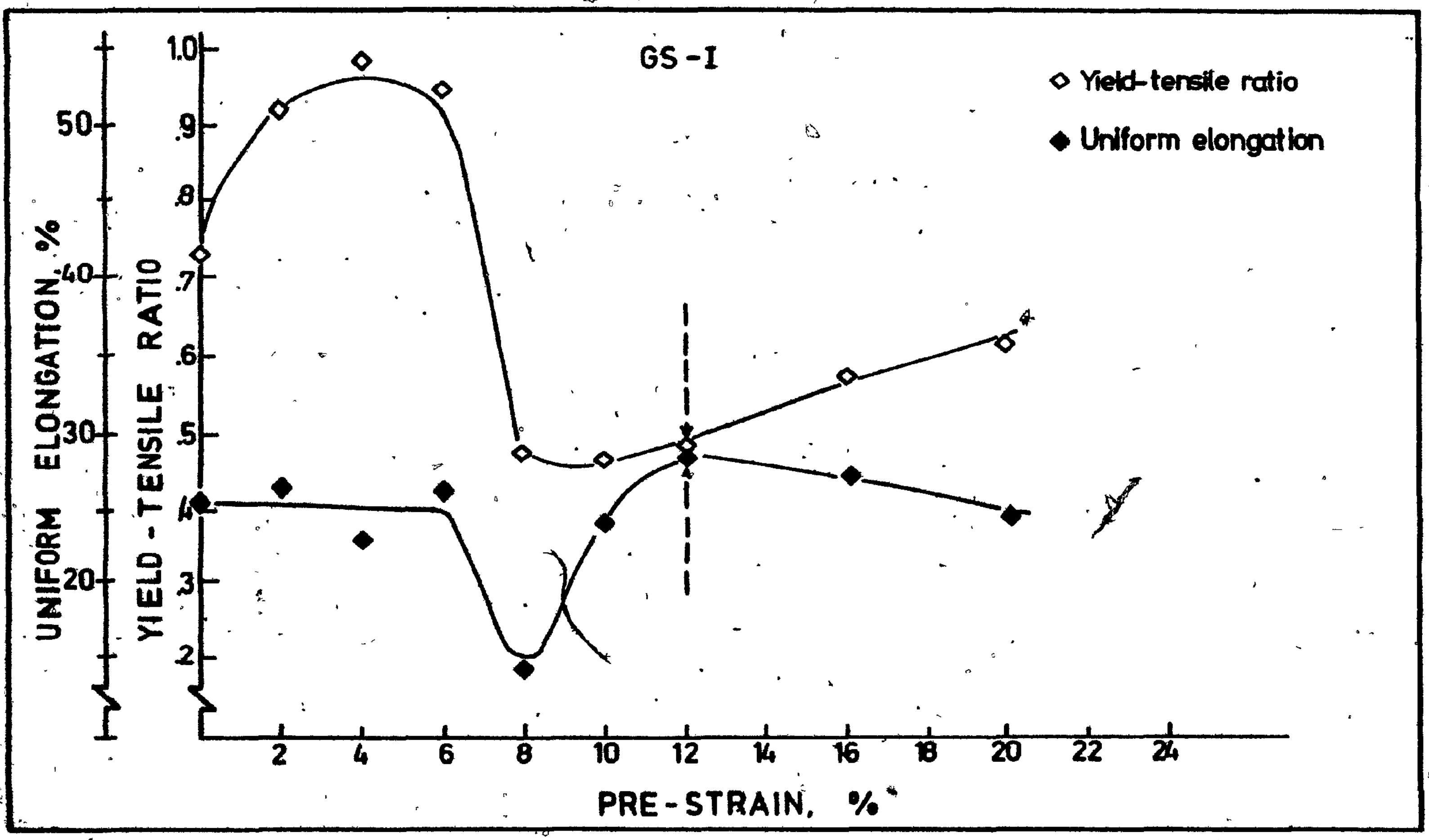


FIGURE 4.18 Specimen selection for metallographic examination.
Annealing temperature: 715°C.
Annealing time: 10 minutes.

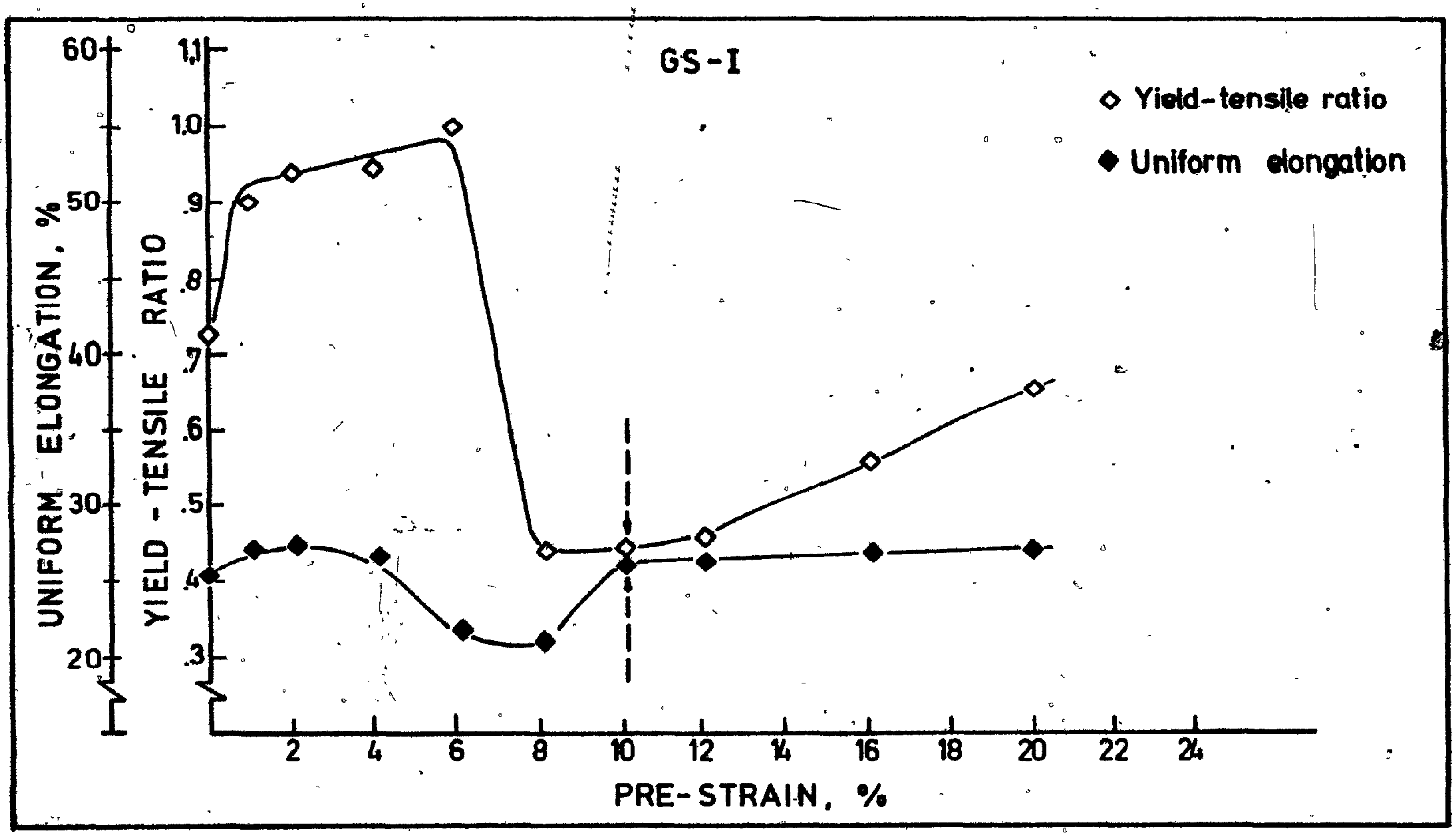


FIGURE 4.19 Specimen selection for metallographic examination.
Annealing temperature: 705°C.
Annealing time: 10 minutes.

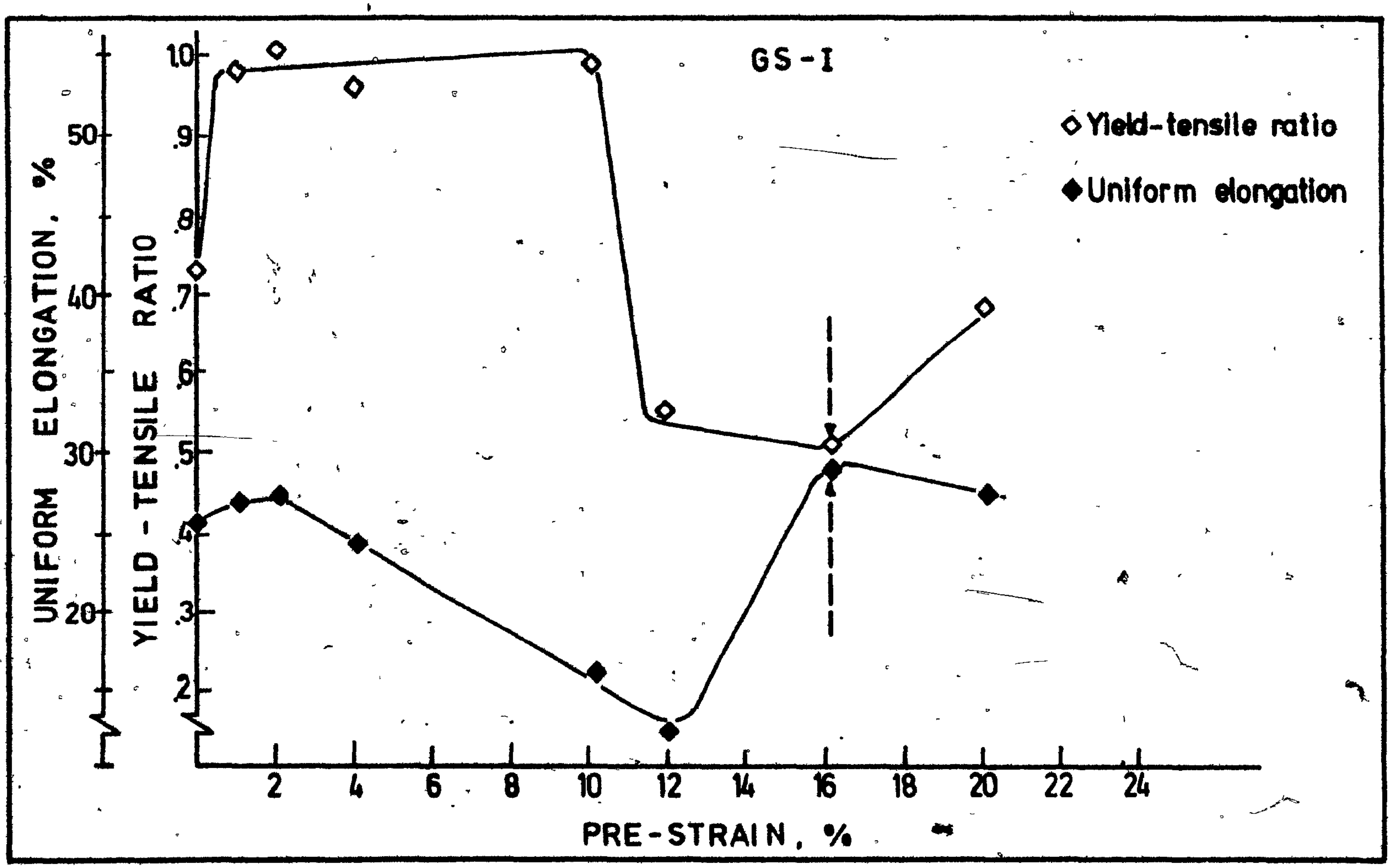


FIGURE 4.20 Specimen selection for metallographic examination.
Annealing temperature: 675°C.
Annealing time: 10 minutos.

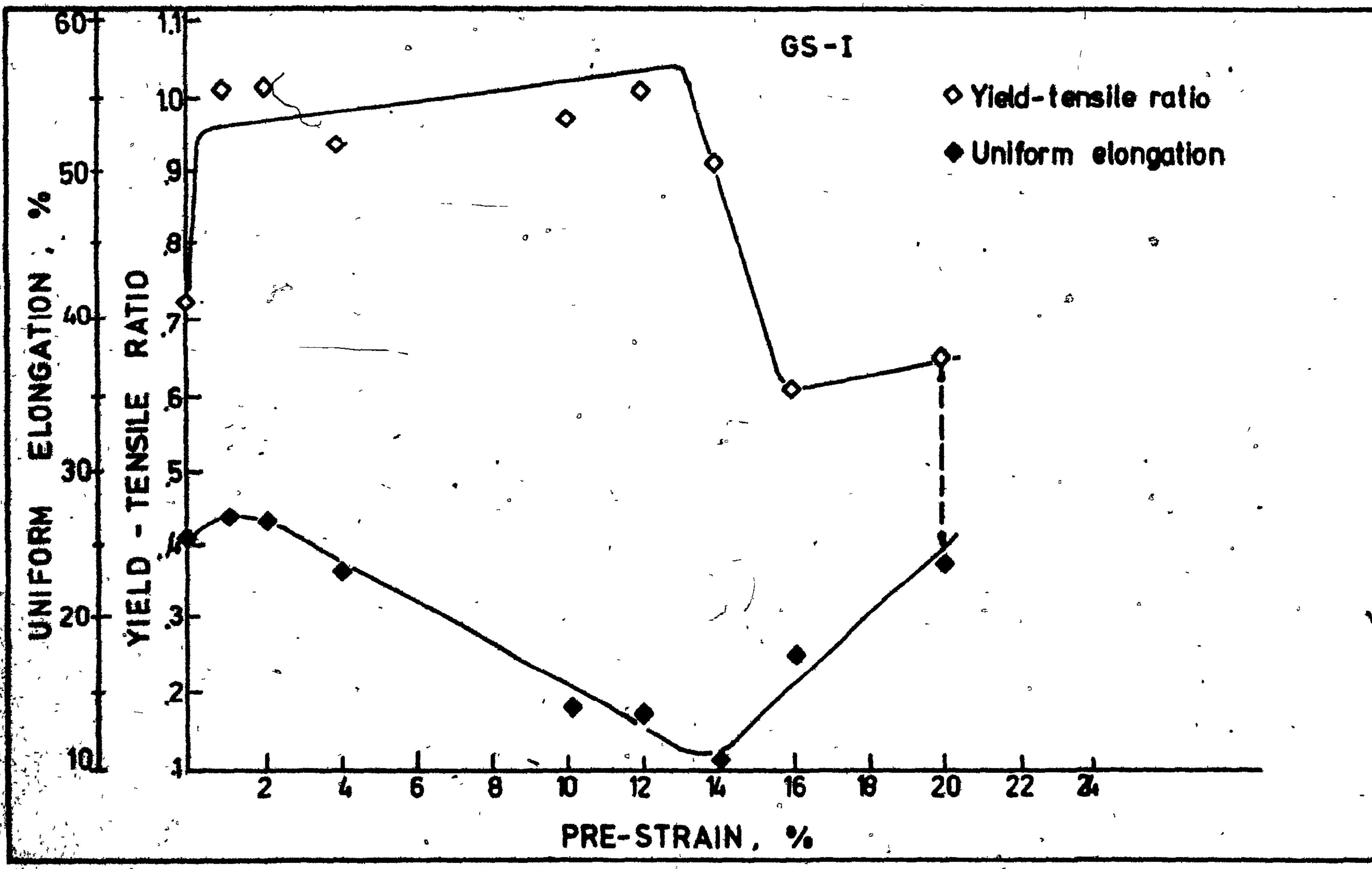


FIGURE 4.21 Specimen selection for metallographic examination.
Annealing temperature: 650°C.
Annealing time: 10 minutes.

