ABSTRACT

THE PARTIAL ANNEALING OF
LOW-CARBON STEEL STRIP

M.Sc. by Metallurgy

Clifford J. Adams

Tinplate stock that had been cold reduced 82% by rolling was partially annealed at various temperatures to produce the full strength spectrum that could be obtained from a continuous annealing line. The mechanical properties of this material were compared to those of material that had been cold rolled by various amounts to equivalent strengths.

It was found that the ductility of partially annealed material was substantially superior to that of cold-rolled material at virtually all strength levels. This difference in ductility between the two materials at equal strengths has been visually correlated, by a series of photomicrographs, to a difference in microstructure. Microstructural recovery and recrystallization are responsible for the increased ductility of partially annealed material over that cold rolled.

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by

Clifford J. Adams

A thesis submitted to the Faculty of Graduate Studies and Research in Partial fulfilment of the requirements for the degree of Master of Science in Metallurgy.

McGill University

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INTRODUCTION

Manufacture of Steel Sheet

(i) Historical Prologue

In 1870 W. Siemens in Wales, began to manufacture steel sheet as the base plate for tinning instead of the long used wrought iron. From that time to about 1930, the Hot-Pack Process[1] was the major method of producing this steel sheet. It consisted of first cutting a long flat bar into sections of length equal to that of the desired finished plate width. Then each section was reheated, rolled, and doubled over on itself; the process was repeated three times until a pack of eight sheets of nearly finished gauge was produced. The individual sheets were separated, pickled to remove the scale and then cold rolled between highly polished rolls to obtain the smoothest surface possible. This cold rolling was followed by "white annealing", i.e. low-temperature heating which produced only slight oxidation. The sheets were stacked in bundles on a bogie, protected with a cast iron cover and pushed into a large pack furnace. Annealing was followed by "white pickling" using dilute sulphuric acid and then the sheets were ready to be tinned by hot dipping.

During the early 1930's, steel plants began to introduce high-speed, high-capacity hot and cold-rolling mills. These mills produced the steel sheet as coils, often weighing up to 10 tons, a development which rendered hot-pack, hand-fed mills and sheet-pack annealing methods obsolete. New methods for handling and annealing coils had to be found.

Before proceeding further, it may be of some advantage to define annealing as the term applies in the manufacturing of low-carbon steel sheet. Annealing is the process of heating steel sheet to a temperature below the lower critical temperature (723°C), holding and then cooling to room temperature, the main purpose being to restore to the steel the ductility lost during the cold-rolling process. This is achieved by ensuring that the annealing temperature is above that at which recrystallization occurs and that the steel is held at temperature long enough for complete recrystallization. This is termed subcritical or process annealing and is essentially a softening process.

(ii) Batch Annealing

The coils produced by the newly installed high-capacity rolling mills were too numerous to be handled by the old pack annealing furnaces. Instead, the coils were stacked four high, with up to eight stacks on a hearth. An inner steel cover was placed over each stack to hold the protective gases that are introduced to prevent scaling by atmospheric oxidation. Then a portable furnace cover was placed over the entire assembly. A full description of conventional batch annealing can be found in the literature (2,3,4,5).

The biggest disadvantage to batch annealing is its complete inflexibility due to the problem of heating and cooling a large mass of steel. This not only leads to a process time of from 6 to 10 days, but also to a non-uniformity of properties throughout the coil. This is because the outside has to be heated to a higher temperature than the inside in order to reach the required recrystallization temperatures at the center.

To alleviate large in-process tonnages due to the long annealing times, open-coil annealing (6,7,8) has been developed since about 1954. In this process, a tightly wound "closed" coil is rewound prior to annealing into a loose or "open" coil by placing a twisted wire spacer between each wrap near the top edge. After annealing, the coil is again rewound into a normal tight coil and is ready for further processing. The spacer permits the circulation of the atmosphere gases between the laminations during the annealing cycle and thus increases the effective surface area of the coil for heating or cooling by about 1000 times over that of a tight coil. Thus, this process permits more rapid heating and cooling (a 20 hour cycle as opposed to 8 days), more uniform temperature distribution, and exposure of the whole steel surface to an atmosphere of known composition to permit utilization of gas-metal reactions such as decarburizing. However, open-coil annealing is not suitable for the thinner gauges of steel used for tinplate because coiling with the wire spacers between the laps tends to distort the material.

(iii) Continuous Strip Annealing

After the introduction of modern strip mills to replace hot-pack mills, it became apparent that the only part of the process in which there was no continuous flow was that of annealing. The batch annealing process seriously hampered increased production due to the very long heating cycle and large in-process tonnage. In the period 1936-40, a few production continuous annealing furnaces were installed but although successful, they made no

great impact on the canmaking trade. The product was considerably harder and stiffer than the equivalent batch annealed material and was generally looked upon with scepticism by the tinplate industry since it was not compatible with existing equipment for forming can bodies. Development was seriously delayed and it took almost 15 years before the canmakers realized that the high degree of uniformity in the mechanical properties of continuously annealed tinplate had a significant beneficial effect on canmaking speeds, once their automatic forming equipment had been adjusted to process the harder plate. Proven advantages for both producer and consumer have led to the very rapid increase since 1955, in the production and use of continuously annealed tinplate. New annealing lines, recently installed, have strip speeds of 2000 ft/min. to give production rates of 60 tons/hr.

Typical strip annealing lines, as illustrated by Figure 1, have been described in detail elsewhere (9,10,11). Briefly, annealing lines consist of three sections; an entry section, comprising strip handling, welding and cleaning equipment; a vertical strand furnace section; and an exit or delivery section in which the annealed strip is re-coiled. Since the strip must move through the furnace continuously and at a uniform rate, looping towers for strip storage are placed at each end of the furnace section. These loopers allow the entry and delivery sections to be stopped independently in order to add or remove coils without affecting the strip moving through the furnace.

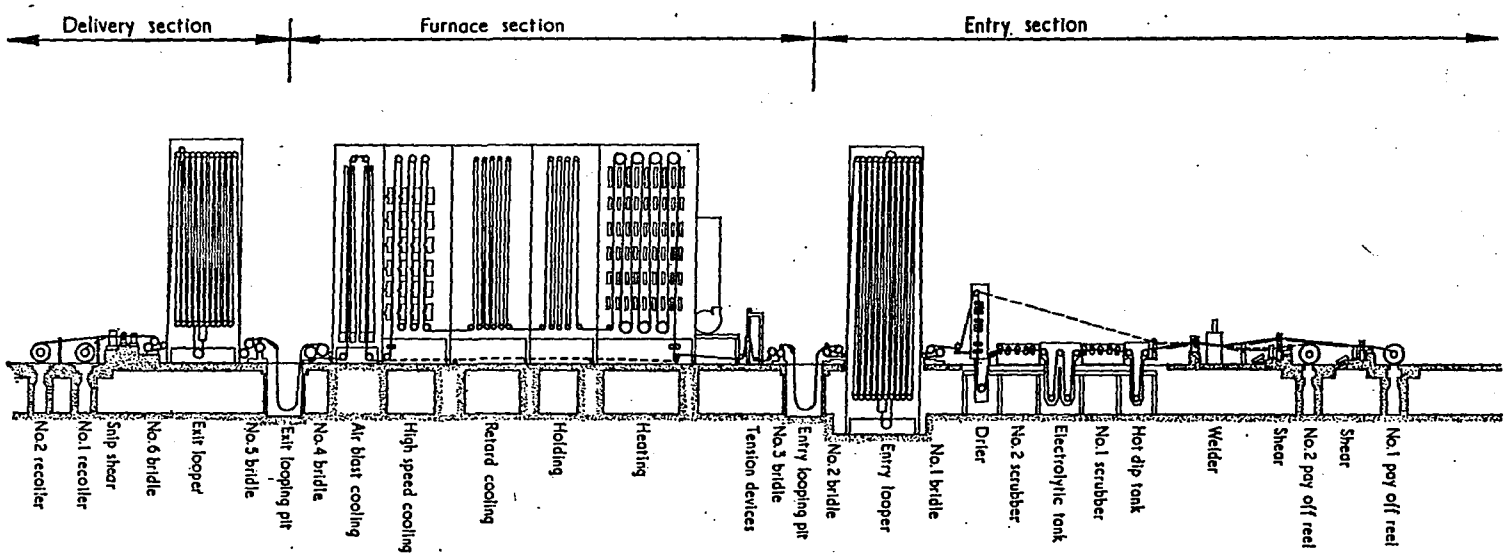

Figure 1. Schematic section of a continuous annealing line (9,10).

The furnace section is divided into four main zones; heating, soaking, slow cooling and fast cooling; and throughout these zones the steel strip is protected from oxidation by an atmosphere gas. The strip enters the furnace and is rapidly heated to nearly 700°C in 20 seconds. It then enters a soaking zone to allow time for complete recrystallization which is another 20 seconds at 700°C. After soaking, the strip is slowly cooled in an insulated chamber to about 480°C over a period of 30 seconds.

The slow cooling is considered necessary because on cooling from 700°C to room temperature, the solid solubility of carbon and nitrogen in the ferrite decreases considerably. If the recrystallized strip is cooled rapidly,the ferrite becomes supersaturated with these elements leading to precipitation of fine carbide and nitride particles at room temperature. This phenomenon, known as quench aging, results in a considerable increase in hardness and decrease in ductility of the steel strip. Slow cooling allows most of the carbon in solid solution to precipitate onto existing carbides and allows at least part of the nitrogen to diffuse out of the steel, thus avoiding quench-aging effects. After slow cooling to below 480°C, the strip is rapidly cooled.

The rapid rates of heating and cooling are made possible by the very thin sections of the strip (approximately 0.010 in.) and by the very nature of the process, all parts of each coil are subjected to exactly the same thermal cycle ensuring a uniform product in respect to mechanical properties and surface conditions. It has been mentioned previously that continuous annealing produces a harder grade

of material than batch annealing. This, as will be discussed later, is due to a fine grain size in the continuously annealed product, a consequence of the very short annealing cycle. For soft, deep-drawing grades, the open-coil batch annealing process appears to be most successful and should in time replace conventional batch annealing, while for the harder grades, especially tinplate, continuous annealing has attained complete acceptance.

Manufacture of Tinplate

(i) Conventional Method

Most of the tinplate now being produced is made from low-carbon steel strip which has been hot rolled to an intermediate gauge, pickled, cold rolled almost to finished gauge, cleaned, process annealed, temper rolled to final gauge, and finally electrolytically coated with tin. It is sold to can manufacturers according to hardness and strength specifications, normally called tempers.

Temper is principally dependent upon steel composition, annealing practice (batch or continuous) and degree of temper rolling. The strengthening of steel strip prior to cold reduction by alloying, e.g. with chromium or nickel, is undesirable from an economic point of view, but the addition or retention of nitrogen or phosphorus has successfully been used to obtain the harder tempers. Continuous annealing, with its inherent fine grain size, is satisfactory for the intermediate tempers of tinplate but batch annealing which produces a larger grain size must be used to obtain the very soft tempers. Temper rolling, or preferably skin-pass rolling, is used to improve strip shape and surface but most important, it imparts to the steel a slight measure of cold working. Two advantages gained from this cold working are: firstly, the temper within the intermediate range can be controlled by the amount of cold reduction which is usually less than 5%, and secondly, the yield extension, which results in defects called stretcher strains, is eliminated for reductions greater than 1%.

The tempers available in tinplate are conveniently indicated by a range of Rockwell superficial hardness values (R30T scale) and these are given with typical applications in the diagram (12) shown in Figure 2.

(ii) Double-Reduced Tinplate

In recent years there has been a strong trend towards tinplate of thinner gauge to enable more economical canmaking so that steel may compete effectively with other materials. As the steel base becomes thinner, its strength must be increased so that it can still serve its intended purpose.

The double-reduction process (13,14) is unique in that thinner as well as stronger material is produced in the same operation. This is a two-stage cold-reduction process with intermediate annealing of the strip. The strengthening is achieved by strain hardening in the second cold reduction that replaces the conventional skin-passing operation. The final hardness and strength are controlled by the steel grade and by an adjustment in the amount of second reduction: in the order of 30% for Temper 7 and 50% for Temper 8. The extra strength of this thinner (0.006 in.) double-reduced strip is obtained at the expense of ductility and thus it can be used only in applications where plastic deformation is at a minimum. Another major disadvantage to this process is that single-stand temper-rolling mills are not designed to cold reduce more than a few percent. The installation, at high capital costs, of two-stand or three-stand mills to handle the final cold reduction, is necessary.

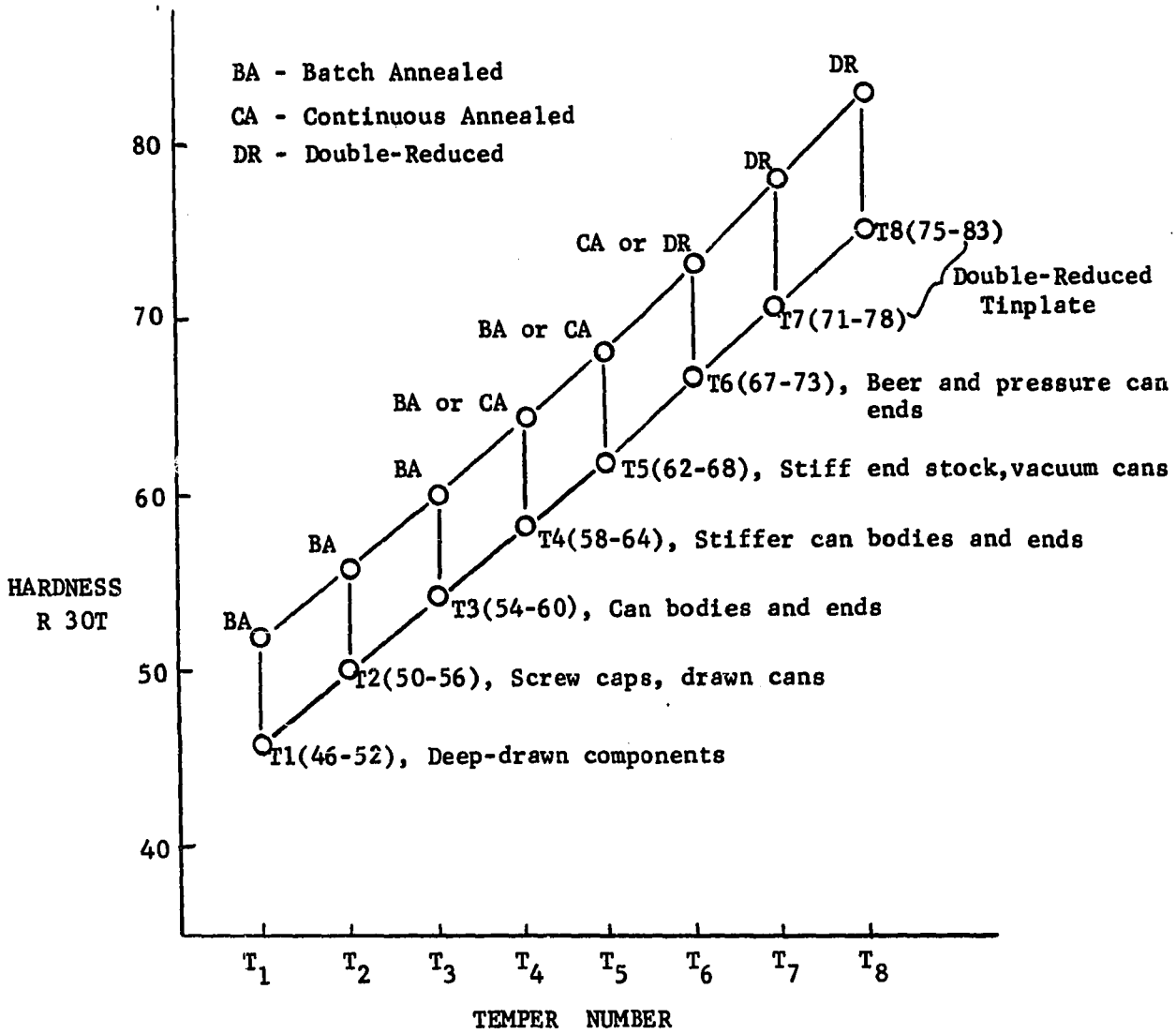

Figure 2. Tinplate temper grades and applications (12,31).

Partial Annealing - Project Objective

In the future it is likely that there will be an ever increasing demand for higher-strength tinplate. Double-reduced material is finding very wide application in pressure packs of the type used for soft drinks and beer. The heavy capital expenditure needed to produce this material which has, at best, only marginal ductility, has led to a search for alternative processes. The ability to produce high-strength tinplate on a continuous annealer, without the use of a second cold reduction would obviously be attractive. One way of achieving this objective is to control times and temperatures of the continuous annealing cycle to enable production of less than fully recrystallized material. This material is then said to be partially annealed.

In a partial annealing process, the high strength obtained from strain hardening the material by a cold reduction of 80-90%, would be retained by only partially annealing this very hard material back to the temper or strength specification. An investigation has been undertaken to compare the mechanical properties of heavily cold-reduced, then partially annealed strip to those properties obtained from cold rolling annealed strip by various amounts. This comparison should determine what, if any, improvement could be made in the ductility of low-carbon steel strip by partial annealing as opposed to the standard temper-rolling or double-reducing processes.

REVIEW OF LITERATURE ON PARTIAL ANNEALING

Non-Ferrous

There are only a few references to partial annealing of materials in published literature, which in itself would indicate that too little attention has been paid to the process. Investigations (15,16,17) into partial annealing of aluminum and aluminum alloys all showed that partially annealed material had higher elongation values for a given tensile strength than material which had been temper rolled. Reduced directionality (18) has also been reported but the same author warns that variables such as metal composition, casting methods and intermediate annealing, affect the rate and extent of recovery during partial annealing. This may be true of batch annealing methods but on a continuous single-strip annealer, another investigator (19) reported that high heating rates and short annealing times minimize the influence of prior history of these aluminum alloys. Parsons (20) has partially annealed aluminum-magnesium alloys and found significant beneficial effects on elongation, yield-to-tensile ratio and directionality.

An investigation (21) into the self-annealing of copper at room temperature and a recent paper by Mima et al. (22) on the annealing spectrum of heavily drawn copper wire, both showed the extent to which the mechanical properties varied with time and temperature, i.e. with degree of recrystallization, but the data were not very helpful in

planning the present series of experiments. A brief mention in a book (23) by Herenguel, states that partial annealing of copper and its alloys has been empirically practised for quite some time as an alternative way in which to produce tempers of 1/2 hard, 1/4 hard, etc., but temperature control in annealing and control of mechanical properties hinder its development. Fritz (24,25) made the first known attempt to compare partially annealed copper with that which had been temper rolled. He showed that partially annealed copper has a greater elongation and a lower yield-to-tensile ratio than does temper-rolled material, but little difference in directionality was found between the two. An investigation (26) into using partially annealed 70/30 brass as an alternative to temper-rolled material showed that "the combination of mechanical properties obtained is better at virtually any desired strength level and that normal mill variations in composition were found to have negligible effects on the annealing behaviour and on the properties of the finished material".

Low-Carbon Steel Strip

Patent rights (27,28) to a partial annealing process were granted in 1965-66 to Frazier and Toth of the Youngstown Sheet and Tube Company. This all-encompassing patent covers several different process paths whereby partially annealed low-carbon steel could be produced. The authors claim economy in production and superior ductility without significant loss in strength for their rolled steel products, especially for their high-strength tinplate which was used as their example. However, they have published no literature relating directly to the process or to the mechanical properties of their partially annealed low-carbon steel strip. A copy of their U.S. patent (27) is presented for perusal in appendix B.

Several other tinplate manufacturers(29,30,31) are known to have investigated partial annealing as an alternative to temper rolling. All have indicated(except Youngstown) that the stringent degree of temperature control that would be needed on a continuous annealing line to produce a uniform product, would make the process impracticable.

Montgomery (29) of the Steel Company of Canada used an internal resistance heating apparatus to simulate the heating cycle of a continuous annealing line running at speeds of 400 and 800 ft/min. Strip which had been cold reduced 85,90 and 95% were given partial annealing treatments in which the peak temperature ranged from 800°F (427°C) to 1200°F (650°C).

He concluded that in the range of peak temperatures wherein the mechanical properties changed most rapidly with respect to temperature, the scatter of measured properties especially ductility, was very large. He further concluded that with this scatter and variations in composition or prior history, it would be difficult to produce a uniform product in ordinary commercial practice.

Bernick and McFarland (30) of Inland Steel Company reported an investigation into the direct cold reduction from hot-rolled strip to double-reduced gauge (0.006 in.), followed by annealing to give the desired strength and ductility. They reported that it became obvious early in the experimental program that continuous annealing was not suitable for partial annealing because even with the best temperature control possible in commercial continuous annealing, recrystallization kinetics are unfavourable to allow control of the mechanical properties. However, by batch annealing for 8-24 hours, they claimed it was possible to obtain a wide range of mechanical properties with reasonably good control.

Wooldridge (31,32) at the Steel Company of Wales carried out laboratory studies of the partial annealing of heavily cold-reduced steels. He used a salt bath to simulate both continuous and batch annealing cycles on steels of 16 different compositions. Hardness and tensile values, elongation, Erichsen cupping and Jenkins bend values were obtained. Isothermal and isochronal plots of hardness versus annealing time and temperature respectively, were the bases on which it was concluded that the degree of temperature control that is necessary to produce higher-

temper tinplate was outside the present capabilities of batch or continuous annealing processes. He also found that the ductility of partially annealed material was only slightly superior to that of double-reduced tinplate of equivalent hardness.

Wooldridge also investigated quench-aging effects, temper-rolling and strain-aging effects, and rate-of-heating effects on partially annealed material. After this, double-reduced material (30-50% cold reduction) was partially annealed using combinations of batch and continuous annealing cycles. Nine more steel compositions were added to investigate aluminum-killed steels, niobium-treated steels and finally nitrogenised double-reduced steels. The investigator concludes, "Some difficulty has been experienced in the meaningful interpretation of the results of mechanical and tensile tests on the material studied for the report". He goes on to suggest that the range of testing should be extended to include flange tests and springback tests as a way out of these difficulties. Parts of this investigation were later published by Jenkins et al.(14).

Richards et al. (33) of John Lysaght (Australia) Ltd. investigated recovery rates of cold-reduced low-carbon steel strip with a view to improving the ductility of light-weight galvanised structural and building shapes without impairing their strength. The paper describes the development of a loop fracture test and its correlation with x-ray

line broadening during recovery and recrystallization, but nevertheless, it does show that partial annealing is being considered for materials other than tinplate.

The tinplate manufacturers seem to be unanimous in their opinions that partial annealing is outside the capabilities of present-day continuous annealing lines. A temperature control of $\pm 5^{\circ}C$ to maintain a hardness range of ± 3 R30T has been mentioned (11). The investigations concentrated mainly on the operational difficulties of partial annealing. Too little attention has been paid to the comparison of mechanical properties of partially annealed material to temper-rolled material. It is in this respect that this project was expected to be most helpful.

THEORETICAL BACKGROUND

Cold Working

A metal may be said to be cold worked if its grains are in a distorted condition when plastic deformation is completed. This will usually be the case when the deformation occurs at a temperature lower than one half of the melting point of the metal, on the absolute scale (34). Certainly, when low-carbon steel is cold worked by rolling at room temperature, its grains are so severely compressed and the normal ferrite lattice is so distorted that it is said to be in a highly strained state or strain hardened.

Cold working increases the number of vacancies and more important, the number of dislocations in a metal. Fully annealed ferrite will contain about 10^7 dislocation lines per sq.cm. while heavily cold-worked ferrite will contain approximately 10^{12}. Since each dislocation represents a crystal defect with an associated lattice strain, increasing the dislocation density increases the strain energy of the metal. Under an applied stress, these dislocation lines move along different crystallographic slip systems, interact with one another and become trapped thereby increasing the resistance to further deformation. A higher applied stress is then necessary to force the dislocations to resume movement. In this manner, the metal becomes progressively stronger and harder with increased deformation, a process known as strain hardening. The flow stress is independent of temperature and is proportional to the square root of the dislocation density.

When polycrystalline iron is deformed at room temperature, the random moving dislocations become entangled with each other and with grain boundaries or other obstacles. They begin to form, at about 5% strain, a cellular structure with a cell size of about 3 microns. These cells are a network of entangled dislocations separated by areas of low dislocation density. The cells decrease in size with increasing deformation, up to about 10% strain, then they maintain a constant diameter of about 1 micron. After the strain has been increased to 70%, the cell walls have become quite densely populated with dislocations, while the interiors of the cells remain at low dislocation density. This process of forming cells by dislocation interaction is described by Leslie et al. (35) and Austin (36). An excellent review of strain-hardening theories is given by Weidersich (37).

The perfection of the cell structure depends upon the impurity content. In zone-refined iron, the cell walls are sharp but in less pure iron such as ferrite the walls are thicker and ill-defined. Also, when there are fine precipitates or solute atoms present in the matrix there is an appreciable density of dislocations within the cells. The lattice misorientation across a cell wall is about 2°, but these cell misorientations are additive, which means that the total range of misorientation within any one grain that had been cold worked is usually large and of the order of 20°, depending upon the amount of strain.

Annealing

(i) Recovery

In recovery, the physical and mechanical properties that suffered changes as a result of cold working tend to recover their original values and occurs before the appearance of any new strain-free grains (38,p.37).

It is the increase in the free energy as a result of plastic deformation that is the driving force for the phenomena observed during annealing. Among the several measures of recovery which have been employed are, the release of stored energy, the decrease in electrical resistivity, the decrease in x-ray line broadening, the changes in mechanical properties and most recently, the changes in vacancy concentration by mass density measurements and the changes in the dislocation density and distribution. Recovery in iron has been reviewed by Leslie et al. (35) and in metals generally by Li (39). In most instances, recovery has been studied in relatively pure materials and in single crystals after small strains. Very little data are available on recovery in polycrystalline iron reduced more than 50%.

Hu (40) has outlined three stages of recovery in his investigations on 3% silicon-iron single crystals which had been cold reduced 80%. These are illustrated schematically in Figure 3 by plotting vacancy

concentration and dislocation density versus annealing temperature. In stage I, the crystal density is the first property to recover with no change in x-ray line broadening, hardness or microstructure, suggesting that this observed density change is largely due to the annealing out of vacancies in the temperature range of 150-190°C. In stage II, from 200-500°C, annihilation and rearrangement of dislocations constitute the main process responsible for x-ray line sharpening and a decrease in microhardness. In stage III, at annealing temperatures above 500°C, major structural differences becomes evident, well-defined subgrains are formed, and recovery of mechanical properties is nearly complete.

It has been found, with the aid of electron transmission microscopy, that the changes in properties during recovery stages II and III are associated with, but not wholly controlled by, a decrease in average dislocation density of the previously cold-worked material. Evidence has been gained to show that dislocations at the recovery temperatures become mobile; straighten; those of opposite sign begin to annihilate each other; and those in the cell walls rearrange themselves. The dislocations move by cross-slip and climb which are thermally activated processes, so that recovery is temperature dependent. Dislocation climb is also dependent on the number of atomic vacancies present in the metal and thus the amount of prior cold working, which determines the amount of vacancies, will also affect recovery rates.

