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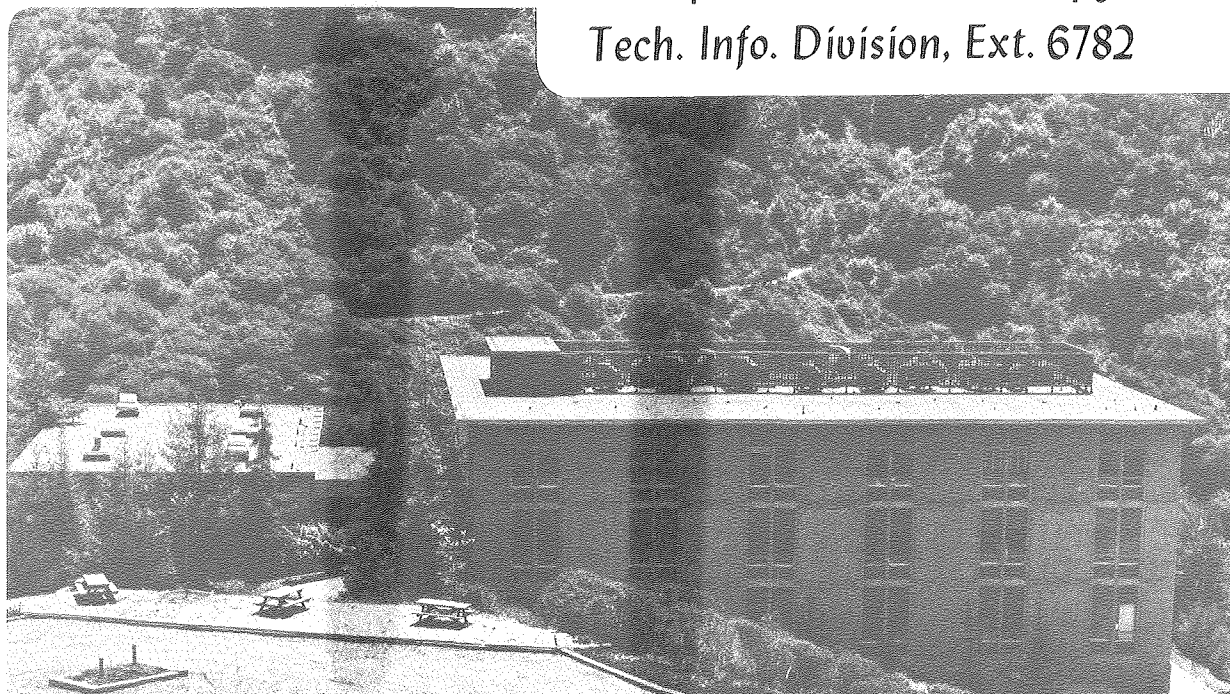
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# THE ROLE OF DISLOCATION THEORY IN THE DESIGN OF ENGINEERING MATERIALS

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## *ABSTRACT*

The science of materials development has progressed to a stage in which it is possible to compose a "recipe" for new materials. The recipe has three steps: given a desirable set of properties and material constraints, one identifies a composition and microstructure to achieve them; given a desirable composition and microstructure, one identifies a processing sequence to achieve them; given a trial alloy, one conducts analytical testing to identify its shortcomings and overcomes them. In effecting each stage of this recipe, it is usually necessary to be aware of and understand the role of the dislocations which determine material properties, define material microstructure, and control its evolution. The role of dislocations is discussed in the following text. The text contains examples of particular alloy development efforts, and suggestions for research in dislocation theory which might contribute to the solution of significant problems in materials development.



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## I. INTRODUCTION

I am reliably informed that when the announcement of this conference reached one national laboratory it was tacked to a bulletin board with the title of this paper highlighted and a marginal note added: "This will be the shortest paper in history." The editorial comment was probably authored by my informant and intended in good fun, but it is symptomatic of the gulf which is widely perceived to exist between the students of dislocation theory as a science and the engineering metallurgists who are their natural technological constituency. I suspect that a similar perception led the organizers of this conference to ask me to write this introductory paper, essentially from the perspective of a consumer advocate, to point out some things that dislocation theorists might do to ease the task of those who are trying to put new engineering materials together.

While we shall always be blessed with physicists to argue that the term materials science is a self-contradiction (and with Oxonians to point out that it is grammatically incorrect), the notion that there should be some underlying science to the business of concocting engineering materials is becoming increasingly prevalent. The impetus for a science of new materials is pragmatic and derives from the engineering revolution in which we find ourselves. A dominant goal of this revolution is the provision of useful energy, which (apologies to Gibbs) will never be free, but should be made available in ample quantity at tolerable cost. The advanced technological schemes which promise to pace the energy revolution invariably involve the operation of engineering devices under extreme conditions or in aggressive environments. The minimum materials specifications for these systems often approach the capabilities of the best available materials; a broad range of new and often narrowly tailored materials will be

needed if the advanced energy technologies are to achieve their theoretical potential.

The challenge to the materials scientist comes not only from the difficulty of the individual materials problems but also from their variety and from the urgent pace of energy technology, which forces a corresponding pace on new materials development. Those engaged in materials development are being asked to do better faster, to proceed successfully in several technologically different directions at the same time, and to accomplish these things from an engineering base which, at least in the United States, bears the scars of a decade-long contraction of real research effort which has still not ended in some of our basic materials industries. The problem in materials development is to do more with less, and, as in any other field of activity, the best way to overcome that problem is to increase the efficiency of the effort.

An efficient materials development effort requires the participation of fundamental science. It is not necessary that basic science provide exact answers to materials development problems; in its present state of development it cannot do so in any case. The fundamental science rather contributes indirectly, and in two ways: it helps the experimenter avoid sterile approaches and unsolvable problems, and it often helps him to short-cut the tedious trial-and-error process which is inherent to a purely empirical approach. Since materials development usually requires the manipulation of microstructural defects, dislocation theory often provides its most useful scientific base.

A specific discussion of the role of dislocation theory in the emerging science of new materials is best phrased in the context of the form such a science may take.

## II. A RECIPE FOR NEW ENGINEERING MATERIALS

The vast majority of the engineering materials which are used in modern technology are either the accidental results of research directed toward other ends (serendipity) or the residue of Edisonian testing: the more or less systematic screening of all materials which might conceivably work in a particular application.

Serendipity is the likely source of the key engineering materials that have come down from the distant past; Cyril Smith (1) makes a strong argument that most of the common metal alloys were

originally found by jewelers and artists rather than smiths, armorers or founders. Serendipity, in the extended sense of fundamental research with no direct engineering goal, continues to be a principal source of new materials with unique properties, such as superconductors, amorphous metals, and shape memory alloys. It is, however, no more than an adjunct to engineering, precisely because the research is undirected; modern technology requires systematic ways to satisfy materials needs which are known and established.

Edisonian testing is the principal way in which existing materials find new uses and, in that sense, become new engineering materials. It will remain dominant for two reasons. First, given the cost, difficulty, and uncertainty of a materials development effort, the relevant management is usually reluctant to support one until it has been established that existing materials are unsatisfactory. Second, materials needs usually become evident during the design or operation of engineering systems and often at a sufficiently advanced stage that the engineering schedule for the system precludes the delay involved in a materials development effort. The usual solution is to modify the design or operating schedule to accommodate the properties of the best available materials. But it is not always possible to match available materials to acceptable designs. Nor is it always wise to gamble that "off-the-shelf" materials can be found to satisfy an increasingly challenging set of known future needs. Edisonian testing is complementary to materials development in the sense that its failure to identify adequate or optimal materials defines the problems the materials scientist is asked to address.

Once a materials need has been identified, in current practice it is approached in one of two ways, whose superficial similarity obscures a fundamental difference which defines the boundary between traditional engineering and engineering science. In traditional engineering one systematically varies composition and processing to obtain a desired set of properties; in materials science one systematically varies composition and processing to obtain a microstructure which imparts the desired set of properties. The distinction between the two approaches is critical. Dislocation theory has virtually nothing to contribute to materials development based on traditional engineering practice but is essential to an approach grounded in materials science.



As a review of the relevant technical and patent literature will make clear, the traditional engineering approach remains dominant in new materials development, not only in metallurgical industries but in advanced electronics as well. Its operational variables are composition and processing, and it draws on a rich experimental base which addresses the interdependence between composition, processing and properties. Materials science is used, if at all, to rationalize the results. The approach is empirical and suffers from the inherent shortcoming that it is doomed to remain so since there is no explicit mechanistic connection between its input variables (composition and processing) and its output parameters (properties). While useful engineering materials have and will continue to emerge from research undertaken along traditional lines, this approach will always involve more art than science.

The operational variables of materials science, on the other hand, are composition and microstructure. Its perspective is that composition and microstructure determine engineering properties, while microstructure is, itself, determined by the processing a material of given composition has undergone. The attempt to develop an engineering material from a base in materials science is hence largely an exercise in microstructural engineering. It focuses on mechanistic connections and has a definable recipe: Given desired engineering properties, one identifies a promising composition and microstructure. Given composition, one devises a processing sequence to establish the desired microstructure. Given the imperfections in the current state of materials science, however, the initial selection of microstructure is rarely ideal and the choice of microstructure must hence be refined through analytical testing. The testing identifies shortcomings in the material; subsequent microstructural analysis is used to clarify the source of these shortcomings and to suggest appropriate changes in the microstructure. In the usual case the procedure must be iterated through several trials before a successful result is obtained.

The scientific foundation needed to implement the "recipe" for an engineering material is, of course, not well developed. What materials science offers is a qualitative outline rather than a quantitative prescription. The development of a new material remains an act of invention which involves a sufficiently complex admixture of empiricism, intuition, blind luck, and after-the-fact rationalization that it is difficult, even when describing one's own work, to be honest and precise about how "scientific"

one has been. Nonetheless, the broad outlines of a science-based microstructural engineering are increasingly apparent in the new materials literature. The science is most apparent in high technology materials such as semiconductors and related materials where the structure-property relations are relatively simple and well understood (Von Hippel (2) described the field as "molecular engineering" almost two decades ago) and in high temperature alloys for use in extreme environments such as turbine engines, where the substantial performance improvements that can be achieved with better materials have inspired researchers to push very hard on the limits of performance (3) . But there is also an increasing scientific emphasis in the development of new alloys for more traditional uses, such as automotive structures (4) and welding (5).

The body of this paper addresses the steps of the recipe for alloy design, with emphasis on the past and potential contributions of dislocation theory. I shall focus on the structure-property and structure-processing connections, leaving the almost equally important area of materials testing and analysis for other papers in this symposium.

### **III. THE CORRELATION BETWEEN STRUCTURE AND PROPERTIES.**

#### *A. The Role of Dislocations.*

The first step in the design of a new material is the identification of a combination of composition and structure which promises to give the desired set of engineering properties. In practice this step is almost never taken from first principles. One rather identifies a material or class of materials which is known to satisfy at least some of the engineering objectives and sets about modifying composition and structure to achieve the remainder.

The structure-property relations underlying this exercise are almost entirely qualitative. Not only are there few, if any, fundamental relations which are capable of predicting the properties of an engineering material from its structure, but limitations on the completeness of structure characterization in real materials would make it difficult to use such relations even if they were available. The developer of new materials looks to fundamental science for guidance rather than detailed instruction. He must select the appropriate level of the microstructure at which the properties of interest to him are controlled, identify the mechanisms of behavior which control those properties, and select the

microstructural modifications which will lead to an improvement in those properties without serious deterioration in other properties that are also of interest. The fundamental information which is of greatest use to him is that which helps in understanding the nature of microstructure, identifying mechanisms, estimating trends in engineering properties with systematic microstructural changes, and anticipating the variation of engineering properties with one another.

The science of materials development must, of course, be concerned with the full structure of a material and with the aggregate of its engineering properties, so it is often impossible to isolate its interest in dislocations. But dislocations are almost always an important element of the problem. They enter the structure-property relation in two ways: as an element of structure and as active defects which influence or control behavior.

As microstructure elements, dislocations are present both as specific defects and as components that may be used to construct or model such other internal defects as interfaces and heterogeneities. As linear defects, dislocations impede the motion of other dislocations, influence the flow of percolating species such as diffusing atoms and conduction electrons and introduce specific local states into the electronic structure. When organized into cells, subgrain, or grain boundaries they affect the distribution of internal stress, modify local cleavage fracture, and introduce planar electronic defects which scatter electrons in semi-conductors and pin magnetic flux lines in superconductors.

As active species, dislocations are most evident in their influence on mechanical properties, including particularly the plastic deformation that dislocations were invented to explain. The dislocations present in structural alloys usually determine their yield and plastic flow, their work-hardening and creep characteristics, and the micromechanics of fatigue and fracture. Relevant chemical-mechanical properties such as environmental embrittlement and stress-corrosion cracking may also be determined by the interaction of dislocations with embrittling species or by their role in the achievement or loss of the coherency of protective surface films. Dislocations tend to degrade the properties of electronic or optical materials by introducing unwanted electrically active sites.

A well-developed dislocation theory is, hence, crucial to the theory of structure-property relations. The development of dislocation theory has, moreover, had an influence on the appreciation of

structure-property relations which goes beyond its specific treatment of particular aspects of dislocation behavior. Particularly to those of us who were educated after the explosion of dislocation theory following the Second World War, dislocation theory is the basis on which mechanical behavior makes sense and the basic concept of a structure-property relation becomes meaty and meaningful.

*B. Examples of the use of structure-property relations in alloy development.*

To illustrate the use (and in one instance the misuse) of structure-property relations in the development of new engineering alloys, I offer three examples of work done at Berkeley. The first concerns the development of ferritic cryogenic steels for structural use at 4K, the second addresses the improvement of the room temperature toughness of very high strength maraging steels, and the third concerns the development of nickel-free cryogenic steels for structural use at temperatures as low as 77K.

*1. Ferritic Cryogenic Steels for 4K Service (6-8).*

The utilization of high field superconducting magnets for advanced energy systems such as fusion energy devices, MHD generators, and high energy accelerators, has created the need for structural alloys that combine high strength and toughness at the temperature of the liquid helium which is used to cool the superconductor. The principal candidate alloys have been austenitic stainless steels, which remain tough at 4K but are both high in cost and low in structural strength relative to the ferritic structural steels. Conventional ferritic structural steels, on the other hand, are unusable because of their low temperature brittleness. Even the best of the available cryogenic steels, such as commercial 9%Ni steel, undergo a ductile-to-brittle transition at temperatures well above 4K. There is, therefore, a need to identify new steel compositions or heat treatments leading to tough ferritic alloys for 4K service.

To phrase this alloy development problem in terms of a structure-property relation, one must look into the mechanism of the ductile-brittle transition. The classic ductile-brittle behavior is shown schematically in Fig. 1. The ductile-brittle transition is always associated with a change in the fracture mode. In the case of Fe-Ni cryogenic steels the mode shift is from classic ductile rupture to a well-

defined quasi-cleavage as the temperature passes through the ductile-brittle transition.