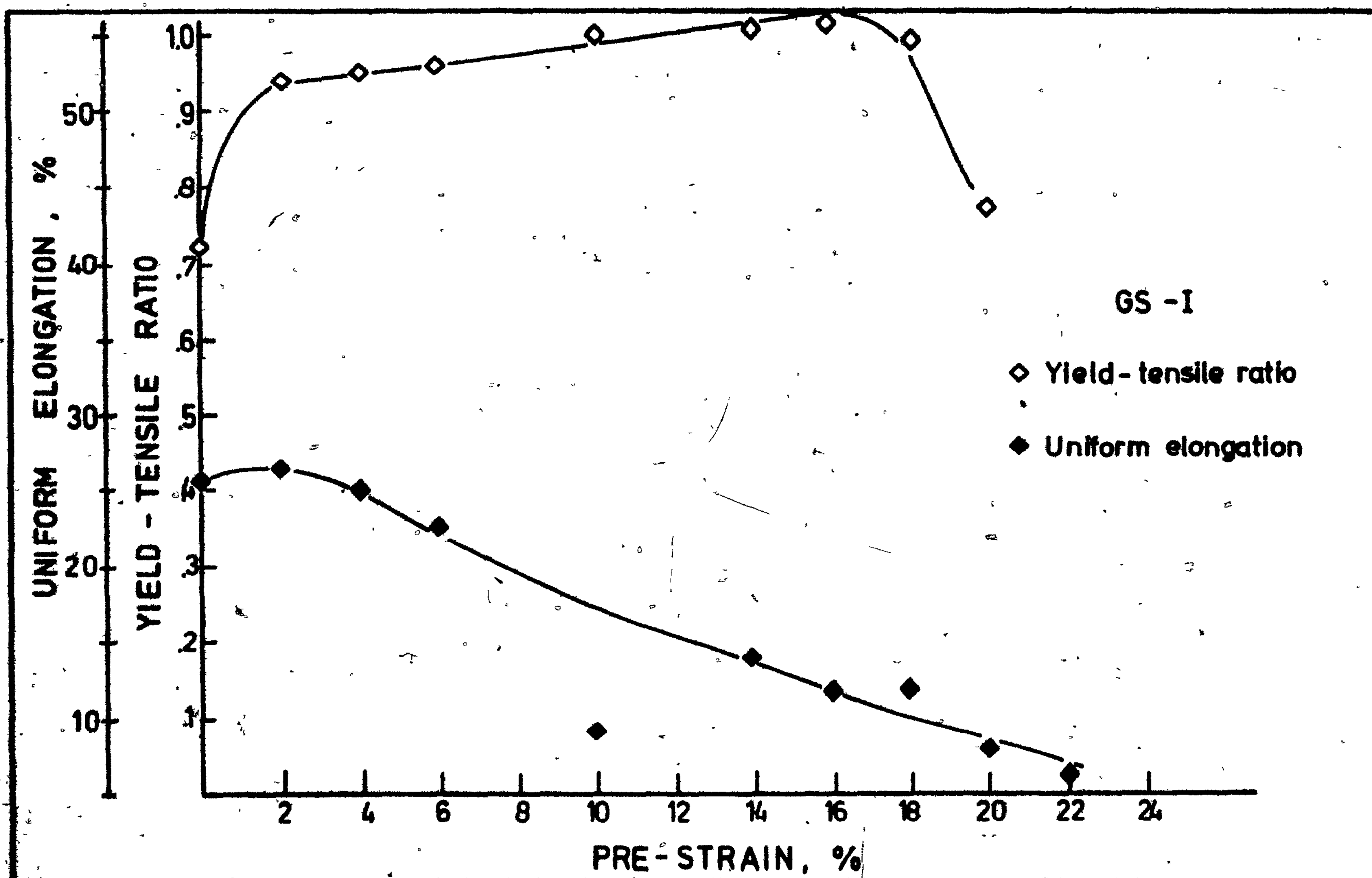


FIGURE 4.22 Specimen selection for metallographic examination.
Annealing temperature: 610°C.
Annealing time: 10 minutes.

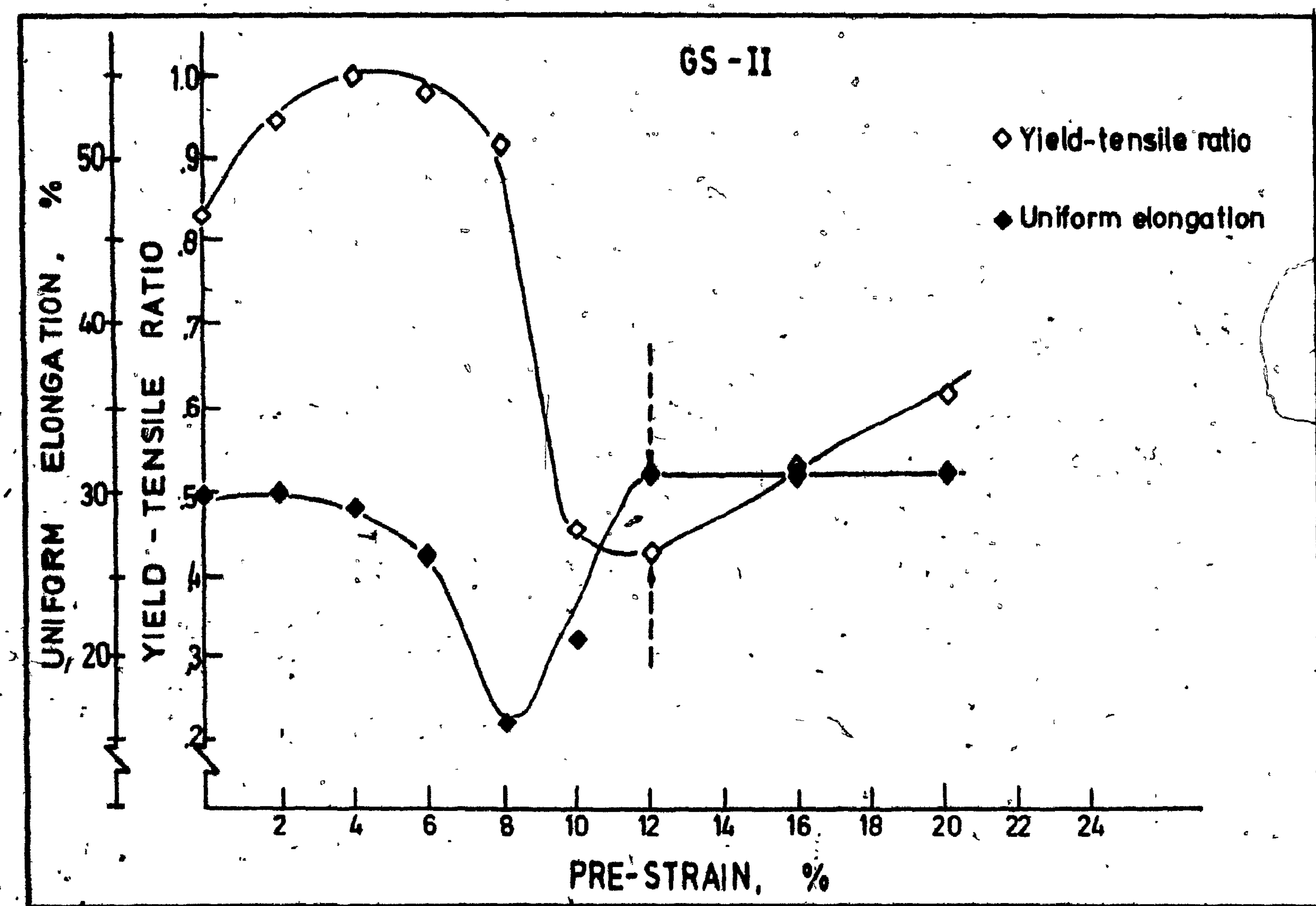


FIGURE 4.23 Specimen selection for metallographic examination.
Annealing temperature: 715°C.
Annealing time: 10 minutes.

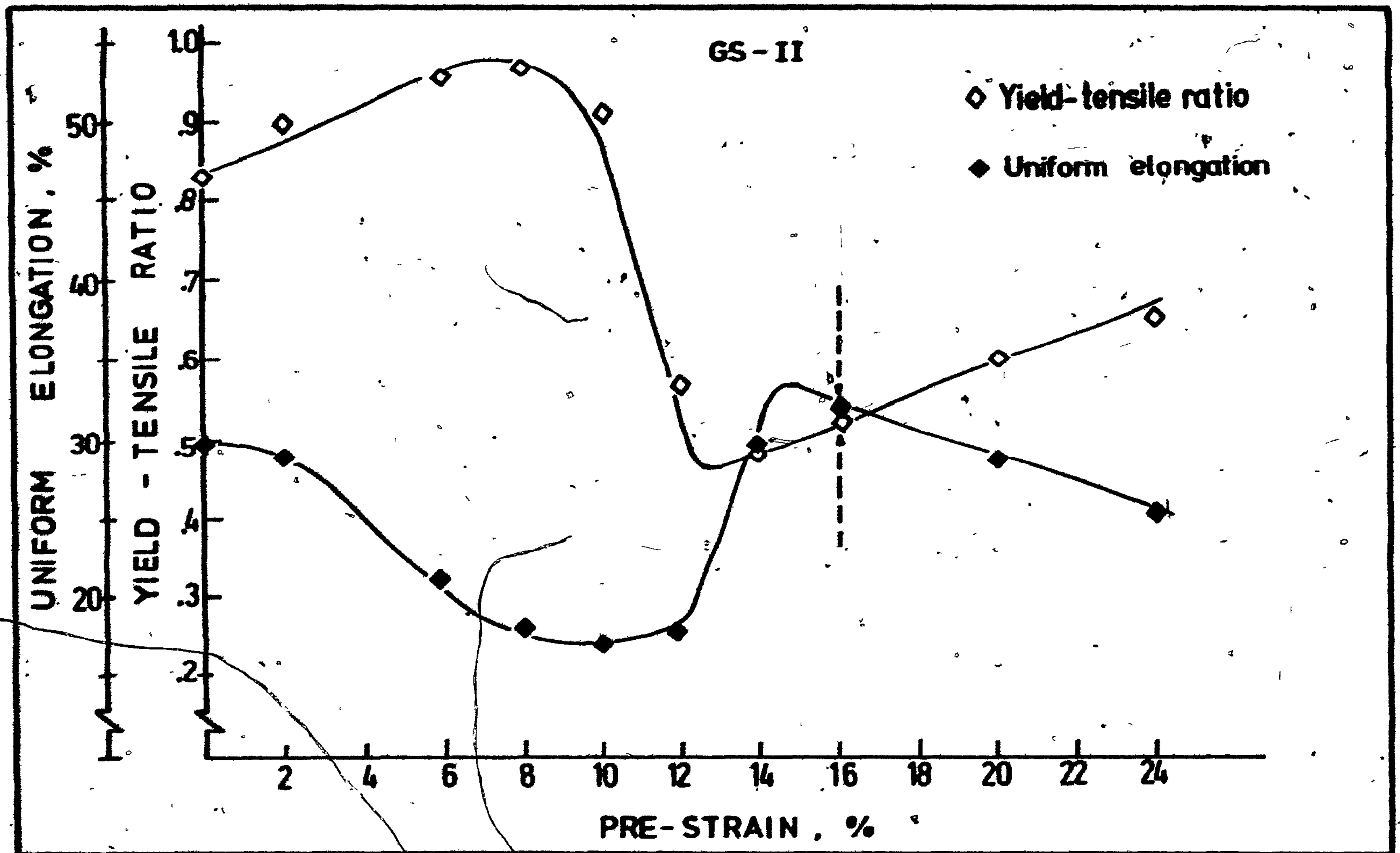


FIGURE 4.24 Specimen selection for metallographic examination.
Annealing temperature: 705°C.
Annealing time: 10 minutes.