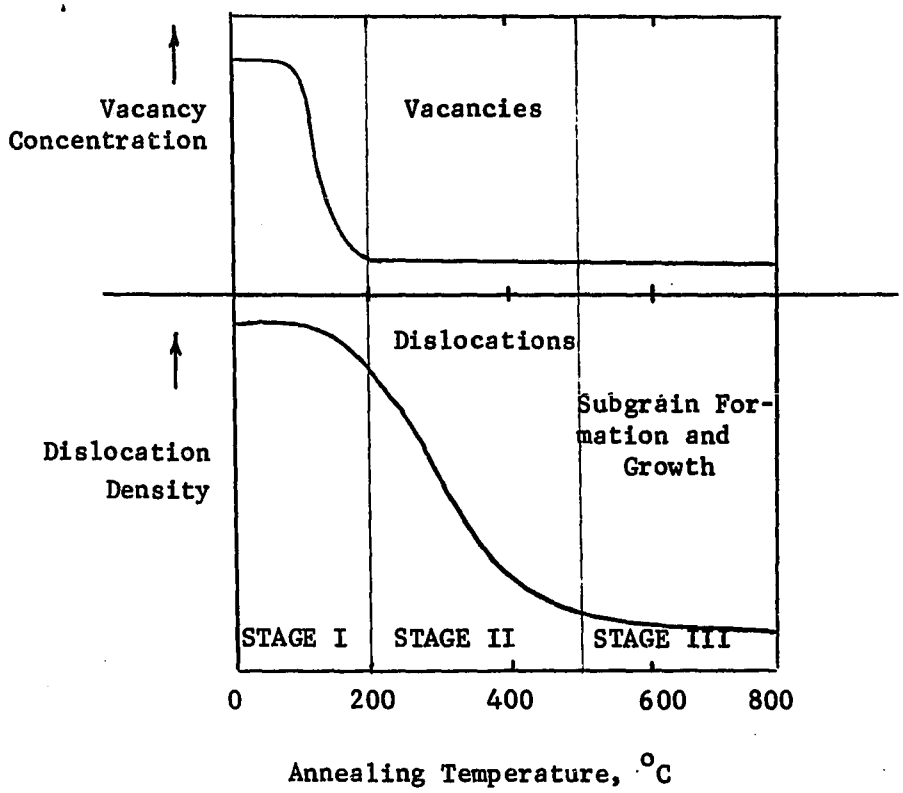

Figure 3. Schematic representation of the three stages of recovery. (40).

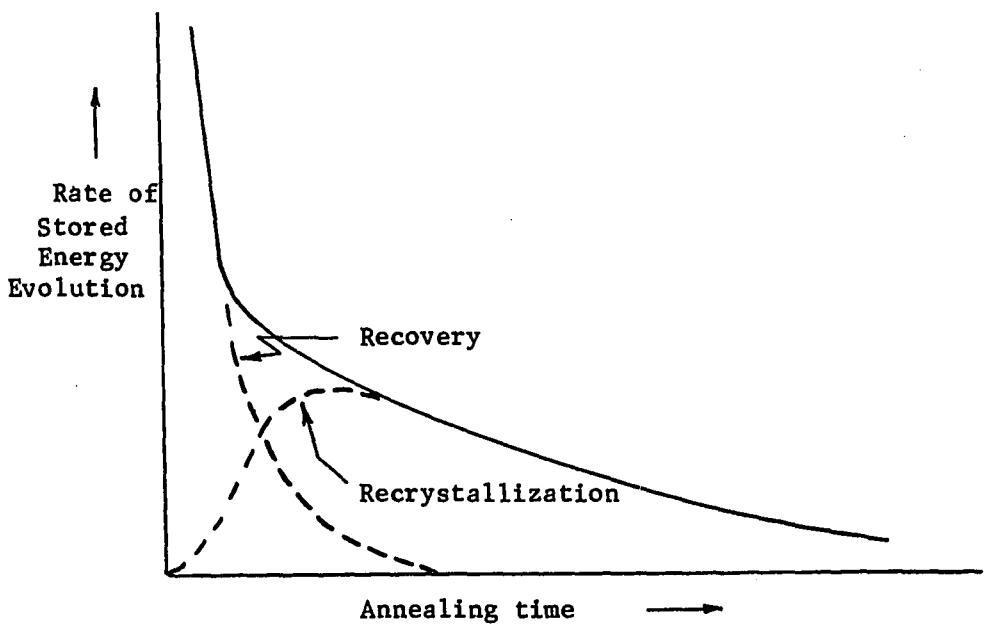

Figure 4. Schematic illustration of the overlapping of recovery and recrystallization in the rate of stored energy evolution versus annealing time. (41).

The cell walls, having become sharper and well-defined, are now termed sub-boundaries. The interiors of the cells, now subgrains, have become nearly dislocation-free but are still the same size as the dislocation cells of the deformed iron.

Dislocation-free subgrains bounded by low-angle sub-boundaries may be defined as the finished product of recovery, while the growth of these subgrains constitutes the incubation period of recrystallization. It must be pointed out that stage III recovery and the starting of recrystallization may overlap each other within the same specimen and thus no distinct line of demarcation would exist between the two processes (41). Figure 4 schematically illustrates this point and thus for simplicity of explanation, each process is described separately.

(ii) Recrystallization

Primary recrystallization is defined as the nucleation and growth of new strain-free grains and the gradual consumption of the cold-worked matrix by the growth of these grains. The growth is accomplished by the migration of the high-angle grain boundaries (38,p.60).

Recrystallization has been shown to be a growth controlled process. There is no random nucleation as in solidification, that is, the formation of nuclei by thermal fluctuations does not occur. The theory of

nucleation advanced by Cahn (39,42) which involves the growth of subgrains to the point at which high-angle boundaries are formed, has been shown to be essentially correct (35,p.153). The growth of these subgrains, which constitutes the incubation period of recrystallization, can occur at three types of sites; in the interior of a ferrite grain, at a high-angle boundary, and at an inclusion or second-phase particle.

Hu (40) has obtained direct evidence that subgrain growth in the interior of ferrite grains is by a process of subgrain coalescence, which he explained as a gradual moving-out of dislocations from a disappearing subgrain boundary into connecting or intersecting boundaries. This process probably requires some dislocation climb along the disappearing subgrain boundary and a rotation of the subgrain itself, by lattice diffusion, to merge into a common orientation. Further coalescence increases the angle of misorientation between the now large subgrain and its neighbours to form a high-angle boundary. Thus the larger subgrain has become a recrystallized grain at an early stage of formation. This is shown schematically in Figure 5, where the subgrains have coalesced, but the original subgrain boundaries within the recrystallized grain R, are still faintly visible. Segments of the interface between R and the matrix consist of highly mobile high-angle boundaries and further growth takes place by migration of these boundaries into the subgrained matrix.

The effectiveness of previous high-angle boundaries as nucleation

sites of recrystallized grains is well established. Beck (43), working with aluminum, observed that migrating boundaries may be traced back to independent nucleation sites at the original grain boundary that existed before cold working. Leslie et al. (35, pp. 157-160) have confirmed this observation using electron transmission microscopy in recrystallization studies of cold-worked dilute solid solutions of iron.

The nucleation of recrystallized grains by second-phase particles, oxide inclusions and copper precipitates in iron, has been observed and

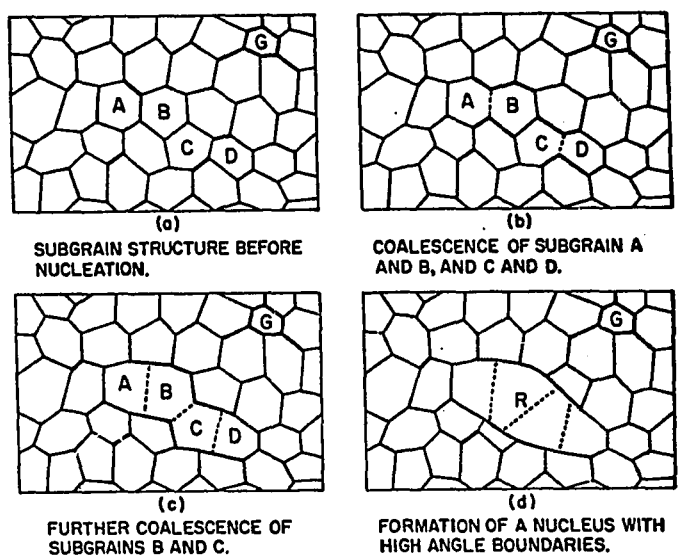

Figure 5. Schematic representation for the formation of a recrystallized grain by the coalescence of subgrains (40).

an investigation (35,p.167) has shown that by increasing the number of inclusions present in a specimen, its rate of isothermal recrystallization is increased. Although inclusions or precipitate particles are known to inhibit the movement of grain boundaries, such inhibition cannot occur when the inclusions themselves are preferred sites of grain nucleation. It is this nucleation at many inclusion sites that will produce a uniform fine-grained structure after recrystallization, as compared with that where nucleation and growth occurred from only a few sites.

The inclusions are assumed to provide regions near their surfaces in which the lattice is relatively undeformed during working but surrounded by highly deformed material. It can be visualized that a streamlined plastic flow of metal around a hard incompressible inclusion will leave small areas, forward and behind the inclusion, wherein the metal is relatively undisturbed when compared to that which flows around the inclusion. In addition, the inclusions provide free surfaces to serve as sinks in which dislocations can run out, thus allowing the perfection of the lattice in cells bordering the inclusion. The embedding of undeformed metal in inclusion microcavities proposed by Burke and Turnbull (44), which will serve as recrystallization nuclei, has been discounted. The effect of second-phase particles in recrystallization is not restricted to oxide or copper inclusions as the presence of cementite particles is the most probable reason for the rapid recrystallization of low-carbon steels.

After the nucleation of new strain-free grains by one or more of the aforementioned mechanisms, they grow into the cold-worked matrix by the outward migration of their high-angle boundaries. Studies of recrystallization kinetics, comprehensively reviewed by Burke and Turnbull (44), showed that recrystallization proceeds by nucleation and growth to yield sigmoidal isothermal reaction curves. It was also shown that the increase in size of any one grain can be measured in terms of a constant linear rate of growth. However, Leslie et al. (35,45) found that the rate of linear growth in zone-refined iron decreased with increasing annealing time and favour the explanation that recovery is competing with growth for the available stored energy. They explain that after small strains, recovery may be substantially complete before recrystallization begins, thus leaving a constant driving force to produce a constant rate of growth; but for heavily cold-worked iron, recovery may not have ceased before the grains have impinged upon each other. There is a great need for more work to be done on the recrystallization of iron and its dilute alloys.

(iii) Grain Growth

Grain growth may be defined as the gradual increase in the average grain size upon further annealing after all the cold-worked structure has been consumed by new recrystallized strain-free grains. When recrystallization is complete, the main driving force, i.e. the retained

energy of deformation, is spent, but the grain structure is not yet stable because the material still contains grain boundaries having interfacial (or surface) energy of about 500 ergs/cm^2. When the grain size increases, the total grain boundary area decreases and consequently the energy of the metal decreases. Grain growth then, is a function of time and temperature and not of strain or cold working. Reed-Hill (34, p.199) and Byrne(38) describe the kinetics of grain growth generally, but literature on grain growth during subcritical annealing of low-carbon steel seems to be lacking, due mainly to the very long experimental annealing times necessary since the temperature must remain below the 723°C transformation temperature. Most investigations were of a practical nature where ASTM grain size of a particular grade of steel was plotted against the soaking time of a batch annealing cycle.

Grain growth is very important in the batch annealing process and enables production of very soft deep-drawing steels and large-grained electrical steels. In continuous strip annealing however, the very short annealing cycle prevents grain growth and results in a material which has a finer grain size and therefore harder than the batch annealed product.

The above description of grain growth is very abreviated and only the main features are outlined because of the irrelevance of this

phenomenon to the work in this thesis. It is self-evident that grain growth will have no part in a partial annealing process.

(iv) Effects of Composition on Annealing Rate

The effects of solute elements upon recrystallization of heavily deformed high-purity metals can be represented qualitatively by the curve of Figure 6. The rate of high-angle boundary migration drops

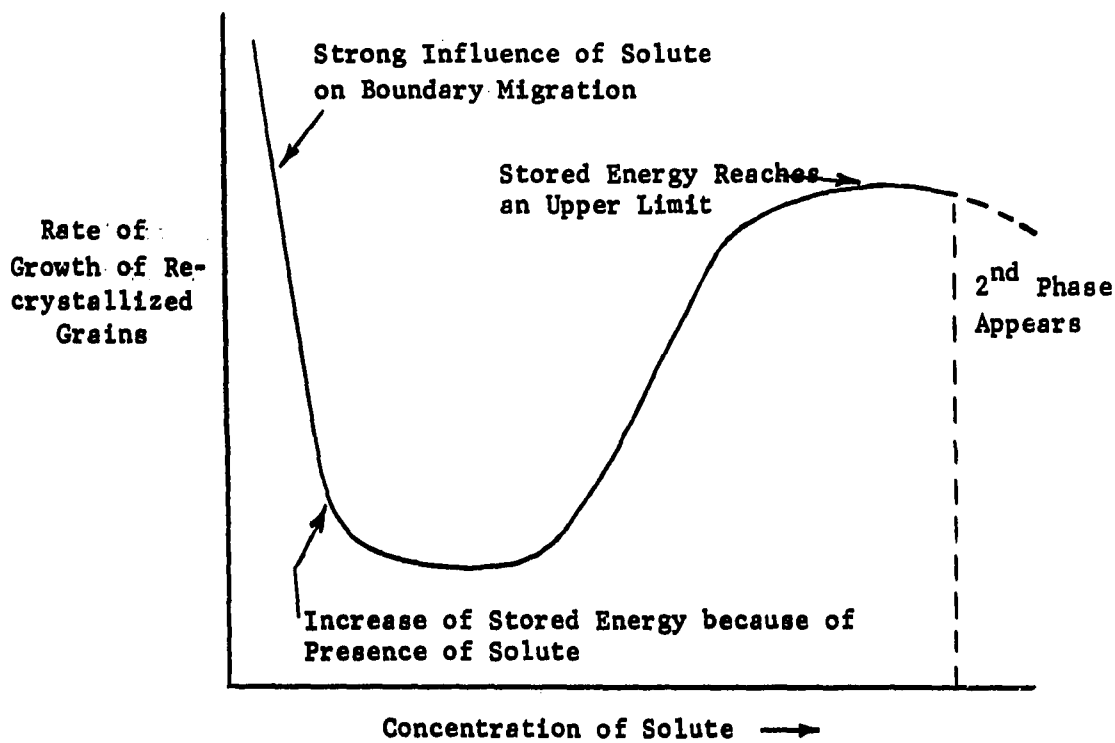

Figure 6. The effects of a solute element upon recrystallization (35).

very rapidly with the first very small additions of one solute element. This is thought to be due to a solute atmosphere drag upon the migrating boundaries and theories on this topic are extensively reviewed by Gordon and Vandermeer (46). At first, the very small additions do not cause any measurable increase in the driving force for boundary migration, but as the concentration increases, the extra stored energy of deformation due to the effect of solute atoms within the matrix on strain-hardening mechanisms (37), is sufficient to offset the drag effect of the solute upon boundary migration. With further increasing solute content, the stored energy for a given deformation may finally reach a nearly constant value and the rate of recrystallization may change very little. Beyond this concentration, the limit of solubility may be reached and an already involved situation is further complicated by the appearance of a second phase (35, p.118).

Abrahamson and Blakeney (47) have systematically investigated the effects of small additions of the transition elements on the recrystallization of iron. Their results show a linear increase in their defined recrystallization temperature with increase in solute up to a "critical concentration" where the slope of the line suddenly lessens. Considerable controversy over theories and experimental techniques exists in the explanation of the role of solute in recrystallization studies.

The effect of carbon on recovery and recrystallization kinetics of iron has been studied by Ventuello et al. (48). These investigators doped high-purity iron with up to 86 ppm carbon and found a marked retardation of recovery but little or no effect on recrystallization. It was explained that the interstitial carbon interacted strongly with dislocations in the subgrain boundaries and hindered their rearrangement during recovery, but after high-angle boundaries began to move, the carbon had little effect on recrystallization. This, according to the Detert and Lücke theory (49), was because the rate of motion of a grain boundary in the presence of impurities is controlled by the rate of motion of these impurities, i.e. their diffusivity. Hence carbon, because of its very high mobility, has little effect on recrystallization. Similar results were obtained when nitrogen was used as the interstitial solute element (50).

Leslie et al.(45) have studied the effect of manganese on the recrystallization of high-purity iron and found that its addition decreases the growth rate of any one grain. It was proposed that the Mn atoms diffuse very rapidly during annealing to occupy favourable sites at the subgrain boundaries and because of this, recovery and subsequent grain growth are both inhibited by grain boundary drag. It was also found that with the addition of manganese in the amount of 0.30 atomic % or more, a change occurs in the

mechanism of recrystallization. Although the rate of grain growth decreases with time, the rate of recrystallization increases with time. The possibility of Mn - O or Mn - S interactions to produce second-phase particles to aid nucleation was considered.

Mechanisms of recrystallization involving second-phase particles in iron alloys are very complicated and investigations into these phenomena are few, but one particular case has received a great deal of interest because of its commercial importance. The formability of aluminum-killed deep-drawing steel is very much superior to rimmed steel. This benefit was traced to an elongated, pancake-shaped grain structure that occurred in Al-killed steel but not in rimmed steel. Investigations (51,52,53) into the control of this desirable elongated grain structure confirmed the presence of precipitated aluminum nitride particles on the recrystallized grain boundaries. Further investigations by Goodenow (54) and Jolley (55) using electron transmission microscopy have led to an explanation of how the elongated grains are formed.

During batch annealing of the killed steel, the slow heating rate allows sufficient time for the aluminum and nitrogen (in solid solution in the ferrite), to segregate to the as-rolled grain boundaries and subgrain boundaries before the onset of recrystallization. Then, when recrystallization nuclei begin to grow, the high-angle boundaries

"sweep up" the aluminum and nitrogen from the subgrain boundaries to form atmospheres which exert a drag on the migrating boundaries. Eventually the recrystallized grains will reach the original as-rolled grain boundaries,where there is already a high concentration of aluminum and nitrogen, and the impurity drag becomes strong enough to stop the boundary movement. The growth then proceeds only in the longitudinal direction (with respect to the as-rolled grains) to produce an elongated recrystallized structure. Shortly after recrystallization is complete, the aluminum and nitrogen atmospheres form aluminum nitride precipitates outlining the original as-rolled grain boundaries.

Inhibition of recrystallization is not restricted to pre-precipitation clusters or atmospheres of aluminum and nitrogen, for Baird and Arrowsmith (57) have shown that sulphur in solution is precipitated as a fine dispersion of manganese sulphide on the dislocation substructure. Leslie et al. (56) found similar results in their work on iron with additions of manganese and oxygen. Second-phase particles have been shown (35,57) to inhibit recrystallization if they are smaller than 0.1 micron but if they are 1 micron or larger in size, they will promote recrystallization by acting as nuclei. No paper has been found that deals specifically with the most common second-phase particle, Fe_3C.

Plastic Anisotropy

Most forming operations using metal sheet are of the nature where the sheet is required to plastically stretch or bend into a specific shape. There are many cases where the metal sheet will not plastically deform with equal ease in all directions and thus a measure of its plastic anisotropy is of great importance.

Low-carbon steel sheet exhibits plastic anisotropy in two forms. The first, planar anisotropy, refers to variations in properties (e.g. in the yield strength and ductility) with directions in the plane of the sheet. Thus, when a disc is drawn into a cylindrical cup, earing of the rim is an undesirable result of planar anisotropy and its elimination would be advantageous. The second form, normal anisotropy, refers to variations in properties between directions in the plane of the sheet and normal to it; its practical importance lies in the fact that the resistance of the sheet to thinning during pressing or drawing operations, is a desirable result of its normal anisotropy.

The width restriction of the steel strip rolled in the present project prevented investigation into planar anisotropy of partially annealed material, but in order to extract as much information as possible from the tensile tests, a parameter of normal anisotropy in

the rolling direction was obtained. The parameter most commonly used is the strain ratio "r" or Lankford coefficient (58), which is defined as the ratio of the natural strains in the width and thickness directions for a specimen extended in tension:

$$r = \frac{\epsilon_w}{\epsilon_t} = \frac{\ln \frac{w_o}{w_f}}{\ln \frac{t_o}{t_f}}$$

Here w_o and t_o are the initial width and thickness while w_f and t_f are the final width and thickness of the specimen. It can be seen that if the width and thickness strains are equal the material is isotropic, but if the width strain is more than the thickness strain, as it is in aluminum-killed deep-drawing steels, the material is anisotropic, the r-value being about 1.5.

Mathematical treatments (59,60,61) of plastic anisotropy and papers (62,63,64,65) relating r-values to forming operations are explicit. There are several excellent papers (66,67,68,69) relating plastic anisotropy to preferred crystallographic orientations of cold-rolled and annealed steel sheet. Cold-rolling and annealing textures (39,67, 70) were beyond the scope of this project.

Instructions (63,65,71) on determining the plastic strain ratio recommend that length strains replace thickness strains (with the

formula suitably rearranged) to enable a more accurate determination of the ratio, but the design of the tensile specimen used in this project prevented this replacement and thickness measurements were used. All of the previous work has been done on fully recrystallized material and therefore the strain ratio was usually determined after 15-20% elongation. Again, it was clearly impossible to comply with earlier "standard" methods to obtain the ratio because partially annealed or cold-rolled material have elongations as low as 1 or 2%.

For this project, the final width and thickness measurements were taken after fracture on cross-sections sufficiently removed from the necked region. This implies that the strain ratio is obtained after maximum uniform strain which varies from 1% to 25% depending upon the degree of cold rolling or partial annealing that the material had received. The usefulness of such a test will depend upon the comparative results between the two types of materials, i.e. partially annealed versus cold-rolled.

EXPERIMENTAL PROCEDURE

Introduction

In order to compare partially annealed tinplate stock to that produced conventionally by temper rolling (or by double reducing), the variation in properties must be found as a function of annealing temperature and cold reduction, respectively.

Cold-rolled low-carbon steel strip of 0.016 in. thickness was prepared so that portions had received cold reductions ranging from 1 to 80%. The mechanical properties of interest; hardness, yield and tensile strengths, r-value, and ductility as measured by percent elongation,were obtained so that they could be plotted against percent cold reduction in order to exhibit the variation in properties of temper-rolled material.

Material to be partially annealed was cold rolled to 0.016 in., i.e. a reduction of 82%, then cut into tensile specimens and annealed in a salt bath at temperatures ranging from 460 to 620°C. The same mechanical properties, outlined above, were obtained so that they could be plotted against annealing temperature to exhibit the variation in properties of partially annealed material. Comparison of say, ductility at equal strength or hardness of the two differently treated materials, was then possible.

Material

The starting material used for the experiments was MRU grade low-carbon steel strip supplied in two lots from the normal production lines of the Steel Company of Canada Limited.

Lot 1, was in the form of hot-rolled strip, 10 ft long, 5 in. wide and 0.085 in. thick. It represented material which would normally be fed to the cold-rolling mill.

Lot 2, the product of the cold-rolling mill, was supplied in the form of strip 36 in. long, 20 in. wide, and 0.016 in. thick. It had been cold reduced 82% from 0.090 in.

The chemical analyses of the two lots, determined by the Steel Company of Canada, are given in Table I. Carbon analyses performed at McGill, using a Leco analyser coupled to a digital readout system, are also included in the table.

TABLE I

Chemical Composition of Material

Element	Lot 1 wt. %	Lot 2 wt. %
C	0.12	0.12
C*	0.13(0)	0.14(5)
P	nil	0.004
S	0.040	0.025
Mn	0.46	0.55
Si	0.018	0.010
Cu	0.026	0.026
Cr	0.012	0.027
Sn	0.002	0.005
Ni	0.015	0.013
Mo	0.010	0.008
N	0.003	0.002
Fe	balance	balance

* Carbon analyses performed at McGill

Test Methods.

This section precedes what might be considered normal chronological order, so as to aid coherency in later sections on temper rolling, partial annealing and results thereof. Each of these sections will refer to Test Methods for procedural details.

(i) Preparation of Tensile Specimens.

Tensile testing specimens were cut from the rolling direction of prepared strip and shaped on a "TENSILKUT" shaping mill to ASTM E8 specifications (72). During milling, when the specimens became warm to touch, the complete template-vice assembly was cooled in a freezer to avoid any possibility of artificial aging. The specimens cut from temper-rolled material were then ready for measurement and testing, while those cut from the highly cold-reduced material were set aside for partial annealing.

The specimens were very lightly scribed with a vernier height gauge at the five positions shown in Figure 7. The template of the "TENSILKUT" shaping mill was designed with a blended taper of 0.003 in. to insure a minimum cross-sectional area at the center of the specimen gauge length, position 3. Width and thickness measurements at position 2,3 and 4 were made with an "ETALON" micrometer. This precise instrument has anvils of 2.0 mm. diameter and a jewelled dial gauge that can measure to $\pm$ 0.00002 in. This allows fourth decimal place accuracy in measurements while the fifth

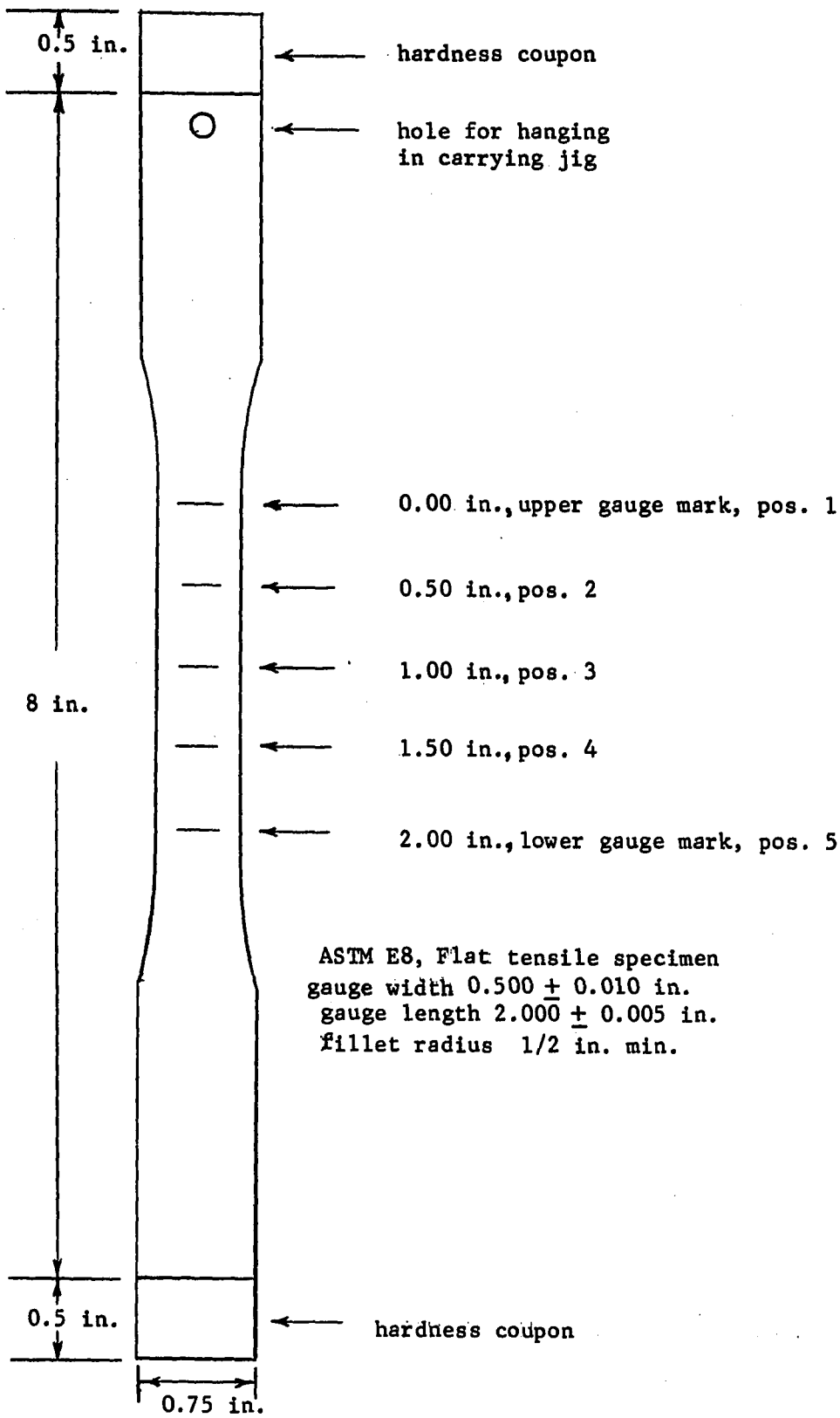

Figure 7. Tensile test specimen (72).

decimal place should be enclosed in brackets to indicate possible error. Each width measurement was an average of three readings while each thickness measurement was an average of five readings taken across the width of the specimen. Measurements at position 3, the center, were used to calculate the cross-sectional area of the specimen, while measurements at position 2 and 4 were used later to calculate the r-values of the material.