A time honored interpretation of the source of the ductile-brittle transition is shown in the "Yoffee diagram" presented as Fig. 2. As the temperature decreases, the thermal contribution to the strength of a ferritic steel causes its yield strength to increase dramatically. If there is a value of the stress at which cleavage will occur if gross plastic yielding does not intervene, and if that stress is relatively independent of temperature, then the ductile-brittle transition is associated with that temperature at which an appropriate measure of yield stress exceeds the cleavage stress. From this perspective there are two ways in which the ductile-brittle transition may be decreased: one may lower the low temperature yield strength, or one may raise the cleavage fracture stress. If one's objective is to create a strong cryogenic steel, the latter is obviously preferable. The cleavage strength may be raised by decreasing the effective grain size of the material, which shortens the mean-free path over which a cleavage crack can propagate in a single step.

A problem in applying this simple reasoning to the ferritic cryogenic steels lies in deciding what the effective grain size of the material is. The substructure of 6Ni ferritic cryogenic steel in the as-quenched condition is shown in the optical micrograph presented in Fig. 3 and in the transmission electron micrograph shown in Fig. 4. The alloy forms a dislocated martensite structure on quenching, which consists of very fine laths of martensite organized in parallel groups that are called packets. In the TEM, the dislocated lath structure appears to have an extremely fine grain size since the laths have submicron width and their boundaries are highly dislocated. However, a closer examination of Fig. 4 reveals that the lath refinement is deceptive. The laths within a packet tend to be in very close crystallographic registry with one another. They share common crystallographic planes including, in particular, the (100) cleavage plane. The effective grain size of the steel with respect to cleavage fracture is hence the dimension of the martensite packet rather than that of the martensite laths. To accomplish grain refinement one must hence seek to break up the internal alignment within packets so as to decrease the cleavage crack length.

In conventional Fe-Ni cryogenic steels the decomposition of the martensite packets is accomplished through an intercritical tempering treatment at a relatively low temperature within the two-

phase  $\alpha+\gamma$  field. This temper introduces distributed particles of thermally stable austenite phase into the interstices of the martensite laths. This austenite is not, in fact, mechanically stable at low temperature. It transforms to martensite during plastic deformation but tends to transform to a variant of martensite compatible with the nominal applied load rather than with the crystallography of the surrounding packet. As a consequence, and as illustrated in Fig. 5, the cleavage crack path through a martensite packet is made tortuous. The intercritical temper causes a substantial decrease in the ductile-brittle transition of the alloy.

The most straightforward way to decrease the ductile-brittle transition of the alloy to below 4K is, hence, to increase the degree of grain refinement through decomposition of martensite packets. However, since cryogenic steels are typically used in the form of relatively thick plates, this decomposition must involve a thermal rather than a mechanical treatment.

Our initial approach was to give the alloy a simple repetitive thermal cycle involving a series of successive martensite transformations. This approach was only partly successful since the packet structure of martensite tended to regenerate after each thermal cycle at approximately the same packet size. To eliminate this "memory-like" effect, we devised what is known as the 2B treatment, diagrammed in Fig. 6. In this treatment thermal cycles into the austenite field are alternated with "intercritical annealing" at a relatively high temperature in the two-phase  $\alpha+\gamma$  region. As illustrated by the sequence of TEM micrographs in Fig. 7, the 2B treatment yields a microstructure of distinct grains which are extremely small and approximately equi-axed.

The first successful application of the 2B heat treatment was to a research alloy whose nominal composition was Fe-12Ni-0.25Ti (the titanium was included to getter carbon). This alloy proved to have exceptional structural properties in the 2B condition, exhibiting a fracture toughness in excess of 200 Ksi $\sqrt{in}$  at 4K at a yield strength level of approximately 200 Ksi. The fracture mode at 4K was fully ductile.

Continuing the research, we then attempted to use the same heat treatment to lower the ductile-brittle transition temperature of commercial 9Ni steel, which contains approximately 0.06C. The 2B treatment by itself was not successful in toughening 9Ni steel at 4K. Microstructural analysis suggested

that there were two reasons for its failure: the grain refinement was not quite as efficient as in the 12Ni alloy, and the presence of carbon and carbides seemed to be lowering the toughness. To overcome these problems we added a final temper in the lower portion of the two-phase  $\alpha+\gamma$  field to introduce a distribution of retained austenite into the structure. This austenite serves the dual function of further refining the grain structure and getting carbon and carbides from the lattice. In the 2BT condition commercial 9Ni steel exhibited a fracture toughness above 170 Ksi  $\sqrt{in}$  at the 200 Ksi strength level at 4K, and fractured in an almost fully ductile manner.

These two 2B- and 2BT-treated Fe-Ni ferritic cryogenic steels are now attracting some interest as candidate alloys for the case structures of high field superconducting magnets in fusion energy devices.

*2. High-strength, high-toughness maraging steel (9).*

The engineering specifications for high-strength structural steels usually require that the steel retain sufficient fracture toughness to provide adequate resistance to brittle fracture in service. Since plane strain fracture toughness decreases with yield strength, it is often necessary to improve the strength-toughness characteristic of the material. To identify a suitable technical approach for toughness improvement one must consider whether the deterioration in toughness is associated with a change in the fracture mode. If the fracture mode changes then the appropriate alloy design approach is to suppress the transition; if no mode change occurs then the alloy designer must improve fracture toughness in the prevalent mode. The failure to recognize this point can result in wasted effort in alloy development, as in the following example:

The problem in this instance was to improve the fracture toughness of maraging steel at approximately the 200 Ksi strength level to achieve a high-strength, high-toughness structural alloy for an aerospace application. For this purpose, we selected a 250 grade maraging steel to insure an adequate strength margin to permit substantial microstructural modification and addressed the problem of improving its toughness at the 200 Ksi strength level.

The 250 grade maraging steel is an 18Ni steel which forms a dislocated lath martensite structure on cooling. Its starting structure bears a strong resemblance to that of 9 and 12Ni steels. Given our earlier success in toughening these lower nickel alloys through grain refinement and intercritical

tempering techniques, we applied the same techniques to the 250 grade maraging steel. A brief research project showed that it is possible to establish an extremely fine grain size in 250 grade maraging steel by appropriate intercritical tempering. Moreover, as in the Fe-Ni cryogenic steels, this fine grained structure can be decorated with a dense distribution of precipitated austenite. We therefore established this structure in the steel and proceeded to mechanical testing .

The results of mechanical testing on the processed steels were disappointing and initially quite surprising. Grain refinement and austenite decoration of the substructure of the 250 maraging steel proved to have no measurable effect whatsoever on the strength-toughness characteristic of the steel over the 180-220 Ksi strength range, as shown in Fig. 8. The reason for the failure became apparent after fractographic analysis of the steels, which showed that the fracture mode of this steel is ductile rupture over the whole relevant strength range, in both its initial state and its reprocessed condition. Since toughness with respect to ductile rupture at a given strength level is controlled by the density and efficiency of void nucleation sites, and since there was no reason to expect that either the density or severity of these nucleation sites would be affected by the microstructural modification we had made, it was not really surprising that the strength-toughness characteristic of the alloy did not improve.

The available understanding of ductile fracture in these high strength steels suggests that the ductile voids are due primarily to fracture or decohesion at inclusions or unusually large precipitates within the structure. This interpretation was consistent with the fracture characteristics of the samples. Improving the toughness of the alloy hence required that we identify techniques to resist ductile fracture from inclusions which were already present. The technical approach we chose to accomplish this involved substantially increasing the dislocation density within the material. Since the alloy is hardened primarily by precipitates, we felt that it should be possible to substantially increase dislocation density without markedly increasing alloy yield strength and, in this case, the high dislocation density would give an improvement in local plasticity which might minimize stress concentration at inclusion sites within the steel.

To engineer an increase in the dislocation density within the steel, we employed the thermal treatment shown in Fig. 9. In this treatment the steel is first aged, then given a rapid cycle to elevated



temperature which causes a reversion to austenite followed by a retransformation to martensite. If the reversion is carried out rapidly, the precipitates are retained and the shear involved in the double martensitic transformation of the decorated alloy leads to a spectacular increase in dislocation density. Mechanical tests on this highly dislocated material showed, however, that despite its very high dislocation density, the yield strength was very low. More careful analysis revealed that the decrease in yield strength is associated with an early, transient yielding phenomena which may be due to a relaxation of the dense dislocation network within the alloy. To eliminate this apparent instability, the alloy was given a second aging treatment to permit thermal relaxation of the dislocation structure and to introduce new precipitates which would hopefully pin the dislocations against premature motion.

The properties of the alloy after aging, reversion, and stabilization are plotted in Fig. 10. It is apparent from the figure that the new heat treatment gives a significant improvement in the strength-toughness characteristic of the 250 maraging steel.

It is not clear that our interpretation of the mechanisms involved in this property-improving treatment are correct. But what is most certainly relevant is that the modification of the alloy addresses the microstructure at the scale pertinent to ductile failure: the dislocation and precipitate distribution. The earlier, unsuccessful heat treatment addressed the microstructure at the level of grain size and phase distribution, features of the microstructure which are not connected to ductile rupture in any necessary mechanistic sense.

### *3. Nickel-free Cryogenic Steels (10).*

The traditional emphasis in the development of new engineering alloys has been on modifying alloy composition rather than on engineering the microstructure. One obvious reason for this emphasis is that chemical composition is relatively easy to quantify while microstructure can be extremely difficult to characterize in a meaningful way. However, the emphasis on chemical composition obscures the fact that composition plays two rather distinct roles in establishing the properties of an alloy. The composition influences both the properties of an alloy having a given microstructure and the response to processing which determines precisely what microstructure the alloy will have. The two effects tend to be folded together in most discussions of the influence of composition on properties, with the

consequence that one often speaks, for example, of the influence of a certain alloying element on the mechanical properties of steel without troubling to clarify whether that influence is purely chemical or microstructural.

The modern metallurgist is increasingly asked to address the problem of material substitution: whether it is possible to replace an element which is strategic or expensive by some alternative element while maintaining reasonable properties. To phrase a viable technical approach to material substitution, it is essential to distinguish between the influence of composition on microstructure and its inherent influence on properties. The following example concerns the replacement of nickel by manganese in ferritic cryogenic steel. The technical approach employed in this case was based on the assumption that the principal influence of nickel is microstructural rather than chemical.

The low ductile-brittle transition temperature of tempered Fe-Ni cryogenic steels appears to have two sources: the dislocated lath substructure of the Fe-Ni martensite and the dense admixture of thermally stable austenite which intercritical tempering introduces along the lath boundaries. To the extent that the good properties of Fe-Ni martensites result from the structure itself, it should be possible to replace nickel by any other alloying element which permits the same substructure to be achieved. At low concentrations, manganese has an influence on phase stability and phase transformation kinetics in steel which strongly resembles that of nickel. Manganese is hence an attractive substitute for nickel. There is, however, an important difference between the influences of manganese and nickel which becomes apparent at higher solute contents. While nickel always promotes the austenite phase in steel, the addition of manganese can lead to the formation of hexagonal epsilon martensite, which intrudes in the Fe-Mn binary for manganese contents between the approximately 10 and 18 wt%. The intrusion of this epsilon martensite phase makes it difficult to achieve structures in Fe-Mn ferritic steels which are comparable to those obtainable in the Fe-Ni alloys. At manganese contents of 8 wt% or higher the intrusion of epsilon phase inhibits grain refinement on thermal treatment. At lower manganese contents, epsilon does not appear, but the precipitation of austenite on tempering is made difficult by the lean solute content.

These considerations suggest that if one is to achieve Fe-Mn substructures comparable to those in good Fe-Ni cryogenic steels, the Mn content must be sufficiently low so that the epsilon phase is avoided while heat treatment must accomplish a sufficient segregation of manganese to permit the formation of a solute-rich, thermally stable austenite in a dense distribution.

We were able to accomplish substructural refinement in Fe-Mn steels by selecting an alloy content of 5Mn and employing the "2BT" treatment, diagrammed in Fig. 11, which combines thermal cycling to refine the effective grain size of the steel with a final temper to introduce a distribution of stable austenite. Two of the steps in the grain refinement treatment, the 1B and 2B steps, involve an intercritical anneal which causes a diffusional decomposition of the parent ferrite. This diffusional decomposition creates a dense distribution of regions within the microstructure which are relatively high in manganese content. The precipitation of austenite during the intercritical temper is then kinetically rapid and is essentially confined to these microstructural regions in which the manganese content is already high. The microstructure achieved in 2BT-treated Fe-5Mn steel is illustrated in Fig. 12. This structure strongly resembles that of 2BT-treated 9Ni steel. If low temperature properties are determined more by microstructure than by composition, this steel should have a very low ductile-brittle transition temperature.

The results of mechanical property tests on Fe-5Mn at 77K are presented in Fig. 13. The alloy has a strength comparable to that of commercial 9Ni steel in the quench-and-temper and double-normalized-temper conditions. Its Charpy impact energy at 77K is also competitive with that of the best commercial 9Ni steels. Its fracture toughness at 77K is slightly below that of commercial 9Ni steel but is nonetheless very high. These results demonstrate that it is possible to replace nickel by manganese in ferritic cryogenic steels provided that the microstructure of the Fe-Mn alloy is engineered to resemble that of the Fe-Ni grades.

The results cited above are also important in that they seem to demonstrate that the ductile-brittle transition temperature of cryogenic steels is determined more by microstructure than by the inherent properties of the matrix composition. But it is not possible to conclude that the matrix composition is unimportant. The Fe-5Mn steel is tough at 77K in a structure resembling that which lowers the

ductile-brittle transition of 9Ni steel to below 4K. The two structures are, moreover, not identical. The question of the precise relative role of matrix composition and microstructure in determining low temperature mechanical properties, which is a very important question affecting the further development of ferritic cryogenic steels, can only be answered by further research involving dislocation theory.

### *C. Future Research on the Structure-Property Relation.*

For the purposes of the present paper we are interested in that research on structure-property relations which may make an important contribution to the science of alloy design. From that perspective, it should be kept in mind that only a small part of current research on the structure-property relation is directed toward alloy development. The bulk of such research is directed toward the successful prediction of the properties of materials whose structure is given.

In the long run research on the structure-property relation which is intended to improve the predictability of material properties and that which is intended to improve the design of materials will converge. But in the short term they are very different, and it is sometimes true that the very best research on the prediction of properties from structure is of very limited value to alloy design. The reason lies in the way the microstructure is parameterized. To be of direct value to the science of alloy design the structure-property relation must parameterize the microstructure in terms of variables which are accessible to direct observation and metallurgical control. Structure-property relations developed for the purpose of predicting properties often use a phenomenological parameterization of structure (such as the "hardness" of the Hart-Li (11) approach). While such models may be of enormous value in the prediction of material properties, from the alloy designer's perspective they only shift the problem to a new level since he is now faced with the difficulty of finding a microstructural foundation for the phenomenological variables which figure into the theory.