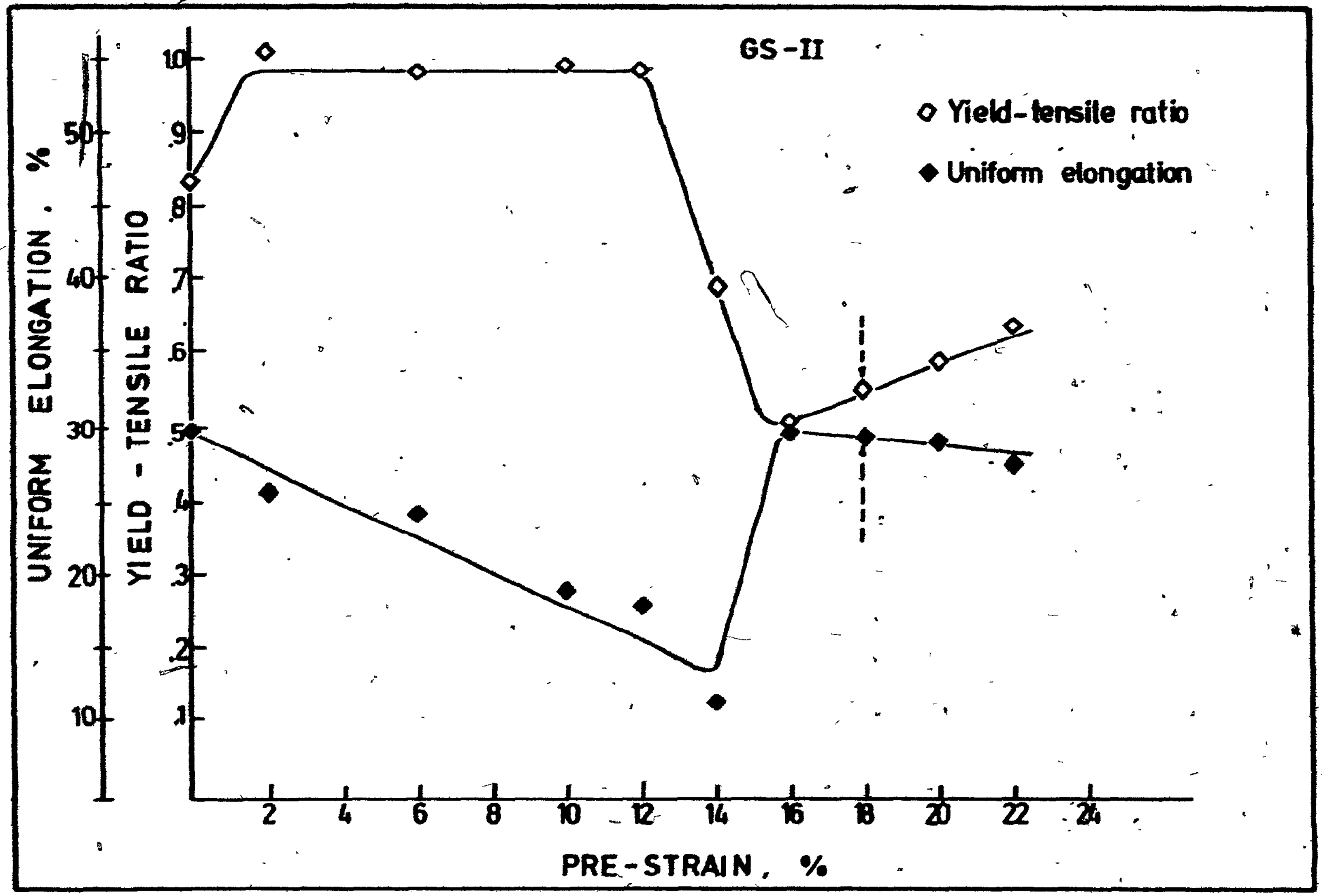


FIGURE 4.25 Specimen selection for metallographic examination.
Annealing temperature: 675°C.
Annealing time: 10 minutes.

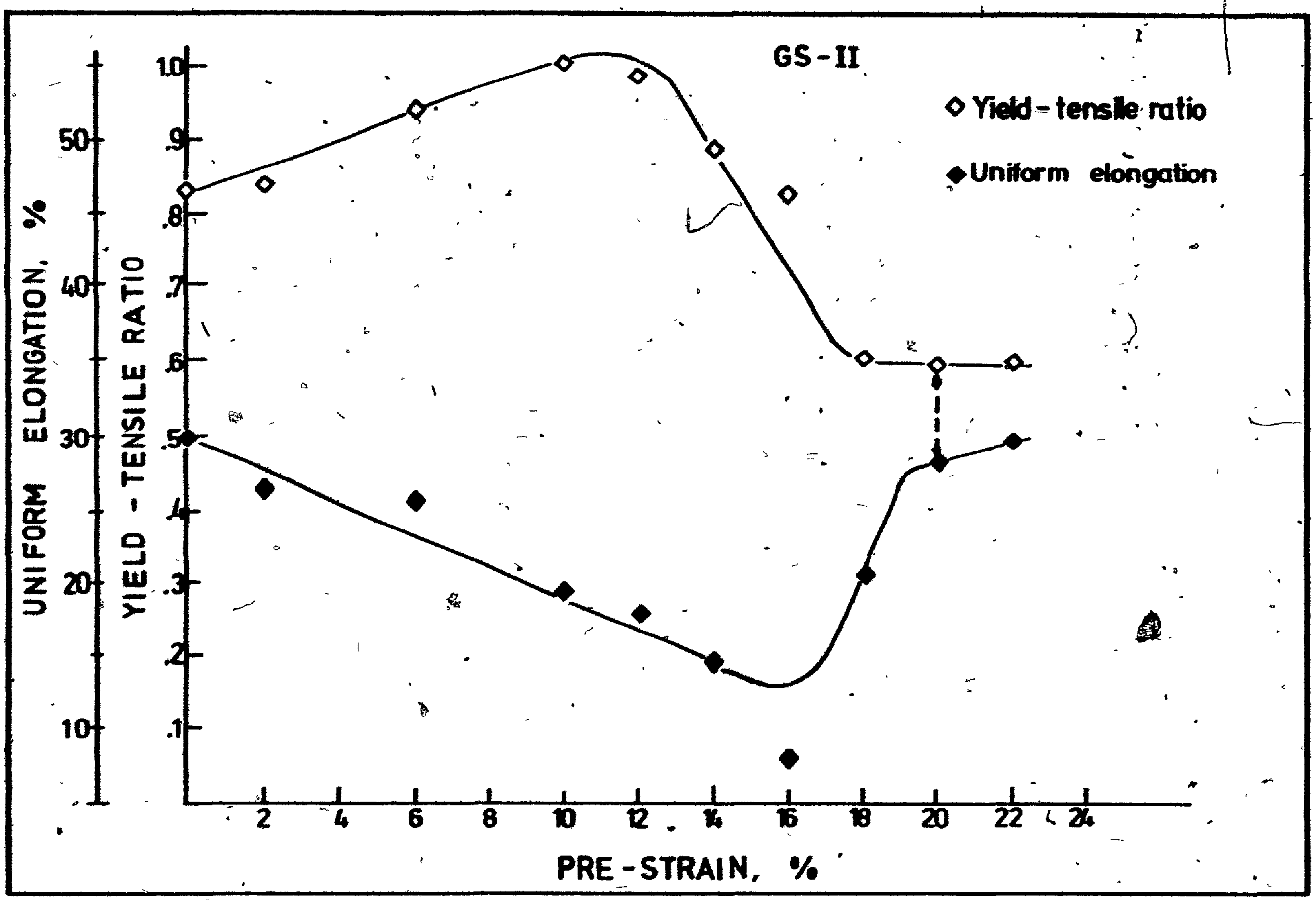


FIGURE 4.26 Specimen selection for metallographic examination.
Annealing temperature: 650°C.
Annealing time: 10 minutes.

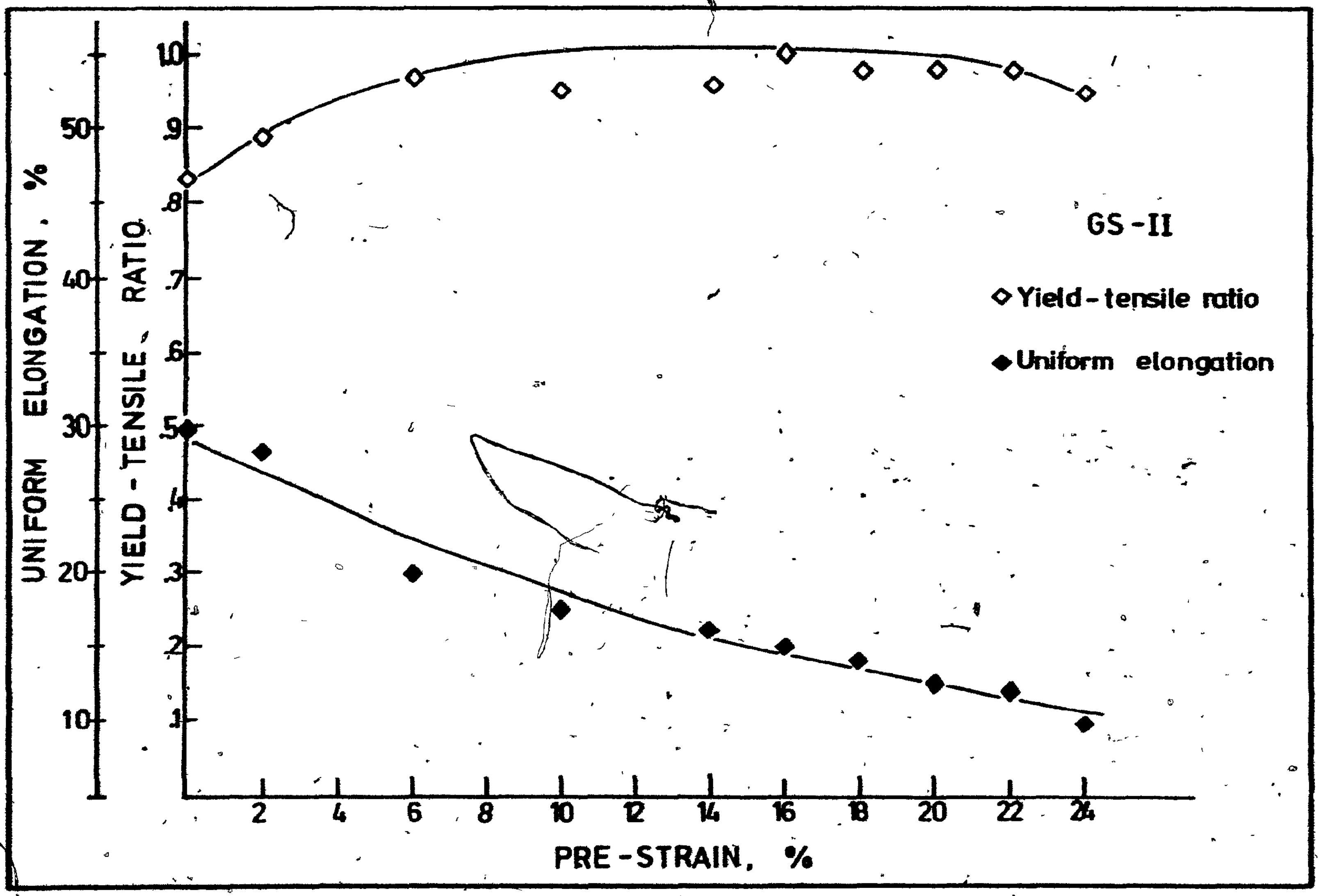


FIGURE 4.27 Specimen selection for metallographic examination.
Annealing temperature: 610°C.
Annealing time: 10 minutes.

The characteristics of specimens which were selected using this criterion are given below:

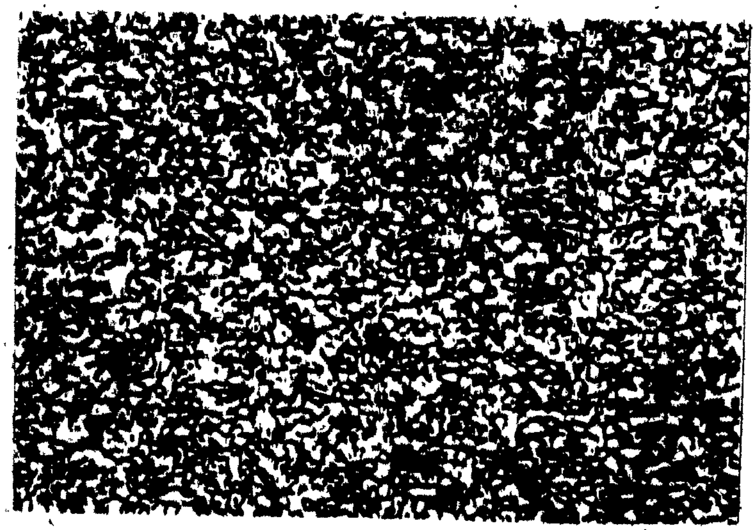
Annealing Temperature (°C)	% Pre-Strain GS-I	% Pre-Strain GS-II
715	12	12
705	10	16
675	16	18
650	20	20
610	-	-

As would be expected, the % pre-strain of the selected samples was always greater than the critical strain. No metallographic specimens were selected from samples annealed at 610°C; no specimen exhibited a desirable combination of properties which was predictable as the critical strain was never exceeded for this annealing temperature (see Figures 4.22 and 4.27).

4.5.2 Microstructures

Figures 4.28 and 4.29 show the starting grain sizes, GS-I and GS-II, employed in the experiments. These and all subsequent photomicrographs were taken at a magnification of X 100.