(ii) Hardness

Small test coupons were cut from each end of the tensile specimens. The hardness of these coupons was measured with a Rockwell Superficial Hardness Machine, fitted with a hardened steel anvil, on the R 30T scale. An average of at least five readings per specimen was recorded as specimen hardness. The hardnesses of the four tensile specimens from each rolling or annealing experiment were again averaged for an over-all hardness value for each experiment.

(iii) Tensile Testing

All tensile tests were carried out in an Instron TT-D Universal Testing Machine at room temperature using wedge-action grips and a cross-head speed of 0.1 in./min. The loads were measured with the standard GR Instron load-cell while the extensions were measured with a strain gauge extensometer designed for a 2 in. gauge length. The Instron cross-head

movement, which was equipped with a control dial, and which could be adjusted and read to $\pm$ 0.002 in., was used to calibrate the extensometer. This gave an extensometer error of $\pm$ 0.1% to all calculations of percent elongation, i.e. elongations quoted as 1.0% or 10.0% should be read as (1.0 $\pm$ 0.1)% or (10.0 $\pm$ 0.1)% respectively, in order to include the extensometer error. This error was considered well within the accuracy range for tensile testing.

An autographic chart recording of load versus extension was obtained for each test using an X-Y chart drive system at the greatest sensitivity consistent with chart size limitations. The extensometer recorded the entire test up to and including fracture.

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 total and uniform extensions were read directly from the chart record of each specimen and were defined in the following way. In Figure 8a, the flat-topped load-extension curve is typical of the higher-strength material and its line of proportionality defines the magnitude of elastic strain of this material under load. The total extension was defined as the horizontal distance E_t, from the point of fracture to the line of proportionality. To exclude all localized deformation in the necked zone, the uniform extension was defined as the horizontal distance E_u,

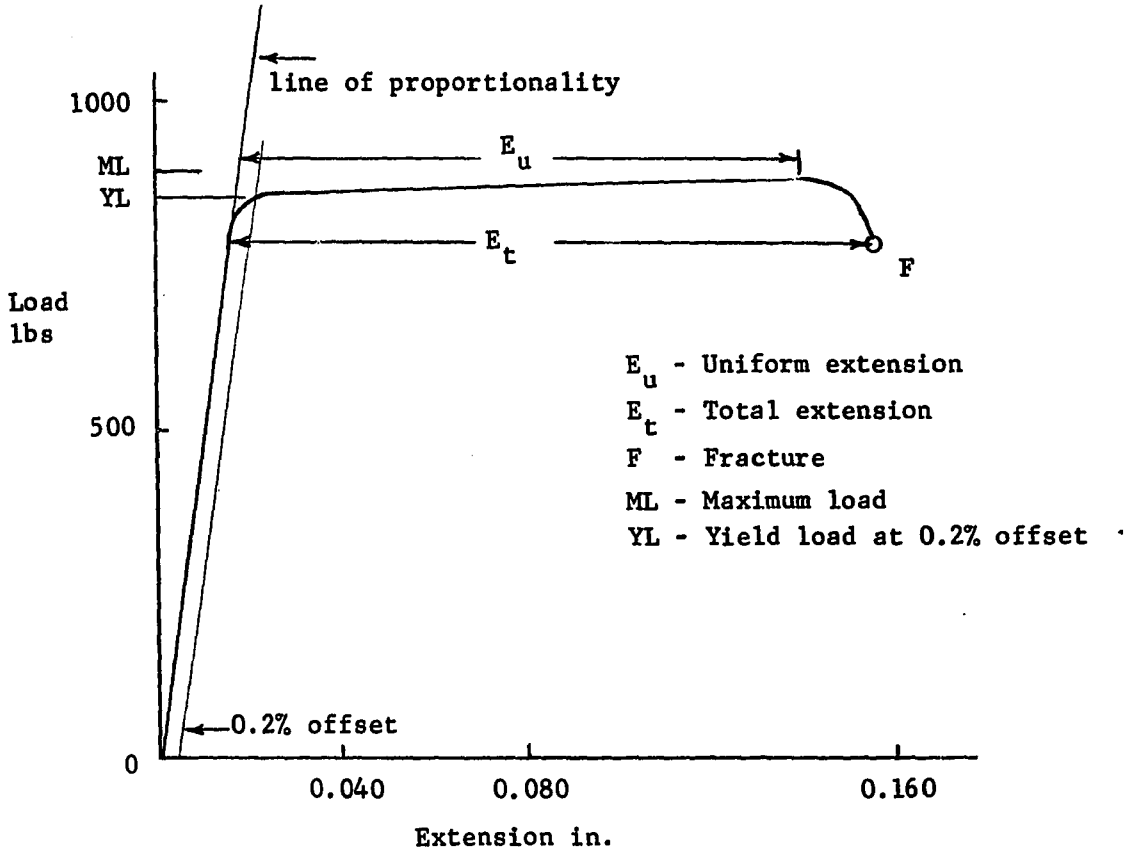

Figure 8a. Load-extension curve typical of high-strength material.

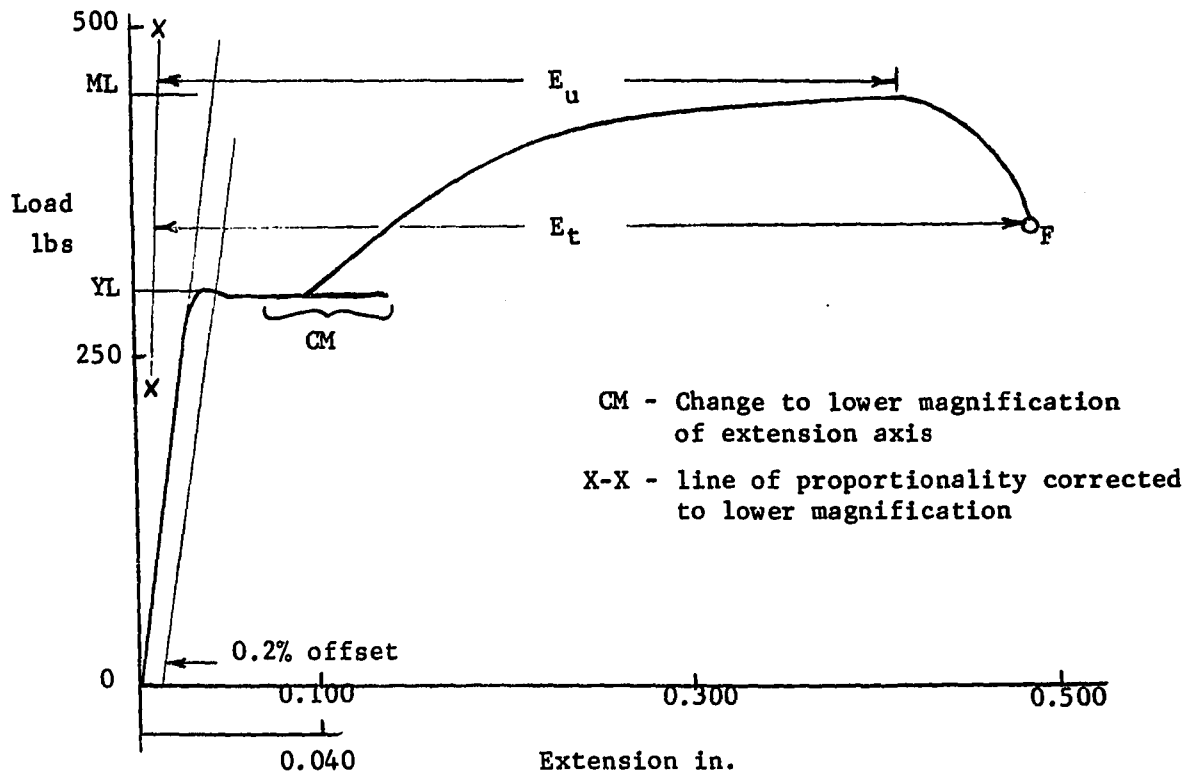

Figure 8b. Load-extension curve typical of the softer material.

from the point where the first perceptible drop from maximum load was observed, to the line of proportionality. These extensions were then converted to percent elongations.

The load-extension curve of Figure 8b is typical of the softer materials. In these more ductile materials—with relatively large elongations — it was necessary to switch to a lower magnification of the extension axis over the plastic range of the curve in order to retain the whole curve on the recording chart. To maintain consistent and accurate calculations of the yield loads, the magnification was not lowered until well after the yield. Because of this change in magnification, the line of proportionality had to be corrected to the slope it would have had at the lower magnification and then the extensions E_t and E_u were calculated from the chart.

Observation of the first perceptible drop from maximum load, especially on the long flat-topped curves, was helped by a particular load-following characteristic of the recording system. This resulted in a slight dip in the ink-line record at the very start of the load descent.

The extensions read from the charts were compared to those obtained from measuring the specimens in order to assess the accuracies of the direct and indirect methods. A cathetometer, accurate to $\pm$ 0.005 cm., was used to measure the five scribe marks on each of thirty specimens, before and after testing to fracture. The results indicated that the

total extensions to fracture, when measured by the cathetometer, were usually a few thousandths of an inch greater than those calculated from the charts. This small discrepancy was considered to be due to errors made in fitting the broken specimens together. Since the specimens necked and broke in the centers of the 2 in. gauge lengths, measurement of uniform extensions proved to be impossible because of the small amount of specimen length unaffected by necking. The extensions E_t and E_u were therefore obtained from the autographic charts.

The yield strengths, tensile strengths, total and uniform elongations of the four tensile specimens from each rolling or annealing experiment were averaged.

(iv) Strain Ratio r

Each tensile specimen was designed to break at the center of its gauge length, position 3, Figure 7. This left positions 2 and 4 sufficiently removed from the necked region to allow accurate measurements of width and thickness after fracture in order to calculate r-values. The two r-values from each specimen were averaged, then the four specimen values were averaged to obtain an overall r-value for each experiment.

(v) Metallography

Representative tensile specimens were chosen from both the temper-rolling and partial annealing experiments in order to correlate mechanical

properties with metallurgical microstructures. Longitudinal cross-sections of each of the specimens were mounted in Lucite, then successively ground on 320,400 and 600 grit silicon carbide papers. This was followed by a polish with a slurry of 5 micron alumina, on a nylon-covered wheel; then a final polish on a low-speed "Microcloth"-covered wheel with a slurry of 0.3 micron alumina. The microstructure was revealed by a light etch in 2% nitric acid in alcohol.

Photomicrographs were taken with a Vickers M55 metallograph on Kodak Metallographic plates. Plate development was with Kodak D-19, diluted 1:1, for 3 min., while Velox contact prints were developed for 1 min. in Dektol diluted 1:2.

Preparation of Temper-Rolled Materials

To compare partially annealed stock to that produced by temper rolling, the mechanical properties of the material must first be found as a function of percent cold reduction. To eliminate specimen thickness as an experimental parameter, it was necessary to produce material of varying thicknesses so that upon cold rolling to 0.016 in., a range of cold reductions could be obtained. The rolling schedule to produce this material from Lot 1 is shown in Table II.

The 0.085 in. hot-rolled material was first cut into 2.5 x 7 in. strips and cold rolled in the longitudinal direction to the thicknesses shown in column 2 of Table II. All cold rolling was performed on a STANAT 2-high rolling mill, with 4 in. diameter rolls.

After cold rolling, the material was cut into 9 in. segments and packed tightly, with the thickest pieces on the outside, into a stainless steel annealing envelope. This envelope of trade name SEN-PAK, allows scale-free heat treatment of steels in furnaces that are not equipped with a protective atmosphere generation system. The rolled steel, within the envelope, was placed in a Leeds and Northrup Hump furnace and given an intermediate anneal for 50 min. at 670°C. Upon cooling from the annealing temperature there was a possibility of oxidation of the steel from air that could be sucked into the sealed envelope. To avoid this, the envelope

TABLE II

Rolling Schedule to Produce Temper-Rolled Material

Starting Material - Lot 1, hot-rolled strip, 0.085 in.
- hardness R 30T 59-61

Column 1	2	3	4	5	6
Desired Cold Reduction	Cold Rolled to	Anneal	Hardness after Anneal	Cold Rolled to	Final Cold Reduction
%	inches		R 30T	inches	%
80	0.0805	Intermediate Anneal at 670°C-50 min.	61	0.0157	80.5
70	0.0538		55	0.0157	71.0
60	0.0400		58	0.0152	62.0
50	0.0320		58	0.0155	51.5
40	0.0266		59	0.0153	42.5
30	0.0229		58	0.0156	32.0
20	0.0198		57	0.0150	24.0
15	0.0177		57	0.0148	16.5
10	0.0177		57	0.0158	10.5
5	0.0168		58	0.0158	6.0
1	0.0158(5)		57	0.0157(2)	1.0

was transferred directly from the furnace into a bath of unstirred quenching oil and left for 30 min. When the steel was removed from the envelope, it was free of oxidation, save for some bluing on the edges. The cooling rate was considered much too slow to have any quenching effect upon the material.

Rockwell superficial hardness measurements using the R 30T scale were made on each of the annealed segments. As shown in column 4 of Table II, the hardnesses were within the range of 55-61 R 30T. The lowest value of 55 R 30T was of the segment destined for 70% reduction. Microscopical examination revealed that all segments were fully subcritically annealed and that the piece with the lowest hardness had the largest grain size. This was in accordance with the long established inverse relationship between hardness and grain size of the same annealed material. The larger grain size of that particular segment was due to the effect of a critical amount of prior cold reduction on grain growth during annealing.

The annealed material was then cold rolled to the finishing thicknesses given in column 5 of Table II. All of the material thicknesses were measured with the ETALON micrometer. The final percent cold reductions which ranged from 1 to 80 percent are tabulated in column 6. This temper-rolled material was then cut and shaped into tensile test specimens. The mechanical properties were obtained (see Test Methods) and plotted against percent cold reduction.

Table II is the culmination of a great deal of trial and error to obtain material that could be tested properly and would be comparable to that manufactured in a plant. The remainder of this section will describe some of the difficulties encountered in producing this material.

To produce thin material, it was at first considered necessary to use a 4-high rolling mill with 0.75 in. diameter work rolls. Rolling of the steel strip under these conditions proved to be extremely difficult: hand feeding of the material to the mill provided no strip tension and thus the resulting cold-rolled strip was rippled. When this material was later tested in a tensile machine, it yielded and necked prematurely in several different spots which were clearly related to the ripples. No reliable testing data could be obtained and the 4-high mill arrangement had to be abandoned. To obtain any quantity of thin ripple-free material, a coiling apparatus should be used with the 4-high mill. Switching to a 2-high mill with 4 in. diameter rolls produced ripple-free material that tested properly.

Difficulty was also encountered in the choice of a temperature for the intermediate anneal. It was desirable—in order to be comparable to industrial practice—to have starting material of the same hardness and grain size as that produced commercially in a continuous annealing line. The hardness range of such material is 58-64 R 30T. This material, in conventional temper-rolling practice, would be reduced in thickness

by amounts ranging from 1% to as much as 50%, depending upon the strength and thickness required in the final product.

However, in order to eliminate grain size variations due to different prior rolling histories within a batch of material, consideration was given to a heat treatment in the austenitic range, i.e. at temperatures above 900°C where the $\alpha \rightarrow \gamma \rightarrow \alpha$ transformation cycle would swamp the prior rolling histories. Batches of material heat treated between 950°C and 900°C produced a hardness range of 47-51 R 30T. This was too soft and it became obvious that no treatment above 900°C would produce a grain size fine enough to enable the required hardness range to be met.

Annealing trials from 720°C to 670°C were however, more successful in producing a finer grain size, thereby increasing the hardness. Thus a final batch of cold-rolled material was intermediately annealed at 670°C to produce what was considered to be an acceptable 55-61 R 30T hardness range, after which, the material was cold rolled to the finishing thicknesses described previously in Table II. The small variation in grain size, within the batch, due to prior rolling histories was tolerated and fortunately its effect on testing results was minimal.

Preparation of Material for Partial Annealing.

Lot 1: To prepare material for partial annealing, the 0.085 in. hot-rolled strip was cut into 2.5 x 7 in. strips and cold rolled, longitudinally, to thicknesses of 0.0155 in. and 0.0073 in. to give reductions of 82% and 92% respectively. Most of the partial annealing work was carried out on 0.0155 in. material. Only five annealing tests on 0.0073 in. material were needed to indicate that the thickness had no bearing upon the response of the material to partial annealing.

Lot 2: This material had been cold rolled in large-scale plant production by the Steel Company of Canada Ltd. It had been cold reduced 82% from 0.090 in. to 0.016 in. and no additional preparation was needed.

In order to compare the properties obtained from partial annealing with those obtained from temper rolling, variables such as chemical composition and rolling procedure should be kept constant. This has been achieved by using Lot 1 for both sets of experiments. However, Lot 2 represented material cold rolled in plant production and its response to partial annealing treatments was also of great interest. Therefore, the temper-rolling experiments used Lot 1 only, while partial annealing was performed on both Lots 1 and 2. The highly cold-reduced material from both lots was cut and shaped into tensile test specimens which were then used for the partial annealing experiments.

Partial Annealing

(i) Technique

To partially anneal tinplate stock, a manufacturer would have to lower the temperature of his continuous strip annealer while maintaining the strip speed as high as possible to ensure maximum production. Since the strip annealer would run at a nearly constant speed, it was thought best to keep the experimental annealing time constant and of a convenient 10 min. duration. This was, of necessity, longer than the conventional annealing time of 60-90 sec. so as to avoid transfer and measurement errors. A reduction of annealing temperature compensated for the increased annealing time and should in no way affect the results of mechanical testing.

The specimens were artificially aged for 10 min. at 200°C after partial annealing in order to precipitate any excess carbon and nitrogen left in the alpha solid solution from the annealing treatment. Aging may not be necessary for specimens annealed below 540°C but to eliminate any chance of an aging factor, all annealed specimens were aged.

A preliminary working curve of hardness versus annealing temperature was first obtained. This entailed taking small test squares of the material and annealing for 10 min. in a salt bath at temperatures ranging from 440°C to 630°C. Using this curve as a guide, five to seven tensile

specimens were hung vertically in a jig and totally immersed in a salt bath. After 10 min. annealing, the jig and specimens were transferred directly to the aging salt bath.

(ii) Thermocouple Calibration

All temperature measurements were made with a portable chromel-alumel thermocouple combined with a Leeds and Northrup potentiometer. The thermocouple was calibrated against the thermal arrest plateaus on the cooling curves of the pure metals tin, zinc and aluminum. A thermos flask of ice and water maintained the cold junction at $0^{o}C$.

(iii) Partial Annealing.

A 5 in. diameter steel cylinder, 14 in. in height, containing the annealing salt, was placed into a Leeds and Northrup Hump furnace that had been fitted with a Honeywell "Brown Electronik" controller. The salt, Houghton Draw Temp 430, was a eutectic mixture of 54% potassium nitrate and 46% sodium nitrate. A controlling thermocouple was placed between the salt container and the furnace wall to ensure maximum sensitivity to temperature changes. Bath temperature measurements were made through a hole in the refractory cover with the portable thermocouple. At the lower temperatures used for partial annealing, there was a difference of $1^{o}C$ between top and bottom of the bath, while at the higher temperatures, thermal convection eliminated this gradient.

A lightly constructed steel jig to carry the tensile specimens, was made to slide down into the salt container. The specimens were hung vertically and spaced so that free circulation of salt around them ensured a maximum heat transfer rate. When the jig and specimens were plunged into the salt bath there was an initial temperature drop of approximately $5^{o}C$ within the first 30 sec. due to the mass of metal to be heated. However, after this initial drop there was always less than $1^{o}C$ variation over the 10 min. annealing time. Temperature measurements at the center of the bath were made each minute and with practised manipulation of the controller variac, nearly steady bath temperatures of $\pm$ $0.5^{o}C$ were achieved. After 10 min. annealing at various temperatures within the range $460^{o}C$ to $620^{o}C$, the jig and specimens were transferred to the aging bath.

(iv) Artificial Aging.

This second, somewhat larger salt bath, 12 in. in diameter, was heated by an electric coil within the bath, making continuous stirring necessary. A Bristol proportional controller maintained the bath temperature at $200 \pm 1^{o}C$. The salt, Houghton Draw Temp 275, was a mixture of 55% potassium nitrate and 45% sodium nitrite. After a 10 min. aging treatment, the jig and specimens were transferred into a bucket of hot water so that the adherent salt would be dissolved from the specimens.

(v) Pickling.

A very thin oxide layer formed on the specimens during annealing and was removed by pickling in 10% sulphuric acid at 45°C for 1 to 2 min. A 600 grit water-proof polishing paper was used to remove the black residue. The specimens were then thoroughly dried to prevent rusting of the newly polished surfaces.

(vi) Skin Passing.

All specimens that were partially annealed — even those annealed at the lower temperatures where only recovery had taken place — had their upper yield point restored. During subsequent tensile testing, plastic yielding in the form of Luder's bands, so thinned the specimens that their cross-sectional areas could not sustain the applied loads and the specimens broke prematurely. It was thus necessary to eliminate the upper yield point and lessen the severity of the Luder's bands by skin passing* the specimens.

Each annealed specimen was measured for thickness in three positions along the gauge length with the ETALON micrometer. The specimen was then carefully passed through the rolls of the 2-high mill several times and remeasured. Lateral curving and excessive reduction, especially in the softer material, frequently led to a loss of one or more specimens in each annealed set. It was found that 1.5% cold reduction was needed to

* Skin passing may be defined as a light cold reduction to eliminate the upper yield point with its associated testing problems, while the term temper rolling is reserved for cold reductions designed to increase the hardness and strength of the steel strip.

eliminate the testing problems on the 0.016 in. material, while 2.5% was needed on the 0.007 in. material. After skin passing, all specimens were stored until testing, in a freezer at -12°C to avoid strain aging.

The mechanical properties of the partially annealed material were obtained (see Test Methods) and plotted against annealing temperature.

RESULTS AND DISCUSSION

Introduction

In this section, the results of the temper-rolling and partial annealing experiments are presented, mainly in the form of graphs. A comparison of the results of partial annealing to temper rolling, especially the ductilities at equal strengths or hardnesses, then follows and lastly, the relationship between the mechanical properties and microstructures of the material is shown by a series of photomicrographs.

Temper Rolling.

The mechanical properties obtained from cold rolling low-carbon steel strip by various amounts up to 80% reduction are shown in Figures 9 and 10. As expected, there was an increase in strength and hardness and a decrease in ductility as the percentage cold reduction was raised. These variations are due to strain hardening which has been discussed previously in the theoretical section. The main purpose of the temper-rolling curves was to serve as standards with which the properties of partially annealed material could be compared.

Each plotted point of Figures 9 and 10, is the average of four tensile test results. The spread of the values that were averaged was very small and of the order of the triangle or square that surrounds the point. The largest spread of either yield or tensile strength was 2100 psi

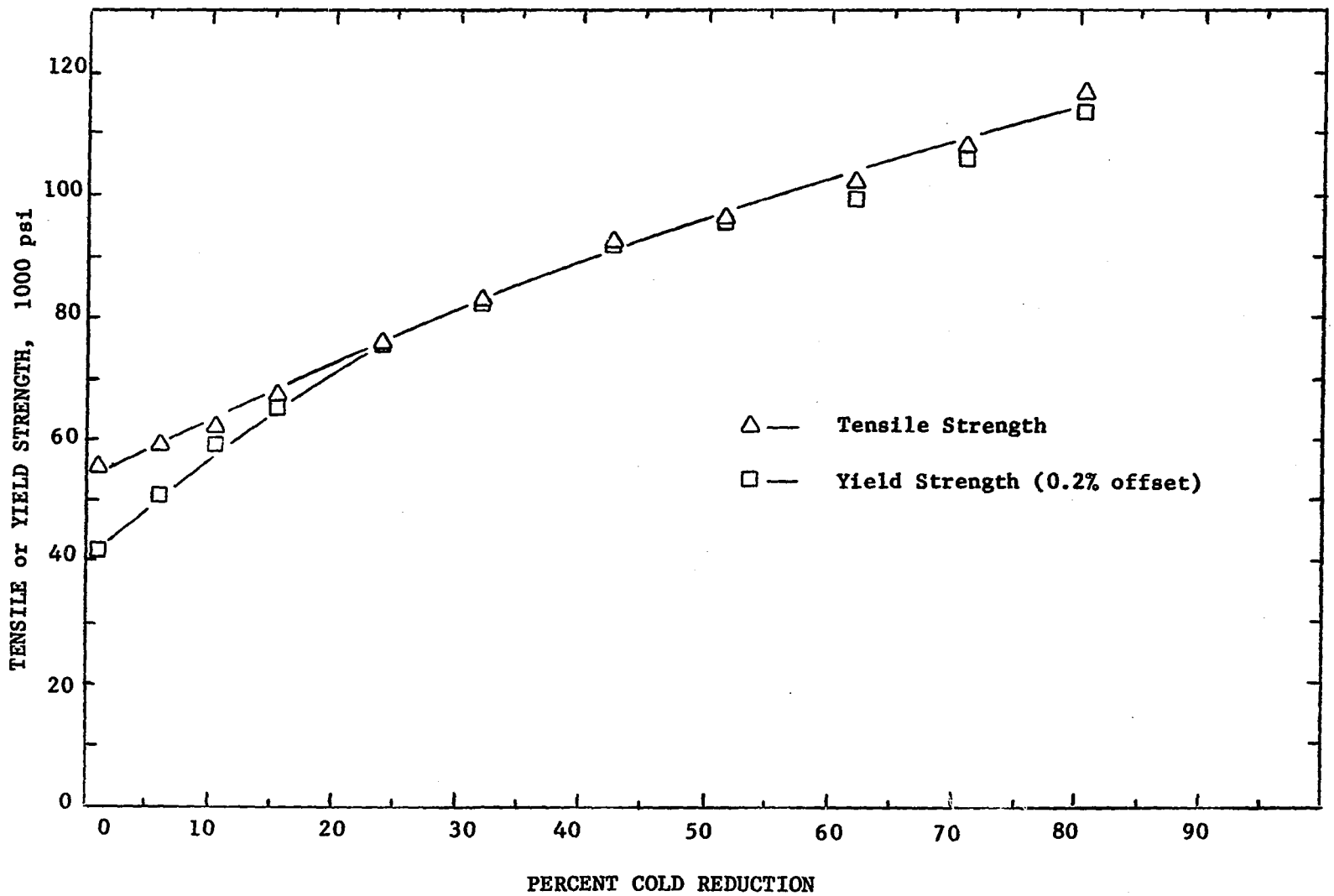

Figure 9. The Effect of Cold Rolling on the Tensile and Yield Strengths of Material from Lot 1.