There are at least three currently active lines of structure-property research which promise to have a short term impact on new materials development. These are: fundamental research in dislocation theory, "equation of state" studies which employ constitutive variables chosen from the microstructure, and research on the nature of microstructure itself.

*1. Fundamental research.*

The ultimate value of fundamental research in dislocation theory lies in the possibility that it will eventually yield an essentially complete quantitative theory of those properties which are determined by the existence and behavior of dislocations. But a quantitative theory is likely to be a long time in coming. The short range impact of fundamental dislocation theory lies in the physical insight and understanding it provides and in the education or re-education of engineers who practice alloy design.

But in addition to its pedagogical function, fundamental research in dislocation theory can provide rather specific short time assistance to alloy development activities. The fundamental research is most helpful when it assists in the identification of dominant or rate-controlling steps in materials behavior which can be readily associated with features of the microstructure or modes of dislocation motion. One example of this kind of valuable input concerns obstacle hardening. Some twenty years of fundamental research have led to a reasonably thorough understanding of the manner in which individual mobile dislocations are impeded by microstructural barriers (12). On the basis of this work the materials scientist who is interested in tailoring precipitation-hardened or dislocation-hardened materials for a specific set of properties can often frame a rather clear statement of the microstructural barrier distributions he would like to achieve. A second example which seems to be emerging from recent research is the association between deformation patterns in structural materials and their sensitivity to hydrogen embrittlement and stress-corrosion cracking (13). In ferritic steels which form in the dislocated martensite structure, hydrogen embrittlement apparently promotes a change in the dominant quasi-cleavage fracture plane from the (100) plane common to steels broken below the ductile-brittle transition to the (110) plane, which is both the dominant slip plane and the dominant interlath plane in these materials. The identification of this change in the fracture mode immediately suggests microstructural modifications which might be used to improve the stress-corrosion cracking resistance of martensitic steels. In other structural alloys the sensitivity to stress-corrosion cracking appears to be associated with a tendency toward planar slip in the material, a tendency which apparently has an inverse in the sense that hydrogen charging seems to promote a more planar slip in certain alloys. Since dislocation theory also suggests microstructural mechanisms and compositional modifications which may minimize the

tendency toward planar slip, the clear identification of mechanistic associations of this type can become the plausible basis for new alloy design efforts.

### *2. The Mechanical Equation of State.*

The second kind of research which promises to make a continuing contribution to materials design is that research which aims to predict behavior from the "mechanical state" of a material, and which associates that state with measurable values of specific microstructural features. The classical examples of this sort of research are the familiar quantitative relations relating the strength of a material to the reciprocal root of its grain size or to the root of its dislocation density. An example of ongoing and very interesting research along these lines is provided by the recent work by Kramer, Weissmann and coworkers (14), who report finding a correspondence between the state of mechanical or environmental degradation of a material and its mean dislocation density as measured by standard X-ray techniques. Still another pertinent example is the apparent association between subgrain size and critical current characteristics in Type II superconductors (15). Even when the precise mechanistic source of such empirical structure-property correlations is unclear, their value to alloy development can be substantial.

### *3. The Nature and Characterization of Microstructure.*

The third line of research which may contribute substantially to the science of new materials development is research on the nature and appropriate characterization of microstructure. A clear notion of the concept of structure is certainly necessary to the understanding of the structure-property relation. However, at the present stage of the development of microstructural science our usual characterization of the microstructure extends only to the measurement of such global properties as mean grain size and overall dislocation density, supplemented by a verbal description of what the microstructure looks like at some level of magnification. It would be very useful to have both more probative concepts of microstructure and more informative characterization schemes.

Current research into the nature of microstructure takes two paths. The first follows quantitative stereology and focuses on identifying meaningful quantitative measures of the structure. The second approach is more verbal and conceptual and principally involves the classification of microstructures on the basis of their similarity to one another. The attractive feature of the quantitative approach is that it

produces numbers of the sort which might naturally fit into constitutive equations. Its disadvantage is that microstructures are sufficiently complicated, and the structure-property relation sufficiently obscure, that it is seldom obvious which aspects of the microstructure one ought to be measuring. The qualitative characterization of microstructure, on the other hand, is widely employed in alloy design. The materials scientist often selects a target microstructure on the basis of its resemblance to the microstructure of another material which is known to have properties resembling those he would like to achieve. Similarly, the most useful classifications of microstructure, such as those which identify the "phases" in steel or the various forms which martensitic products may take in steel (16) deal in qualitative descriptions of the overall characteristics of microstructure rather than in quantitative measures of structural detail. These qualitative characterizations of microstructure are often enormously useful but do not form a basis for quantitative prediction.

A useful future continuation of research on the characterization of microstructure might address the problem of joining the quantitative and the qualitative approaches. While the microstructures of real engineering materials are enormously complex, it is clear that they may be cast into categories with some reasonable precision, that the categories of principal interest are finite in number, and that they do not cover the whole possible range of microstructural arrangements but only some subset which is selected by the mechanisms of the relevant phase transformations. There are then quantitative substructural differences between various examples of each microstructural type. One might, therefore, attempt to construct a science of microstructure which is based on an ontological division of existing microstructures into classes and subclasses, with an appropriate quantitative stereology identified for each and used to quantify the salient differences between them.

A formulation of this type may prove useful in phrasing quantitative constitutive relations. Most materials scientists share an imprecise but quite definite empirical belief that materials that are similar in their microstructure are also similar in most characteristics of their behavior. If this belief stands quantitative scrutiny, then it can be translated into the mathematical statement that materials of a given type belonging to a given microstructural class will have their properties determined by constitutive relations having a common mathematical form. The quantitative microstructural measures which will

then find their place in such constitutive equations will be those which determine the comparative properties of different examples of the microstructural class. If microstructural classes are defined with sufficient precision, as in the case of the Johnson-Mehl or cellular microstructures (17), the appropriate constitutive parameters need only be the geometric scaling parameters which measure the difference between examples of a given microstructural class.

In pursuing these or other approaches to a more precise formulation of the structure-property relation, it should above all be kept in mind that there are three very different ways in which the structure-property relation may be phrased to assist in the development of new materials. If one possessed the ultimate solution to the structure-property problem, he could identify precisely what microstructure he should attempt to achieve. In the absence of an exact solution, qualitative and mechanistic reasoning can be used in conjunction with similarity arguments to suggest the general type of microstructure which should be achieved. At a still more primitive level one may indicate directions which promise to improve the properties of an existing microstructure.

#### IV. THE CORRELATION BETWEEN MICROSTRUCTURE AND PROCESSING.

##### *A. Introduction.*

The first step in the development of a new engineering alloy is the identification of an appropriate microstructure. The second step is the achievement of that microstructure through appropriate processing. The processing steps employed are thermal or mechanical. The former establish microstructure through the control of phase transformations or defect relaxation processes in the solid state. The latter are used to introduce defects into the material either to directly influence final properties or to provide a suitable intermediate microstructure for further processing.

The role of dislocations in processing is twofold. In some cases the dislocations are the intended product of the process; the processing steps are designed to introduce a certain dislocation content, or dislocation distribution, so as to control final properties. In other cases, dislocations are used as a part of the processing. Dislocations are intentionally introduced into the material to provide an intermediate



structure from which the desired final microstructure can easily be achieved.

*1. Dislocations as an element of microstructure.*

The common processing treatments which have the dislocation structure as their intended product include dislocation- hardening processes such as work-hardening or mechanical strengthening and softening processes such as recovery tempering and annealing. These processes are, of course, common in the processing of structural metals. While there is little relevant theory, the design of processes of this sort draws on a large body of experimental research and industrial experience.

Dislocation hardening processes are intended to bring structural alloys to a certain strength level. Their immediate objective is hence to introduce a sufficient quantity of defects into the material. The choice of processing techniques is usually dictated by economic and operational constraints. Sheet materials are usually cold rolled to high strength levels. Thicker pieces are difficult to cold roll, both because of the large forces required and because of the difficulty of establishing a uniform deformation structure in the work piece. These may be mechanically processed through warm or hot rolling processes or forging, though thermal recovery processes occur during deformation, and it is hence not always possible to obtain the desired strength levels. In the case of ferritic steels one may use the phase transformation as an alternative to mechanical processing. If the steel naturally forms a dislocated martensite substructure on quenching and if the hardenability of the steel is sufficient to establish the structure through the piece, then very high dislocation densities can be obtained. In materials such as aluminum alloys which do not undergo phase transformations on cooling, transformation processing is not readily applicable. More elaborate treatments such as shock treatments and explosive forming have been devised to achieve very high and uniform defect densities in the interior of these alloys.

As the science of materials processing develops, however, materials engineers increasingly recognize that the achievement of superior properties requires some control over the distribution of dislocations as well as over the total dislocation content. Even in thin sheet materials, cold deformation processing rarely establishes a defect structure which is uniform on the scale of the microstructure. At higher strength levels cold work tends to embrittle the alloy and the embrittlement is often believed to result, in part, from the non-uniformity of the deformation structure. Experience shows that an

improved combination of strength and toughness is often obtained if high strength alloys are either overworked cold and then given a recovery annealing treatment to lower their strength to the desired level, or if they are warm worked rather than cold worked. While the science underlying these observations is not well developed, the reason presumably lies in the redistribution of dislocations during warm working or recovery tempering which relaxes regions of very high internal stress within the material which might otherwise act as sources of early fracture. These experimental observations suggest that it may become possible to fine-tune the deformation processing of alloys if fundamental research can create a deeper and more specific understanding of the relevant reactions.

In the case of superconducting materials, an elementary theory exists to guide deformation processing. The relevant theoretical work suggests that isolated dislocations do not provide efficient pinning sites for magnetic flux lines in Type II superconductors, but that cell or grain boundaries are very effective. The commercial processing of important Type II superconductors, such as Nb-Ti, involves a severe deformation of the material in the course of drawing it into a multifilamentary wire, which is followed by a tempering treatment that improves its critical current characteristic. While detailed microscopy has not been done, the tempering treatment presumably serves as the recovery temper which polygonizes the dislocation structure into cell boundaries. Cell boundaries then act as more efficient pinning sites for the magnetic flux lines.

## *2. Dislocations as an element of processing.*

Materials processing techniques which employ dislocations as an integral part of processing are of many different types, and are widespread in materials engineering. The dislocations are used for at least three different purposes: to promote phase transformations by increasing the thermodynamic driving force for the transformation, to increase the transformation rate, and to control the morphological features of the phase transformation. In many processing techniques the dislocations perform more than one of these functions simultaneously.

The classic case in which dislocations are used to increase the thermodynamic driving force for a phase transformation is the recrystallization of metals. Modern controlled rolling processes for high strength, low alloy (HSLA) steels, for example, combine hot deformation, recrystallization, and

quenching in a continuous process on the production line to achieve a fine grained sheet steel product. In the simplest such process the steel is rolled to its final desired thickness at a temperature which permits rapid recrystallization after the sheet of steel passes through the rolls. At the completion of the recrystallization process, but before any significant grain growth can occur, the steel is quenched rapidly to room temperature to preserve the fine grain structure.

A common example of the use of dislocations to increase the kinetics of phase transformation is in the thermomechanical processing of precipitation hardening steels. In these kinds of processes severe deformation is used either concurrently with or immediately before the aging treatment. The specific influence of dislocations on the kinetics of precipitation reactions is not fully understood theoretically, but it involves some combination of at least three effects: an increase in the kinetics of diffusion, both through the action of dislocations as diffusion pipes and through supersaturation of point defects induced into the lattice by dislocation motion; an increase in the thermodynamic driving force for precipitation due to internal strains developed by the deformation process; and the provision of efficient precipitate nucleation sources, particularly at nodes and pileups in the dislocation structure.

Dislocations can also be useful to control the morphological characteristics of precipitation reactions. Deformation processes are often helpful in insuring that the precipitates form in the interior of grains rather than along grain boundaries. The reason for the change of the precipitation site after deformation is presumably the provision of new and efficient nucleation sites within the interior of the grains.

*B. Examples of the use of dislocations in the processing of new materials.*

The two examples below are taken from recent work at Berkeley and include a case in which thermal processing was used to create a high dislocation density in an austenitic steel for the purpose of improving its strength, and a case in which thermomechanical processing was used for the treatment of a superconducting material to control the precipitation of the A15 superconducting phase.

*1. Transformation-strengthening a high strength austenitic steel (18).*

A principal reason for the high strength of ferritic or martensitic steels comes from the fact that these steels undergo a phase transformation on cooling and hence have high defect densities in the as-cooled state. Since there is no comparable phase transformation in austenitic steels, these steels must be mechanically worked in order to achieve a defect density conducive to high strength. However, mechanical working is often an undesirable processing step, particularly when the steel is to be used as a relatively thick piece.

A method for obtaining transformation-strengthening in austenitic steels was suggested and developed some years ago by Krauss and Cohen (19). Their approach was to modify the composition of the steel so that the martensite start temperature  $M_s$  lies between room temperature and liquid nitrogen temperature. In this case the steel can be partially transformed to martensite by chilling it in liquid nitrogen and then retransformed to austenite by heating at moderate temperature. If the reheat temperature is sufficiently low and the reheat time sufficiently short, the double martensitic transformation introduces a high density of defects into the austenitic lattice which increases the strength of the alloy. In its original form, however, the Krauss and Cohen procedure does not impart ultra high strength to the alloy, and is difficult to apply to thick plates because of recovery of the dislocation structure during the necessarily slow heating steps.

To create an ultra high strength austenitic steel through transformation strengthening, we sought to combine the benefits of transformation strengthening with those obtainable from precipitation hardening. The alloys used were iron-based superalloys essentially from the Fe-Ni-Ti ternary. An example composition was Fe-34Ni-3Ti-0.5Al-1Mo-0.3B-0.01V. The function of the Fe-Ni-Ti group in the composition was to make the alloy age-hardenable through precipitation of the  $\gamma'$   $Ni_3Ti$  precipitate. The function of the Mo-V-B group was to suppress grain boundary precipitation during the age-hardening step so that efficient age-hardening could be achieved. The alloys were processed through a three step sequence: The alloys were age-hardened so as to create a fine precipitate structure and achieve high strength. The alloys were then transformation-strengthened by cooling to liquid nitrogen then reheated to above the austenite finish temperature  $A_f$  to introduce a high density of dislocations into the decorated matrix. The alloys were finally given a low temperature secondary aging treatment to

stabilize the austenite and improve the defect structure to achieve a higher strength-toughness combination. The thermal processing sequence is diagrammed in Fig. 14. The initial composition of the alloy was chosen so that after the aging process the  $M_s$  temperature of the alloy was between room temperature and liquid nitrogen temperature. The  $M_s$  must, in fact, lie below approximately  $-70^\circ\text{C}$  so that the  $M_d$  temperature, the temperature at which martensite begins to appear on mechanical deformation does not lie too close to room temperature. If the  $M_d$  is too low then the yield strength of the alloy will be controlled by the martensite transformation rather than by normal plastic deformation processes.