The next set of figures, 4.30-37 illustrate the final recrystallized grain size of the selected pre-strained and annealed samples. To enable the correlation of properties with microstructure, each photomicrograph is accompanied by the following data: initial grain size, amount of pre-strain, annealing temperature, as well as final mechanical properties and recrystallized grain size. The



GS-I

YS = 40.7×10^3 Psi

UTS = 55.9×10^3 Psi

UE = 25.4%

TE = 29.7%

$\bar{\ell}$ = 0.0096 mm

ASTM No. = 10.1 ± 0.208 (95% C.L.)

FIGURE 4.28 GS-I, as recrystallization I annealed. 100 X

GS-II

YS = 36.9×10^3 Psi

UTS = 44.5×10^3 Psi

UE = 29.6%

TE = 39.6%

$\bar{\ell}$ = 0.0185 mm

ASTM No. = 8.28 ± 0.215 (95% C.L.)

FIGURE 4.29 GS-II, as recrystallization I annealed. 100 X

GS-I

YS = 18.2×10^3 Psi

UTS = 37.4×10^3 Psi

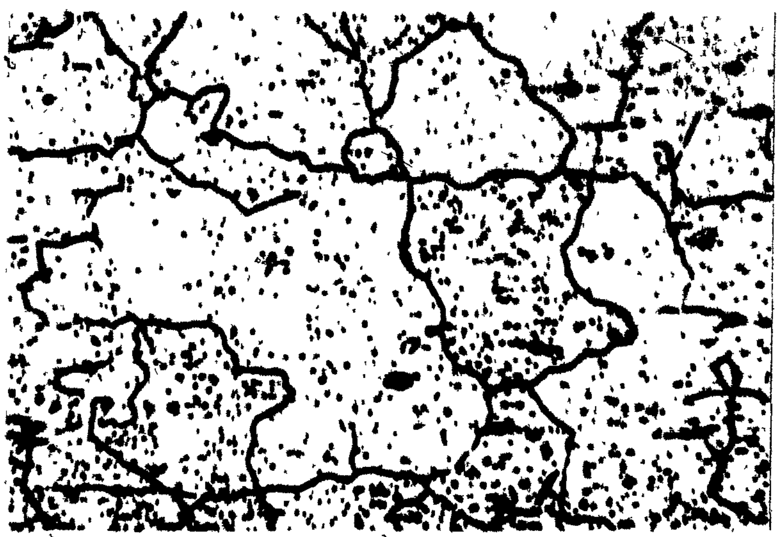
UE = 28.5%

TE = 37.8%

$\bar{\ell}$ = 0.096 mm

ASTM No. = 3.35 ± 0.183 (95% C.L.)

FIGURE 4.30 Pre-strained 12%.
Ann. temperature: 715°C
100 X



GS-II

YS = 15.0×10^3 Psi

UTS = 35.0×10^3 Psi

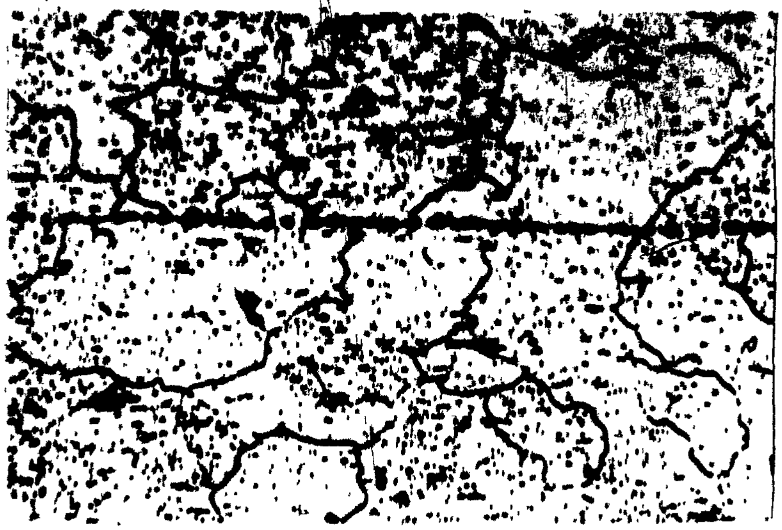
UE = 31.1%

TE = 37.9%

$\bar{\ell}$ = 0.096 mm

ASTM No. = 3.45 ± 0.426 (95% C.L.)

FIGURE 4.31 Pre-strained 12%.
Ann. temperature: 715°C.
100 X



GS-I

YS = 16.2×10^3 Psi

UTS = 36.3×10^3 Psi

UE = 26.3%

TE = 36.5%

$\bar{\ell}$ = 0.135 mm

ASTM No. = 2.7 ± 0.67 (95% C.L.)

FIGURE 4.32 Pre-strained 10%.
Ann. temperature: 705°C.
100 X

GS-II

YS = 19.2×10^3 Psi

UTS = 36.7×10^3 Psi

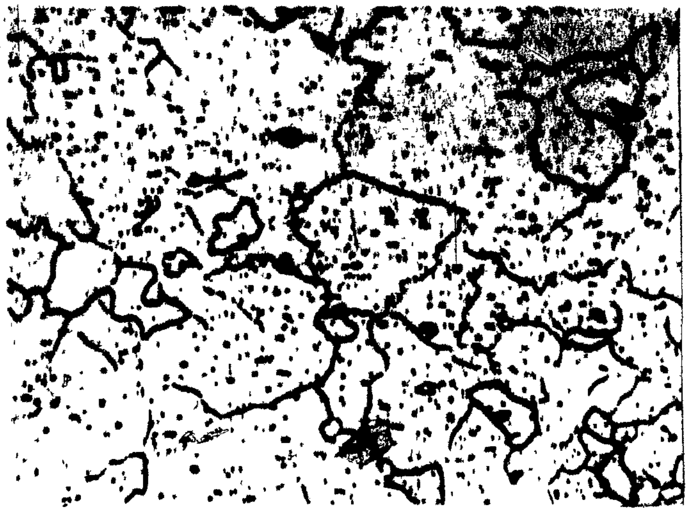
UE = 32.0%

TE = 40.0%

$\bar{\ell}$ = 0.096 mm

ASTM No. = 3.48 ± 0.5 (95% C.L.)

FIGURE 4.33 Pre-strained 16%.
Ann. temperature: 705°C.
100 X



GS-I

YS = 19.7×10^3 Psi

UTS = 38.4×10^3 Psi

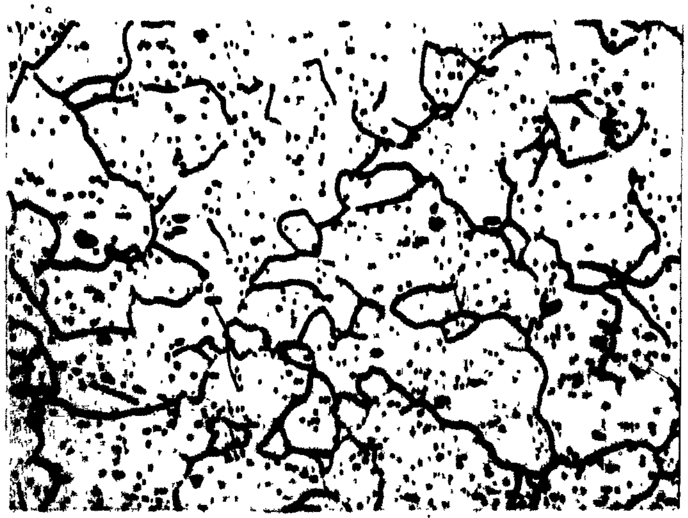
UE = 28.9%

TE = 38.5%

$\bar{\ell}$ = 0.081 mm

ASTM No. = 3.95 ± 0.468 (95% C.L.)

FIGURE 4.34 Pre-strained 16%.
Ann. temperature: 675°C.
100 X



GS-II

YS = 20.1×10^3 Psi

UTS = 36.8×10^3 Psi

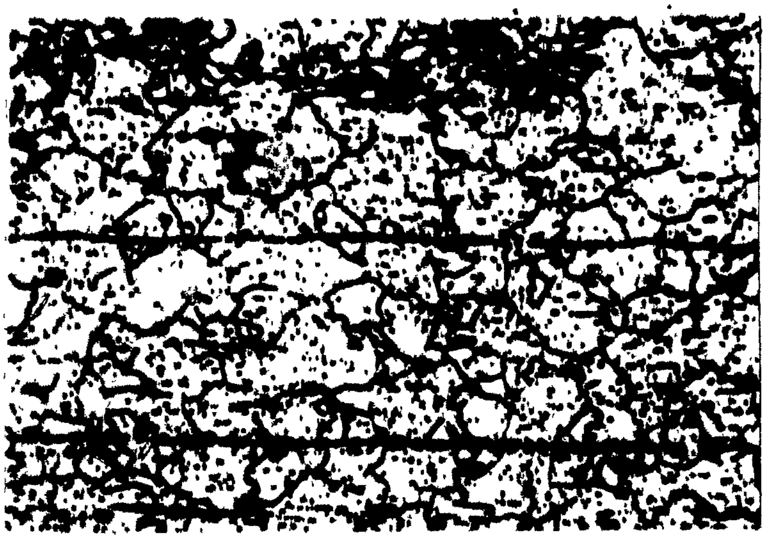
UE = 29.2%

TE = 38.0%

$\bar{\ell}$ = 0.072 mm

ASTM No. = 4.28 ± 0.19 (95% C.L.)

FIGURE 4.35 Pre-strained 18%.
Ann. temperature: 675°C.
100 X



GS-I

YS = 25.7×10^3 Psi

UTS = 39.5×10^3 Psi

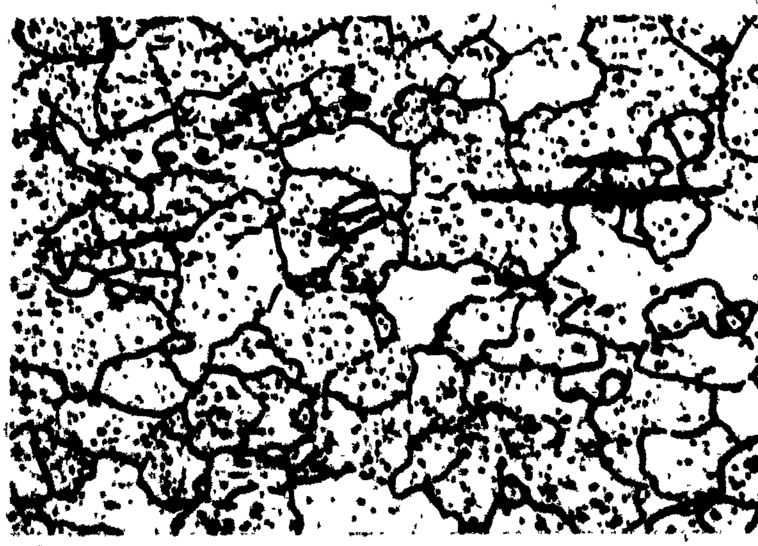
UE = 23.8%

TE = 28.3%

$\overline{\ell}$ = 0.047 mm

ASTM No. = 5.47 ± 0.2
(95% C.L.)

FIGURE 4.36 Pre-strained 20%.
Ann. temperature: 650°C.
100 X



GS-II

YS = 22.8×10^3 Psi

UTS = 38.6×10^3 Psi

UE = 27.8%

TE = 37.5%

$\overline{\ell}$ = 0.053 mm

ASTM No. = 5.25 ± 0.18
(95% C.L.)

FIGURE 4.37 Pre-strained 20%.
Ann. temperature: 650°C.
100 X

mechanical properties listed are yield strength (YS), ultimate tensile strength (UTS), uniform elongation (UE) and total elongation (TE).

There was a striking difference between the grain size of samples following recrystallization I and that obtained after the pre-strain - recrystallization II cycle. Very large grained material was produced by the latter treatment, even at the lowest annealing temperature of 650°C. It was interesting to note that GS-I samples did not fully recrystallize at 650°C (see Figure 4.36) while in material with the larger starting grain size GS-II, recrystallization was complete at this annealing temperature.

4.6 Rolling Mill Deformation Results

Because it was not possible to deform the tensile samples beyond the onset of plastic instability without creating regions of highly distorted material, it was decided to use rolling deformation to produce higher pre-strains.

Some problems were encountered during the rolling experiments. It was found that hand feeding of the material did not provide sufficient back-tension to prevent ripple formation in the cold reduced material. Thus, when the material was tested in tension following the recrystallization anneal, necking occurred preferentially at sites that were clearly related to these ripples. Hence, the tensile test data included with the photomicrographs must be viewed in the light of this limitation.

The overall results of grain size measurement are presented in Figure 4.38 and plotted against tensile pre-strain or, (in the case of deformation by rolling), equivalent tensile pre-strain.* As expected, the grain size continued to decrease with increasing deformation but the rate of decrease tapered off with increasing deformation.

Photomicrographs of the resulting recrystallized grain size are shown in Figures 4.39-43 corresponding to 24.6, 25.9, 28.3, 30.4 and 32.1% cold reduction, respectively (i.e. 21.3, 22.4, 24.5, 26.3 and 27.8% tensile strain). The grain size refinement is easily seen when the figures are viewed in sequence.