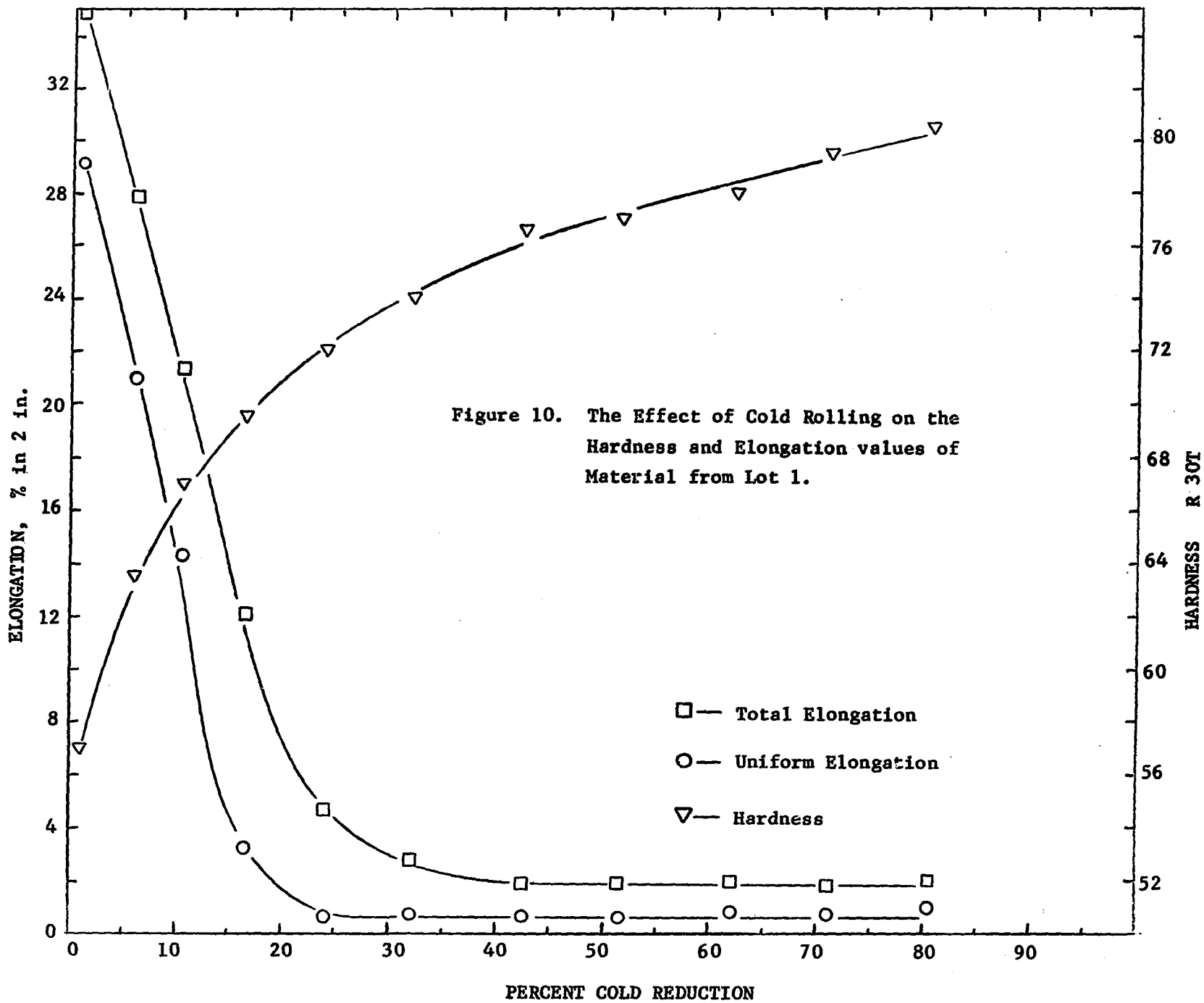

Figure 10. The Effect of Cold Rolling on the Hardness and Elongation values of Material from Lot 1.

and the spreads in the total and uniform percent elongations were 1% at the high values and 0.2% at the low values. Thus, limit bars on the graphs to indicate the spread of testing results are not necessary. The results for temper rolling are tabulated and shown in Table I of appendix A.

From Figure 9, it may be seen that the yield strength approaches the tensile strength as the percent reduction increased and that they became nearly equal after 24% cold reduction. The yield strength in the range of 60 to 80% reduction can be seen to drop slightly from the tensile strength; this behaviour appears to be a testing anomaly where the radius of the yielding knee of the load-extension curve, Figure 8a, p.44, becomes slightly larger for materials cold rolled to the higher reductions.

Figure 10, shows that the ductility drops very rapidly with cold reduction and that the uniform elongation reached its minimum value after only 24% reduction, i.e. at the same reduction as where the yield strength approached the tensile strength. By way of explanation, consider a tensile test on a soft material (it may be helpful to refer to Figure 8b, p.44), where two opposing factors operate to determine the load required for a given <u>plastic</u> extension. The first is the strain-hardening rate, which is dominant at the smaller extensions and leads to an increase in load, while the second is the decrease in cross-sectional area of the specimen as it elongates and leads to a decrease in the

required load. At larger extensions, the strain-hardening rate decreases and can no longer compensate for the decrease in cross-sectional area so that the load reaches a maximum and the specimen becomes plastically unstable (73). At this maximum load, any portion of the specimen that is slightly weaker will elongate slightly more than the rest of the specimen. This will tend to decrease the cross-sectional area and increase the local stress so that further local elongation will occur, quickly forming a thinned section or neck.

Consider now, rather than a tensile test, a cold-rolling operation which strain hardens the material and increases its yield strength at a higher rate than its tensile strength. Eventually, with increased percent reduction, the load needed to cause yielding in a subsequent tensile test becomes nearly equal to that which causes necking, i.e. to the maximum load from which the tensile strength is calculated. Remembering that uniform elongation is defined as the plastic elongation up to the onset of necking, it is evident that when the yield strength becomes nearly equal to the tensile strength, the specimen has very little capacity left for uniform elongation.

The positions of the curves in Figures 9 and 10, mostly depend upon the grain size of the starting material. A larger grain size would shift the strength and hardness curves downwards with respect to their present positions, while the elongations at the lower cold reductions, would increase (74). As discussed previously in the temper-rolling procedure, much experi-

mentation was needed in order to obtain the correct grain size (about 0.008 mm. average diameter) to give the desired hardness range of the starting material. This was necessary if the temper-rolling curves were to serve as comparative standards for partially annealed material.

Partial Annealing

The mechanical properties obtained from the partial annealing of 82% cold-reduced low-carbon steel strip, at various temperatures for 10 minutes, are shown in Figures 11 and 12 for Lot 1, and in Figures 13 and 14 for Lot 2. The strengths, both yield and tensile, and hardnesses fell as the annealing temperature was raised while the corresponding elongations increased.

As in the case of the temper-rolling curves, each plotted point on the partial annealing curves, Figures 11 to 14, was the average of four tensile test results. The spread of these averaged results was again very small and thus alleviated the need for limit bars on the graphs. In the range from 545 to 570°C, where the properties change most rapidly and are most difficult to control, the spread was always less than 3100 psi for the strengths, and 2% for the elongations, i.e. (20 ± 1)%. The partial annealing results for Lots 1 and 2 are tabulated and shown in Tables II and III respectively, of appendix A.

Inspection of the partial annealing curves shows that with increasing temperature, the material was recovering from its as-rolled condition up to about 520°C and then recrystallization began — as is evident from the steeper descent of the strength curves. One aspect that may be of commercial importance is that there is a significant measure of recovery before recrystallization begins. This may be seen from the slopes of the recovery portions of the curves up to 520°C. Estimates of percent recovery of the mechanical properties up to the onset of recrystallization are listed in Table III.

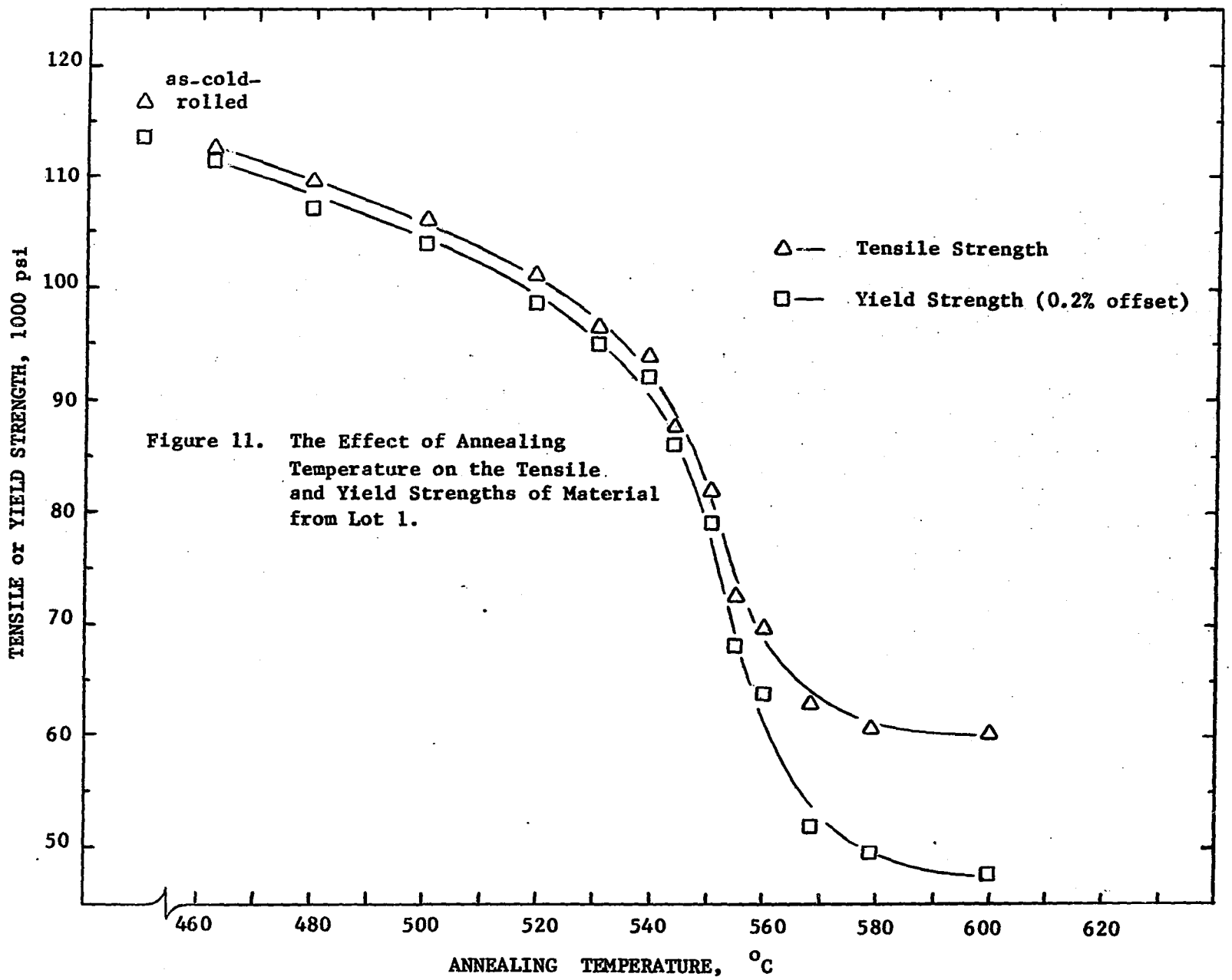

Figure 11. The Effect of Annealing Temperature on the Tensile and Yield Strengths of Material from Lot 1.

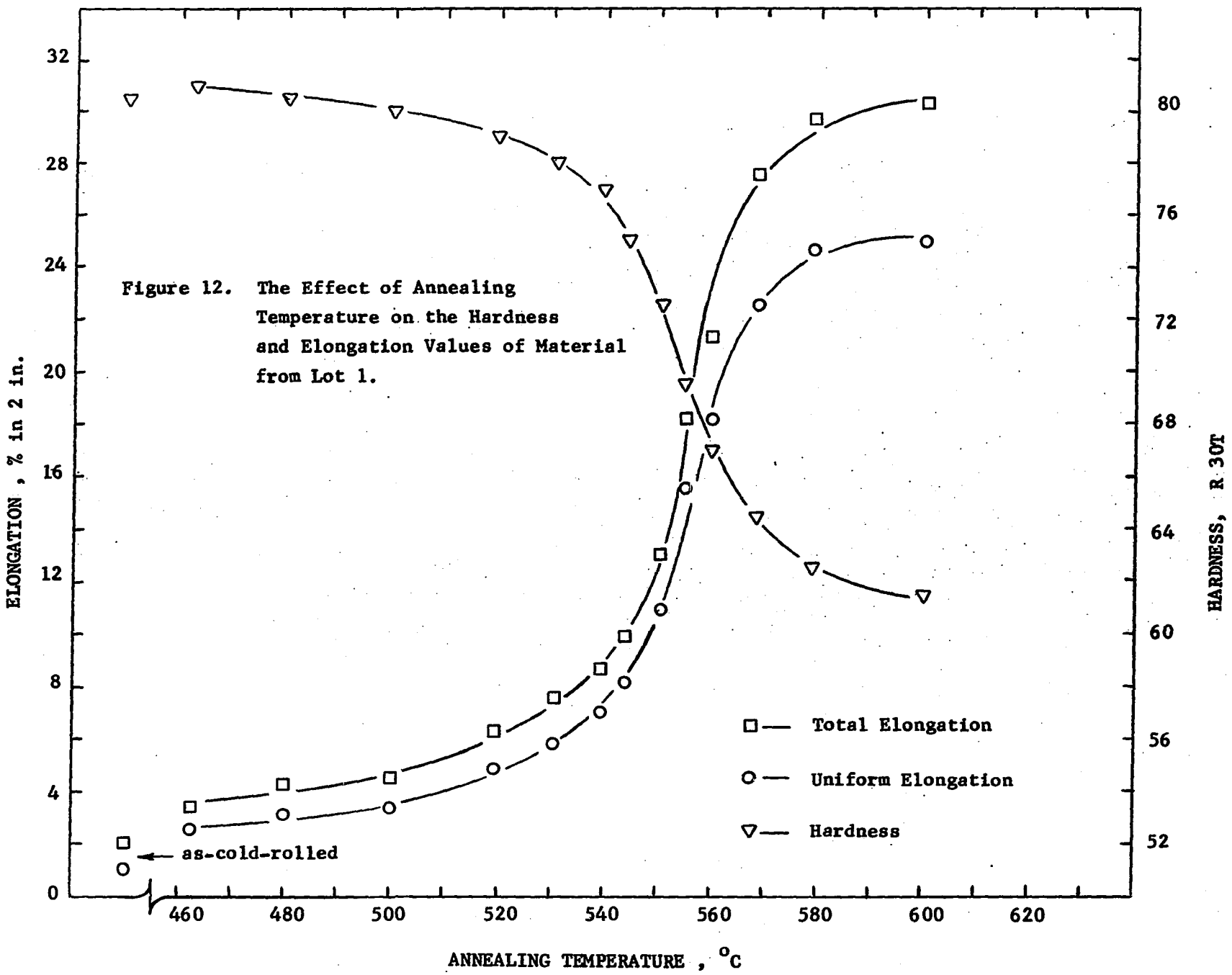

Figure 12. The Effect of Annealing Temperature on the Hardness and Elongation Values of Material from Lot 1.

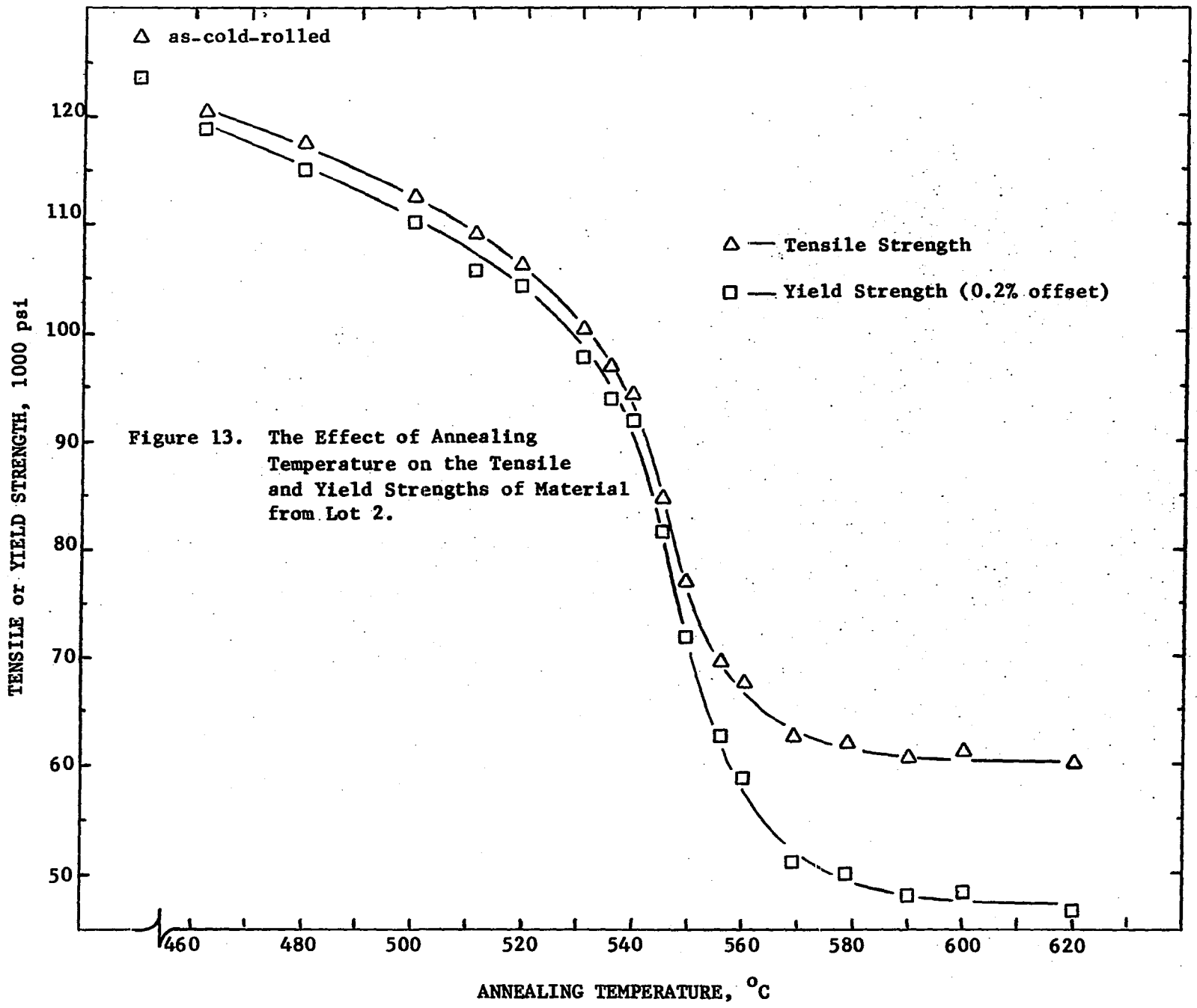

Figure 13. The Effect of Annealing Temperature on the Tensile and Yield Strengths of Material from Lot 2.

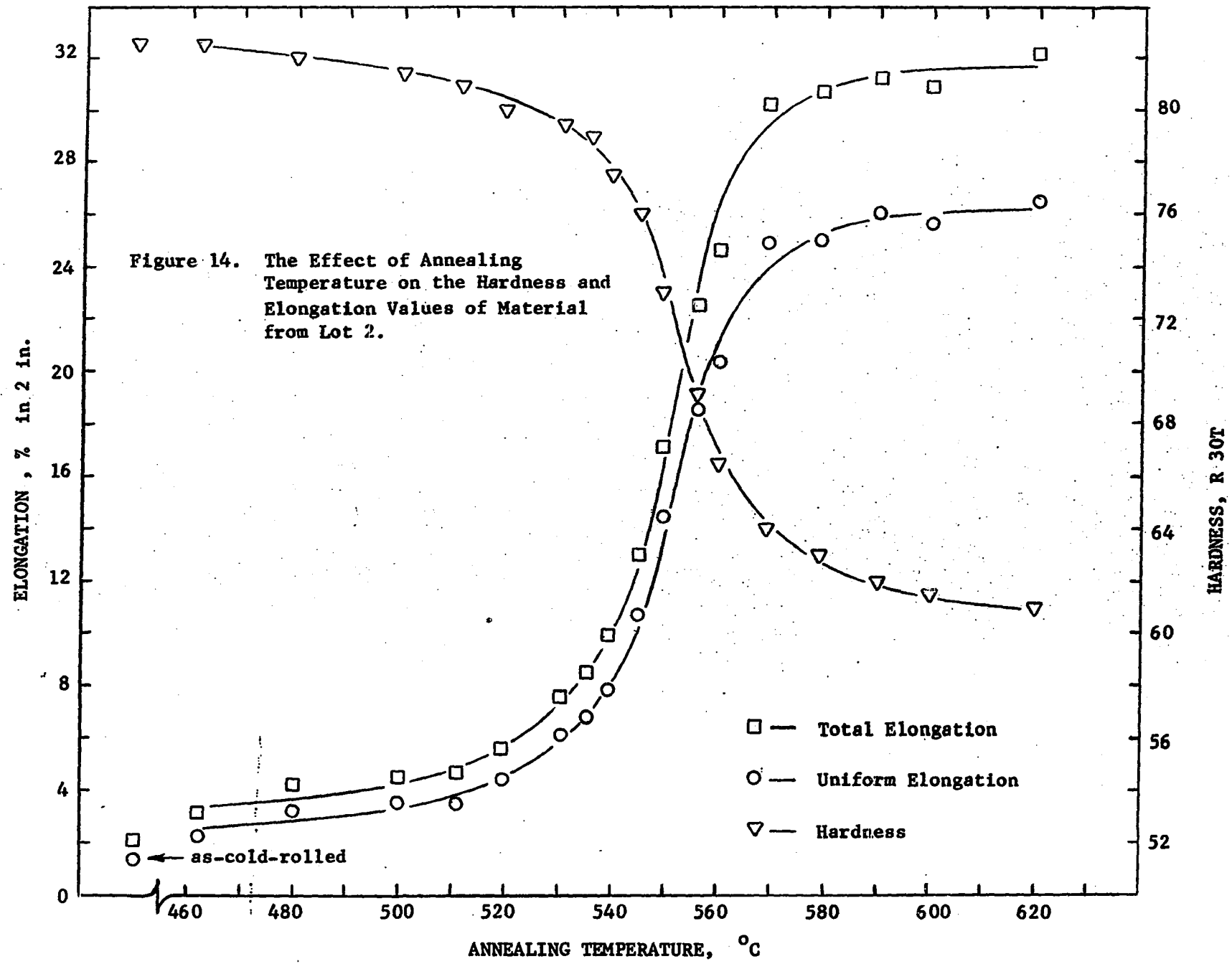

Figure 14. The Effect of Annealing Temperature on the Hardness and Elongation Values of Material from Lot 2.

TABLE III

Percent Recovery Up To 520°C

Property, average of Lots 1 and 2	After Cold Rolling	At 520°C	Fully Annealed	Percent Recovery
Tensile Strength, psi	121,000	104,000	60,000	30
Yield Strength, psi	118,000	102,000	48,000	23
Hardness, R 30T	82.5	80	61	12
Total Elongation, %	2	6	31	14
Uniform Elongation, %	1	5	26	15

The percent recoveries of both the total and the uniform elongations up to the onset of recrystallization do not convey two important facts: firstly, the total elongation rose from 2 to 6%, a 3 to 1 increase, and secondly, the uniform elongation increased from 1 to 5%. It thus appears possible to produce recovered, high-strength material with a greatly increased ductility over double-reduced material. The range of cold-fabricated products using recovered (partially annealed) high-strength material could be expanded because the increased ductility would enable more severe cold forming.

Recrystallization occurred above 520°C where new strain-free grains were nucleated and grew into the recovered matrix. This was accompanied by major changes in mechanical properties as shown by the steepness of

the curves, but when recrystallization was virtually complete, by about 580°C, the curves become nearly horizontal. These curves are typical of the shapes expected from isochronal recovery and recrystallization of metals.

When the strength curves of Lot 1 are compared to those of Lot 2, Figures 11 and 13, it may be seen that Lot 2 had a higher initial cold-rolled strength than Lot 1, although each had been cold reduced 82%. The spread in strengths narrowed with increased annealing temperature up to 540°C, beyond which temperature the curves superimposed.

The difference in cold-rolled strength between the two lots could be due to a difference in chemical composition, in grain size prior to rolling, or in the rolling procedure itself. Table I, page 39, shows that the carbon, manganese and chromium may have caused a minor increase in the cold-rolled strength of the material by an increased particle and solid solution hardening effect. There is no reason to suspect that there was a significant difference between the two lots of material in grain size (or hardness) of the hot-rolled strip prior to cold rolling. Thus, the extra strength of Lot 2 was not considered to be due to a difference in hot-rolled grain size. However, a major difference between the two lots was in the cold-rolling procedure where Lot 1 was rolled with a laboratory mill using 4 in. diameter rolls, while Lot 2 was rolled in large-scale plant production using say, 20 in. diameter rolls. The effect of roll diameter, strain rate, and reduction per pass during cold rolling on the strength of the ensuing steel strip is considered the major reason for the strength difference between the two lots.

Comparison of Partially Annealed to Temper-Rolled Materials.

(i) Comparison of Ductilities

The main objective of the present work was to compare mechanical properties, especially the ductility, of partially annealed to temper-rolled tinplate stock. The uniform and total elongations have been compared on the bases of equal tensile strengths, Figures 15 and 16, equal yield strengths, Figures 17 and 18, and equal hardnesses, Figures 19 and 20. It is evident from each of these comparisons that partially annealed material has elongations substantially higher than the equivalent temper-rolled material. Particular attention should be paid to the extraordinary improvement in the uniform elongation. Included in Figures 15 and 17 are the results of partially annealed 0.007 in. material (as opposed to 0.016 in.) which show that thickness had no bearing upon the response of the material to partial annealing.