The development of the alloy microstructure during the processing sequence is illustrated in Fig. 15. Figure 15a shows the starting microstructure, which is a large-grained and clean austenite. Aging (Fig. 15b) develops a dense distribution of fine gamma prime precipitate particles. Refrigeration in liquid nitrogen (Fig. 15c) accomplishes a partial transformation into the martensite phase. The martensite plates contain a high density of internal twins. Reversion to austenite and cooling to room temperature produces a final austenite structure (Fig. 15d) whose grains are decorated by regions of very high dislocation density. These dislocated regions are the "ghosts" of the previous martensite phase.

Transmission electron microscopy (Fig. 16) reveals both the high dislocation density and the pinning of the dislocation structure by the precipitate phase. Because of the interaction between the transformation-induced defects and the precipitate structure, the defect structure is extremely stable and persists even after several hours of annealing at temperatures of several hundred degrees centigrade. The final brief aging appears to relax the dislocation structure and to cause a very fine scale secondary precipitation. This fine scale secondary precipitation lowers the  $M_s$  temperature of the alloy, which increases the stability and also adds slightly to the strength.

The change in the properties of the alloy with processing is presented in Table I. Alloy strengths as high as 180 Ksi yield have been achieved in iron-based superalloys processed in this way. The central element of the processing is the use of the combination of precipitation and martensitic phase transformation to introduce a dense and stable dislocation structure.

In the context of this paper it should be pointed out that the strengths obtained in these transformation-strengthened austenitic steels were high but were not sufficiently high to meet the target

objectives of the alloy development on which we were working at the time. The objective of that program was to develop an austenitic steel which might be used in the retaining rings of large electrical generators, and the strength requirement was for a yield strength approaching 200 Ksi. Our inability to achieve target values relates to difficulties in controlling the martensitic transformation which have their origin, we believe, in the role of dislocations. To achieve higher strength it is necessary to increase the fractional transformation to martensite at 77 K. This cannot be done by raising the  $M_s$  temperature because the  $M_d$  will then become too high and the yield strength will decrease due to premature transformation to the martensite phase under mechanical load. What is needed is to narrow the temperature difference between the  $M_d$ ,  $M_s$ , and  $M_f$  temperatures for the martensite transformation. A survey of all likely alloy element additions suggests that these increase the temperature difference between  $M_s$  and  $M_f$ , and hence lead to a decrease in achievable strength. This undesirable result is almost certainly due to a chemical influence on the behavior of dislocations in the lattice, since the nucleation of martensite is believed to be dislocation dominated. It may be possible to find some clever means for overcoming these problems and achieving very high strengths in transformation-strengthened austenitic steels but such a breakthrough is, in our opinion, unlikely until dislocation theory sheds more light on the source and control of the martensitic transformation.

*2. A15 superconductors through direct precipitation processes (20).*

The hard superconductors preferred for use in high-field superconducting magnets are  $A_3B$  compounds in the A15 structure, where the major component is Nb or V and the minor component is Sn, Al, Si, Ga, or Ge. These A15 compounds are brittle intermetallics. Wires based on these A15 compounds are manufactured by indirect means, such as the "bronze" process now used for the commercial manufacture of multifilamentary superconductors containing  $Nb_3Sn$  or  $V_3Ga$  (21). The bronze process is, however, inapplicable for thermodynamic reasons to other A15 superconducting compounds such as  $Nb_3Al$ .

One promising alternative process for the manufacture of A15 superconducting wires is the "direct precipitation" process which is under development in our laboratory. In this process a homogeneous solid solution, which is supersaturated with respect to precipitation of the A15 phase, is formed by

solidification and homogenization at high temperature. The solid solution is quenched to room temperature rapidly enough to suppress the formation of the A15 phase. The product is a ductile metal, which may be formed into a wire or tape through deformation processing at relatively low temperatures. The wire or tape is then aged at an intermediate temperature to precipitate the A15 phase. To create a superconducting wire having good current-carrying characteristics through the direct precipitation process one must, of course, insure thermodynamic conditions which result in an A15 precipitate with good inherent characteristics. Good properties also require a suitable morphology of the precipitation reaction. One wishes to obtain a high volume fraction of the precipitate phase and to have this phase configured into a continuous network of very fine grained particles.

To achieve a high volume fraction of fine grained particles in a continuous network, we utilized two characteristic behaviors of the high density of dislocations introduced by mechanical deformation. First, if the deformed wire is heat treated at intermediate temperature, the dense distribution of dislocations will rapidly polygonize into a fine cell structure. Second, the cell walls of the structure provide good catalytic sites for nucleation of the A15 phase. An example of the microstructure of fully processed Nb-18.8 at% Al is shown in Fig. 17. The figure illustrates the essentially continuous precipitation of fine grained  $Nb_3Al$  along the network of subgrain boundaries formed by polygonization of the dislocation structure introduced during the deformation process.

The direct precipitation process is under continuing development. The experimental samples produced to date show promising superconducting properties, though these are not yet competitive with the best available commercial superconducting wires. In the case of  $Nb_3Al$  the major problem concerns the inherent properties of the A15 superconducting phase, which must be brought closer to their known values at stoichiometric composition.

*C. Directions for Future Research.*

The field of material processing is so broad that it is difficult to identify an organized set of general research areas in which fundamental research in dislocation theory would be helpful. What I shall do in the following section is to identify four more specific research directions which could provide the needed background for specific breakthroughs in new materials development. The topics are a personal selection; other authors will undoubtedly identify equally important areas which are not included in this brief list.

*1. The mechanism of martensite transformation.*

While virtually everyone involved in the field of metallurgy would agree that the mechanism of martensitic phase transformations is one of the outstanding problems of the subject, most would place this problem in the field of phase transformations rather than dislocation theory. It is, however, well known that dislocations play an extremely important role in martensitic transformations, and it is becoming increasingly apparent that the understanding and engineering control of martensitic transformations in steel will require a rather large input from those whose basic expertise is in the behavior of dislocations. Dislocations appear to be critical to the nucleation of martensite, the autocatalytic development of the martensitic transformation, the morphology of the martensitic product, and the enhancement of martensitic transformations by plastic deformation. Virtually all current theories of martensite nucleation hypothesize that the nucleation is assisted by dislocations. The major qualitative discrepancy between the models which are presently most active concerns the specific mechanism of dislocation participation. In some models, for example that of Olson and Cohen (22), the principal role of dislocations is geometric. A specific geometric configuration of appropriate dislocations splits into partials so as to create an island of the transformed phase. This model is easy to visualize in the case of FCC-HCP transformations, such as the formation of epsilon martensite in steel, but it is less clear in the case of the FCC-BCC transformation such as the transformation to the usual  $\alpha'$  martensite. The second class of models which have been proposed, and are supported by Suzuki (23) and Khachatryan (24) among others stress the role of dislocations in providing regions of severe elastic strain in which the initiation of martensite may become relatively easy. These models gain particular experimental



support from the work of Suzuki (23) and others on strain-induced transformations in steel in which the martensite phase is found to nucleate at the intersections of active slip bands in variants which are compatible with the local state of internal stress.

*a. Metastable austenitic steels.* A sound theory of the nucleation of martensite under both thermal and strain conditions would be enormously helpful in the development of both new alloy steels and other materials which undergo martensitic transformations. One specific example was given earlier and concerns the possibility of obtaining high strength in austenitic steels through transformation strengthening. A second set of examples concerns the design of austenitic structural alloys that are stable with respect to martensitic transformations during plastic deformation.

Interest in the stability of austenitic steels has been stimulated by the recent demonstration (25) that martensitic transformations in the highly stressed regions near internal flaws in austenitic steels contribute to their sensitivity to hydrogen embrittlement. Hydrogen, in fact, appears to promote martensitic transformation near propagating fatigue cracks. The design of hydrogen-resistant steels would therefore benefit from a more detailed understanding of the strain-induced martensitic transformation and the apparently catalytic role of hydrogen, which may have its source in the influence of hydrogen on the behavior of the dislocations which promote martensite nucleation.

The understanding and control of strain-induced martensitic transformations is also important to the development of structural materials for use at deep cryogenic temperatures, for example, in the structure of high-field superconducting magnets for magnetic fusion energy devices. Metastable austenitic steels are attractive for use in these superconducting magnets because of their availability and their good low temperature properties in conventional mechanical tests. However, in the operation of such systems these steels will see a combination of cryogenic loading, fatigue, and thermal cycling in a high magnetic field. Current understanding of strain-induced transformations in metastable austenites does not permit a reasonable forecast of the response of candidate alloys to this sort of complex deformation history. It is important to know whether potential martensitic transformations pose a threat to the structural integrity of these alloys.

*b. Martensitic steels.* Dislocations also influence the internal state and the morphology of the product of martensitic transformations. This influence is most pronounced in the shift from "dislocated" to "twinned" martensite which occurs with increasing carbon content in many alloy steels. The identification of composition or processing modifications which promote the formation of dislocated martensites is of interest because of the better mechanical properties usually exhibited by steels having this structure. On a finer scale, moreover, dislocations influence the size of the martensite laths within dislocated martensite, their alignment into lath "packets", and the internal state of these packets. These detailed features of the microstructure affect both the properties of the steel and its response to further heat treatment.

An improved understanding of this behavior may well lead to the development of new martensitic steels having excellent low temperature properties after simple thermal treatments. As discussed above, the microstructural element which appears to determine the ductile-brittle transition temperature of lath martensite steels is the dimension of the martensite packet. This packet consists of a number of individual martensite laths which are in an almost perfect crystallographic registry with one another, and hence share a common crystallographic cleavage plane. In current practice, one lowers the ductile-to-brittle transition temperature of the steel by heat treating it so as to break up the packet alignment. Simpler heat treatments, and perhaps better steels, could be obtained if it were possible to establish a direct metallurgical control over the formation of packets so as to prevent the crystallographic alignment of laths in the first place. Since both the initial establishment of the packet structure and the retention of the packet structure through cyclic heat treatments is certainly influenced by the dislocation distribution associated with the transformation, the problem of understanding and controlling lath alignment is a problem in dislocation theory.

## *2. Thermomechanical processing.*

A second set of processing techniques which could benefit from the serious attention of dislocation theorists comes in the area of thermomechanical processing and particularly in those processing techniques which utilize mechanical work to improve the structural properties obtainable through subsequent precipitation hardening. The enhancement of properties by thermomechanical treatment can be

quite spectacular. Figure 18, for example, shows the strength-toughness characteristic of a class of high-strength austenitic alloys developed in our laboratory for possible use in the retaining rings of large electric generators (25). The alloy was in some cases aged directly after high temperature forging and was in other cases aged after having been given an intermediate anneal to remove the forging damage. As can be seen from the figure, the materials processed from the as-forged condition have a strength-toughness characteristic which offers approximately 20 Ksi higher strength at a given toughness level than does that of the materials processed after intermediate anneal.

The role of dislocations in this kind of thermomechanical processing is at least twofold: dislocations improve the kinetics of the precipitation process by enhancing diffusion of the precipitating species and influence the morphology of the precipitate distribution by providing nucleation sites for precipitate particles. If the dislocations are retained in the structure through the aging treatment, then they may also contribute a dislocation hardening component to the final alloy strength. None of these contributions of dislocations to thermomechanical processing is well understood at this time. Consequently the development of thermomechanical processing sequences is almost purely empirical. Fundamental contributions by dislocation theorists to the the understanding of thermomechanical processing will almost certainly lead to the development of new structural materials having exceptional properties.

### *3. Semiconducting materials.*

Dislocations in semiconductors are almost always undesirable as these introduce linear arrays of electronically active sites which are unwanted and which interfere with the electrical performance of the piece. Moreover, dislocations have the particularly nasty habit of appearing in arrays near interfaces and junctions where the control of electrical properties is particularly important. The dislocations are, in this case, induced by the stresses caused by lattice mismatch if the boundary is coherent or by differences in thermal contraction characteristics if the boundary is, for example, an electrical connection made by soldering or joining a metallic conductor at some higher temperature.

The variety of ways in which dislocations appear and unfavorably influence electrical or optical characteristics is almost as great as the variety of semiconducting devices itself. The considerations which influence the engineering control of the problem are, however, similar in most of these cases.

One must either prevent the formation of dislocations in the first place, or one must somehow render these dislocations relatively innocuous to the electrical properties .

Preventing the formation of dislocations requires at least two types of information: a knowledge of the stresses developed at the site, usually an interface, at which the dislocations are likely to form and a knowledge of the specific mechanism involved in dislocation nucleation. The computation of relative stress distributions in multi-layer devices which may have been subjected to rather complex thermal histories is not simple and remains unsolved in many situations of interest to the electronics industry. The principal mechanisms of dislocation nucleation, vacancy condensation into dislocation loops, emanation from free surfaces, and dislocation punching from inclusion particles, are reasonably well known. There is, however, considerable room for profitable research directed toward the suppression or control of these dislocation sources in situations of particular engineering interest.

If the nucleation of dislocations cannot be entirely prevented, then research must also address the question of how those dislocations can be rendered relatively innocuous in their influence on electrical properties. The most direct solution to this problem would be to find means for titrating or altering the electronic state of the dislocations so that the electronic defect was removed or rendered less harmful. It is, unfortunately, difficult to identify plausible means for doing this. An alternative solution is to process the material so that the dislocations are removed to less harmful positions within the semiconductor.

#### *4. Stress relaxation processes.*

Material processes which involve the solidification of metal under constrained conditions often lead to high residual stresses in the finished piece. These stresses may cause quench-cracking or microfissuring in the cooled part. Even if the piece remains intact the presence of high residual stresses make it liable to cold cracking or premature fracturing in service. Since it is often not practical to heat treat welded or cast materials after they have been manufactured, it is desirable to find some means for minimizing or eliminating severe concentrations of internal stress during cooling or after the part has returned to room temperature.

One method for relieving internal stress concentrations which has been frequently suggested, though used with mixed results, is the use of mechanical vibration or the introduction of acoustic waves either as the part cools or after it has reached room temperature. When this technique is successful the most plausible interpretation for its success is that the vibration liberates dislocations which flow in response to the internal stress so as to relieve it. In other words, the vibration induces an accelerated relaxation creep of internally stressed piece.

My own research group has recently looked into this type of processing as a means of relieving internal stresses in welds in parts which are intended for use at cryogenic temperatures. As far as we have been able to discover, there is very little relevant fundamental research to assist one in designing a technological process of this sort, though there is a vast literature on the subject of internal friction which addresses some of the relative fundamental interactions. Given the technological importance of the residual stress problem, and the possibility that internal stresses may be relieved through fine tuned redistribution of the dislocation structure at low temperature, this would also seem to be a very fruitful area for fundamental research.

## V. CONCLUSIONS

In the foregoing sections of this paper I have tried both to provide some specific examples of where a knowledge of the behavior of dislocations is beneficial to new materials development and to indicate some useful directions for further research. But I would, in conclusion, like to reemphasize that the influence that dislocation theory and other types of fundamental research in materials science will have on the development of new materials is almost wholly dependent on the technical approach of those who control the materials development. As new materials development becomes increasingly scientific in its approach and increasingly microstructural and mechanistic in its orientation, as I believe it will and must, then fundamental investigations and basic understanding will become increasingly important to it. As long as the act of new materials development remains empirical in its approach and chemical in its orientation, the best fundamental research can be no more than a useful adjunct to it.