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* Equivalent tensile pre-strain was calculated from the rolling reduction by multiplying rolling strain by $\sqrt{3/2}$.(46)

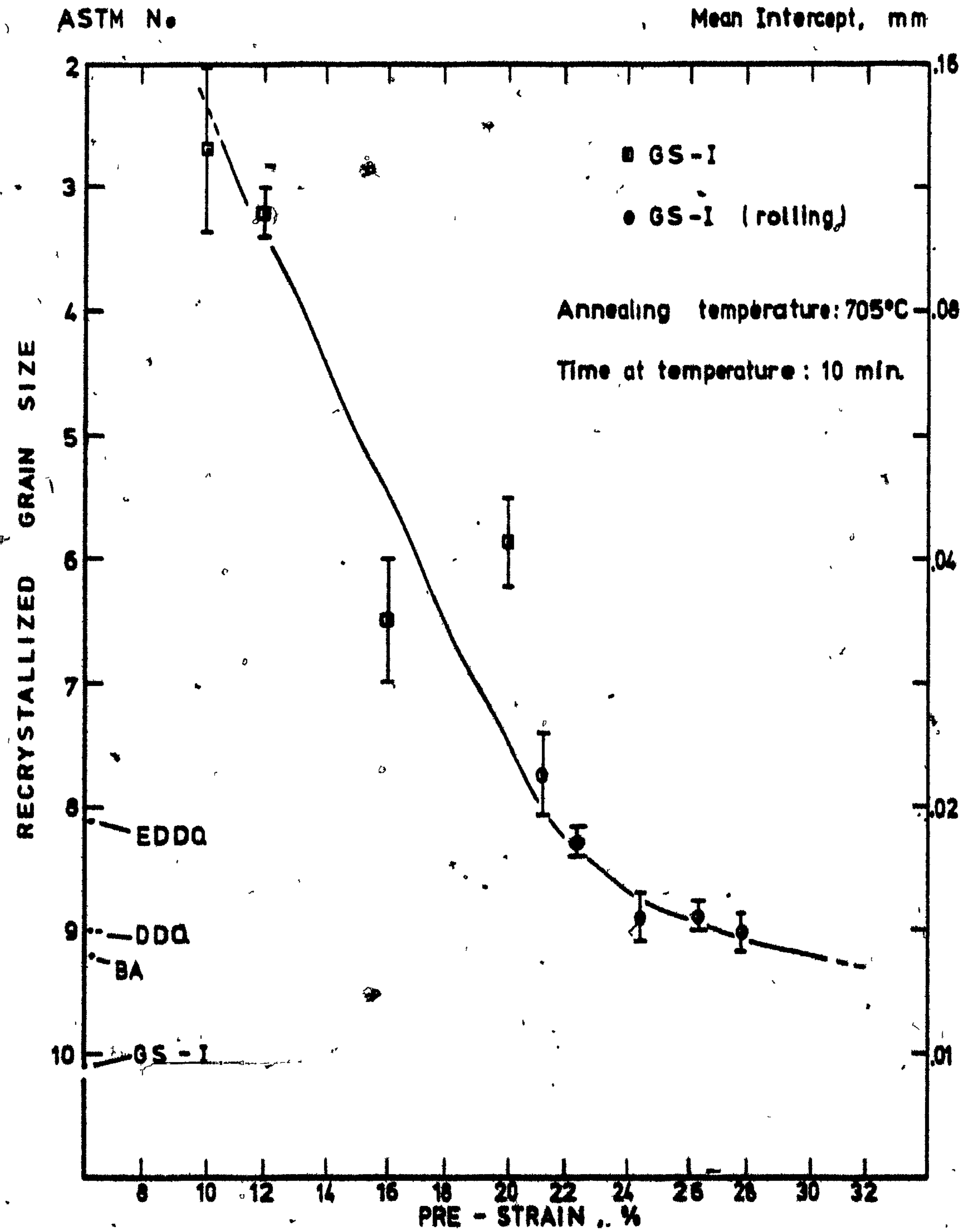


FIGURE 4.38 Relation between the final grain size and pre-strain expressed as tensile strain.

GS-I

YS = 28.4×10^3 Psi

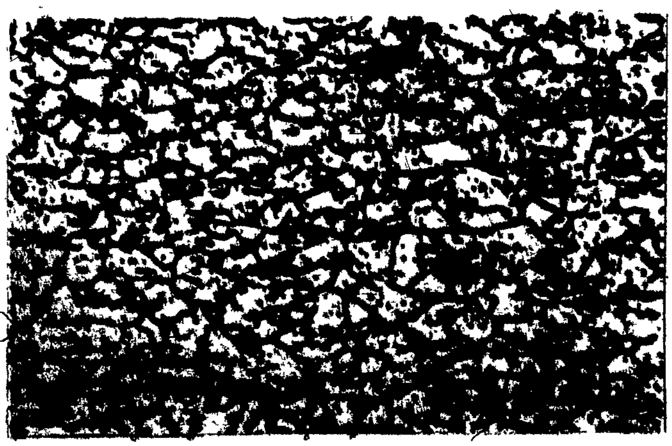
UTS = 42.0×10^3 Psi

TE = 31.6%

$\bar{\ell}$ = 0.022 mm

ASTM No. = 7.75 ± 0.327 (95% C.L.)

FIGURE 4.39 24.6% cold-reduction.
21.3% tensile strain.
Ann. temperature: 705°C.
100 X



GS-I

YS = 27.8×10^3 Psi

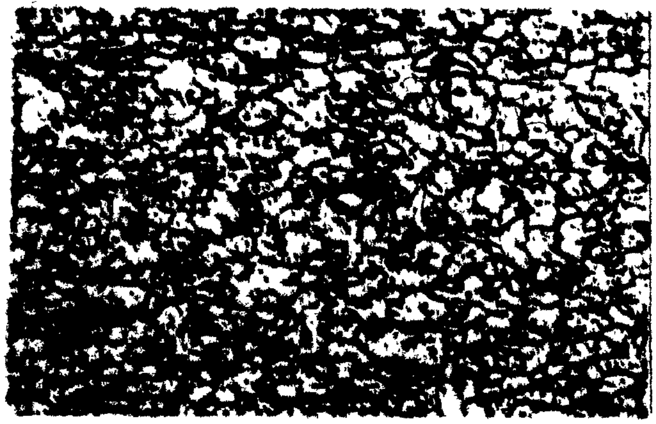
UTS = 42.9×10^3 Psi

TE = 44.4%

$\bar{\ell}$ = 0.018 mm

ASTM No. = 8.3 ± 0.12 (95% C.L.)

FIGURE 4.40 25.9% cold-reduction.
22.4% tensile strain.
Ann. temperature: 705°C.
100 X



GS-I

YS = 29.0×10^3 Psi

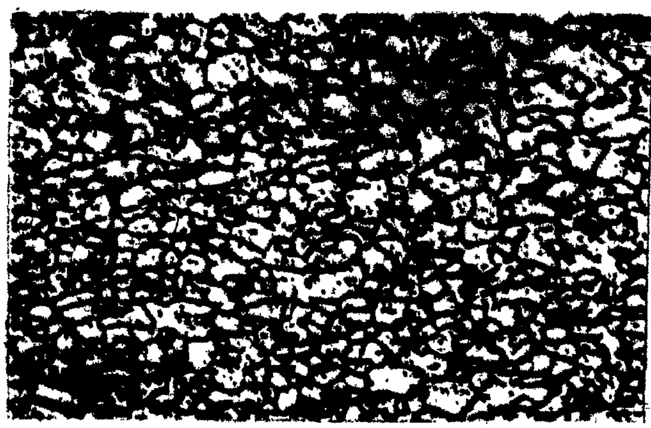
UTS = 42.8×10^3 Psi

TE = 33.0%

$\bar{l}$ = 0.014 mm

ASTM No. = 8.9 ± 0.183 (95% C.L.)

FIGURE 4.41 28.3% cold-reduction.
24.5% tensile strain.
Ann. temperature: 705°C.
100 X



GS-I

YS = 29.62×10^3 Psi

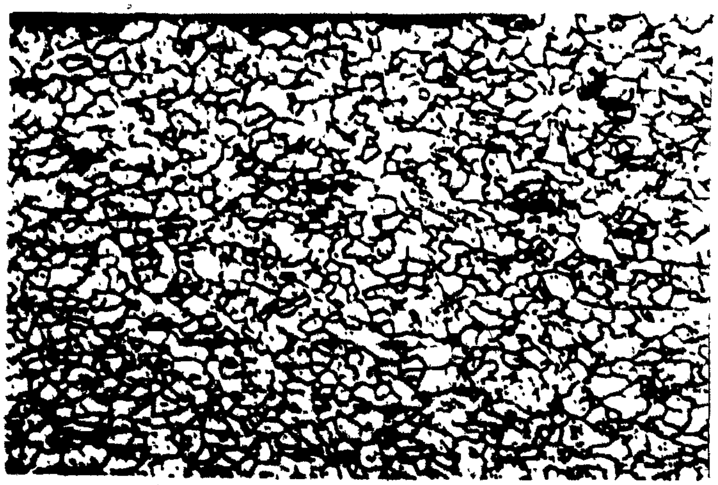
UTS = 42.4×10^3 Psi

TE = 24.6%

$\bar{l}$ = 0.0135 mm

ASTM No. = 8.9 ± 0.12 (95% C.L.)

FIGURE 4.42 30.4% cold-reduction.
26.3% tensile strain.
Ann. temperature: 705°C.
100 X



GS-I

YS = 31.5×10^3 Psi

UTS = 43.1×10^3 Psi

TE = 40.0%

$\bar{\ell}$ = 0.014 mm

ASTM No. = 9.0 ± 0.105 (95% C.L.)

FIGURE 4.43 32.1% cold-reduction.
27.8% tensile strain.
Ann. temperature: 705°C.
100 X

CHAPTER 5

5. DISCUSSION

A discussion of the results outlined in the previous chapters can be approached from two directions. Firstly, the results themselves can be assessed and, secondly, they can be related to the properties of commercially annealed materials so that the applicability of the results to the commercial production of deep drawing quality sheet can be evaluated.

5.1 Discussion of Results

5.1.1 Recrystallization I

The mechanisms by which metals and alloys repair the structural damage caused by mechanical deformation (tension, rolling, torsion, compression, etc.) are thermally activated. Hence, the deformed material has to be heated before the recrystallization can occur. Recrystallization is characterized by the replacement of the deformed grains which are characteristic of the cold-worked state, i.e. high strain energy state, by new equiaxed deformation-free grains with a consequent change in physical and mechanical properties. A population of new deformation-free grains is nucleated and grows at the expense of the deformed structure until it is all consumed. Thereafter, grain boundaries continue to migrate as the recrystallized grains grow, leading to grain growth which is an important aspect of batch annealing (but not of continuous annealing).

The temperature at which recrystallization occurs is known as the recrystallization temperature which may be defined as the temperature at which a particular material with a specific amount of cold deformation will completely recrystallize in a specified period of time. The recrystallization temperature depends upon material characteristics such as the amount of prior deformation, grain size before deformation and purity.

When (60% cold reduced) mild steel (0.07% C) sheet was annealed for 10 minutes in a lead bath a softening related to the recrystallization process occurred at annealing temperatures greater than approximately 600°C; the only difference observed between material annealed at 600°C and 700°C was the final grain size. Thus, a temperature range of 600-723°C was established in which the material would recrystallize, and a temperature approximately 700°C was selected on this basis as a suitable annealing temperature for Recrystallization I treatments.

When the material was isothermally annealed at 700°C in a metal foil container, the softening occurred at between 2 and 4 minutes and after four minutes at 700°C the material was completely recrystallized (Figure 4.3). An increase in annealing times beyond four minutes was reflected in an increase in the final grain size and hence times of 30 minutes and 24 hours at temperature 705°C were selected for the annealing of the cold-rolled mild steel to produce the starting material with recrystallized grain sizes GS-I and GS-II, respectively.

5.1.2 Pre-Strain - Recrystallization II

It should be emphasized that "pre-strain" refers only to the deformation, produced either by tension or rolling, of the sheet between

Recrystallization I and Recrystallization II annealing treatments. The main aim of this investigation was to utilize this intermediate deformation to control grain size, i.e. to produce a specific final grain size on recrystallization annealing (recrystallization II).

It is known that the rate of recrystallization is determined by the rate at which nuclei form, N, and the rate at which they grow, G, and that it is greater the higher the annealing temperature, and the greater the degree of prior deformation.[47,48] The rates of nucleation and growth also influence the grain size of a recrystallized material.[49] Both N and G increase with temperature at all degrees of deformation and they also increase with the degree of deformation at all temperatures. The nature of this variation is of singular importance to this investigation: N increases with deformation more rapidly than G and since the final grain size after the completion of recrystallization varies inversely with the ratio N:G, then the final grain size decreases with increasing deformation. For small deformations a point will be reached where very few nuclei are formed and since the rate of variation of N with deformation is low at small deformation, the probability of one nucleus forming in the material becomes infinitely small. Thus, there will be a minimum deformation at which recrystallization will commence; this deformation yields, as a consequence, large recrystallized grains: this is the critical deformation, also known as the critical amount of cold work. The existence of this relationship allows the control of recrystallized grain size through manipulation of the degree of prior deformation and hence of N and G.

The critical deformation should decrease with increasing annealing times, since increasing time increases the probability of the appearance of a nucleus.[49] Nevertheless, the annealing time in

Recrystallization II treatments was kept constant at 10 minutes since samples that were deformed at small (less than 8%) deformations and annealed for longer times, recrystallized unevenly as nucleation originated in Luder's bands regions resulting in a non-uniform recrystallized grain size; 10 minutes was employed as it was found that for this period of time, recrystallization was uniform in character.

These treated samples, i.e. pre-strained and annealed samples, were then tensile tested to failure and microstructurally examined. Observations on the various mechanical properties are discussed below.

Yield Strength

Yield strength is very significant in assessing formability; a low value is desirable if other properties, such as tensile strength and elongation, are favorable.

It is clear from the yield strength results (Section 4.3.1) that the amount of pre-strain and the annealing temperature strongly influence the yield strength behaviour. Three different regions of behaviour were observed in each yield strength curve. Firstly, small amounts of deformation, i.e. less than the critical strain, did not impart sufficient strain energy to the material to create appropriate regions for nucleus formation and growth, regardless of the annealing temperature, and hence the yield stress increased with increasing deformation as a consequence of work hardening. However, a limiting pre-strain existed beyond which increases in pre-strain increased the strain energy of the material sufficiently to allow the formation of recrystallization nuclei and their growth. At this point the strain was close to the critical strain and only a few nuclei formed resulting in a coarse grained microstructure. Beyond the critical strain, further increases in pre-strain resulted in higher nucleation rates and the resulting microstructure became increasingly fine-grained, with a resultant increase in yield stress.