Most successful forming operations using metal sheet depend upon the ductility of the metal before the onset of necking. This ductility, the uniform elongation, is frequently not determined even though it may be more significant than the total elongation to fracture in assessing the ductility of a particular material. Only in applications, say in flanging or in the forming of lock seams, where the elongations are very local, would there be a use for ductility beyond the onset of necking. This present work emphasizes uniform elongation as a more important measure

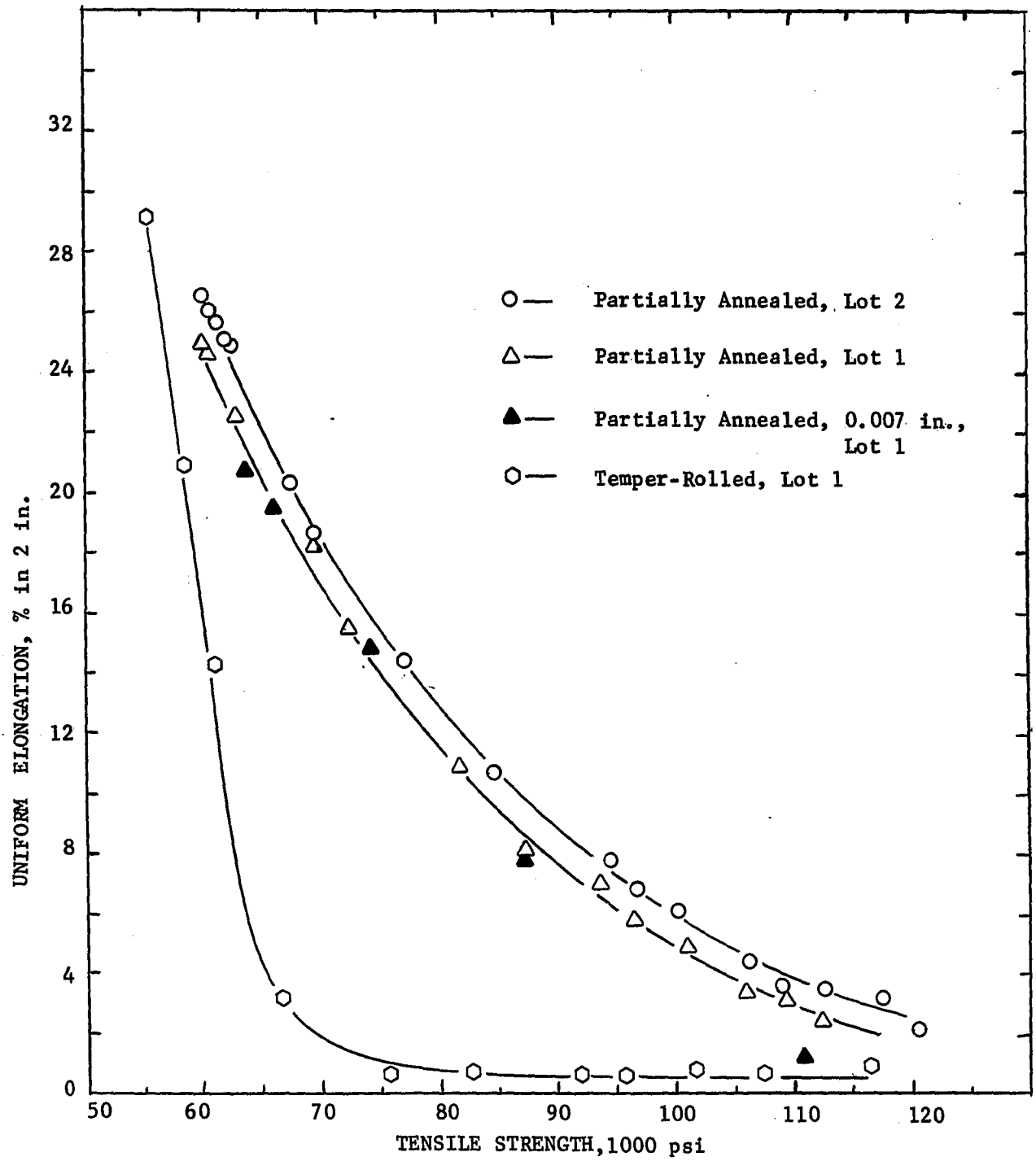

Figure 15. Comparison of Uniform Elongations of Partially Annealed to Temper-Rolled Tinplate Stock at Equal Tensile Strengths.

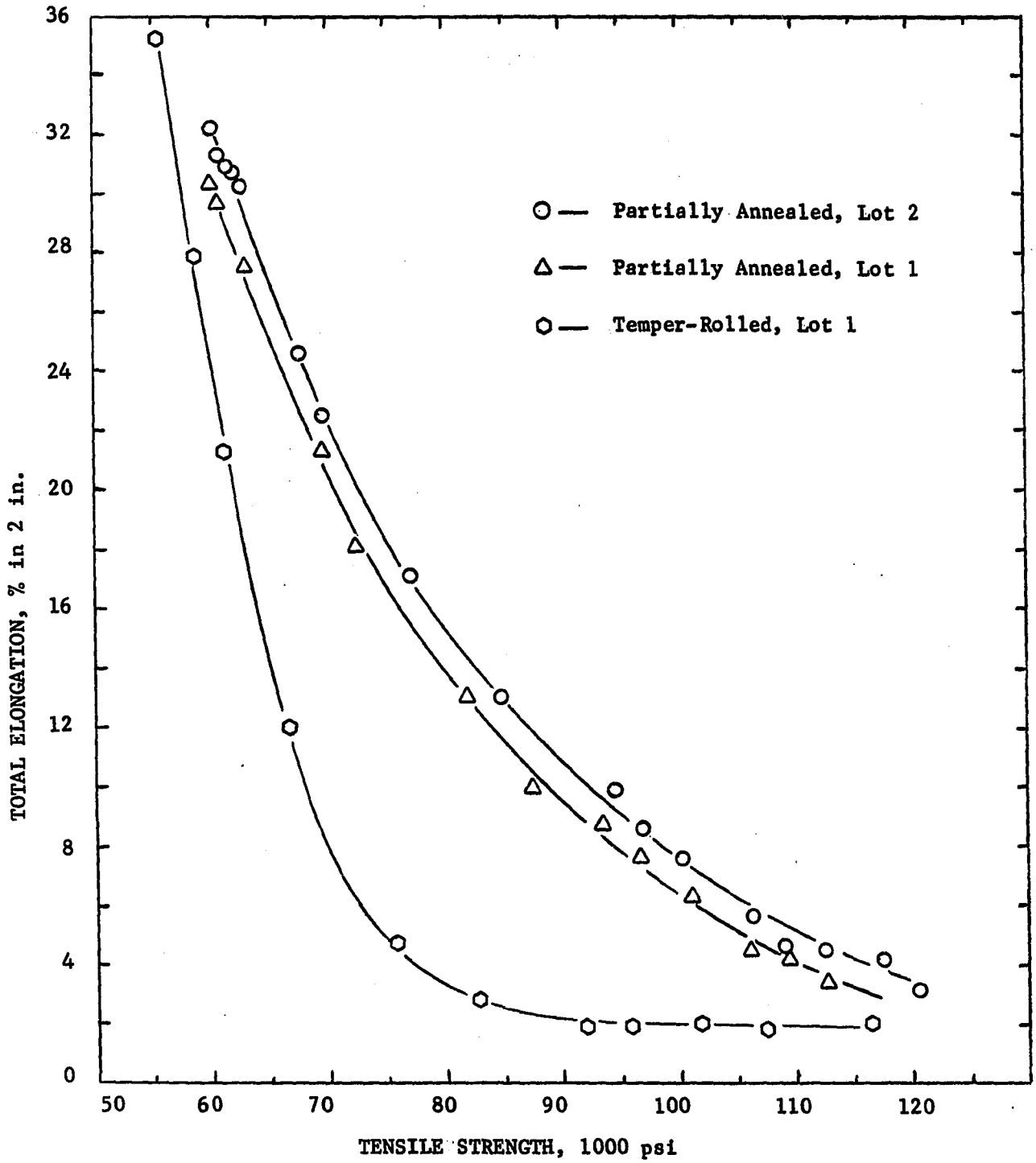

Figure 16. Comparison of Total Elongations of Partially Annealed to Temper-Rolled Tinplate Stock at Equal Tensile Strengths.

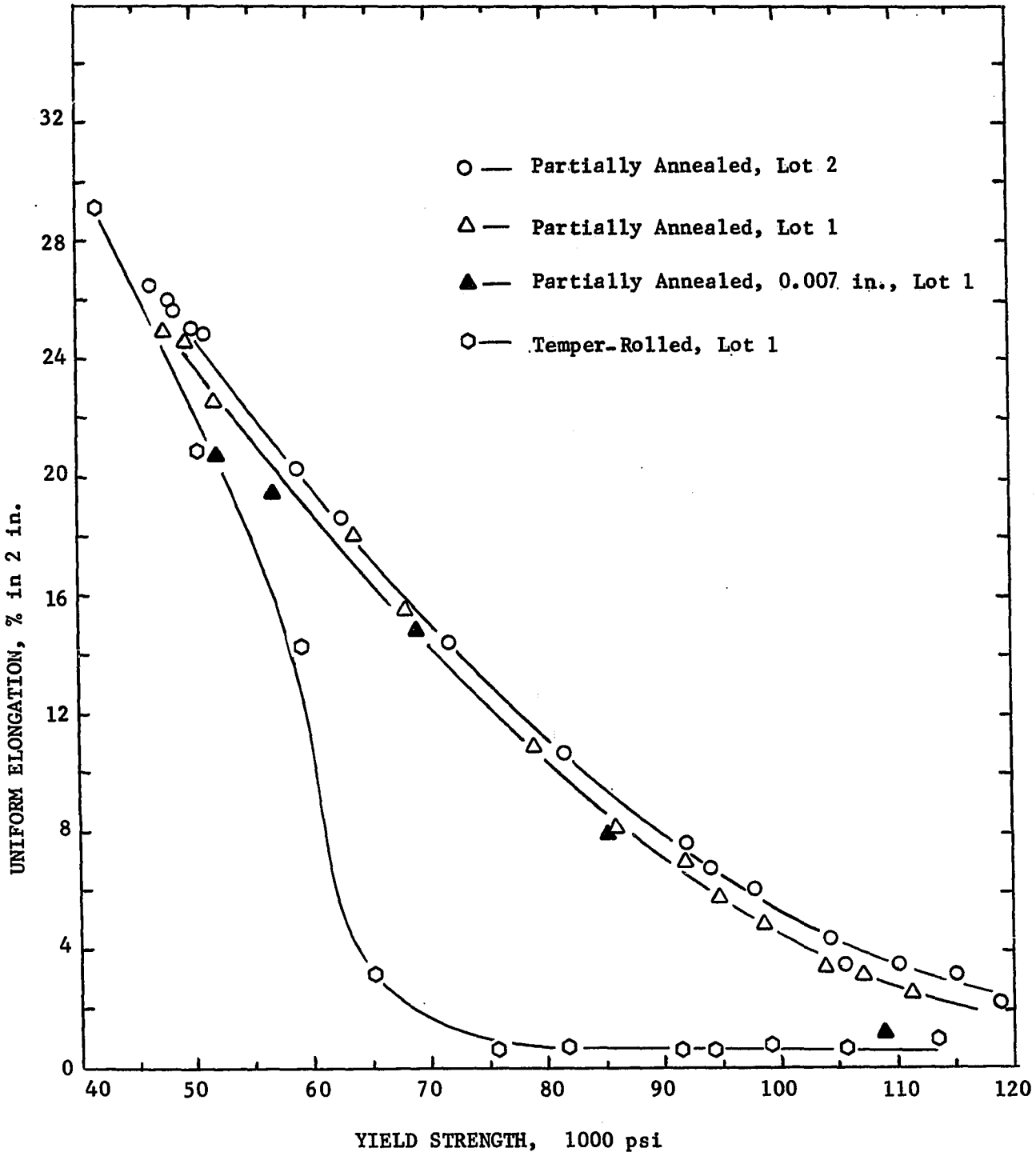

Figure 17. Comparison of Uniform Elongations of Partially Annealed to Temper-Rolled Tinplate Stock at Equal Yield Strengths.

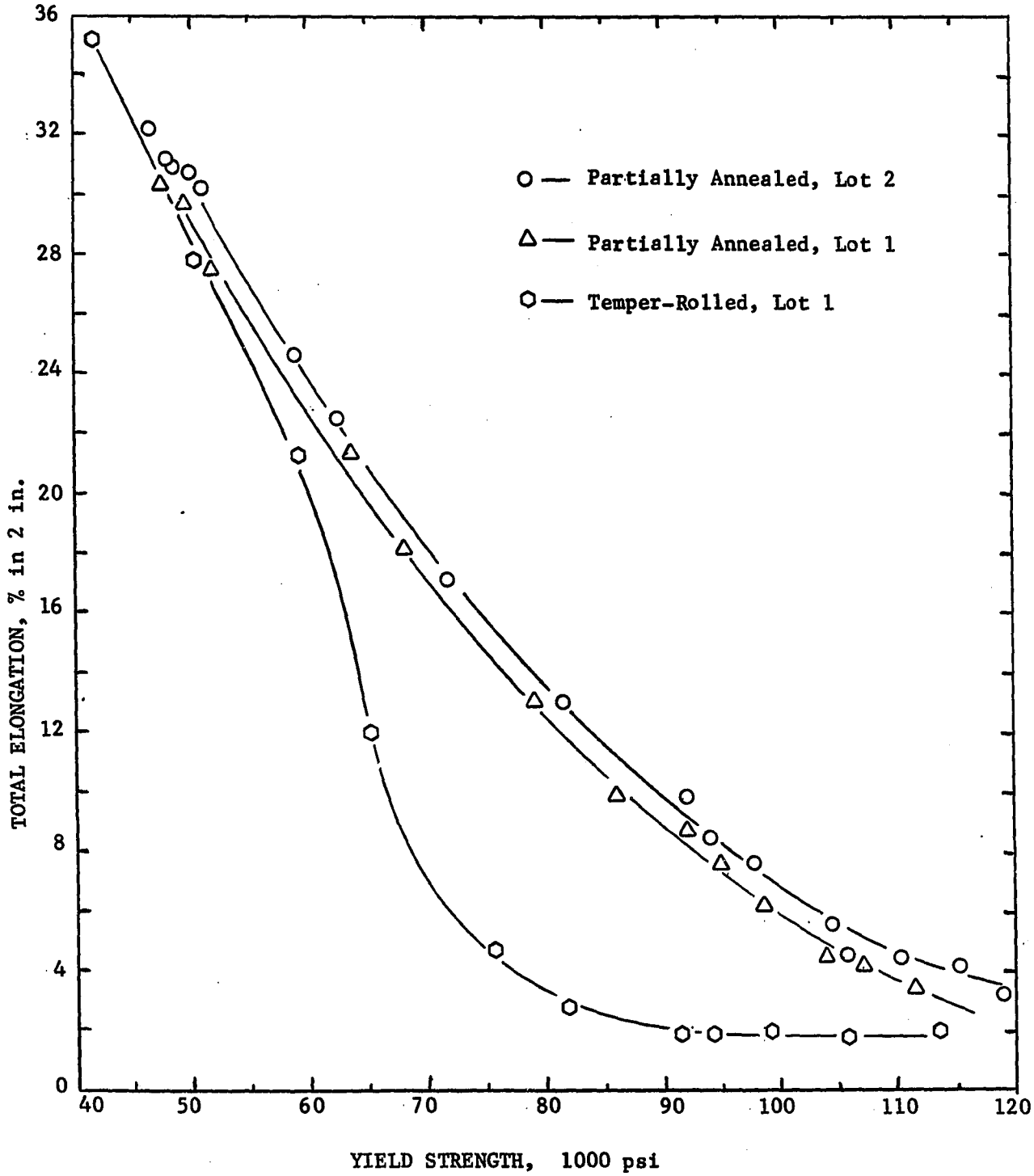

Figure 18. Comparison of Total Elongations of Partially Annealed to Temper-Rolled Tinplate Stock at Equal Yield Strengths.

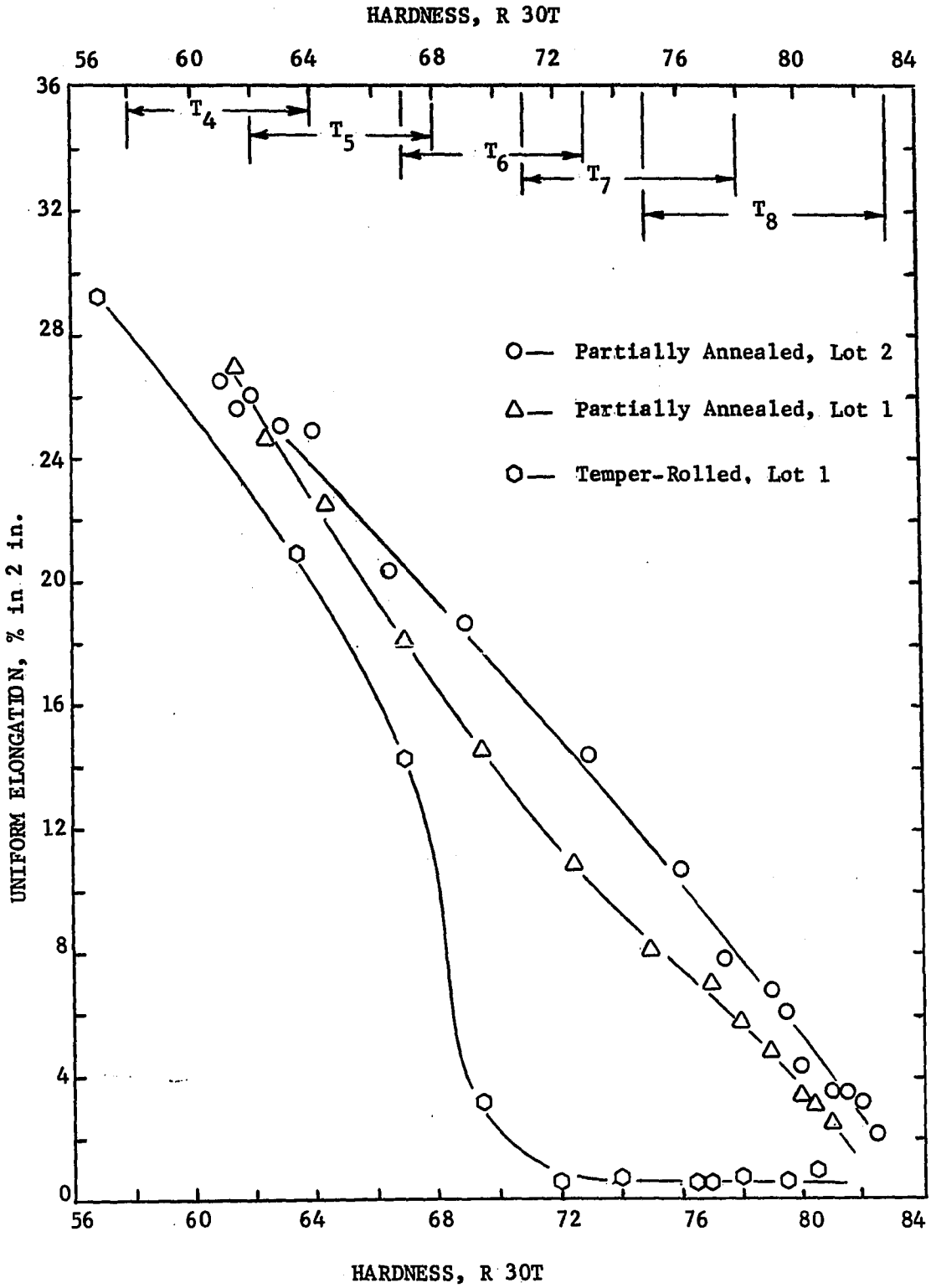

Figure 19. Comparison of Uniform Elongations of Partially Annealed to Temper-Rolled Tinplate Stock at Equal Hardness Values.

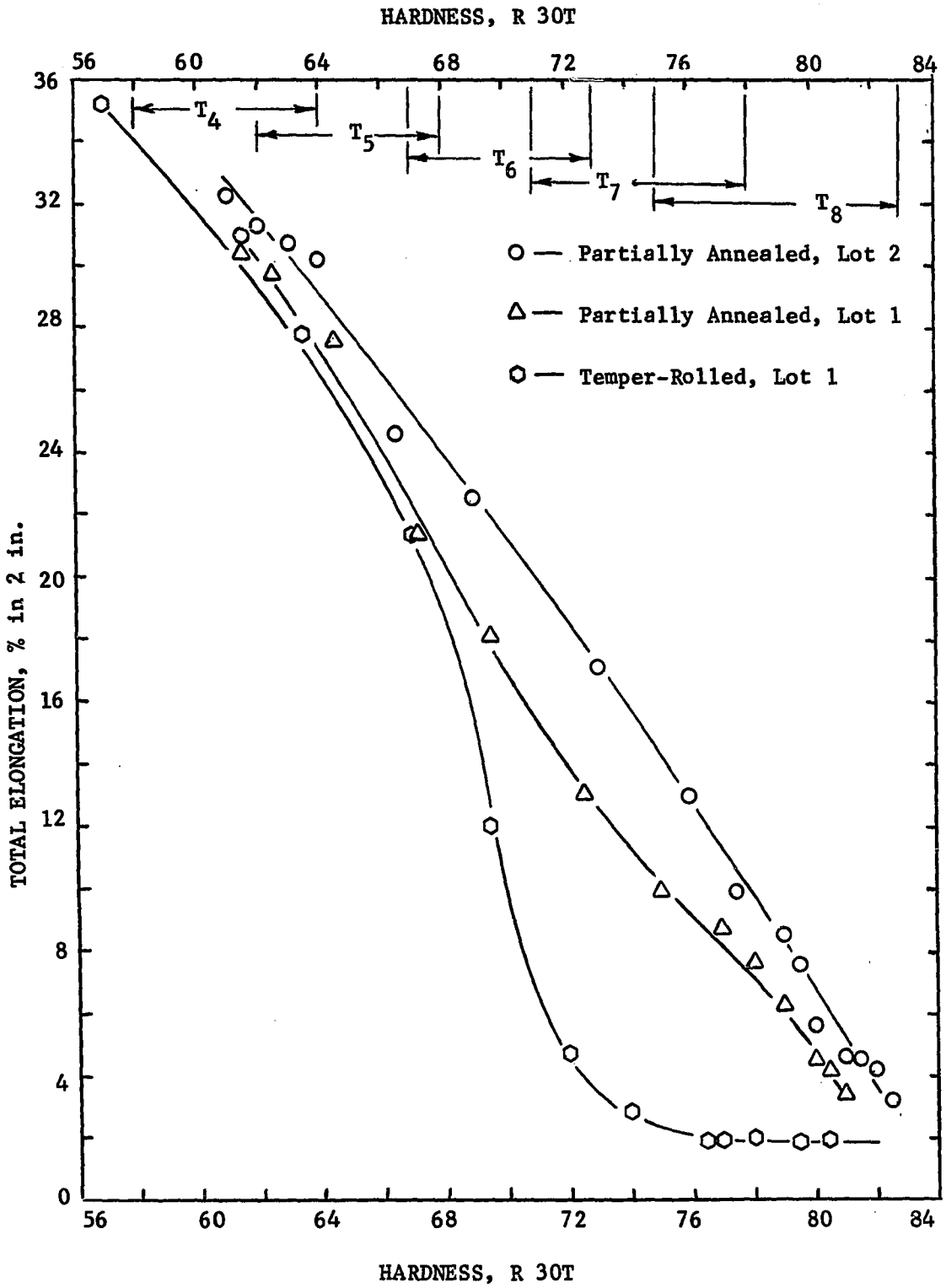

Figure 20. Comparison of Total Elongations of Partially Annealed to Temper-Rolled Tinplate Stock at Equal Hardness Values.

of ductility but since total elongations are traditionally quoted, they have also been used to compare the ductilities.

In comparing the elongations of each type of material, three bases were used; tensile strength, yield strength and hardness. The validity and usefulness of these criteria depend upon the needs of the tinplate producer and of the canmaker. Equal tensile strength was considered an important criterion for comparison of ductility, since uniform elongation and tensile strength are dependent upon the strain-hardening ability of the material. Clearly, in cold-forming operations using metal sheet, the uniform elongation ceases at the point of maximum load from which the tensile strength is calculated, and thus a comparison at equal tensile strengths could indicate relative cold-forming ability. However, it is the yield strength of the material that is the major consideration in applications such as pressure pack cans, where yielding or expansion of the can during use is undesirable. A canmaker then, would be interested in both equal tensile and equal yield strengths as comparative bases for ductility.

Hardness measurements (R 30T), form the basis of production control and temper classification of continuously annealed strip. It was primarily for these reasons that equal hardness was also used as a comparative basis for the ductility of temper-rolled and partially annealed material. The hardness ranges for tempers 4 to 8 are indicated on the elongation

versus hardness graphs of Figures 19 and 20 mainly to serve as a guide to the comparative ductilities for each temper. There are serious doubts however, about the usefulness of the equal hardness basis as an experimental method for research. Superficial hardness tests (R 30T) do not appear particularly sensitive and if there was any amount of scatter in either the hardness or elongation results, Figure 20, one might be led to an erroneous conclusion about the comparative ductilities. Uniform elongations at equal tensile or equal yield strengths appear to be the best ways to compare partially annealed to temper-rolled material.

The curves in Figure 15, form an almost closed loop and would have done so exactly if the grain size of the starting material for temper rolling had been equal to that which resulted from the annealing experiments. Starting material grain sizes larger than that resulting from the annealing experiments, open the top end of the loop by changing the slope of the temper-rolling curve in the manner shown in Figure 21. A material with a large grain size strain hardens more uniformly over its plastic range and so loses its ductility during cold rolling at a lower rate than material with a fine grain size (69,74). There is also the Hall-Petch equation (74) which shows that the strength of the material will decrease with increasing grain size. Thus, from a combination of the above two facts, the shape of the temper-rolling curve, Figures 15 and 21, is related to the grain size of the starting material. It was later realized after an examination of all of the results, that a completely

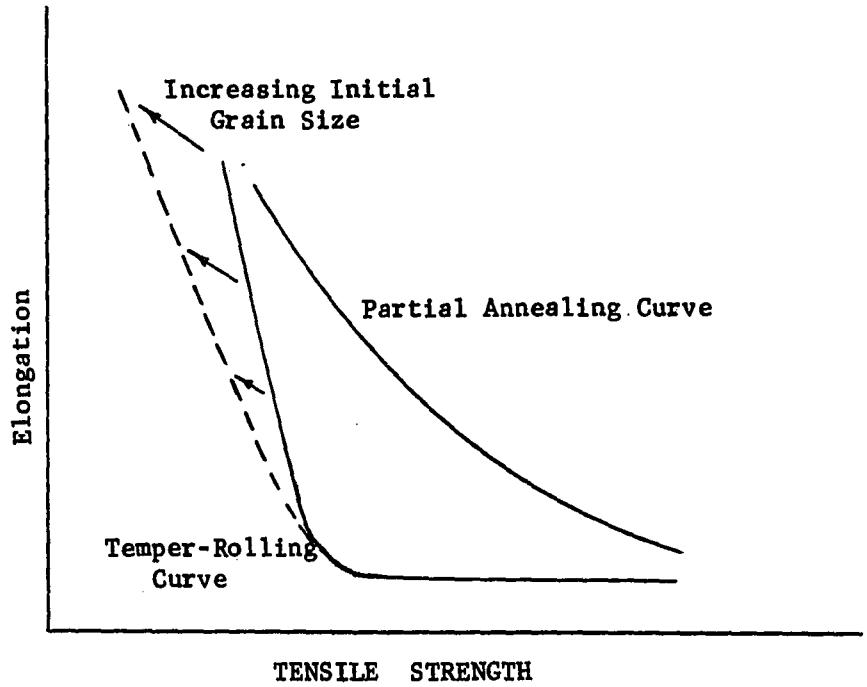

Figure 21. Effect of Grain Size on Temper-Rolled Material.

closed loop would have been ensured if the intermediate anneal given to the starting material for temper rolling (Table II, p.49) had been at a somewhat lower temperature than 670°C.