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REFERENCES

1. C. S. Smith, A History of Metallography, University of Chicago Press, Chicago (1960)
2. A. R. Von Hippel, Molecular Science and Engineering, Technology Press of MIT and J. Wiley, N.Y. (1959).
3. C. T. Sims, Superalloys, Wiley Interscience, N.Y. (1972).
4. R. G. Davies and C. L. Magee, Proc AIME Symp. on Structure and Properties of Dual Phase Steels, New Orleans, 1979. ed. by R. A. Kot and J. W. Morris, Jr.
5. K. E. Dorschu, Welding Research Council Bulletin 231, Welding Research Council, N.Y. (1977)
6. S. Jin, J. W. Morris, Jr., and V. F. Zackay, Met. Trans., A., 6A, 141 (1975); S. Jin, S. K. Hwang, and J. W. Morris, Jr., *ibid.*, A, 6A, 1569 (1975).
7. C. K. Syn, S. Jin, and J. W. Morris, Jr., Met. Trans., A., 7A, 1827 (1976) C. K. Syn, B. Fultz, and J. W. Morris, Jr., *ibid.*, A, 9A, 1635 (1978); J. I. Kim, Ph.D. Thesis University of California and Lawrence Berkeley Lab. LBL Report 9956 (1979).
8. J. W. Morris, Jr., C. K. Syn, J. I. Kim, and B. Fultz, Proc. Int'l Conf. on Martensitic Transformations (ICOMAT-79), MIT, Cambridge, 1979, p 572; J. I. Kim and J. W. Morris, Jr., Met. Trans., A, 11A, 1401 (1980).
9. S. Jin, D. Huang, and J. W. Morris, Jr., Met. Trans., A, 7A, 637 (1976).
10. S. K. Hwang and J. W. Morris, Jr., Met. Trans., A., 10A, 545; *ibid.*, 11A, 1197 (1980); M. Niikura, J. W. Morris, Jr., *ibid.*, 11A, 1531 (1980).
11. E. W. Hart, Acta Met., 18, 559 (1970); E. W. Hart and Che-Yu Li, Proc. 3rd Int'l Conf. on Zirconium in Nuclear Industry, Quebec, 1976, ed. by A. L. Low, Jr., and G. W. Parry, ASTM STP No. 633 p315.
12. U. F. Kocks, A. S. Argon and M. F. Ashby, Thermodynamics and Kinetics of Slip, Progress in Materials, Science, Vol. 19; J. W. Morris, Jr., and D. H. Klahn, J. Appl. Phys., 44, 4482 (1973).

13. Proc. 3rd Int'l Conf. on Effect of Hydrogen on Behavior of Materials, Jackson Hole, Wyoming, 1980, AIME (in press).
14. I. R. Kramer, *Met. Trans.*, 5, 1575 (1974); R. N. Pangborn, S. Weissmann and I. R. Kramer, *Scripta Met.*, 12, 129 (1978).
15. A. M. Campbell and J. E. Evetts, *Adv. Phys.*, 21, 199 (1972); A. D. McInturff and G. G. Chase, *J. Appl. Phys.*, 44, 2378 (1973).
16. T. Maki, K. Tsuzaki, I. Tamura, *Trans. Iron and Steel Inst., Japan*, 20, 207 (1980)
17. W. A. Johnson and R. F. Mehl, *Trans. AIME*, 135, 416 (1939); K. W. Mahin, K. Hanson, and J. W. Morris, Jr. *Acta Met.*, 28, 443 (1980).
18. S. Jin, J. W. Morris, Jr., Y. L. Chen, G. Thomas, and R. I. Jaffee, *Met. Trans.*, A, 911A, 1825 (1978); J. W. Morris, Jr. and G. Thomas, High Strength Austenitic Alloys for Generator Retaining Rings, EPRI Report FP-1061, April, 1979, Electric Power Research Inst., Palo Alto, California.
19. G. Krauss and M. Cohen, *Trans. AIME*, 224, 1212 (1962).
20. M. Hong and J. W. Morris, Jr., *Appl. Phys. Letters*, 37, 1044 (1980); M. Hong, Ph.D. Thesis University of California, Berkeley, Lawrence Berkeley Laboratory Report 11021, 1980.
21. A. R. Kaufmann and J. J. Pickett, *Bull. Am. Phys. Soc.*, 15, 838 (1970); K. Tachikawa, Proc. 3rd Int'l Cryogenic Eng. Congress, Berlin, 1970, Illife Science and Technology, Surry, 1970.
22. G. B. Olson and M. Cohen, *Met. Trans.*, A, 7A, 1897-1923 (1976).
23. T. Suzuki, H. Kojima, K. Suzuki, T. Hashimoto, S. Koike and M. Ichihara, Proc. Symp. on New Aspects of Martensitic Transformation, Kobe, 1976, Japan Inst. of Metals, p 339.
24. A. G. Khachaturyan and G. A. Shatalov, *Soviet Phys., JETP*, 29, 557, (1969).
25. R. B. Benson, Jr., R. K. Dann, and L. W. Roberts, Jr., *Trans., AIME*, 242, 2199, (1968); K. Chang and J. W. Morris, Jr., Proc. Int'l Conf. on Effect of Hydrogen on Behavior of Materials, Jackson Hole, Wyoming, 1980, AIME (in press).



FIGURE CAPTIONS

1. An example of ductile-brittle behavior showing fracture surfaces.
2. The Yoffee Diagram.
3. Optical micrograph of martensite in as-quenched 6Ni steel.
4. Transmission electron micrograph of the martensitic substructure in as-quenched 6Ni steel.
5. Fracture path profile in tempered 9Ni steels.
6. The 2B thermal cycling treatment in Ni steels.
7. The evolution of microstructure in 9Ni steel during the 2B treatment.
8. The fracture toughness-yield strength relationship in 250 grade maraging steels.
9. A heat treatment to increase and pin dislocations in 250 grade maraging steel which involves aging, rapid austenitization (the middle step) and stabilization (the last step).
10. The improvement in fracture toughness accomplished by the heat treatment shown in Fig. 9.
11. The 2BT treatment as applied to 5Mn steels.
12. Transmission electron micrographs of the 2BT-treated 5Mn steels. The time noted on the micrographs represents the holding time during the final tempering (T) treatment.
13. Comparison of the mechanical properties of 2BT-treated Mn steels with those of other cryogenic alloys at 77K.
14. The transformation strengthening and precipitation-hardening treatment used for in Fe-34%Ni-3%Ti alloys.
15. The evolution of microstructure in an Fe-34Ni-3Ti alloy during the thermal processing sequence shown in Fig. 14.

16. Transmission electron micrograph of Fe-34Ni-3Ti alloy after the heat treatment shown in Fig. 14.
17. Transmission electron micrograph of fully processed Nb-18.8 at% Al.
18. Strength-toughness characteristics of iron-based superalloys. A286 and Inconel 718 are commercial alloys. "AN" and "Af" represent specimens thermally processed with and without annealing after the initial homogenization and hot forging.