The dependence of yield stress on grain size is well established (50-52) and is given by the Hall-Petch relationship:

$$\sigma_{ys} = \sigma_o + Kd^{-\frac{1}{2}}$$

where σ_{ys}, is the yield stress; σ_o and K are constants; and d is the average grain diameter. This dependence allows the sudden drop in the yield stress (Figures 4.4 and 4.5) to be related to a dramatic increase in grain size with a small increment of strain, i.e. it permits a distinction between recrystallized and unrecrystallized material.

With increasing temperature, the amount of pre-strain that imparted the sufficient strain energy to the material to allow nuclei formation decreased appreciably. This was observed for both starting grain sizes, but the amount of pre-strain necessary for promoting recrystallization was found to be less for the fine starting grain size (GS-I) than for the coarser starting grain size (GS-II) (see Figure 4.6).

Tensile Strength

Similar behaviour to that observed with respect to yield strength was also followed by the tensile strength.

Uniform Elongation

Elongation is one accepted measure of ductility and therefore, if other properties are favourable, the steel with the highest elongation is the most formable. Uniform elongation can be closely and directly correlated with the amount of deformation that a material can undergo before localized necking occurs, beyond which forming operations become impossible.

As with other mechanical properties, the ductility is dependent upon the grain size of the material.[53] Fine-grained materials have high yield and tensile strengths but low ductilities while very coarse grained materials exhibit a similar lack of ductility, as shown in Figure 5.1.

Reference to Figures 4.9 and 4.10 shows that the uniform elongation curves exhibit a minimum when the critical strain value has just been exceeded, i.e. when the recrystallized grains are at their maximum size. For GS-I starting grain size these minimum uniform elongation values were approximately 14, 21, 12 and 11% for annealing temperatures of 715, 705, 675 and 650°C, respectively. At 610°C a minimum value of 6% was registered at 22% pre-strain. With starting grain size GS-II, these minimum uniform elongation values are generally higher in comparison to those of GS-I.

The increase in uniform elongation beyond the minimum values of critical strain, which was observed at all temperatures except 610°C, and for both starting grain sizes, was attributable to the decrease in the recrystallized grain size as the pre-strains became greater. Uniform elongation values continued increasing with increasing

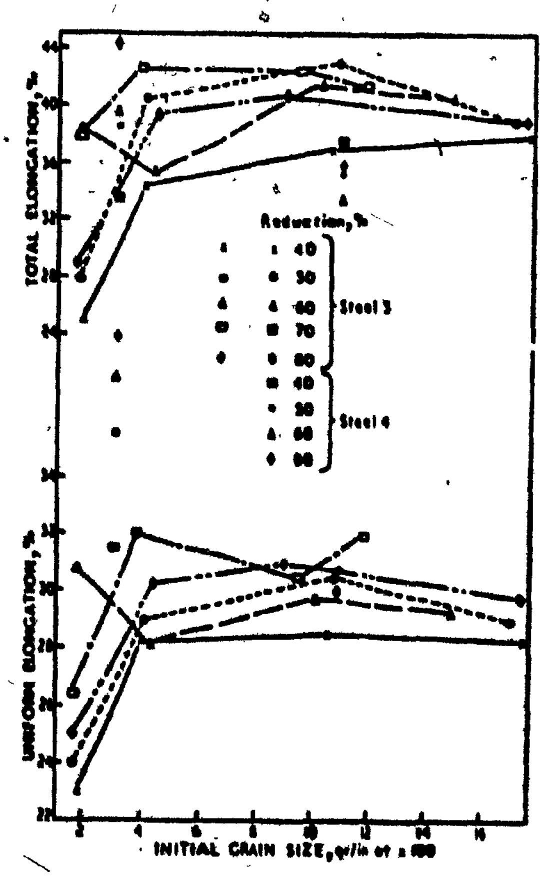


FIGURE 5.1 Effect of grain size on ductility of annealed material.(23)

pre-strain up to approximately the value found in material not pre-strained at all. When this value is reached, uniform elongation either remained constant or exhibited a smooth decrease.

One characteristic of the experimental method may have had a significant influence on the values reported for total and uniform elongation. Recrystallized tensile specimens, all of the same original thickness, were pre-strained, annealed and tested in tension; samples that had been strained to a greater degree were consequently thinner than those pre-strained at lower levels. This difference in thickness undoubtedly affected the uniform and total elongation results: elongation decreases with decreasing sheet thickness, the elongation decrease becoming more evident as the thickness becomes smaller as shown in Figure 5.2. The starting sheet thickness in the present work was 0.035 in.,

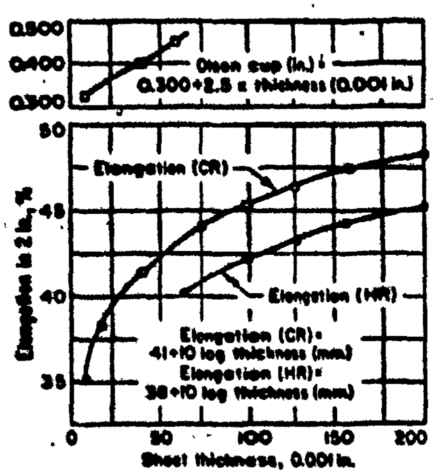


FIGURE 5.2 Effect of thickness on elongation values.[53]

which places the thickness range of the reduced material in the most steeply sloping portion of the elongation versus sheet thickness curve (Figure 5.2). But the effect is not large as even the maximum thickness reductions used in the experimental work, i.e. approximately 30%, will result in a decrease in ductility of less than 5%.

Yield-Tensile Ratio

The yield-tensile ratio is a common parameter for appraising the formability of sheet steel. The lower the value of this ratio the more suitable is the steel for severe forming operations, i.e. there is a large stress range in which the plastic deformation of the forming operations can be carried out. The yield-tensile ratio values determined for all recrystallized sheets in the present work lay in the range 0.43-0.68 for both starting grain sizes, i.e. they are comparable with those of batch annealed sheet.

Grain Size Variation

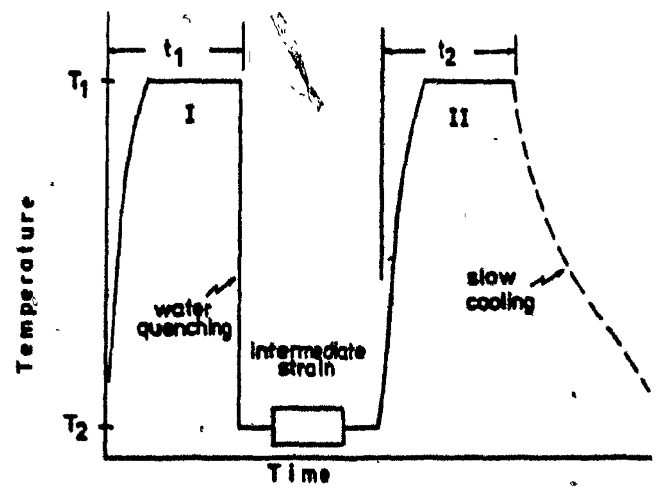
Previous investigators[54] have shown that the recrystallized grain size depends upon the amount of deformation given to the material before annealing. Deformation slightly greater than the critical strain introduces only a relatively low level of strain into the material which creates few sites at which a nucleus can form and grow. This results in a large grain size when the material is annealed. Increasing deformation allows progressively more nuclei to form resulting in smaller grain sizes. The grain size of the material prior to deformation also influences the grain size after annealing and the coarser the starting grain size, the coarser the recrystallized grain size.[25,54]

In the deformation range investigated (0-24%) the results agreed well with this theory. When the pre-strain was only slightly greater than the critical strain, the recrystallized grain size was very large; increasing pre-strain resulted in a steady decrease in recrystallized grain size. The results also indicate that at 715°C the effect of prior grain size had negligible effect on the annealed grain size but the influence of starting grain size became measurable at lower temperatures: a coarser starting grain size resulted in a coarser recrystallized grain size at constant deformation, temperature and annealing time. For the coarse starting grain size, GS-II, recrystallization did not take place at the lowest annealing temperature, 610°C, even with large deformations.

5.2 Evaluation of Results

Continuous annealing has been highly successful for the production of tinplate stock but if it is to become an accepted method for the production of drawing quality sheet, methods will have to be found to ensure that continuously annealed sheet develops a proper grain size and is not susceptible to aging. The two Japanese processes already in use, i.e. CAPL and NKK ensure sufficient grain growth during continuous annealing through close control of composition and prior history of the material while aging is minimized by incorporating an overaging section into the annealing line. By these methods it has been reported[33,43] that steel with comparable properties to batch annealed material can be produced.

However, the present experimental programme has concerned itself with heat treating a mild steel from a normal cold-rolling production run, i.e. with no close control on chemical composition (see strain-heat cycle illustrated in Figure 5.3). An initial recrystallization treatment is followed by deformation at room temperature and then a second recrystallization treatment. According to the results of this investigation the initial recrystallized grain size should be as fine as possible to ensure the amount of pre-strain is minimal. Thus the first recrystallization treatment can be performed at the maximum strip speed compatible with obtaining a fully recrystallized material and cooling rates can be high. The recrystallization temperature in the second heat treatment should also be as high as possible, but less than 723°C, to minimize furnace size. The influence of cooling rate from the final recrystallization treatment on sheet mechanical properties



T_1 : Annealing temperature (650°C-705°C)
T_2 : Intermediate strain temperature (room temp.)

t_1 : Heating and soaking time-part I-
t_2 : Heating and soaking time-part II-

FIGURE 5.3 Strain-anneal cycle proposed.

was not examined but it is clear that either slow cooling or cooling with an incorporated overaging step as in CAPL and NKK processes would be necessary to allow carbide precipitation and thus to ensure the most desirable sheet properties.

The amount of pre-strain is the critical factor determining the final recrystallized grain size and it could be altered to suit the specification of the end product, i.e. relatively fine-grained steel could be produced using a large pre-strain (say 21-28%) while the coarse-grained steels necessary for deep drawing could be pre-strained by a smaller amount.

Table VIII enables a comparison to be made between the properties of low-carbon sheet steel annealed by this technique and those annealed by other methods. It is clear that the strain-anneal method compares very favourably with other production methods and it would also appear to have some advantages over the other processes: (1) close chemical control is not essential to obtaining a specified annealed grain size, and (2) a wide range of annealed grain sizes, and consequently a wide range of mechanical properties, can be produced from the same material solely by varying the amount of pre-strain.

The suggested process does have some drawbacks, noticeably the increment in cost over conventional continuous annealing necessitated by the intermediate deformation and second anneal steps but these may be offset by the increased throughput of the large tonnages of sheet destined for forming operations. Additionally, a close control would have to be kept on the amount of intermediate deformation and in order to ensure a uniform product it might need to be adjusted to accommodate variations in the initial recrystallized grain size. However, it was found that pre-strains in the range 21-28% gave final recrystallized grain sizes comparable with those obtained from batch annealing and the experimental results (see Figure 4.38) showed that control in this region would be less critical than would be necessary with lower pre-strains.

TABLE VIII

Comparison of Mechanical Properties of Material Annealed by Different Methods

Mechanical Properties	Batch Anneal	Continuous Anneal		Present** Investigation
		CAPL*	NKK*	
Yield strength (Psi)	28.2×10^3 max	27.9×10^3	31.3×10^3	27.8×10^3
Tensile strength (Psi)	46.9×10^3	46.6×10^3	46.8×10^3	42.9×10^3
Total elongation, in 2 in. (%)	44	44.2	45	44.4
Yield-tensile ratio	0.60	0.60	0.67	0.64
Grain size (mm)	-	-	0.019	0.018

* Tested with JIS specimens.

** Tested with 8 in. long, 0.6 in. wide strip specimens. 26% cold-reduced.

CHAPTER 6

6. CONCLUSIONS

For the 1008 steel used in this work, and within the limits of pre-strain and annealing times and temperatures employed, the following conclusions can be drawn from the present work:

1. There was a substantial difference in response to final annealing of material with different starting grain sizes, particularly as evidenced by the mechanical properties. In particular, for a given annealing temperature, the amount of pre-strain at which the yield and tensile strengths sharply decreased was smaller the finer the initial grain size.
2. Annealing temperature had a strong influence on critical strain which decreased as the annealing temperature increased.
3. Pre-strains at or slightly higher than the critical strain resulted in a very coarse annealed grain size, which decreased rapidly as the amount of pre-strain increased. Uniform and total elongation values had their lowest values when the grain size was at its maximum.
4. The yield-to-tensile ratio for strain-annealed recrystallized material was always in the range 0.43-0.68 for both starting grain sizes investigated and was lowest near the critical strain. The ratio increased as the annealing temperature decreased.

5. By ensuring an appropriate annealed grain size it was found possible to produce sheet steel conforming to forming operation specifications by using strain-annealing.
6. The amount of pre-strain which had to be applied to the material to produce an appropriate annealed grain size was in the 21-28% range.
7. In the proposed strain-anneal cycle it was not necessary to have close control of chemical composition to ensure the development of the desired annealed grain size.
8. By controlling the amount of pre-strain, it was possible to produce a wide range of final grain sizes and consequently, a wide range of mechanical properties, without any change in annealing times and temperatures.

CHAPTER 7

7. SUGGESTIONS FOR FURTHER WORK

The usefulness of the strain-annealing method for the continuous production of drawing quality sheet is clear. Nevertheless, it is felt that more conclusive results could be obtained and the method further investigated using the following suggestions.