Comparison of the ductilities of the very soft materials resulting from either temper rolling or partial annealing is sensitive to a difference in grain size between the starting and finished materials of the two processes. An error resulting from non-closure of the loop is a distinct possibility but it would be significant <u>only</u> if the gap was wide and if the very soft materials were being compared. However, no error due to a grain size difference is encounted when comparing the stronger materials above a tensile strength of 70,000 psi which are of more practical interest.

The increased ductility of partially annealed material may be theoretically explained in terms of dislocation interactions during cold working, recovery and recrystallization. Consider for example, (a) temper-rolled material cold reduced 50% to obtain a tensile strength of 96,000 psi and a yield strength of 95,000 psi in comparison with (b), partially annealed material which had been initially cold reduced 82% to a tensile strength of 116,000 psi, then annealed for 10 min. at 530°C to produce the equivalent material.

(a) During temper rolling, the material developed a cold-worked cellular structure with a high density of dislocations in the cell walls and in the cell interiors. This high dislocation density, especially in the interiors of the cells, impeded further dislocation motion and as a result, during tensile testing or any other type of cold forming, the material could not strain harden rapidly enough to offset the start of necking. This material had less than 1% uniform elongation.

(b) After an initial cold reduction of 82%, this second material was stronger, had a higher dislocation density and also had less than 1% uniform elongation. During partial annealing, recovery mechanisms reduced the dislocation density of the cells and formed subgrains with nearly dislocation-free interiors. The tensile strength of the material was reduced by recovery to the desired 96,000 psi and the yield strength, while reduced accordingly, remained near the tensile strength at 95,000 psi. When this material was tested in tension (or cold fabricated), the sub-

grains had the capacity to strain harden at a rate which prevented necking for a considerable amount of strain. Thus, this material of equal tensile strength and equal yield strength had a uniform elongation of 6% as opposed to less than 1% for temper-rolled material.

If the partial annealing process had been continued past recovery to enable new strain-free recrystallized grains to nucleate and grow into the recovered matrix, the dislocation density would decrease even further. Also, since growing strain-free grains increase in size and volume, the yield strength would fall from the tensile strength in proportion to the percent recrystallization. This lower dislocation density and increased strain-free volume would allow the material to flow plastically at a lower stress level; to strain harden at a more uniform rate; and to elongate uniformly over a greater stress range than temper-rolled material of equal tensile strength.

(ii) Comparison of Yield-to-Tensile Strength Ratios

The yield-to-tensile strength ratio can be associated with the amount of plastic flow that can occur in a metal sheet before fracture. In forming operations which require considerable plastic flow of the metal, a low value of the ratio is desirable in order that the material may yield at a relatively low stress level, then work harden during elongation (forming) up to its tensile strength. Conversely, a high value of the ratio indicates that the material yields near the tensile strength and has very little capacity for uniform elongation.

When the yield-to-tensile strength ratios of partially annealed and temper-rolled materials are plotted against tensile strength, as in Figure 22, it may be seen that the ratio for partially annealed material is always less than that for temper-rolled material at equal tensile strengths. This lower ratio supports the theoretical explanation for increased ductility put forward in the previous section. The ratio for the temper-rolled material may be seen to decrease from nearly unity at the higher strength levels, but this is due to the testing anomaly discussed previously in the section on the results of temper rolling. Also, the fact that the elongation values at these high strength levels remain at their minimum, indicates that the curve should have continued horizontally at a value near unity.

Thus, with a lower yield-to-tensile strength ratio at equal tensile strength, it could be expected that partially annealed material would have an improved capacity for cold fabrication over temper-rolled material of any strength level.

(iii) Comparison of Strain Ratios

The strain ratio r, a parameter of normal plastic anisotropy, defined previously in the theoretical section as the ratio of natural strains in the width and thickness directions for a specimen extended in tension, is a direct measurement of the resistance of the sheet to thinning during a forming operation. The r-values for the temper-rolled and partially

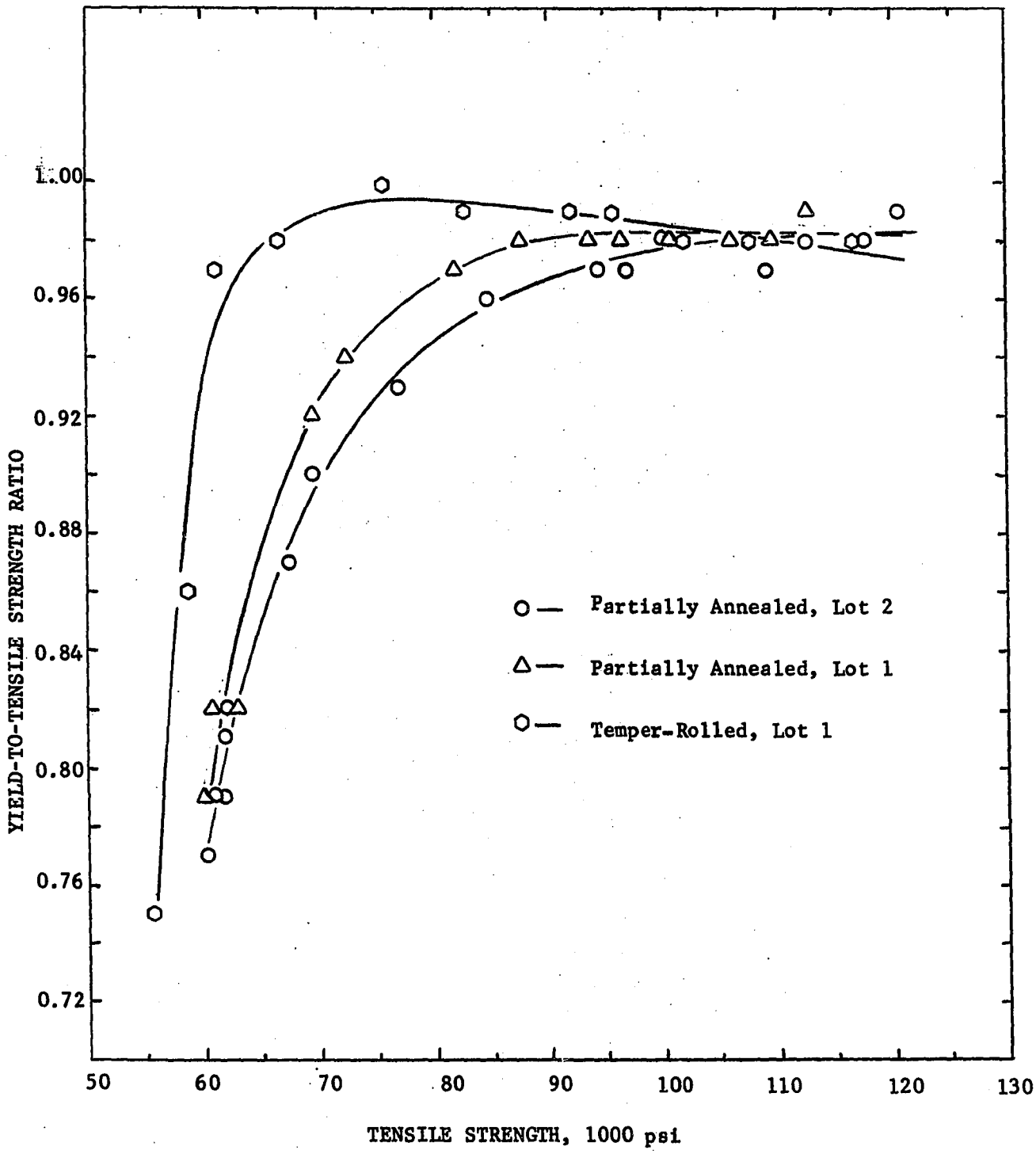

Figure 22. Comparison of Yield-to-Tensile Strength Ratios of Partially Annealed to Temper-Rolled Tinplate Stock at Equal Tensile Strength

annealed materials are listed in Tables I,II and III of appendix A.

A comparison of the strain ratios of partially annealed to temper-rolled material at equal tensile strengths is shown in Figure 23. It may be seen that only the very soft, fully annealed materials behave in a nearly isotropic manner ($r \approx 1$); both partially annealed and temper-rolled materials have r-values between 0.4 and 0.7, i.e. they thin more readily in the thickness direction than in the width direction. From these results, it must be concluded that partial annealing holds no advantage over temper rolling so far as the resistance to thinning is concerned*.

The two points designated CR in Figure 23, represent the cold-rolled starting material from each lot and it may be seen that the r-value decreased after a small amount of recovery. This may be explained by the fact that all of the heat-treated specimens had their upper yield point restored and that even after skin passing, several small Lüder's bands travelled along the specimen during tensile testing. These Luder's bands decreased the cross-sectional area of the specimen homogeneously, i.e. they thinned the specimen in the width and thickness

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* Blickwede (69) indicates that an r-value below 1.0 should be expected for the fine-grained material produced in this work. Burns and Heyer (66) found an r-value of 0.50 for 60% cold-reduced rimmed steel recovered at 455°C. Since the r-values of this present work agree with values obtained by others, the unconventional method used in this project to obtain these values may be said to present a fair and just assessment of the materials.

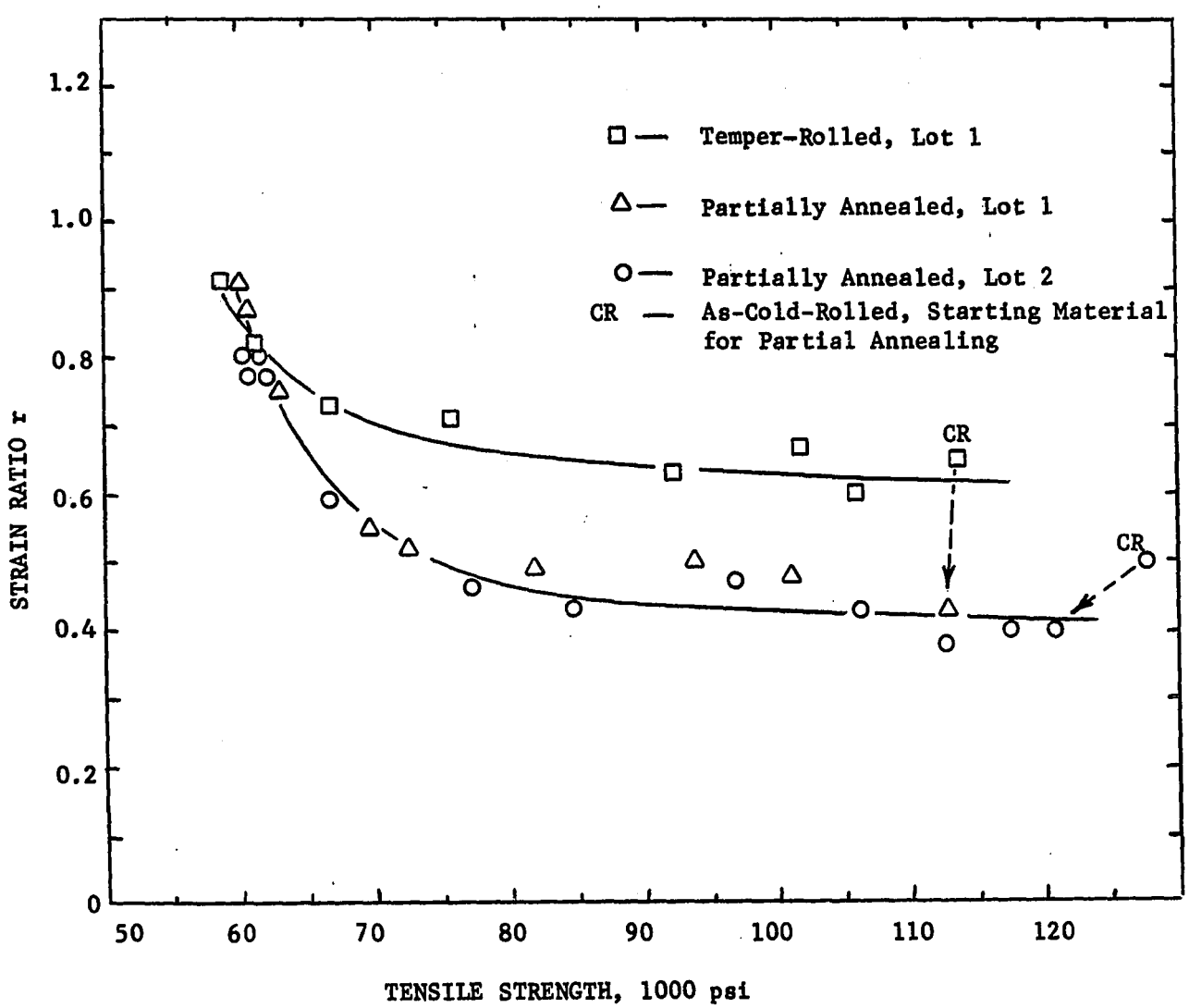


Figure 23. Comparison of Strain Ratios of Partially Annealed to Temper-Rolled Tinplate Stock at Equal Tensile Strengths.

directions by the same amount. Since the specimen thickness was very small when compared to its width, an exaggerated thickness strain resulted and thus a lower r-value was recorded. The temper-rolled material, on the other hand, did not exhibit Luder's bands and thus the r-values for this material could be considered more correct.

(iv) Comparison of Microstructures

Representative tensile specimens from Lot 1 were chosen to correlate mechanical properties with microstructures and to compare the microstructures of partially annealed to temper-rolled material at similar tensile strengths. The specimens were chosen in the manner shown in Figure 24, where pairs nearly equal in strengths, have been matched and identified by the letters a to g. Figures 25 (a-g), upper photomicrographs, illustrate the partially annealed microstructures of materials decreasing in strength. Figures 26 (a-g), lower photomicrographs, are of the equivalent temper-rolled materials.

The partially annealed specimens show the various stages of recovery and recrystallization. Attention is drawn to the facts that the recovered material etches faster and darker than cold-worked material and that the inclusion shown in Figure 25b, nucleated the first strain-free grain which had begun to grow into the recovered matrix. The temper-rolled specimens are seen to consist of grains that become increasingly distorted in the rolling direction as the amount of cold reduction is increased.

Figures 25g and 26g illustrate the difference in grain size between the finished material from the annealing experiments and the starting material for temper rolling. The average grain diameters were estimated by the intercept method and are given with each photomicrograph. These microstructures are typical of subcritically annealed low-carbon steel strip in which the grain size is very fine and the cementite particles appear as stringers parallel to the cold-rolling direction.

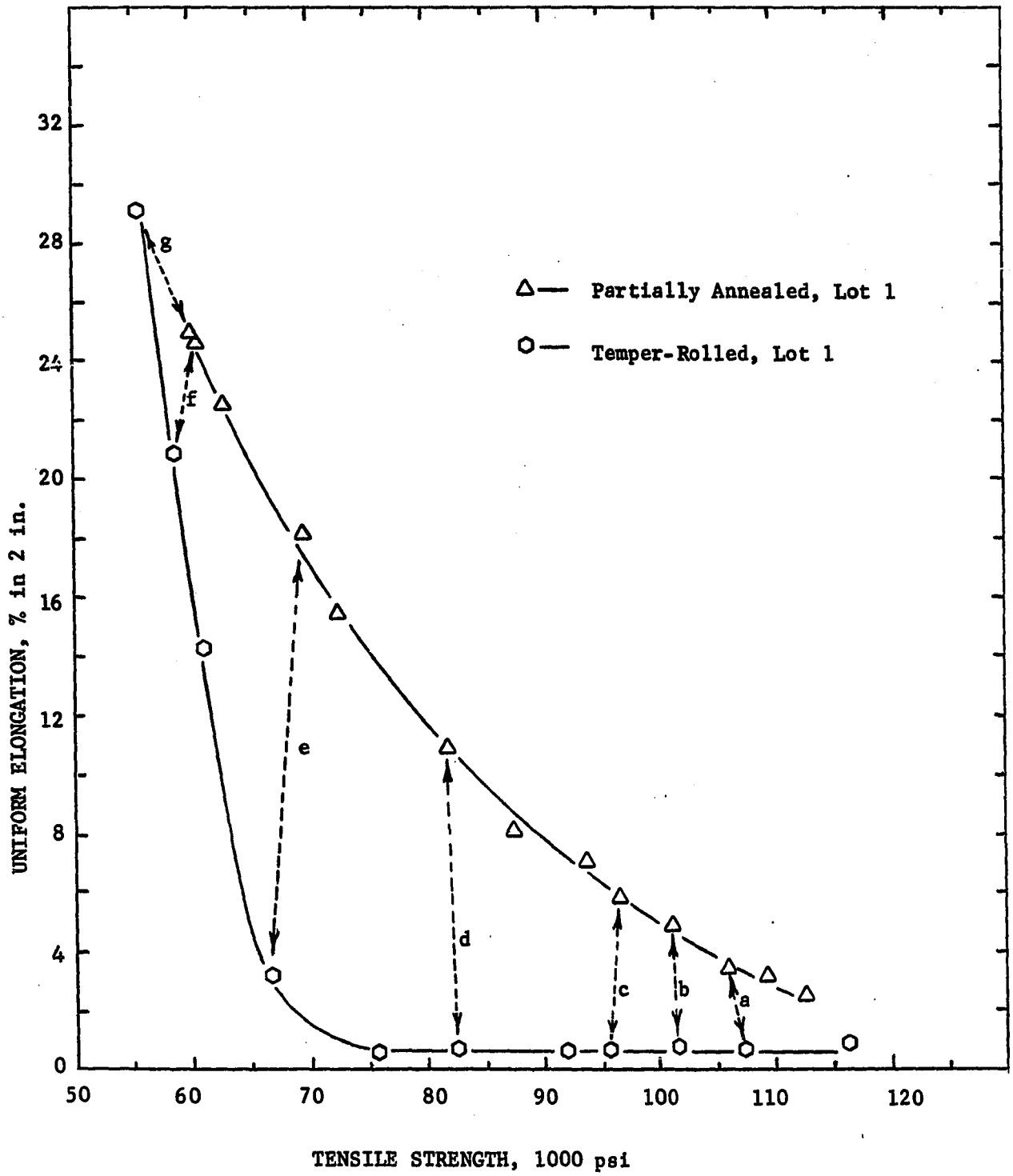
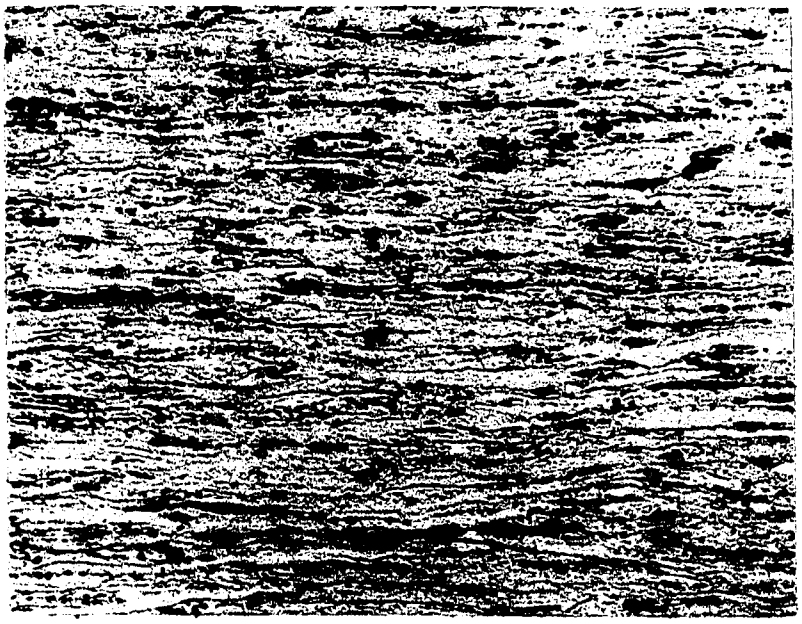


Figure 24. Selection of Specimens for Metallographic Examination.



YS 103,900 psi
TS 106,000 psi
Tot.Elong. 4.5%
Unif.Elong. 3.4%
Hardness 80R30T

Figure 25a. Annealed at 500.0°C. x 750

YS 105,700 psi
TS 107,600 psi
Tot.Elong. 1.8%
Unif.Elong. 0.7%
Hardness 80R30T

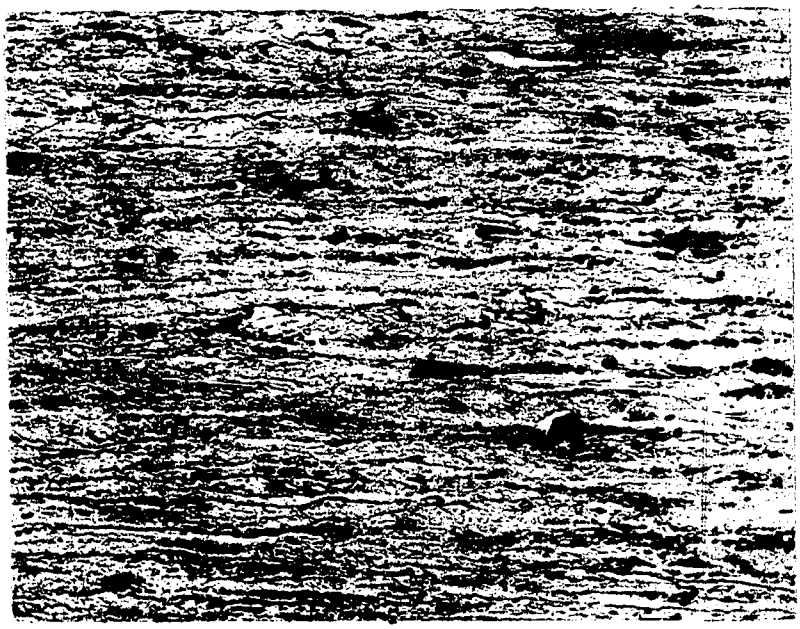
Figure 26a. Temper Rolled 71.0% x 750

YS 103,900 psi
TS 106,000 psi
Tot.Elong. 4.5%
Unif.Elong. 3.4%
Hardness 80R30T

Figure 25a. Annealed at 500.0°C. x 750

YS 105,700 psi
TS 107,600 psi
Tot.Elong. 1.8%
Unif.Elong. 0.7%
Hardness 80R30T

Figure 26a. Temper Rolled 71.0% x 750



YS 98,500 psi
TS 101,000 psi
Tot.Elong. 6.3%
Unif.Elong. 4.9%
Hardness 79R30T

Figure 25b. Annealed at 519.5°C. x 750

YS 99,200 psi
TS 101,800 psi
Tot.Elong. 2.0%
Unif.Elong. 0.8%
Hardness 78R30T

Figure 26b. Temper Rolled 62.0% x 750

YS 98,500 psi
TS 101,000 psi
Tot.Elong. 6.3%
Unif.Elong. 4.9%
Hardness 79R30T

Figure 25b. Annealed at 519.5°C. x 750

YS 99,200 psi
TS 101,800 psi
Tot.Elong. 2.0%
Unif.Elong. 0.8%
Hardness 78R30T

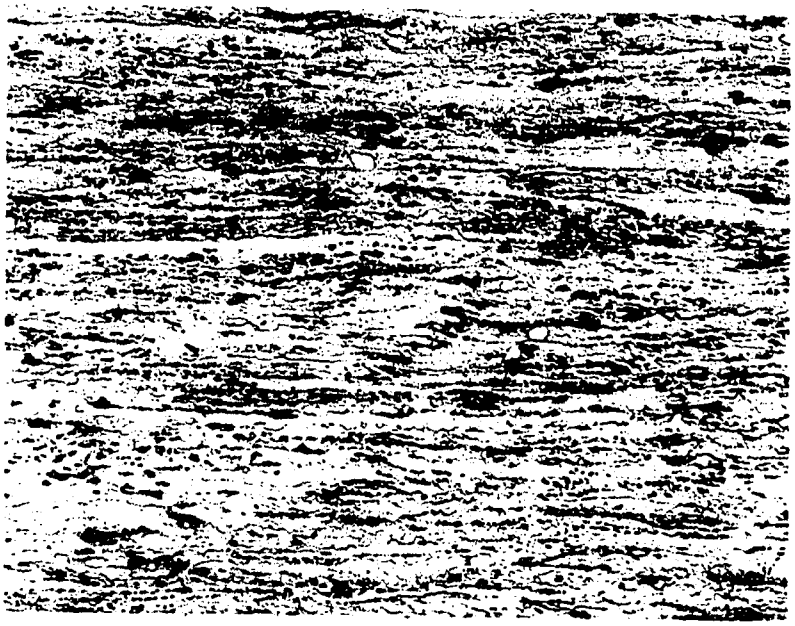
Figure 26b. Temper Rolled 62.0% x 750

YS 94,900 psi
TS 96,500 psi
Tot.Elong. 7.6%
Unif.Elong. 5.8%
Hardness 78R30T

Figure 25c. Annealed at 530.5°C x 750

YS 94,300 psi
TS 95,600 psi
Tot.Elong. 1.9%
Unif.Elong. 0.6%
Hardness 77R30T

Figure 26c. Temper Rolled 51.5% x 750

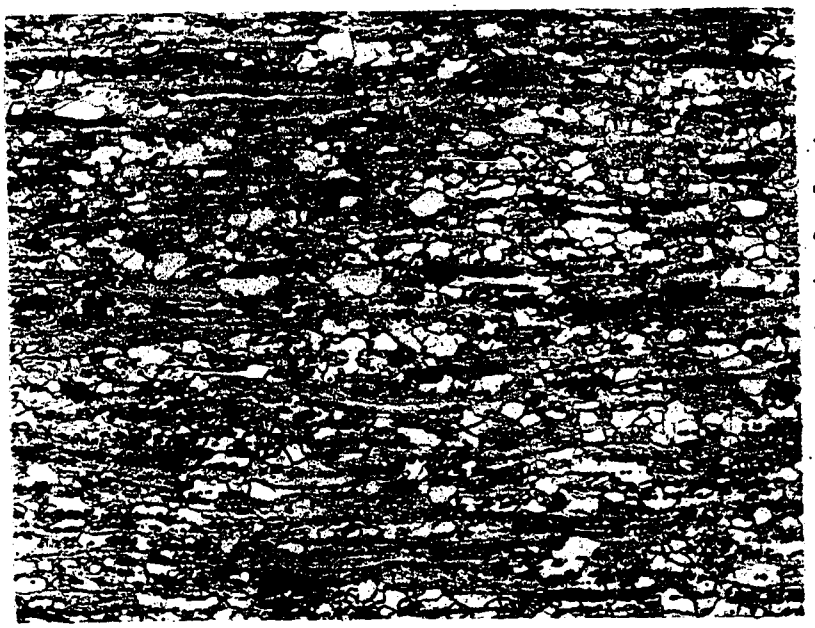


YS 94,900 psi
TS 96,500 psi
Tot.Elong. 7.6%
Unif.Elong. 5.8%
Hardness 78R30T

Figure 25c. Annealed at 530.5°C x 750

YS 94,300 psi
TS 95,600 psi
Tot.Elong. 1.9%
Unif.Elong. 0.6%
Hardness 77R30T

Figure 26c. Temper Rolled 51.5% x 750

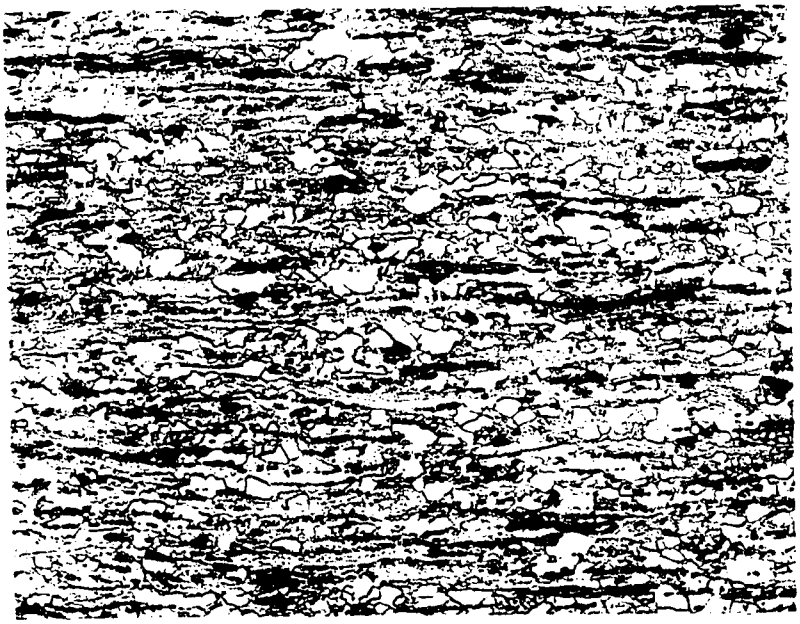


YS 79,000 psi
TS 81,900 psi
Tot.Elong. 13.0%
Unif.Elong. 10.9%
Hardness 73R30T

Figure 25d. Annealed at 550.5°C. x 750

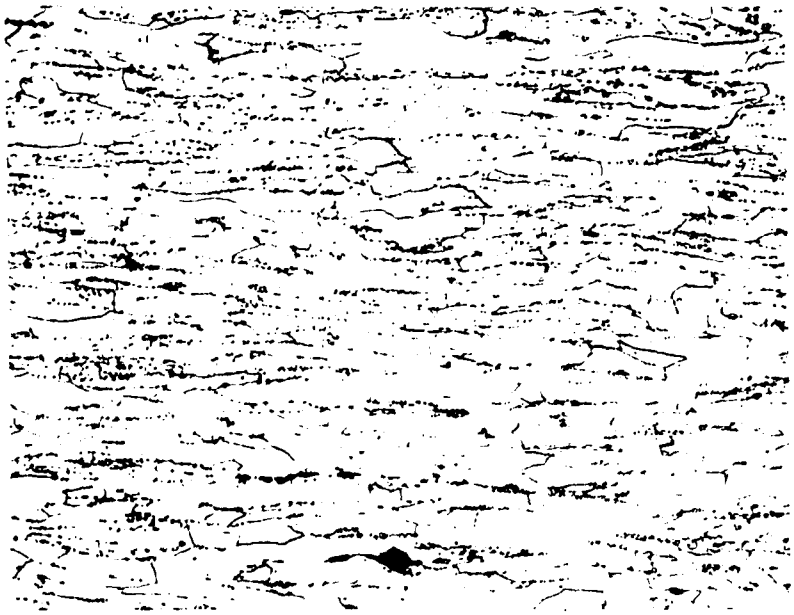
YS 81,800 psi
TS 82,700 psi
Tot.Elong. 2.8%
Unif.Elong. 0.7%
Hardness 74R30T