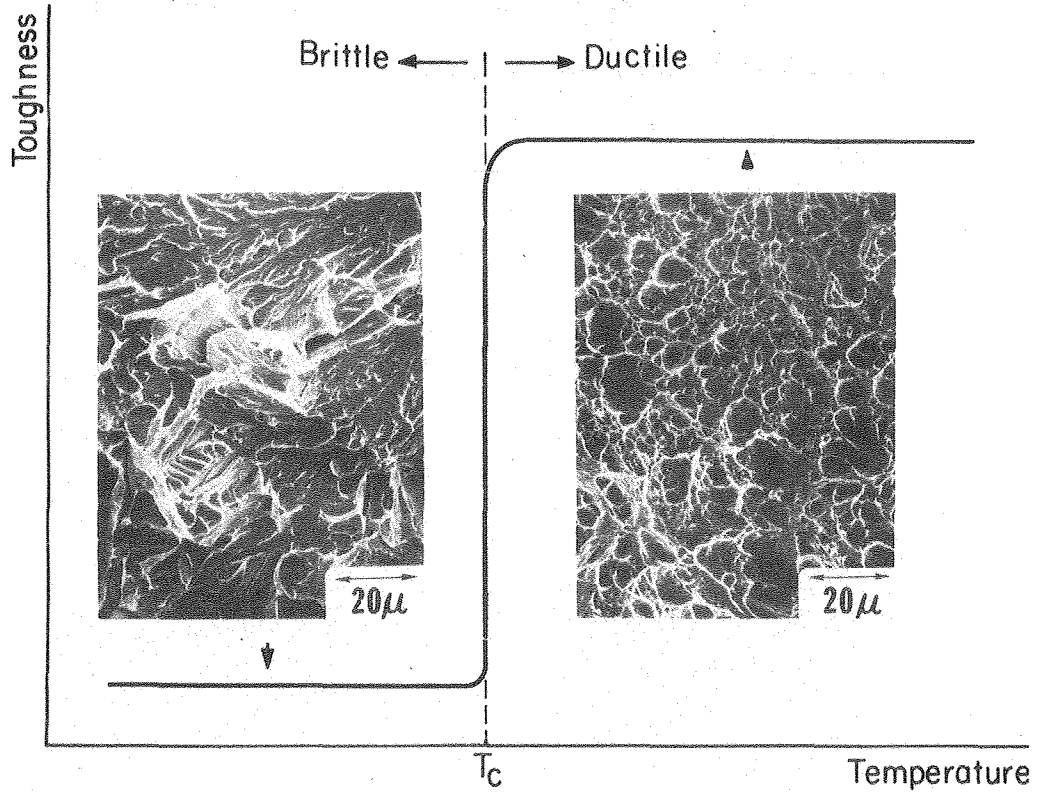


Figure 1

XBB800-14845

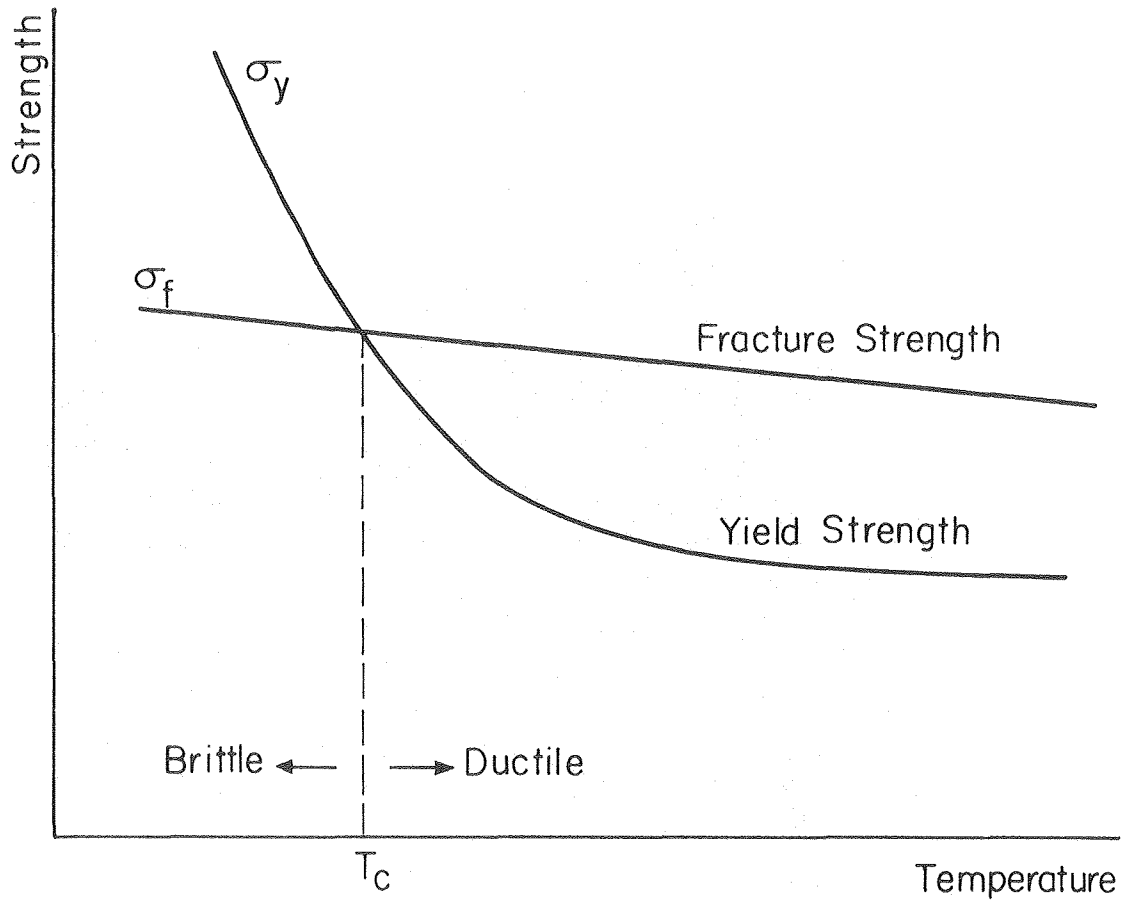


Figure 2

XBL8012-13404

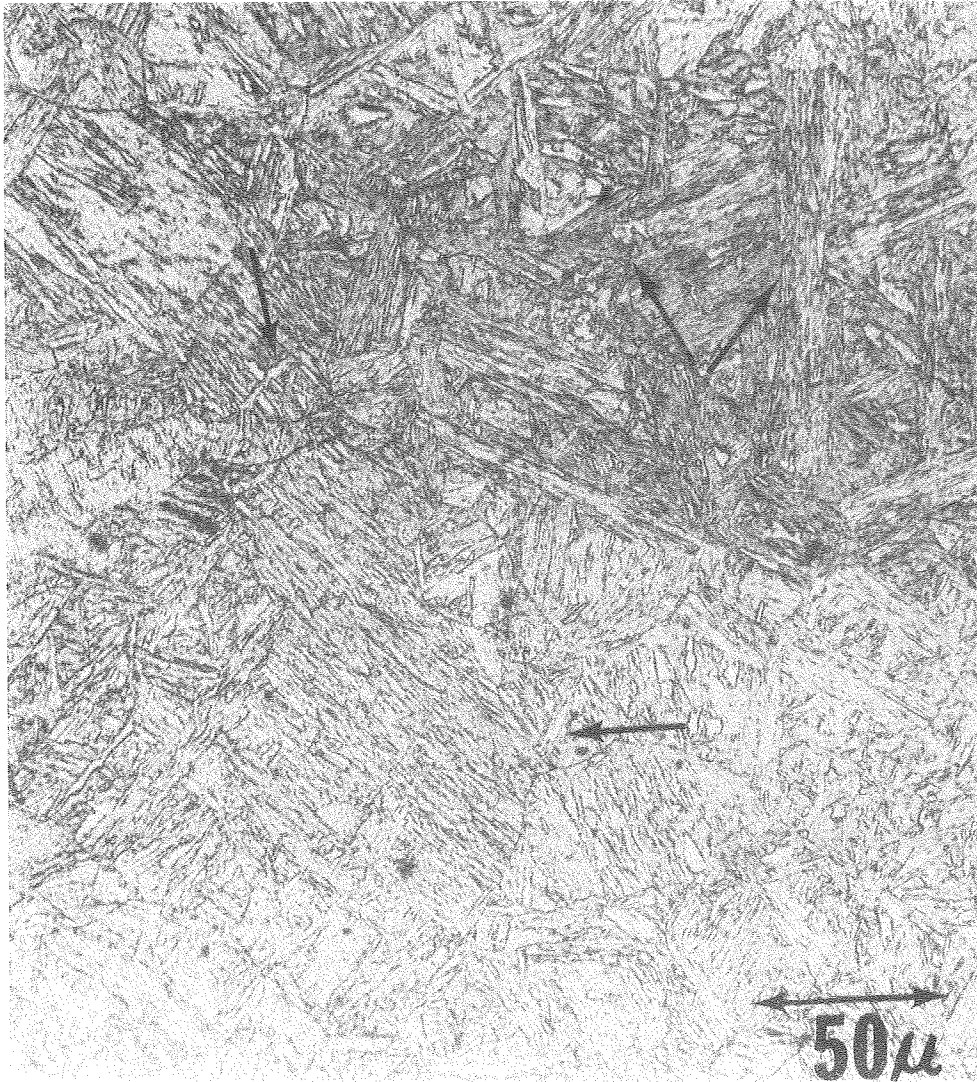


Figure 3

XBB796-7883

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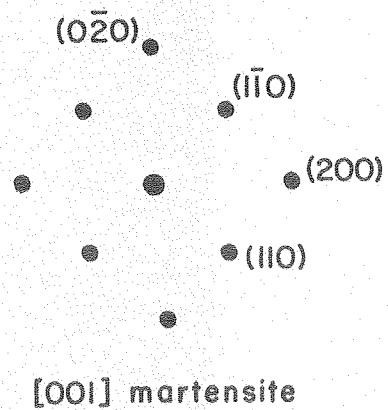
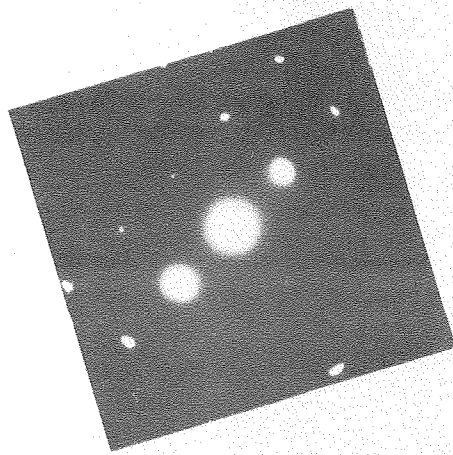
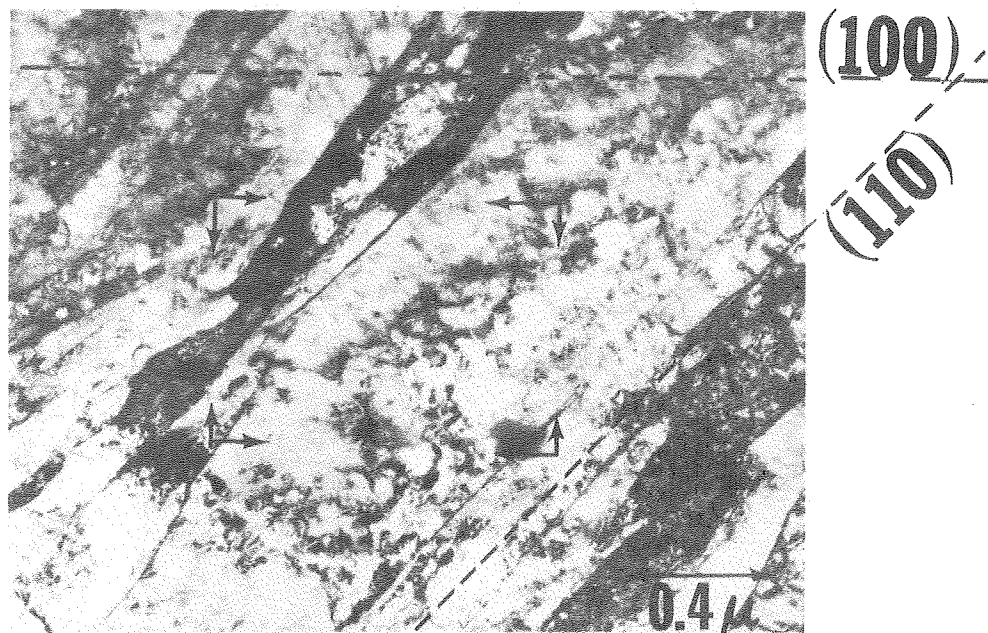
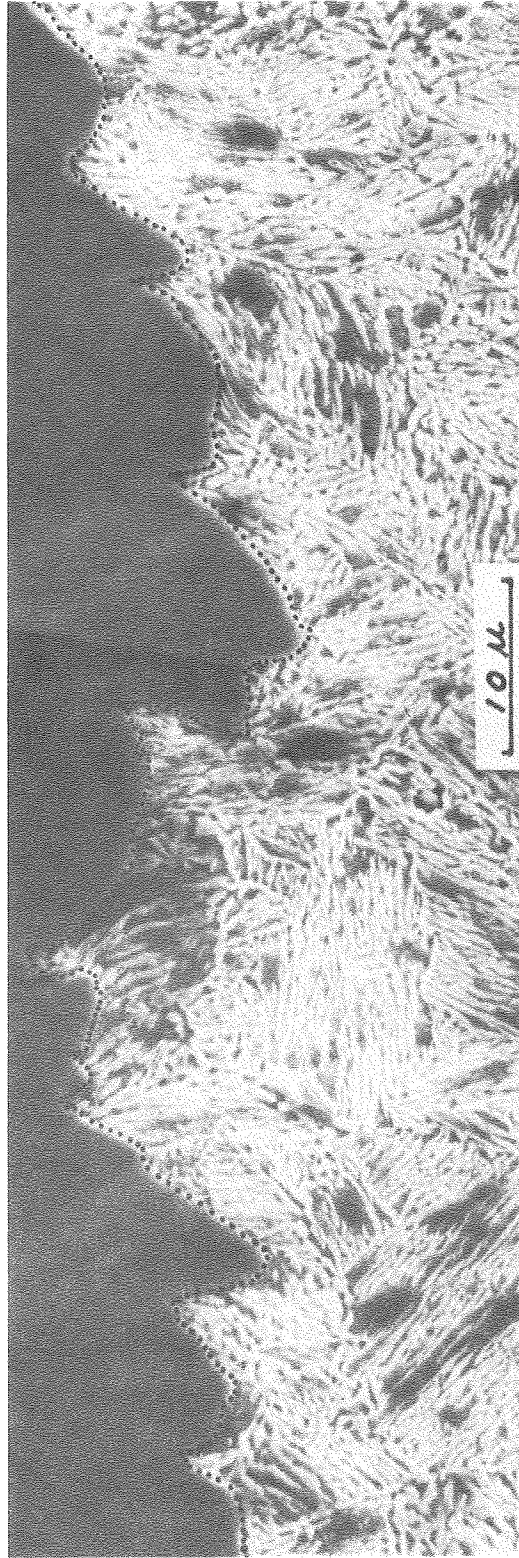