1. Forming operations using metal sheet require the sheet to stretch plastically and uniformly to take a desired shape and thus a measure of anisotropy of the material is of great importance. Thus, both the planar and normal anisotropies of strain-annealed low-carbon sheet steel should be investigated by using (for example) the Olsen cup test and evaluating the strain ratio value "r" (Lankford coefficient).
2. Further strain-anneal experiments should be carried out on other grades of steel which are normally batch annealed. Additional experiments should also be designed to eliminate one important source of error inherent in the present work (Section 5.1) which was due to all starting material being of the same thickness and in the size range where small changes in thickness exerted a large influence on measured ductility number.
3. The effect of heating rate modifying the final recrystallization treatment should be investigated, since it is possible that this parameter could have an effect upon the amount of pre-strain necessary to develop a suitably annealed grain size.

APPENDIX A

APPENDIX A

AGING OF THE ANNEALED STEEL

Introduction

Aging (quench aging or age hardening) in plain carbon steels is the result of precipitation of the interstitial solute carbon and nitrogen from supersaturation in ferrite. Aging is readily observed through the change in mechanical properties and occurs in steels if the cooling rate from the annealing temperature has been sufficiently fast so that equilibrium carbide precipitation has not occurred.

Experimental Procedure

It was found that a steel envelope and its enclosed specimens required approximately 20-30 minutes to cool to room temperature from 700-705^{o}C, i.e. the cooling rate was approximately 23-35^{o}C/min.

In order to ascertain whether this cooling rate was sufficiently slow to allow equilibrium carbide precipitation, tensile tests (see Section 3.5.2) were performed on annealed samples which had been held at room temperature ($\sim$ 24^{o}C) for varying periods of time up to a maximum of 30 days. In addition, one batch of samples was placed in a freezer (- 10^{o}C) after annealing and these samples were also tested after 30 days.

Experimental Results

The aging results are shown in the form of graphs. Figure A.1 shows the variation of the strength properties (i.e yield and tensile

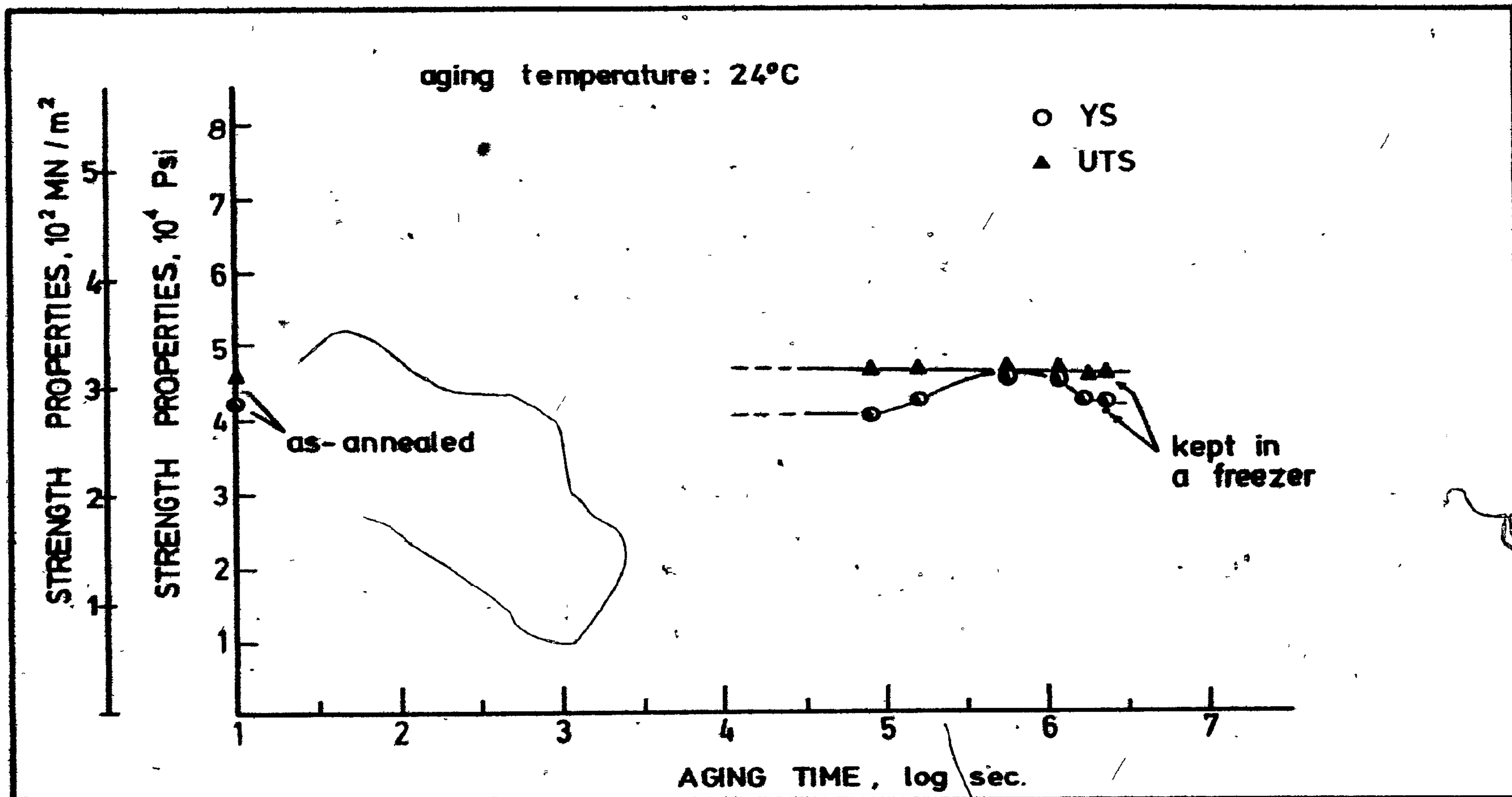


FIGURE A.1 Strength properties variation of samples aged at 24°C for the shown times. Approximate cooling rate: 35°C/min. Heat treatment: heating to 700°C; heating and holding time: 30 min.; air cooling.

strength) with respect to aging time at room temperature of samples annealed at 700°C for a period of time of 30 minutes followed by air cooling (at a cooling rate of 35°C/min.). The values plotted for zero aging time correspond to as-annealed samples. From this figure it may be seen that the yield strength increased after 24 hours aging reaching a maxima after one week then decreasing to the value of the as-annealed material after approximately one month aging. The yield strength maxima represented an increment of about $4x10^3$ psi. The tensile strength did not appear to vary with aging time. Figure A.2 shows the variation in the total elongation and as expected it decreased slightly as the aging time increased to reach a minimum of 42%, i.e. a decrease of about 8% then increased to a value approximately equal to that of the as-annealed material.

The results obtained from the annealed and air cooled samples and kept in a freezer (∿ - 10°C) for periods of time of 30 days showed no significant difference from those results obtained from the samples aged for the same period of time at 24°C.

Conclusions

From these experiment results the following conclusion could be drawn: the cooling rate of 35°C/min., i.e. air-cooling from 700°C was observed to be sufficiently slow to permit most of the carbon to precipitate as only minor aging effects were noted.

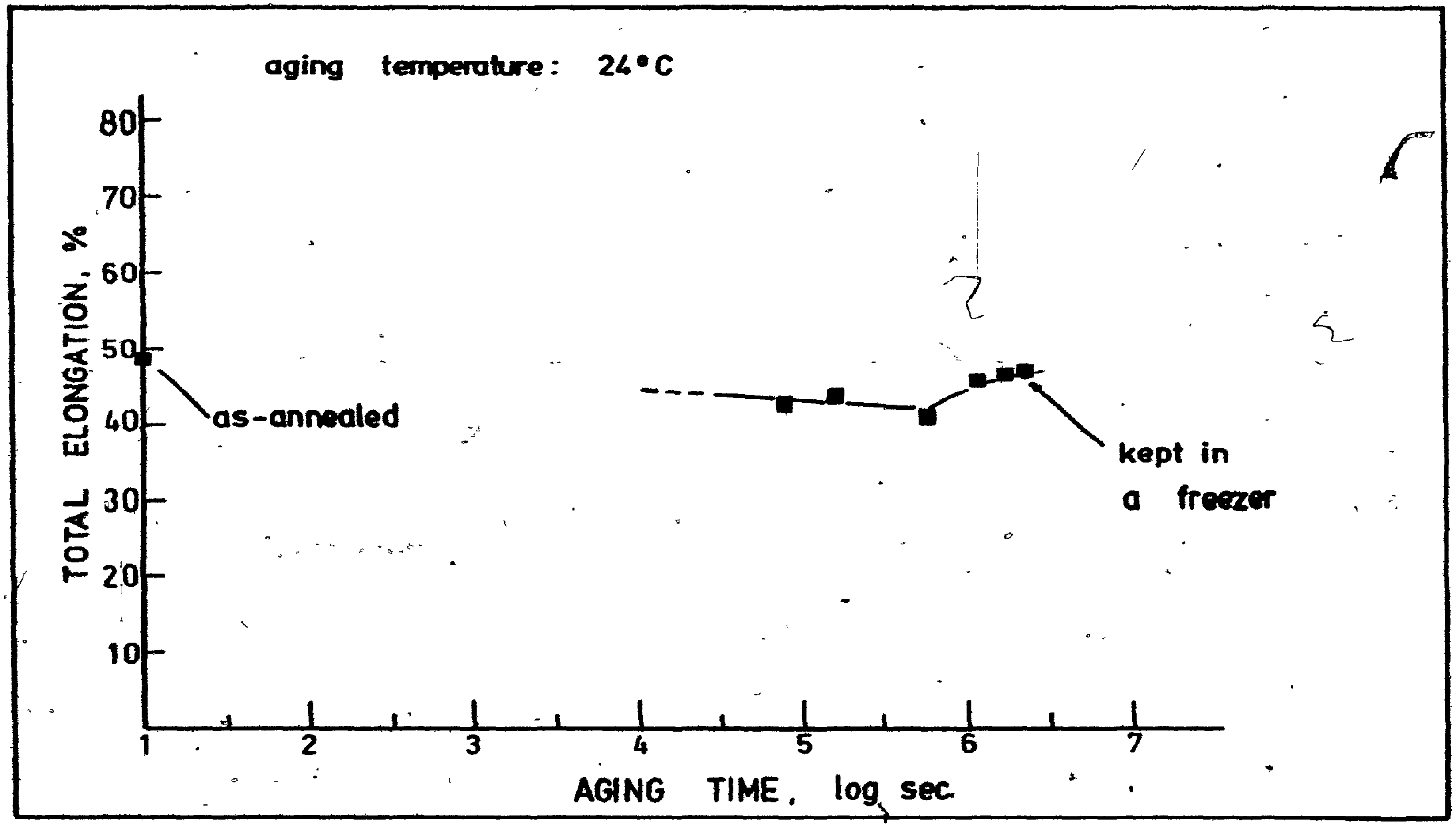


FIGURE A.2 Total elongation variation of samples aged at 24°C for the times shown. Approximate cooling rate: 35°C/min. Heat treatment: heating to 700°C; heating and holding times: 30 min.; air cooling.

APPENDIX B

TABLE B-I

Yield Strength of Pre-Strained and Annealed Material

Starting grain size: GS-I
ASTM No.: 10.1 ± 0.21

Pre-Strain (%)	YIELD STRENGTH$_{0.2}$ (Psi)				
	Annealing Temperature (^{o}C)				
	715	705	675	650	610
1	-	41,186	44,737	44,768	-
2	40,693	43,945	47,660	49,134	43,571
4	45,877	43,401	45,113	42,949	45,029
6	44,392	50,379	-	-	47,990
8	17,076	15,994	-	-	-
10	16,387	16,166	50,522	50,639	53,026
12	18,179	17,533	22,439	55,014	-
14	-	-	-	44,934	57,871
16	21,841	21,704	19,651	23,621	58,313
18	-	-	-	-	57,935
20	23,539	25,161	26,945	25,714	36,083
22	-	-	-	-	47,391

TABLE B-II

Yield Strength of Pre-Strained and Annealed Material

Starting grain size: GS-II
ASTM No.: 8.28 ± 0.215

Pre-Strain (%)	YIELD STRENGTH$_{0.2}$ (Psi)				
	Annealing Temperature (^{o}C)				
	715	705	675	650	610
1	-	-	-	-	-
2	41,593	38,916	45,262	35,268	38,324
4	44,724	-	-	-	-
6	45,285	44,414	45,077	42,980	45,410
8	42,366	44,993	-	-	-
10	16,484	42,078	48,942	47,233	46,568
12	14,948	22,130	47,051	47,914	-
14	-	17,251	29,649	43,048	49,213
16	19,671	19,176	18,584	39,172	52,036
18	-	-	20,127	22,441	51,670
20	23,657	22,003	21,457	22,814	53,572
22	-	-	23,712	23,587	51,860
24	-	24,569	-	-	50,344
26	-	-	-	-	-

TABLE B-III

Ultimate Tensile Strength of Pre-Strained and Annealed Material

Starting grain size: GS-I
ASTM No.: 10.1 ± 0.21

Pre-Strain (%)	TENSILE STRENGTH (Psi)				
	Annealing Temperature (^{o}C)				
	715	705	675	650	610
1	-	45,491	45,595	44,597	-
2	44,545	46,485	45,497	45,628	46,377
4	46,543	45,806	47,038	45,992	47,210
6	46,908	49,097	-	-	49,670
8	36,227	36,132	-	-	-
10	35,043	36,278	51,146	52,304	53,220
12	37,347	37,797	40,701	52,974	-
14	-	-	-	52,164	55,631
16	37,856	38,669	38,441	39,248	56,631
18	-	-	-	-	58,422
20	37,855	38,548	39,285	39,465	47,021
22	-	-	-	-	52,128
24	-	-	-	-	-