Figure 26d. Temper Rolled 32.0%. x 750



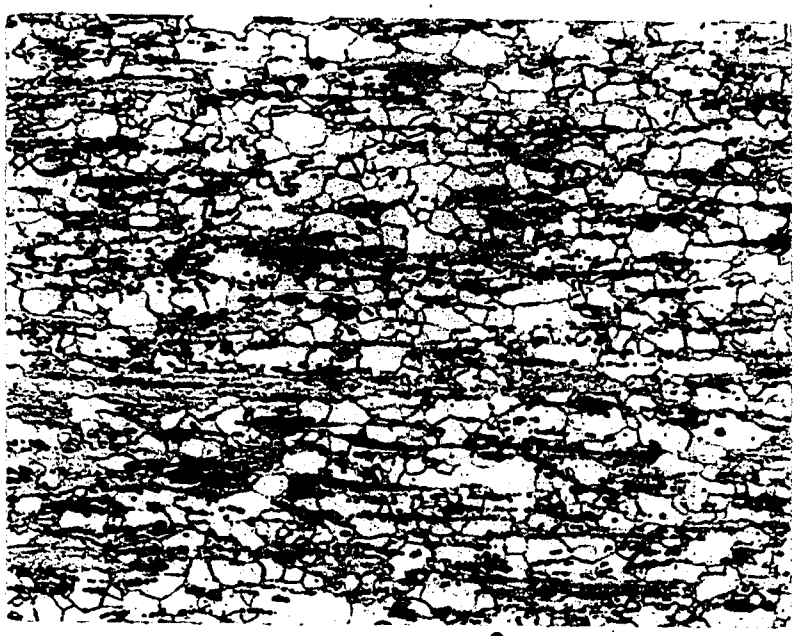
YS 79,000 psi
TS 81,900 psi
Tot.Elong. 13.0%
Unif.Elong. 10.9%
Hardness 73R30T

Figure 25d. Annealed at 550.5°C. x 750



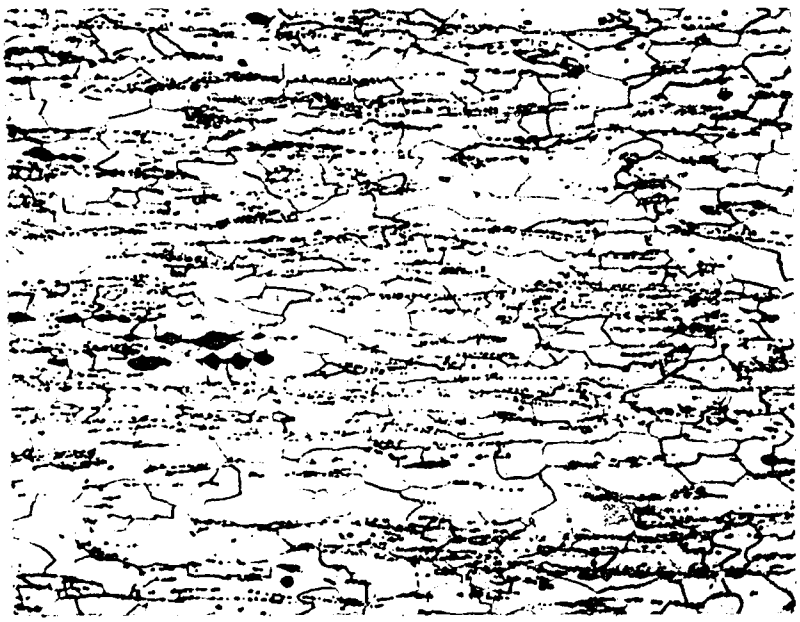
YS 81,800 psi
TS 82,700 psi
Tot.Elong. 2.8%
Unif.Elong. 0.7%
Hardness 74R30T

Figure 26d. Temper Rolled 32.0%. x 750



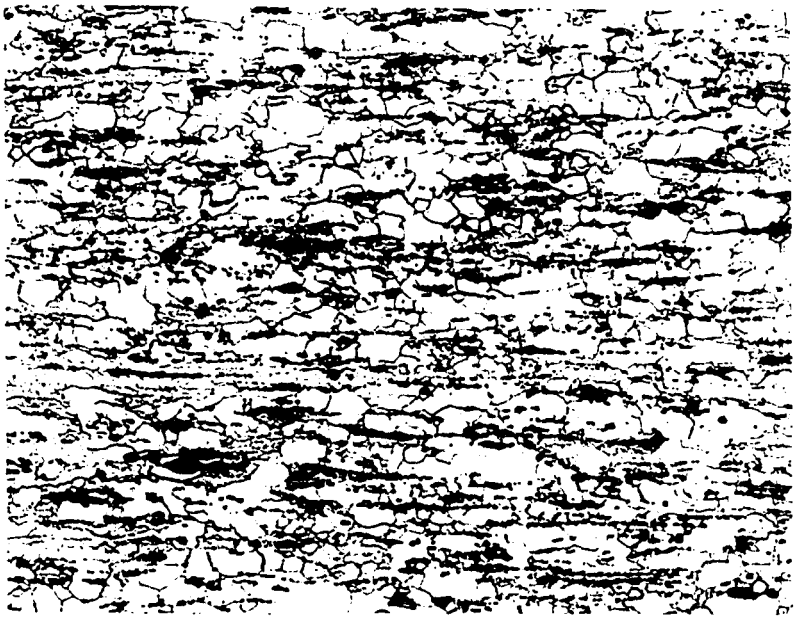
YS 63,700 psi
TS 69,700 psi
Tot.Elong. 21.3%
Unif.Elong. 18.1%
Hardness 67R30T

Figure 25e. Annealed at 560.0°C. x 750



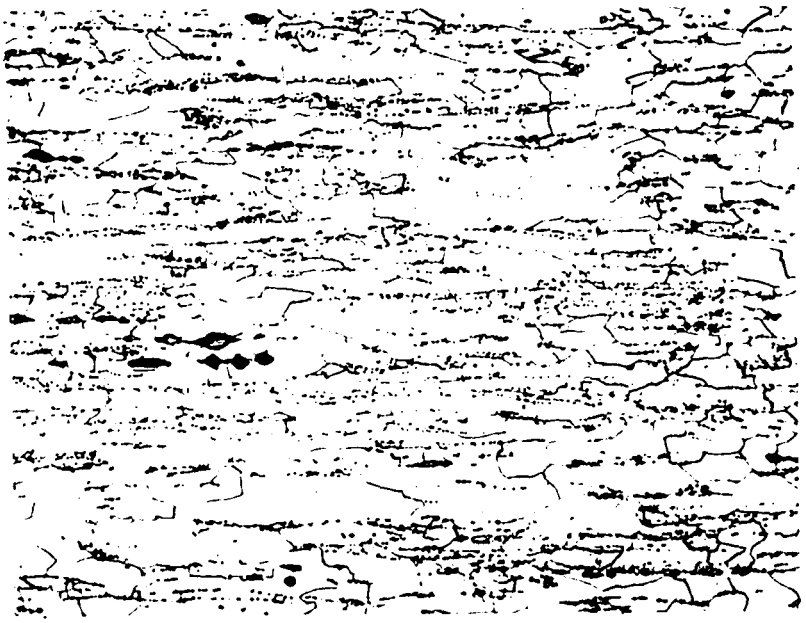
YS 65,200 psi
TS 66,800 psi
Tot.Elong. 12.0%
Unif.Elong. 3.2%
Hardness 69R30T

Figure 26e. Temper Rolled 16.5% x 750



YS 63,700 psi
TS 69,700 psi
Tot.Elong. 21.3%
Unif.Elong. 18.1%
Hardness 67R30T

Figure 25e. Annealed at 560.0°C. x 750

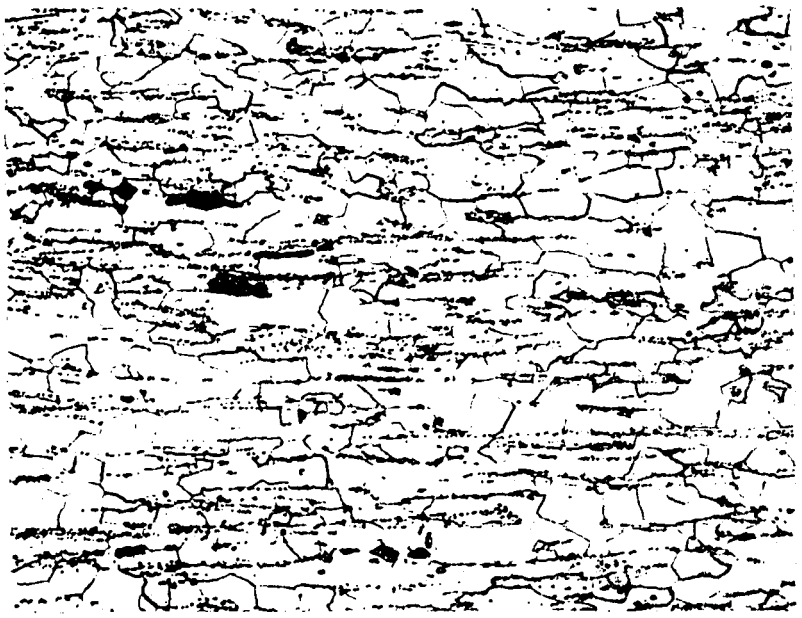


YS 65,200 psi
TS 66,800 psi
Tot.Elong. 12.0%
Unif.Elong. 3.2%
Hardness 69R30T

Figure 26e. Temper Rolled 16.5% x 750

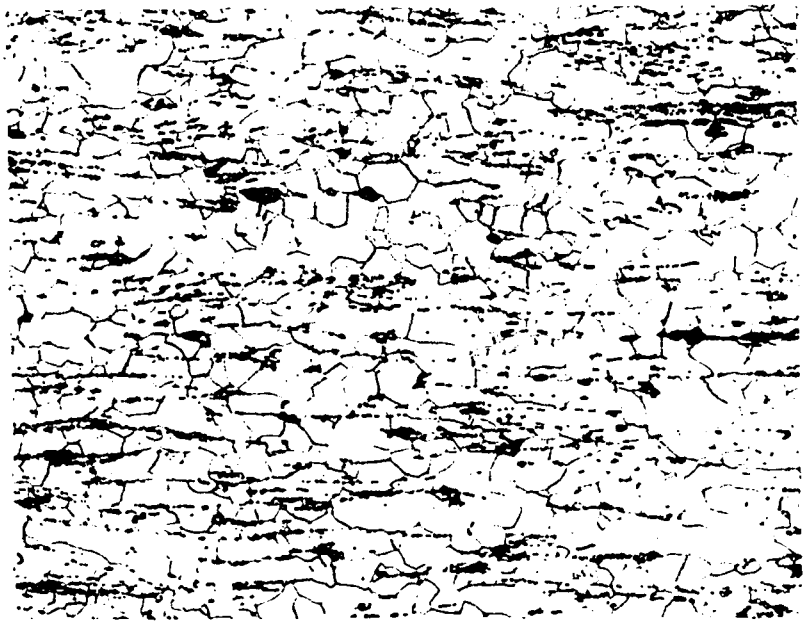
YS 49,500 psi
TS 60,600 psi
Tot.Elong. 29.7%
Unif.Elong. 24.6%
Hardness 63R30T

Figure 25f. Annealed at 579.0°C. x 750



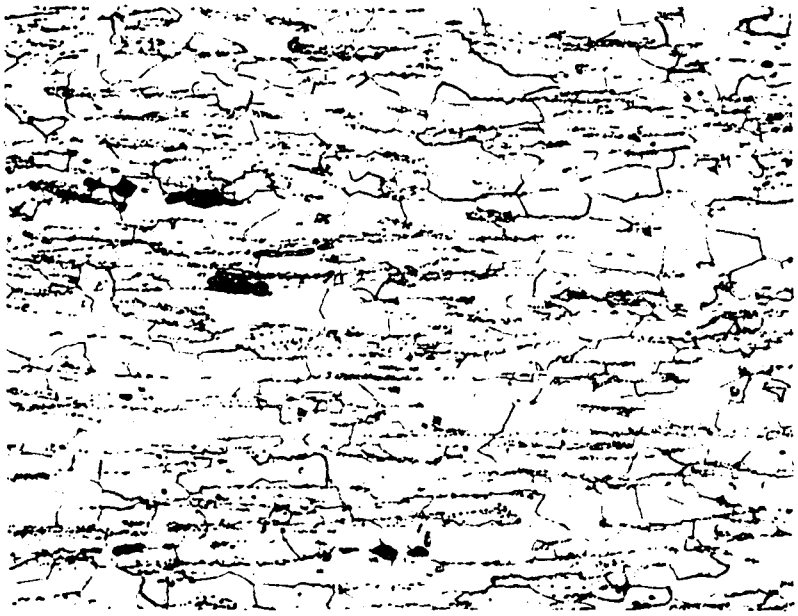
YS 50,500 psi
TS 58,800 psi
Tot.Elong. 27.8%
Unif.Elong. 20.9%
Hardness 63R30T

Figure 26f. Temper Rolled 6.0%. x 750



YS 49,500 psi
TS 60,600 psi
Tot.Elong. 29.7%
Unif.Elong. 24.6%
Hardness 63R30T

Figure 25f. Annealed at 579.0°C. x 750

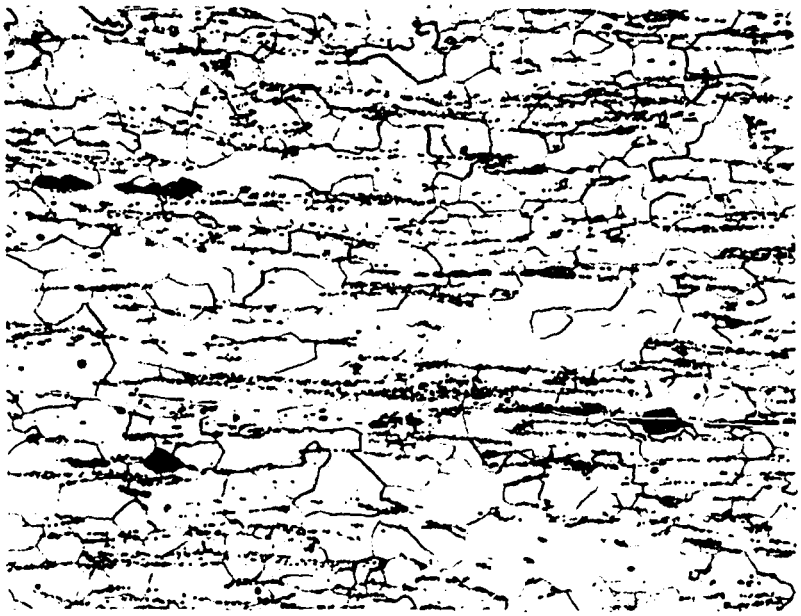


YS 50,500 psi
TS 58,800 psi
Tot.Elong. 27.8%
Unif.Elong. 20.9%
Hardness 63R30T

Figure 26f. Temper Rolled 6.0%. x 750

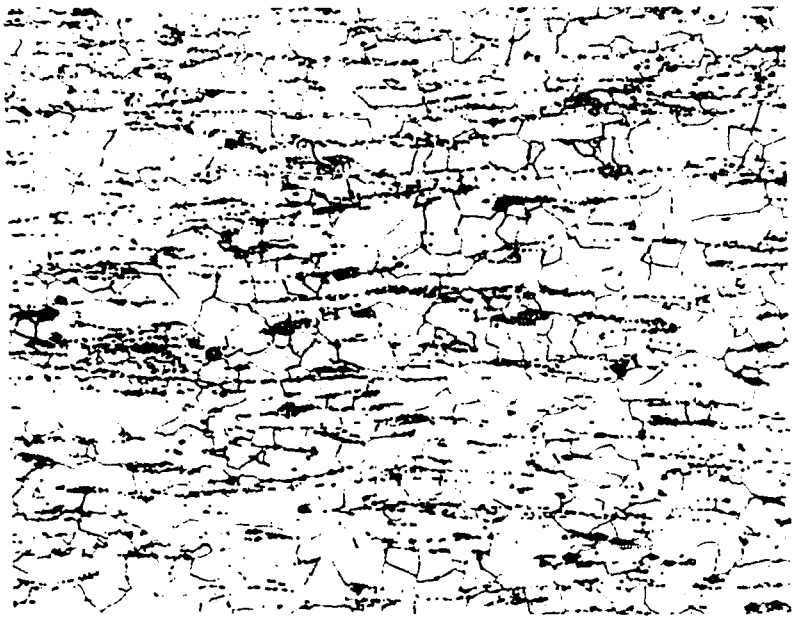
YS 47,600 psi
TS 60,100 psi
Tot.Elong. 30.3%
Unif.Elong. 24.9%
Hardness 61R30T
Average Grain
Diameter 0.006 mm
ASTM No.12

Figure 25g. Annealed at 600.0°C. x 750



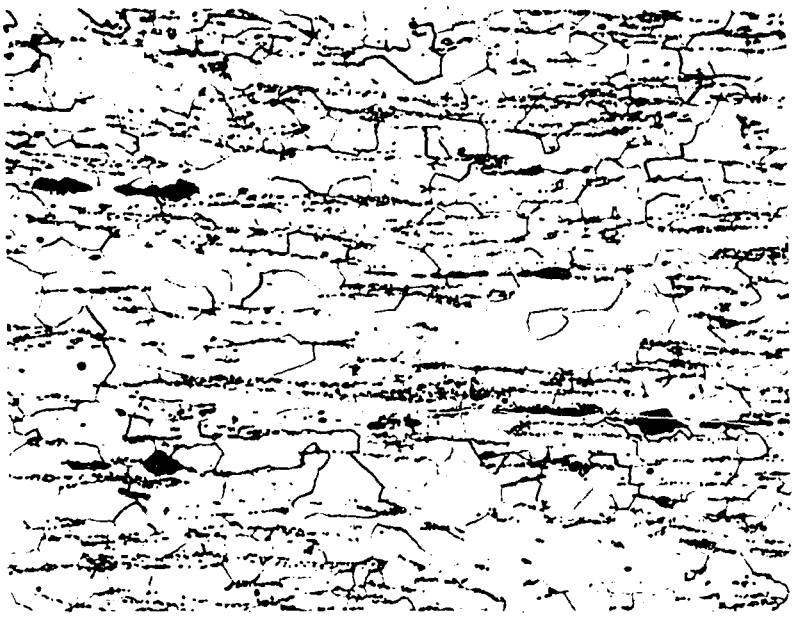
YS 41,800 psi
TS 55,600 psi
Tot.Elong. 35.2%
Unif.Elong. 29.1%
Hardness 57R30T
Average Grain
Diameter 0.008 mm.
ASTM No. 11

Figure 26g. Temper Rolled 1%. x 750



YS 47,600 psi
TS 60,100 psi
Tot.Elong. 30.3%
Unif.Elong. 24.9%
Hardness 61R30T
Average Grain
Diameter 0.006 mm
ASTM No.12

Figure 25g. Annealed at 600.0°C. x 750



YS 41,800 psi
TS 55,600 psi
Tot.Elong. 35.2%
Unif.Elong. 29.1%
Hardness 57R30T
Average Grain
Diameter 0.008 mm.
ASTM No. 11

Figure 26g. Temper Rolled 1%. x 750

SUMMARY AND CONCLUSIONS

Summary

Material that had been cold reduced 82% by rolling was annealed at various temperatures to produce the full strength spectrum that could be obtained from a continuous annealing line. The mechanical properties of this partially annealed material were compared to those of material that had been temper rolled by various amounts to equivalent strengths.

It was found that:

1. As the percentage cold reduction of the temper-rolled material was raised, there was an increase in strength and hardness but a very rapid decrease in ductility. After 24% reduction, the ductility remained at its minimum of less than 1%.

2. The strength and hardness of partially annealed material fell from the as-rolled values as the annealing temperature was raised, while the corresponding ductility increased. Microstructural recovery before the onset of recrystallization accounted for a 30% recovery of strength and a three to five times increase in ductility, over the as-rolled values.

3. There was an extraordinary improvement in ductility, especially uniform elongation, of partially annealed material over that temper rolled to an equivalent tensile strength. Similar results were obtained when yield strength and hardness were bases of comparison.

4. The yield-to-tensile strength ratio for partially annealed material was always less than that for temper-rolled material of equal tensile strength. This would indicate an improved capacity for cold fabrication.

5. Partial annealing holds no advantage over temper rolling so far as the resistance to thinning during forming is concerned. The strain ratio values for each process were about 0.6.

6. The difference in ductility between the two materials at equal strengths can be visually correlated to a difference in microstructure.

Industrial Implications.

Thin high-strength tinplate is being produced by the double-reduction process. The capital cost of say, a three-stand mill to handle the second cold reduction, may prove for many producers to be prohibitive. The most important practical result of this investigation is that superior material can be produced using the existing rolling equipment without the need of a heavy second cold reduction. In fact, it is the elimination of this second reduction in favour of partial annealing that is responsible for the superiority of the material.

Temper-rolled material, similar to that double-reduced, is compared to partially annealed material at strength levels of 90,000 and 100,000 psi, in Table IV.

TABLE IV

Comparison of Mechanical Properties: - Lot 1

		Temper-Rolled	Partially Annealed
TS	psi	101,800	101,000
YS	psi	99,200	98,500
Tot. Elong.	%	2.0	6.3
Unif. Elong.	%	0.8	4.9
Hardness	R 30T	78	79
TS	psi	92,100	93,700
YS	psi	91,500	91,900
Tot. Elong.	%	1.9	8.7
Unif. Elong.	%	0.6	7.0
Hardness	R 30T	76.5	77

Tinplate stock has been used as an appropriate and timely example to illustrate the advantages gained from partial annealing. However, an expanded range of cold-fabricated products using partially annealed high-strength material is possible because the greatly increased ductility would enable more severe cold forming. Corrugated sheet, large bulk containers, light structural or curtain-type steel walls are but just a few future applications.

Production of partially annealed material on present-day continuous annealing lines appears possible. Referring to Figure 14, page 69, it can be seen that at a hardness of 73 R30T, the mechanical properties of Lot 2 change most rapidly with annealing temperature. If a very stringent hardness control of $\pm$ 1 R30T at this point was to be applied, then the annealing temperature would have to be controlled to $\pm$ 2°C ($\pm$ 4°F). However, material of this hardness has yield and tensile strengths of 72,000 and 77,000 psi respectively, whereas it is the higher-strength material with strengths above 90,000 psi which are of more practical interest. With the same hardness restriction of $\pm$ 1 R30T at 79 R30T, annealing temperatures would have to be controlled to $\pm$ 8°C($\pm$ 14° F), a range more likely to be met.

Just as continuous strand annealing eventually replaced batch annealing for tinplate stock, new innovations, such as partial annealing, will continue to refine the process of tinplate manufacturing.

SUGGESTIONS FOR FURTHER WORK

This investigation indicates several areas for further work.

1. Planar anisotropy or directionality of partially annealed low-carbon steel sheet should be investigated to obtain a clearer indication of formability.

2. Recrystallization kinetics are influenced by variations in chemical composition. It should be established whether normal mill variations in C, Mn, etc. will affect the mechanical properties of partially annealed material. Further work may also indicate whether certain chemical combinations will produce a superior product by influencing the recrystallization behaviour.

3. It is the author's opinion that a reduction of the annealing time from 10 min. to 1 min. would not change the shapes of the response curves but would only shift the temperature axis. The effects of higher temperatures, shorter annealing times and a time error of say, $\pm$ 5 sec. in 1 min. on the mechanical properties of partially annealed material should be established.