Figure 4

XBB796-7884

**QUENCHED**



XB5800-14489

Figure 5

Fe - Ni PHASE DIAGRAM

HEAT TREATING CYCLES

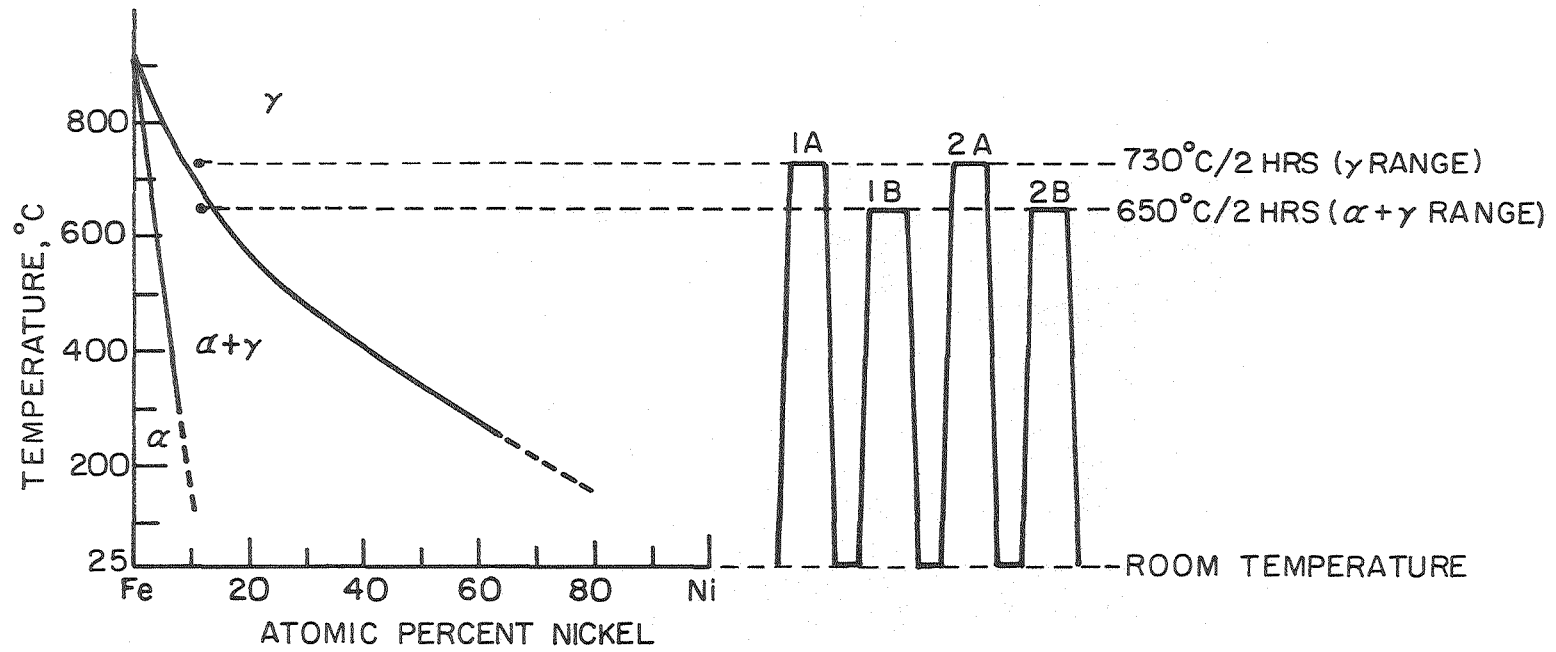


Figure 6

XBL 739-1884



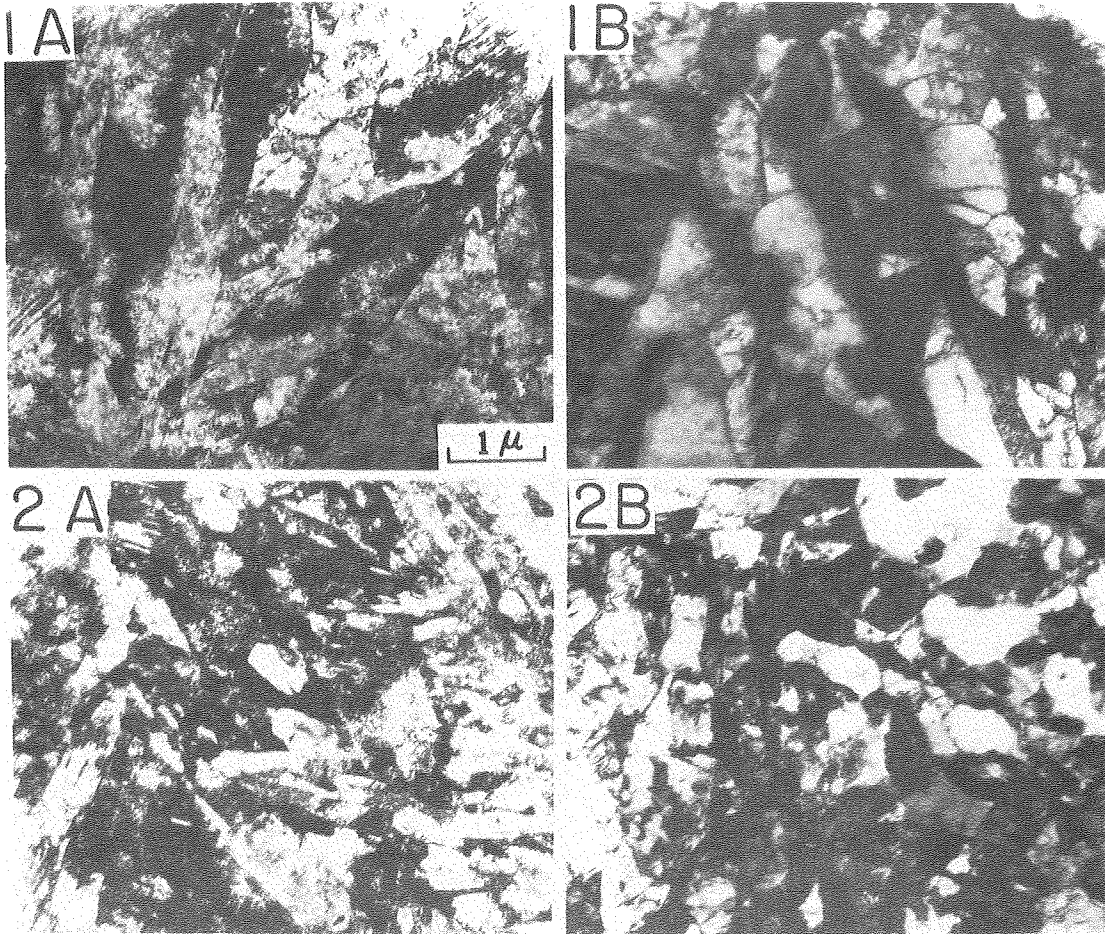


Figure 7

XBB769-8361

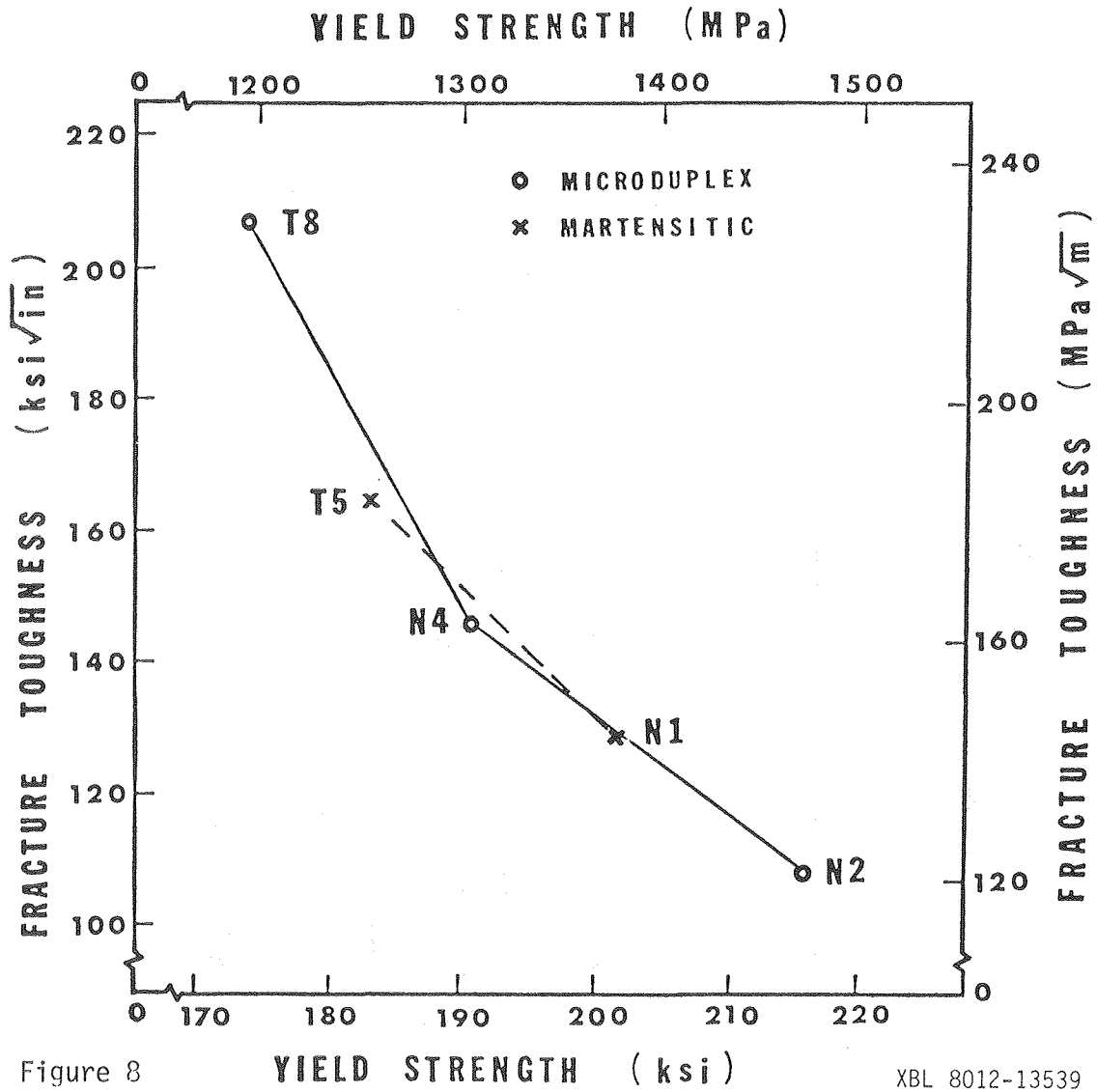


Figure 8

XBL 8012-13539

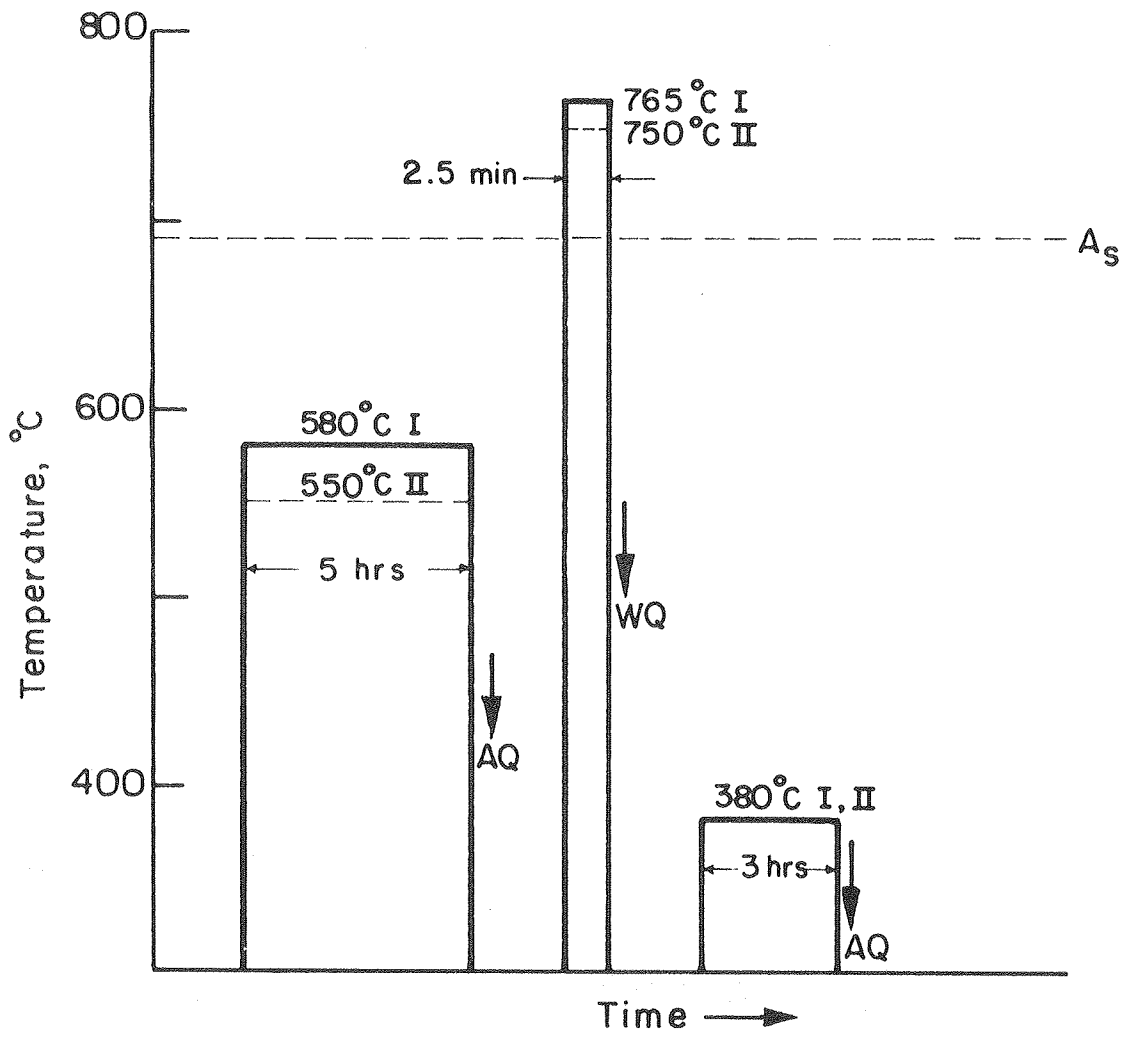


Figure 9

XBL 8012-13434

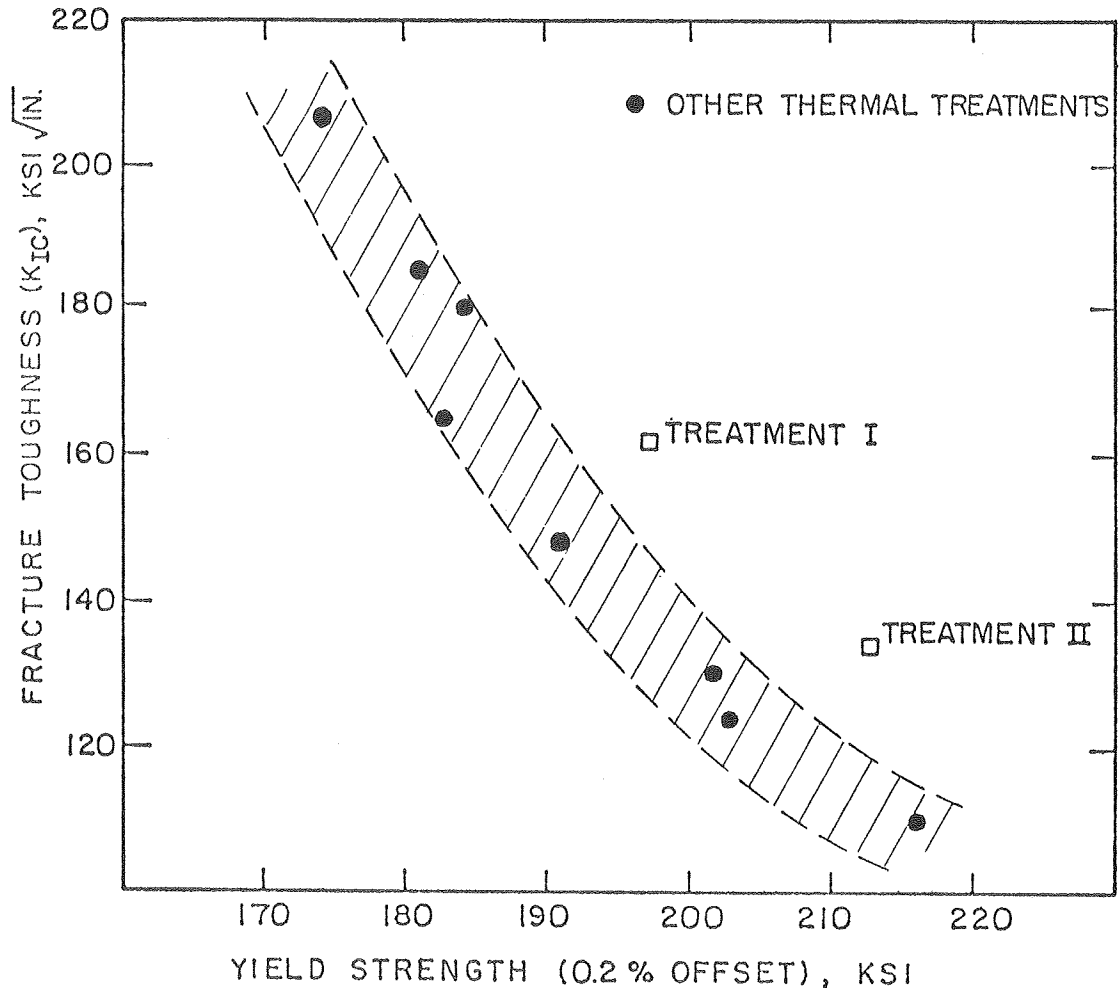


Figure 10

XBL 8012-13540