TABLE B-IV

Ultimate Tensile Strength of Pre-Strained and Annealed Material

Starting grain size: GS-II
ASTM No.: 8.28 ± 0.215

Pre-Strain (%)	TENSILE STRENGTH (Psi)				
	Annealing Temperature (^{o}C)				
	715	705	675	650	610
1	-	-	-	-	-
2	44,045	43,488	43,731	42,082	43,141
4	44,993	-	-	-	-
6	46,152	46,699	46,301	45,845	46,518
8	46,237	46,289	-	-	-
10	35,874	46,132	49,752	47,656	48,580
12	35,005	38,555	48,150	48,693	-
14	-	36,007	43,306	48,537	51,027
16	36,926	36,696	36,402	47,440	51,909
18	-	-	36,768	37,403	52,474
20	37,674	36,929	36,903	38,553	54,107
22	-	-	37,172	39,424	52,586
24	-	37,683	-	-	52,815

TABLE B-V

Uniform Elongation of Pre-Strained and Annealed Material

Starting grain size: GS-I
ASTM No.: 10.1 ± 0.21

Pre-Strain (%)	UNIFORM ELONGATION (%)				
	Annealing Temperature (^{o}C)				
	715	705	675	650	610
1	-	27.0	27.2	27.0	-
2	26.4	27.4	27.5	26.5	26.4
4	23.0	26.6	24.3	23.25	25.1
6	26.0	21.8	-	-	22.4
8	14.5	21.0	-	-	-
10	24.3	26.3	16.2	14.3	8.0
12	28.5	26.25	12.2	13.8	-
14	-	-	-	7.5	13.8
16	27.3	27.0	28.9	17.8	12.7
18	-	-	-	-	11.7
20	24.8	27.0	27.7	23.8	7.7
22	-	-	-	-	6.1
24	-	-	-	-	-

TABLE B-VI

Uniform Elongation of Pre-Strained and Annealed Material

Starting grain size: GS-II
ASTM No.: 8.28 ± 0.215

Pre-Strain (%)	UNIFORM ELONGATION (%)				
	Annealing Temperature (°C)				
	715	705	675	650	610
1	-	-	-	-	-
2	30.1	28.7	25.6	26.4	28.1
4	28.9	-	-	-	-
6	26.2	21.1	24.2	25.5	20.0
8	16.2	18.2	-	-	-
10	21.1	17.2	19.1	19.6	17.9
12	31.1	17.9	18.2	18.1	-
14	-	29.7	11.3	14.5	16.4
16	31.0	32.0	29.2	8.2	15.25
18	-	-	29.2	20.5	14.2
20	30.3	28.4	29.0	27.8	12.7
22	-	-	27.5	29.5	12.4
24	-	25.4	-	-	10.2

TABLE B-VII

Yield-Tensile Ratio of Pre-Strained and Annealed Material

Starting grain size: GS-I
ASTM No.: 10.1 ± 0.21

Pre-Strain (%)	YIELD-TENSILE RATIO (YTR); (YS/UTS)*				
	Annealing Temperature (^{o}C)				
	715	705	675	650	610
1	-	0.905	0.982	1.004	-
2	0.914	0.945	1.047	1.077	0.939
4	0.986	0.948	0.959	0.934	0.953
6	0.946	1.026	-	-	0.966
8	0.471	0.443	-	-	-
10	0.467	0.445	0.988	0.968	0.966
12	0.487	0.464	0.552	1.0385	-
14	-	-	-	0.861	1.040
16	0.577	0.5613	0.511	0.602	1.029
18	-	-	-	-	0.991
20	0.622	0.653	0.686	0.652	0.768
22	-	-	-	-	0.903
24	-	-	-	-	-

* YS/UTS = yield strength/ultimate tensile strength.

TABLE B-VIII

Yield-Tensile Ratio of Pre-Strained and Annealed Material

Starting grain size: GS-II
ASTM No.: 8.28 ± 0.215

Pre-Strain (%)	YIELD-TENSILE RATIO (YTR); (YS/UTS)*				
	Annealing Temperature (°C)				
	715	705	675	650	610
1	-	-	-	-	-
2	0.944	0.895	1.035	0.838	0.888
4	0.994	-	-	-	-
6	0.981	0.951	0.974	0.937	0.976
8	0.916	0.972	-	-	-
10	0.459	0.910	0.984	0.991	0.959
12	0.427	0.568	0.977	0.984	-
14	-	0.479	0.685	0.886	0.964
16	0.532	0.523	0.511	0.829	1.002
18	-	-	0.547	0.600	0.985
20	0.628	0.596	0.581	0.592	0.990
22	-	-	0.634	0.599	0.986
24	-	0.652	-	-	0.953

* YS/UTS — yield strength/ultimate tensile strength.

TABLE B-IX

Total Elongation of Pre-Strained and Annealed Material

Starting grain size: GS-I
ASTM No.: 10.1 ± 0.21

Pre-Strain (%)	TOTAL ELONGATION (%)				
	Annealing Temperature (°C)				
	715	705	675	650	610
1	-	36.0	36.7	36.0	-
2	33.9	38.3	37.0	36.5	33.7
4	30.2	38.3	35.2	33.0	32.7
6	36.7	29.0	-	-	29.8
8	19.9	28.0	-	-	-
10	32.1	36.5	23.7	21.6	17.5
12	37.8	37.2	17.9	22.0	-
14	-	-	-	10.9	21.6
16	36.6	37.3	38.5	25.0	19.6
18	-	-	-	-	18.1
20	31.8	33.1	35.0	28.3	11.3
22	-	-	-	-	8.72
24	-	-	-	-	-

TABLE B-X

Total Elongation of Pre-Strained and Annealed Material

Starting grain size: GS-II
ASTM No.: 8.28 ± 0.215

Pre-Strain (%)	TOTAL ELONGATION (%)				
	Annealing Temperature (^oC)				
	715	705	675	650	610
1	-	-	-	-	-
2	38.2	38.4	32.7	33.7	35.6
4	37.9	-	-	-	-
6	36.4	32.4	31.9	30.1	32.0
8	18.8	22.7	-	-	-
10	26.5	23.3	25.2	26.7	27.7
12	37.9	24.3	26.8	25.8	-
14	-	40.5	17.7	22.1	23.5
16	41.37	40.0	37.7	10.0	22.7
18	-	-	38.0	28.8	21.0
20	36.65	36.9	38.7	37.5	18.9
22	-	-	35.0	36.0	18.1
24	-	32.5	-	-	15.8

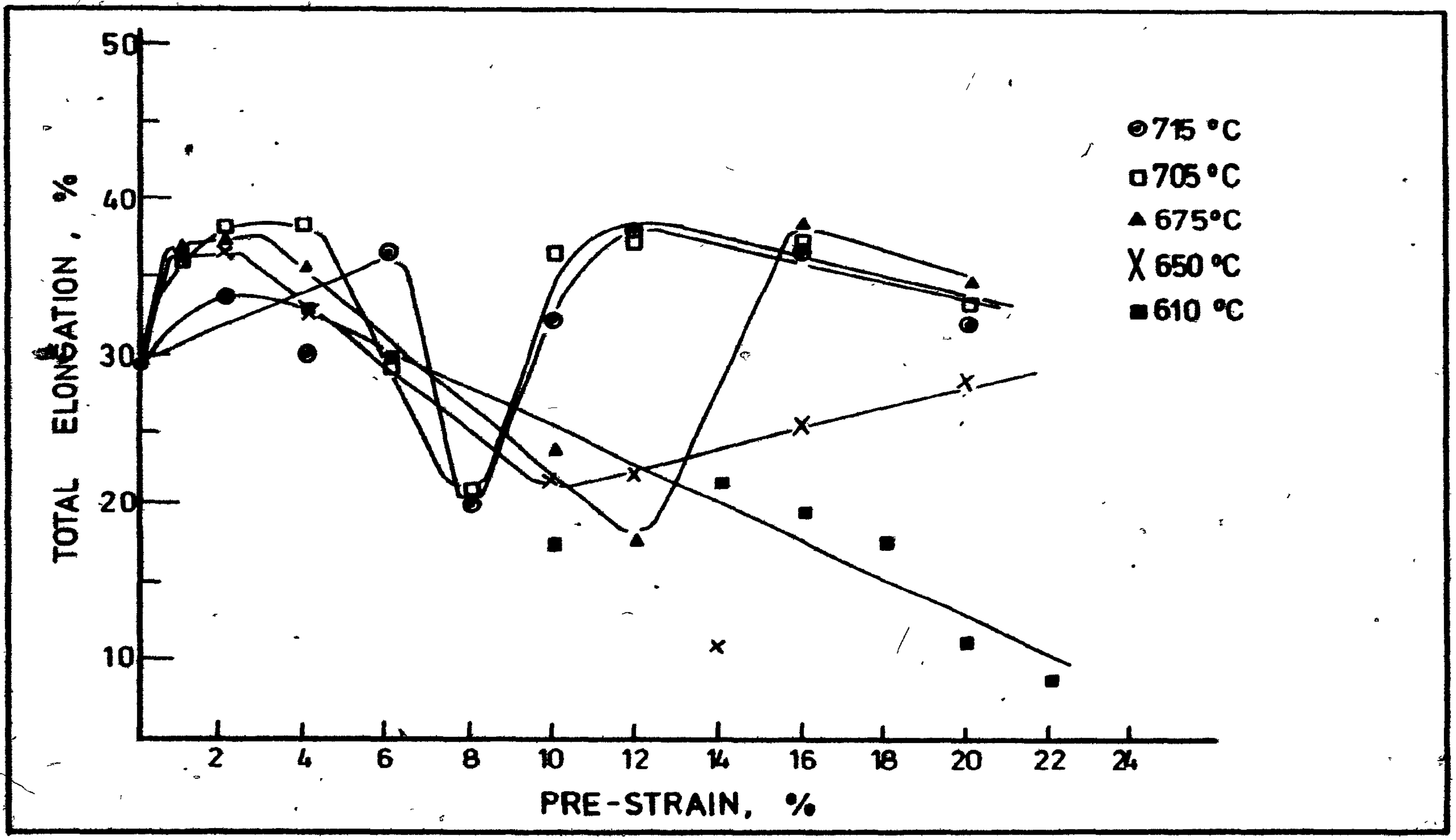


FIGURE B-IV(a) Effect of pre-strain and annealing temperature on total elongation. Starting grain size: GS-I; annealing time: 10 minutes.

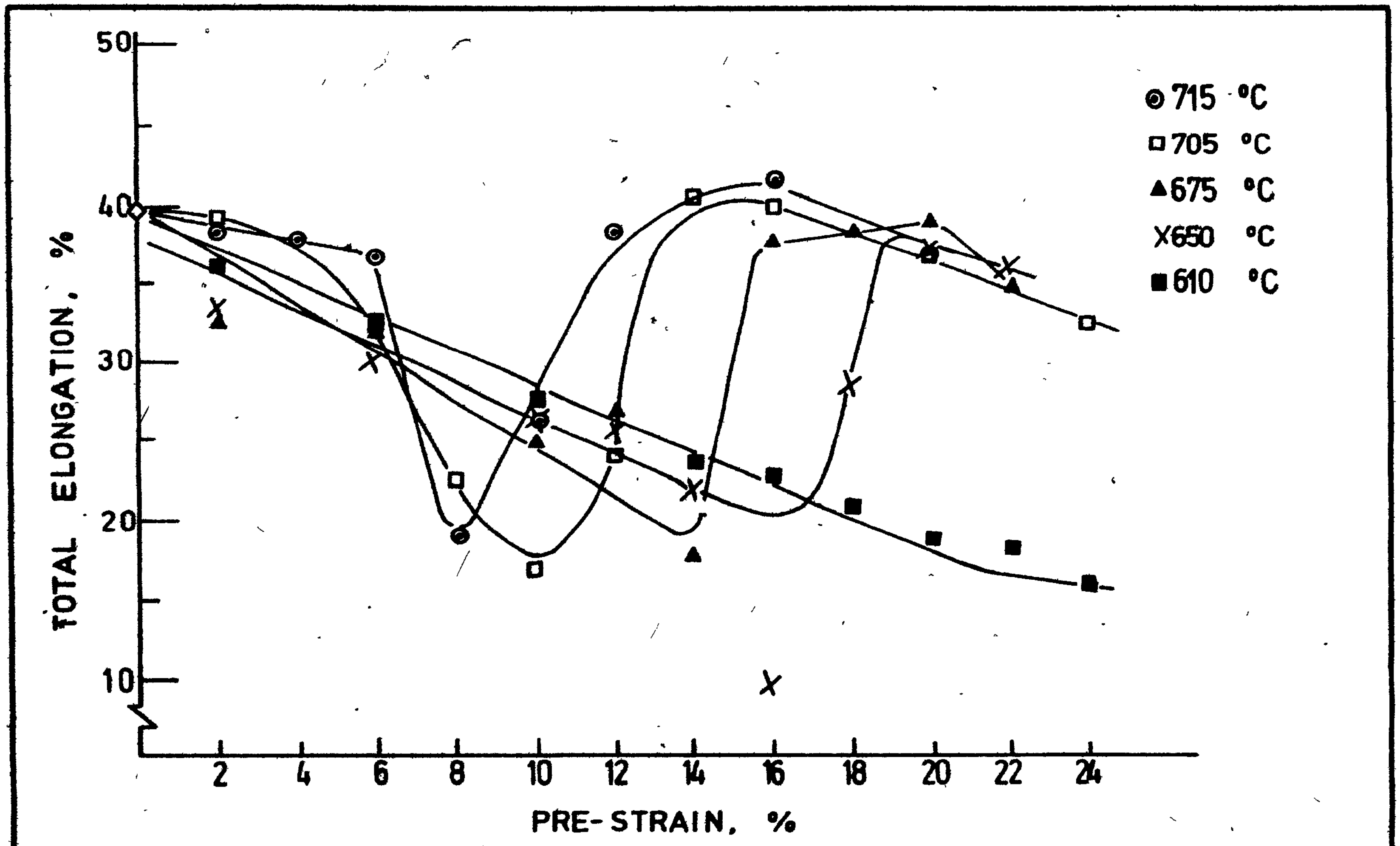


FIGURE B-IV(b) Effect of pre-strain and annealing temperature on total elongation. Starting grain size: GS-II; annealing time: 10 minutes.

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