4. The carrying out of well-prepared, well-documented plant trials will show the feasibility of the partial annealing process. There is no doubt that continuous annealing lines are advancing towards a feedback control which will allow very stringent operating conditions.

APPENDIX A

TABLE I

Mechanical Properties of Temper-Rolled Material

Lot - 0.016 in. Temper-Rolled Material							
Cold Reduction	Hardness	Yield S. 0.2% offset	Tensile Strength	YS/TS	Strain ratio r	Total Elong.	Uniform Elong.
%	R 30T	1000 psi	1000 psi			% in 2"	% in 2"
1.0	57.0	41.8	55.6	0.75	-	35.2	29.1
6.0	63.5	50.5	58.8	0.86	0.91	27.8	20.9
10.5	67.0	59.3	61.2	0.97	0.82	21.3	14.3
16.5	69.5	65.2	66.8	0.98	0.73	12.0	3.2
24.0	72.0	75.6	75.7	1.00	0.71	4.7	0.6
32.0	74.0	81.8	82.7	0.99	-	2.8	0.7
42.5	76.5	91.5	92.1	0.99	0.63	1.9	0.6
51.5	77.0	94.3	95.6	0.99	-	1.9	0.6
62.0	78.0	99.2	101.8	0.98	0.67	2.0	0.8
71.0	79.5	105.7	107.6	0.98	0.60	1.8	0.7
80.5	80.5	113.5	116.5	0.98	0.65	2.0	1.0

TABLE II

Mechanical Properties of Partially Annealed Material

Thickness	Lot 1 - 0.0155 in. - 82% cold reduced; 0.0073 in. - 92% cold reduced							
	Annealing Temp 10 min.	Hardness	Yield Str. 0.2% offset	Tensile Str.	YS/TS	Strain ratio r	Total Elong.	Uniform Elong.
	°C	R 30T	1000 psi	1000 psi			% in 2"	% in 2"
0.0155 in	as-rolled	80.5	113.5	116.5	0.98	0.65	2.0	1.0
	462.5	81.0	111.4	112.6	0.99	0.43	3.4	2.5
	480.0	80.5	107.1	109.4	0.98		4.2	3.1
	500.0	80.0	103.9	106.0	0.98		4.5	3.4
	519.5	79.0	98.5	101.0	0.98	0.48	6.3	4.9
	530.5	78.0	94.9	96.5	0.98		7.6	5.8
	539.5	77.0	91.9	93.7	0.98	0.50	8.7	7.0
	544.0	75.0	86.0	87.5	0.98		9.9	8.1
	550.5	72.5	79.0	81.9	0.97	0.49	13.0	10.9
	555.0	69.5	68.1	72.5	0.94	0.52	18.1	15.5
	560.0	67.0	63.7	69.7	0.92	0.55	21.3	18.1
	568.5	64.5	51.8	63.0	0.82	0.75	27.5	22.5
	579.0	62.5	49.5	60.6	0.82	0.87	29.7	24.6
	600.0	61.5	47.6	60.1	0.79	0.91	30.3	24.9
0.0073 in.	499.0	80.0	109.0	111.0	0.98		2.5	1.1
	544.0	75.0	85.4	87.3	0.98		9.4	8.0
	555.0	70.5	69.0	74.4	0.93		17.0	14.8
	568.5	*	56.9	66.3	0.86		21.8	19.5
	590.5	*	52.1	63.9	0.82		22.2	20.7

* unreliable

TABLE III

Mechanical Properties of Partially Annealed Material

Lot 2: 0.016 in. - 82% cold reduced							
Annealing Temp	Hardness	Yield Str. 0.2% offset	Tensile Str.	YS/TS	Strain ratio r	Total Elong.	Uniform Elong.
°C	R 30T	1000 psi	1000 psi			% in 2"	% in 2"
as-rolled	82.5	123.5	127.2	0.97	0.50	2.1	1.3
462.0	82.5	118.8	120.5	0.99	0.40	3.2	2.2
480.0	82.0	115.1	117.4	0.98	0.40	4.2	3.2
500.0	81.5	110.2	112.5	0.98	0.38	4.5	3.5
511.0	81.0	105.6	109.0	0.97		4.6	3.5
519.5	80.0	104.4	106.2	0.98	0.43	5.6	4.4
530.5	79.5	97.7	100.1	0.98		7.6	6.1
535.5	79.0	93.9	96.8	0.97	0.47	8.5	6.8
539.5	77.5	91.9	94.4	0.97		9.9	7.8
545.0	76.0	81.6	84.7	0.96	0.43	13.0	10.7
549.5	73.0	71.8	77.1	0.93	0.46	17.1	14.4
556.0	69.0	62.6	69.6	0.90		22.5	18.6
560.0	66.5	58.8	67.6	0.87	0.59	24.6	20.3
569.0	64.0	51.1	62.6	0.82		30.2	24.9
579.0	63.0	50.0	62.1	0.81	0.77	30.7	25.0
590.0	62.0	48.0	60.8	0.79	0.77	31.2	26.0
600.0	61.5	48.4	61.4	0.79	0.81	30.9	25.6
620.0	61.0	46.4	60.2	0.77	0.80	32.2	26.5

APPENDIX B

United States Patent Office

3,264,144

Patented August 2, 1966

1

3,264,144
METHOD OF PRODUCING A ROLLED STEEL PRODUCT

Robert H. Frazier and Robert G. Toth, Poland, Ohio, assignors to The Youngstown Sheet and Tube Company, Boardman, Ohio, a corporation of Ohio

Filed Aug. 21, 1963, Ser. No. 303,540
6 Claims. (Cl. 148—12)

This application is in part a continuation of our copending application Serial No. 223,400, filed September 13, 1962, now abandoned.

This invention relates to a method of producing a rolled steel product. The invention has advantages both in economy of production and quality of product and also in developing selected properties in the product. Our method produces with improved economy a rolled steel product having properties similar to the properties of rolled steel products heretofore produced only by relatively complex multistep operations at relatively great cost. Certain of the properties of our product, notably ductility, are improved. By our method the properties of the product produced may be controlled to produce optimum products for particular uses.

While our invention is not so limited in all of its aspects, it has especial application to the production of tin plate, and for purposes of explanation and illustration the invention will be described in connection with the production of tin plate.

As is well known, there is severe economic competition between packaging materials in the container field. One of the large tonnage items in that field is so-called thin tin plate. Thin tin plate has prior to our invention been typically produce in thicknesses ranging from .005" to .007" (most commonly .0066") with an ultimate strength of 65,000 to 100,000 pounds per square inch by cold reducing hot rolled strip approximately 90% to a thickness of .009", subjecting the cold reduced strip to a full anneal, further cold reducing the annealed strip by approximately 25–35% to final gauge and electrotinning; the product of such a process is known as double-reduced thin tin plate. The second cold reduction strengthens the strip while preserving some ductility in the end product. Conventional (as distinguished from double-reduced thin) tin plate is produced by cold reduction of hot rolled strip to final gauge, subjecting the cold reduced strip to a full anneal in a continuous annealing line, temper rolling the annealed strip to improve its shape and surface finish and electrotinning the strip.

The advantage of thin tin plate over conventional tin plate lies in the lower weight per unit area, coupled with high strength, of the thin tin plate. However, the double cold reduction heretofore considered necessary to develop sufficient strength in the end product for certain uses, such as can making, together with the reduction in production tonnage occasioned by the smaller weight per unit area factor, adversely affects production cost. The increased cost of producing double-reduced thin tin plate tends to destroy the competitive advantage of thin tin plate over certain other packaging materials.

We have discovered how to produce thin tin plate at greatly reduced cost without sacrificing the properties required for tin plate, especially in container manufacture. Indeed we produce at reduced cost a product superior in quality to products produced by the double reduction method heretofore employed in the production of thin tin plate. Our product exhibits superior ductility without significant loss in strength. Our product may be used otherwise than for the production of tin plate; for example, the product in unplated form may be used for making containers such as oil drums.

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We hot roll low carbon steel to form hot rolled strip, continuously cold roll the hot rolled strip to reduce its thickness at least 75% to a thickness not greater than .025" and continuously heat treat the cold rolled strip at a temperature and for a time, not exceeding four minutes, such that the steel is not fully recrystallized. The product of our process may exhibit varying degrees of recrystallization or may not exhibit any recrystallization. Generally it does exhibit some recrystallization in the form of small ferrite grains in a striated matrix. Recrystallization in the product of our process may vary from 0 to 95%. The amount of recrystallization may be controlled by control of the time and temperature of heat treatment to produce rolled steel products especially adapted for particular uses.

We may temper roll the heat treated strip, depending upon the use to which the strip is being adapted. The heat treated strip, temper rolled if desired, is plated with tin when the ultimate product is to be tin plate. Tin plate thus produced has superior ductility and strength equal to or better than that of thin tin plate produced by the double reduction method heretofore employed as above described, yet due to complete elimination of the second cold reducing step heretofore deemed necessary in the production of thin tin plate our product is much less costly than thin tin plate heretofore produced. Also, as above indicated, by varying the heat treatment and hence the degree of recrystallization we can produce specific products "tailored" to particular needs, as, for example, can body stock and can end stock.

Other details, objects and advantages of the invention will become apparent as the following description of a present preferred method of practicing the same proceeds.

In the accompanying drawings we have illustrated diagrammatically a present preferred method of practicing our invention and have shown by photomicrographs examples of products produced by our invention and comparison with the prior art. In the drawings,

FIGURE 1 is a diagrammatic illustration of our invention;

FIGURE 2 is a photomicrograph of a rolled steel product produced by our invention evidencing no substantial recrystallization;

FIGURE 3 is a photomicrograph of another rolled steel product produced by our invention, the product of FIGURE 3 evidencing a small amount of, but substantial, recrystallization;

FIGURE 4 is a photomicrograph of still another rolled steel product produced by our invention, the product of FIGURE 4 evidencing a somewhat increased amount of recrystallization as compared with the product of FIGURE 3 (recrystallization of the order of 40–50%);

FIGURE 5 is a photomicrograph of yet another rolled steel product produced by our invention, the product of FIGURE 5 evidencing a further increased amount of recrystallization as compared with the product of FIGURE 4 (recrystallization of the order of 90–95%):

FIGURE 6 is a photomicrograph of a full hard (unannealed) rolled steel product of compositon similar to that of the products of FIGURES 2, 3, 4 and 5;

FIGURE 7 is a photomicrograph of a fully annealed (100% recrystallized) rolled steel product of composition similar to that of the products of FIGURES 2, 3, 4, 5 and 6;

FIGURE 8 is a photomicrograph of a rolled steel product of composition similar to that of the products of FIGURES 2, 3, 4, 5, 6 and 7 produced by cold reducing hot rolled strip approximately 90% to a thickness of .009", subjecting the cold reduced strip to a full anneal in a continuous annealing line and further cold reducing the annealed strip by approximately 25%; and

FIGURE 9 is a master hardness curve for annealing low carbon steel cold reduced 91% to 0.0066" in thickness illustrating applications of our invention. Hardness of the product varies with the degree of recrystallization and is an indication of the strength of the product.

Low carbon steel is first hot rolled to a gauge such that, when it is subsequently cold reduced at least 75%, the thickness of the cold reduced strip will be not greater than .025". The hot rolled strip is cold reduced at least 75% to a thickness of .025" or less. When tin plate is to be produced the cold reduction is preferably of the order of 90–95% and the thickness of the cold reduced strip is preferably of the order of that of thin tin plate as heretofore produced, a standard thickness being .0066". Thus we effect the entire cold reduction in a single cold reduction operation (without intermediate anneal) in contradistinction to two separate cold reduction operations with a full anneal in between as has heretofore been the practice in producing thin tin plate.

We have found that by continuously heat treating the cold reduced strip at a temperature in the range 900–1175° F. for a time, not exceeding four minutes, such that the steel is not fully recrystallized we can produce a rolled steel product which may be plated with tin to produce thin tin plate having properties like those of the previously produced double-reduced thin tin plate, and in most cases having superior properties, while saving the cost of the second cold reduction step. While the annealing temperature will generally be in the range 900–1175° F. it varies depending upon the chemical composition of the steel. The time of heat treatment is limited to four minutes or less and the steel will either not be recrystallized at all or if it is recrystallized will not be fully recrystallized but will contain small ferrite grains in a striated matrix.

A specific example of the practice of our process will now be described. A steel of the following composition may be used:

	Percent
Carbon	0.03 to 0.15.
Manganese	0.20 to 0.60.
Phosphorus	0.20 max.
Sulphur	0.40 max.
Silicon	0.010 max.
Copper	0.20 max.

The steel may be teemed into 32" x 46" bottle top ingot molds and then mechanically capped after a rimming action for not longer than 2½ minutes, 1½ to 2 minutes being preferred. The steel may be hot rolled in steps to a final gauge of 0.074". The strip so produced may be finished at an aim temperature of 1650° F. and coiled at 1150–1200° F. The strip may next be continuously pickled in sulphuric acid and may then be cold reduced 91% to a final thickness of 0.0066". The procedure may vary for the production of end products having other final thicknesses as will be understood by those skilled in the art.

The steel may next be continuously heat treated at 1075° F. for 1½ minutes. The steel is not fully recrystallized and may contain small ferrite grains in a striated matrix. The continuous heat treatment is conducted in a reducing or neutral atmosphere. We prefer to employ a dry gas consisting of 93% nitrogen and 7% hydrogen.

The microstructure of our rolled steel product is shown by the photomicrographs constituting FIGURES 2, 3, 4 and 5. All seven of the photomicrographs constituting figures of drawings herein are at 1000 diameters magnification and of specimens which are nital-etched. The microstructure of our product is in between that of full hard or unannealed steel as shown in FIGURE 6 and that of fully annealed (100% recrystallized) steel as shown in FIGURE 7. The product of FIGURE 2 produced in accordance with our invention evidences no substantial recrystallization. The product of FIGURE 3 evidences a small amount of, but substantial, recrystallization. The product of FIGURE 4 evidences an increased amount of recrystallization, of the order of 40–50%. The product of FIGURE 5 evidences further recrystallization, the recrystallization of that product being of the order of 90–95%. The small ferrite grains in a striated matrix in our product are plainly evident in FIGURES 3, 4 and 5. The fully annealed steel shown in FIGURE 7 contains randomly oriented well developed substantially equiaxed ferrite grains. FIGURE 8 shows a rolled steel product produced by cold reducing hot rolled strip approximately 90% to a thickness of .009". subjecting the cold reduced strip to a full anneal in a continuous annealing line and further cold reducing the annealed strip by approximately 25%.

The following table shows comparative properties of double-reduced thin tin plate produced by the method heretofore employed and thin tin plate produced by our method:

Material	Hardness Rockwell 30 T Mean.	Longitudinal Ultimate Tensile Strength, Mean. 1,000 p.s.i.	Olsen Cup Value Mean. Inches	Transverse Amsler Bend No. of 90° Bends to Failure Mean.
Double-Reduced Thin Tin Plate 25% Cold Reduced After Continuous Annealing (0.0066")	76	85.0	0.13	13
New Thin Tin Plate Produced by the Method Constituting the Present Invention	73	89.0	0.15	17

FIGURE 9 is a master hardness curve for annealing of low carbon steel of the type which we employ. The hardness of the full hard or 91% cold reduced steel is shown at the upper left hand corner of FIGURE 9. The hardness curve is broken at this point to indicate that this is the "as cold reduced" hardness of the full hard material without an annealing treatment. The range of our invention is shown in FIGURE 9 and also specific areas in which the invention has found specific application. Below 66 Rockwell 30T 100% recrystallization is indicated in the figure.

The time-temperature parameter $T(C+\mathrm{LOG}_{10}t)$ is derived from a rate equation for diffusion; see Larson and Salmas, "A Time-Temperature Relation for Recrystallization and Grain Growth," Transactions of the American Society for Metals, vol. 46, p. 1377. Since it is generally believed that recrystallization and grain growth are dependent upon diffusion this time-temperature parameter is applicable to our invention. The letter C is a material constant and for steel of the type which we have used as the example, a value of 20 has been established for C.

FIGURE 9 shows a band rather than a single hardness curve. The width of the band is related to the variation in annealing response of heats of steel within the composition range above specified yet with minor variations in composition. Data from five heats were plotted to produce the hardness band shown.

Thus we produce with improved economy a product which can be used in place of previously produced products which could be produced only at much higher cost. Generally the product of our process is superior in ductility to that of thin tin plate as heretofore produced. The product of our process is competitive with materials other than steel which can be produced economically and with which the previously produced steel products have been competitive only at the sacrifice of profit to the steelmaker.

While we have described and illustrated a present preferred method of practicing the invention it is to be

distinctly understood that the invention is not limited thereto but may be otherwise variously practiced within the scope of the following claims.

We claim:

1. A method of producing a rolled steel product comprising hot rolling low carbon steel to form hot rolled strip, continuously cold rolling the hot rolled strip without intermediate anneal to reduce its thickness at least 75% to a thickness not greater than .025" and continuously heat treating the cold rolled strip at a temperature and for a time, not exceeding four minutes, such that the steel is recrystallized 0–95%.

2. A method of producing a rolled steel product comprising hot rolling low carbon steel to form hot rolled strip, continuously cold rolling the hot rolled strip without intermediate anneal to reduce its thickness at least 75% to a thickness not greater than .025" and continuously heat treating the cold rolled strip at a temperature and for a time, not exceeding four minutes, such that the steel is recrystallized 0–95% and contains small ferrite grains in a striated matrix.

3. A method of producing a rolled steel product comprising hot rolling low carbon steel to form hot rolled strip, continuously cold rolling the hot rolled strip without intermediate anneal to reduce its thickness at least 75% to a thickness not greater than .025", continuously heat treating the cold rolled strip at a temperature and for a time, not exceeding four minutes, such that the steel is recrystallized 0–95% and temper rolling the heat treated strip.

4. A method of producing a rolled steel product comprising hot rolling low carbon steel to form hot rolled strip, continuously cold rolling the hot rolled strip without intermediate anneal to reduce its thickness at least 75% to a thickness not greater than .025", continuously heat treating the cold rolled strip at a temperature and for a time, not exceeding four minutes, such that the steel is recrystallized at 0–95% and plating the heat treated strip with tin.

5. A method of producing a rolled steel product comprising hot rolling low carbon steel to form hot rolled strip, continuously cold rolling the hot rolled strip without intermediate anneal to reduce its thickness at least 75% to a thickness not greater than .025", continuously heat treating the cold rolled strip at a temperature and for a time, not exceeding four minutes, such that the steel is recrystallized 0–95%, temper rolling the heat treated strip and plating the temper rolled strip with tin.

6. A method of producing a rolled steel product comprising hot rolling low carbon steel to form hot rolled strip, continuously cold rolling the hot rolled strip without intermediate anneal to reduce its thickness at least 75% to a thickness not greater than .025", continuously heat treating the cold rolled strip at a temperature and for a time, not exceeding four minutes, such that the steel is recrystallized 0–95% and contains small ferrite grains in a striated matrix, temper rolling the heat treated strip and plating the temper rolled strip with tin.

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Aug. 2, 1966 R. H. FRAZIER ET AL 3,264,144

METHOD OF PRODUCING A ROLLED STEEL PRODUCT

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Fig. 1.

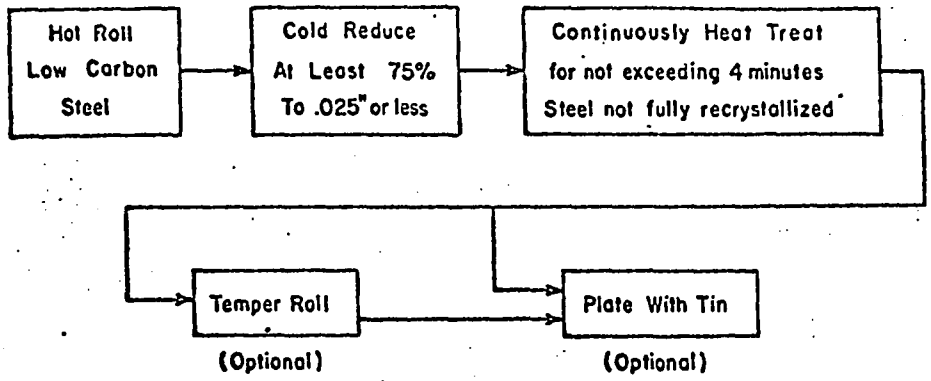
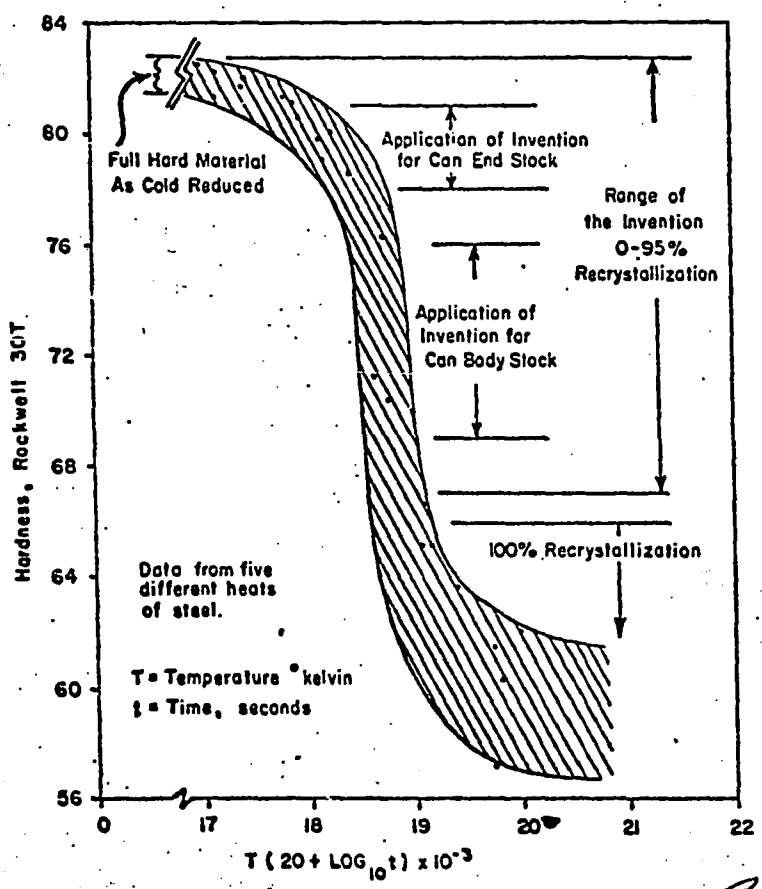


Fig. 9.



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METHOD OF PRODUCING A ROLLED STEEL PRODUCT

Filed Aug. 21, 1963 3 Sheets-Sheet 2

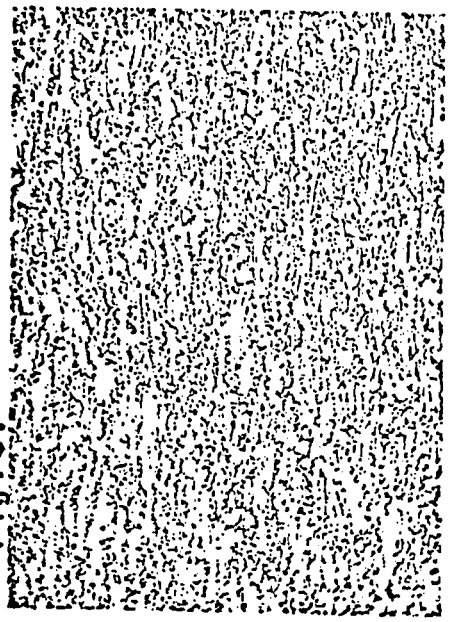

Fig. 3.

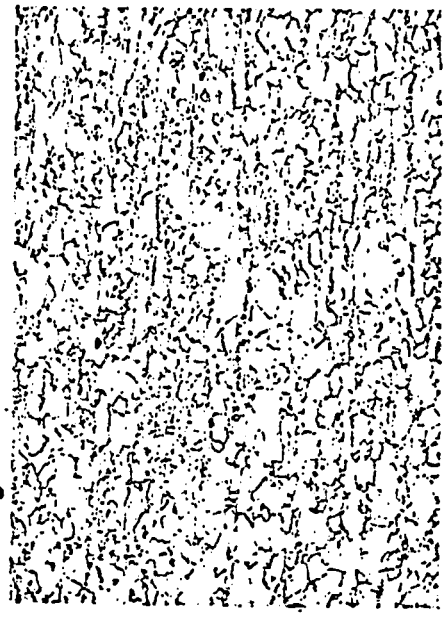

Fig. 5.

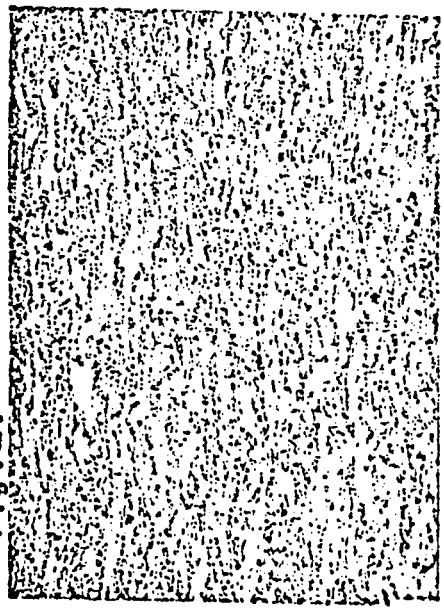

Fig. 2.

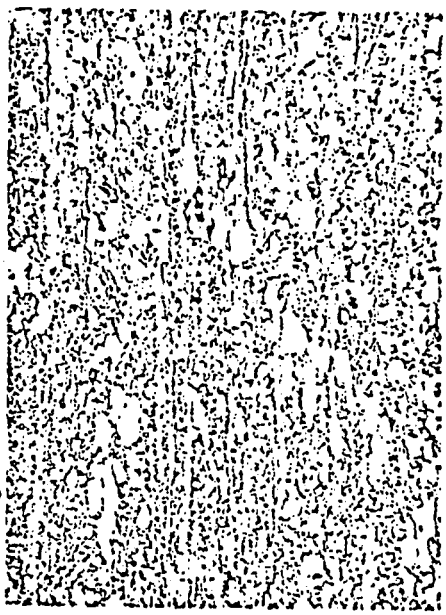

Fig. 4.

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Fig. 7.

Fig. 6.

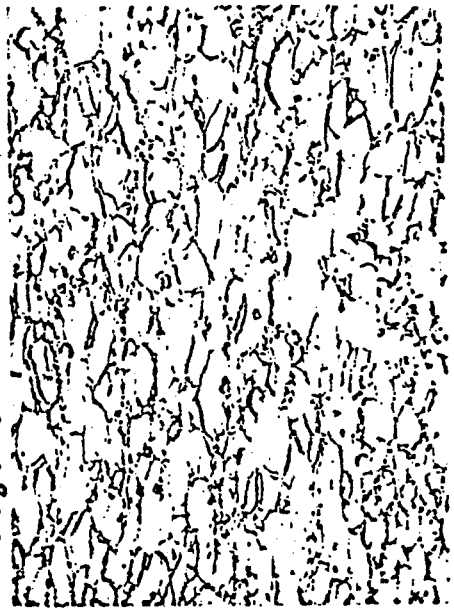

Fig. 8.

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