# THERMAL CYCLING - T ( 2BT )

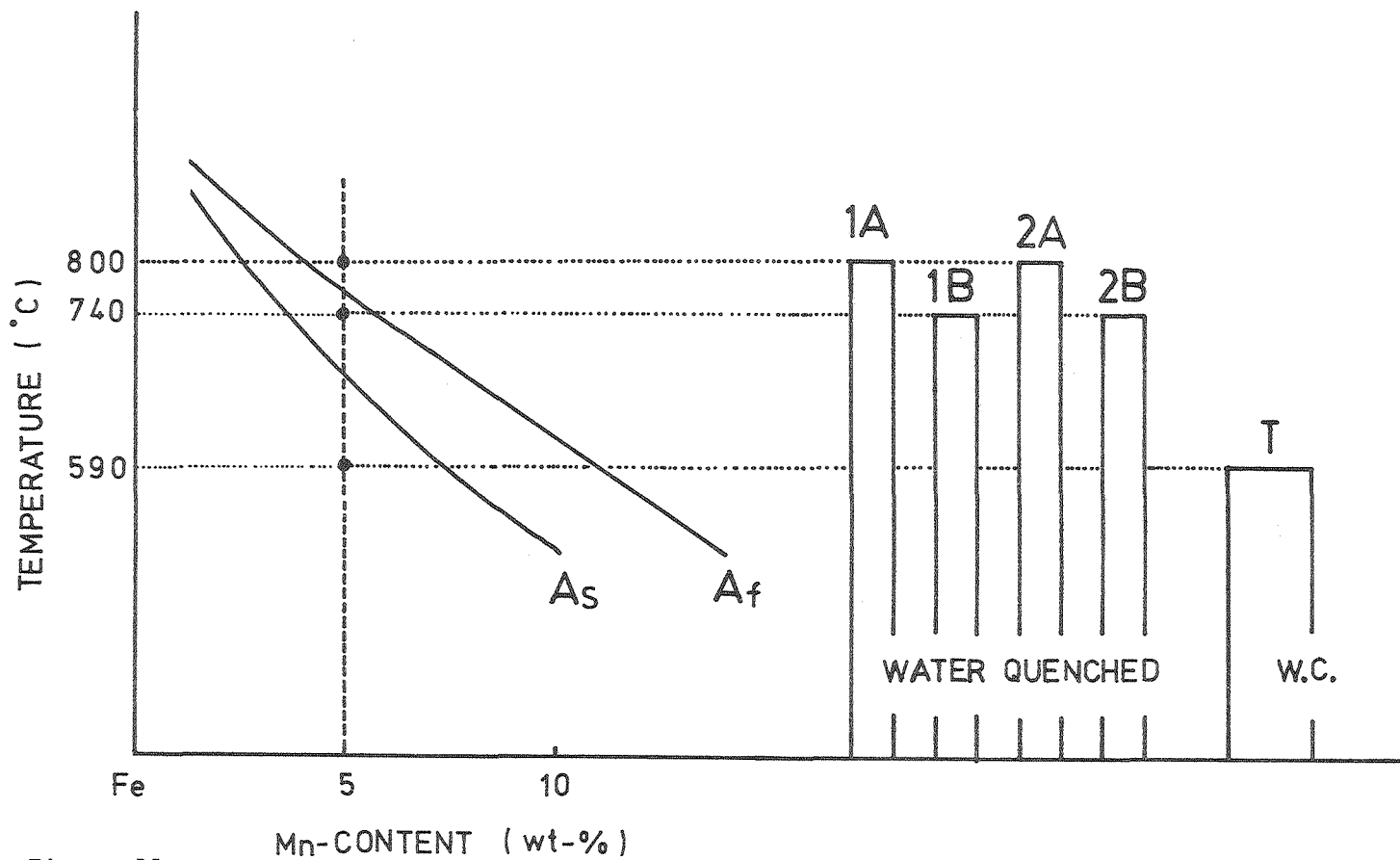


Figure 11

XBL 792-8393

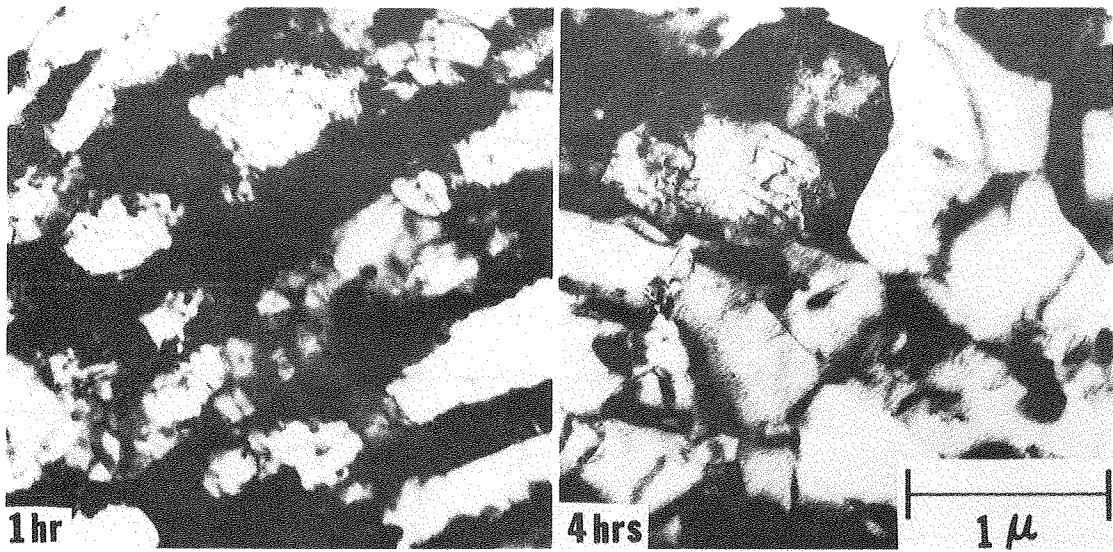


Figure 12

XBB799-11379

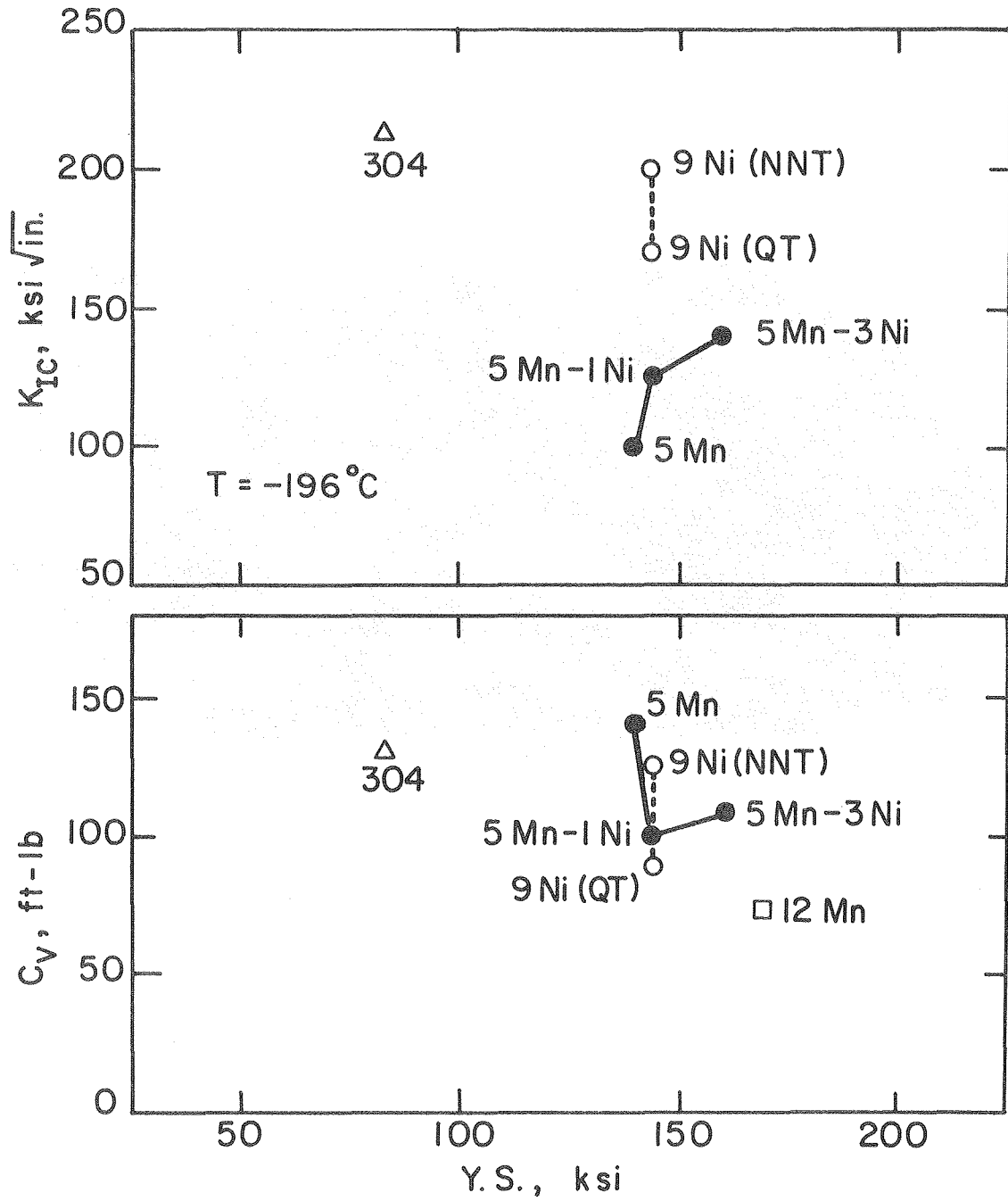


Figure 13

XBL 798-6766

# THERMAL PROCESSING

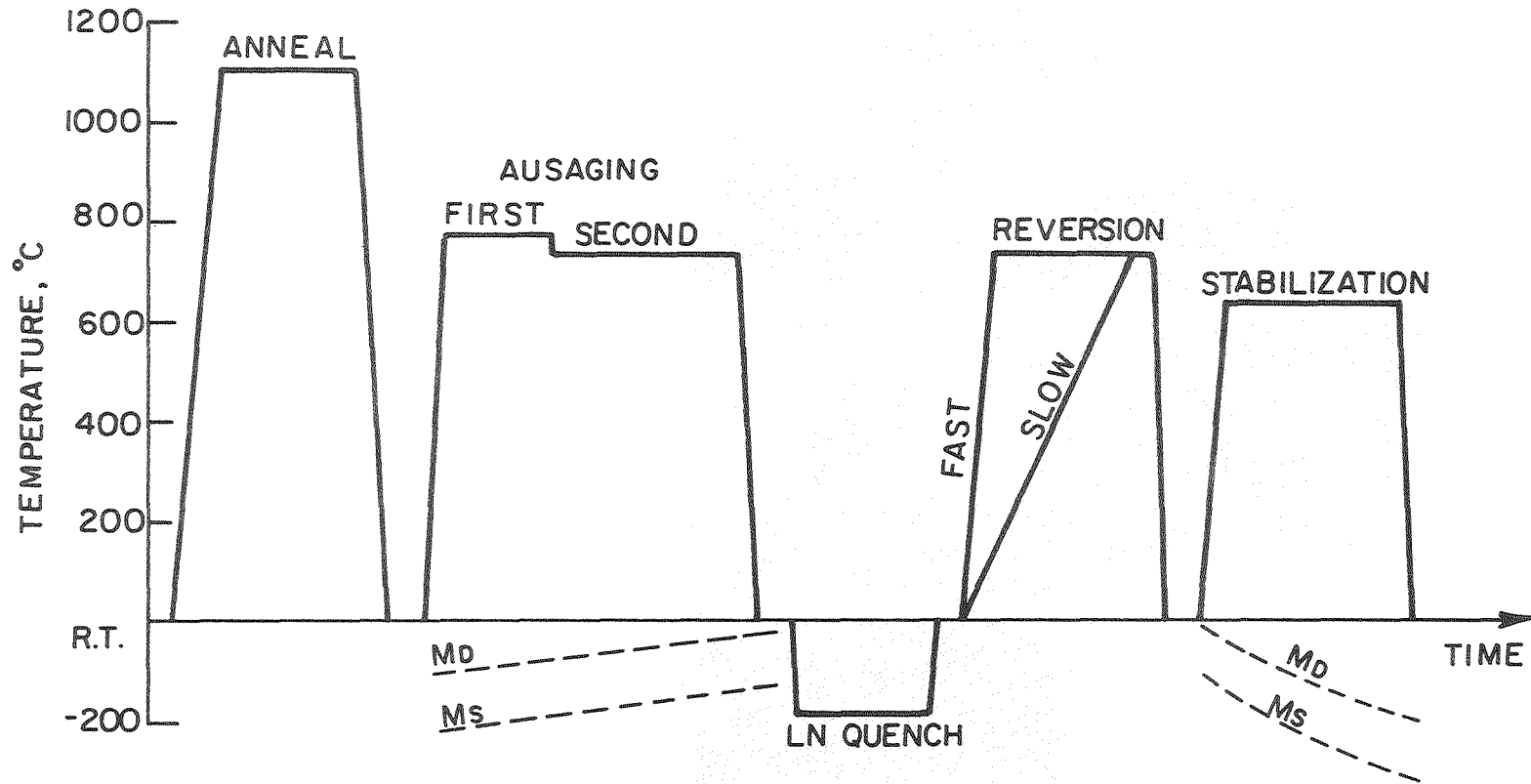


Figure 14

XBL 7710-6283



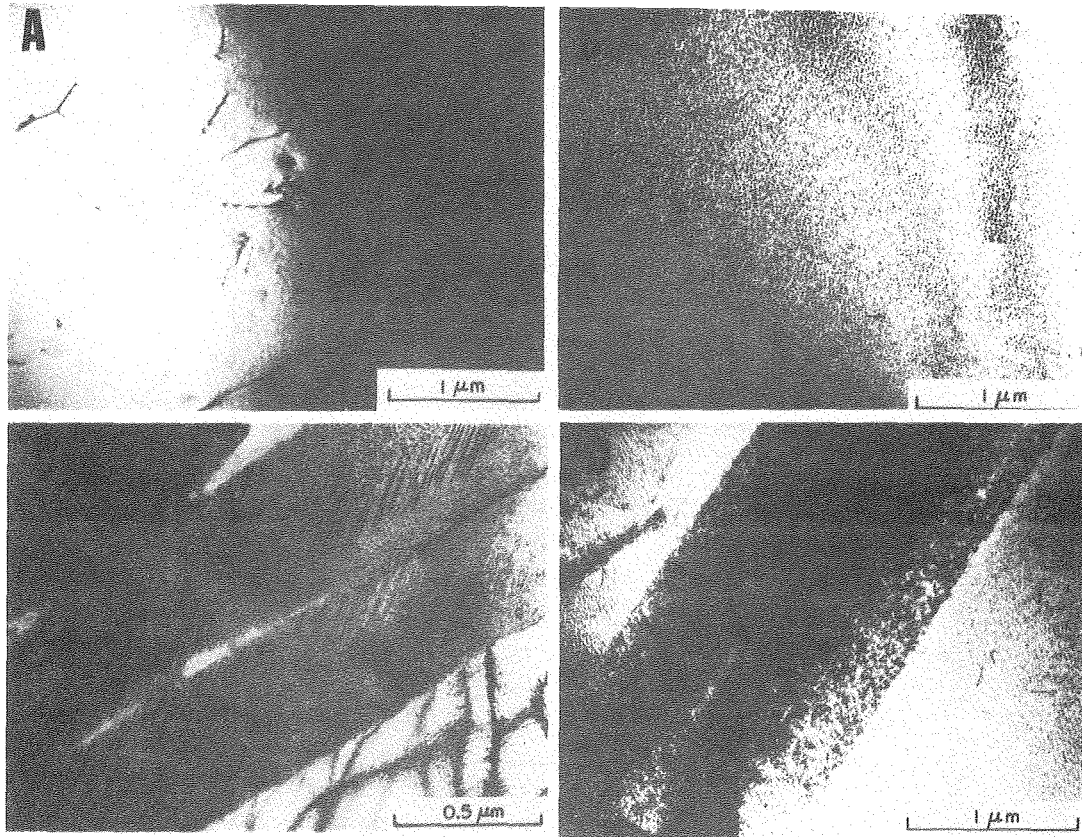


Figure 15

XBB800-14846

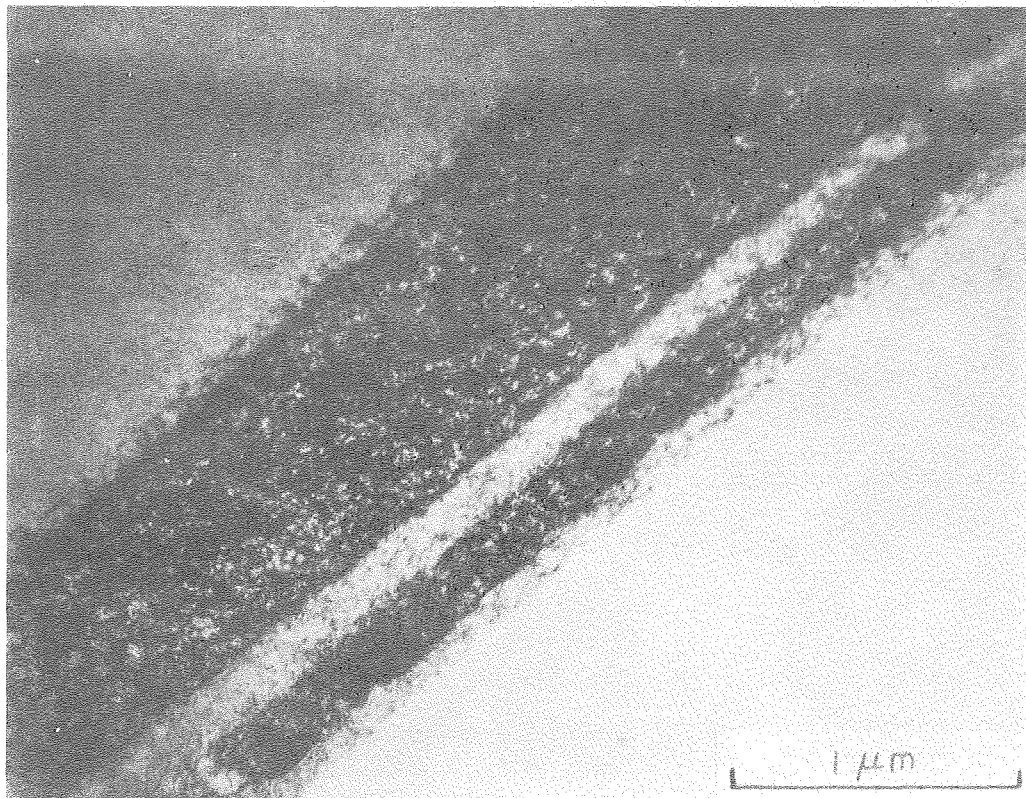


Figure 16

XBB766-5830

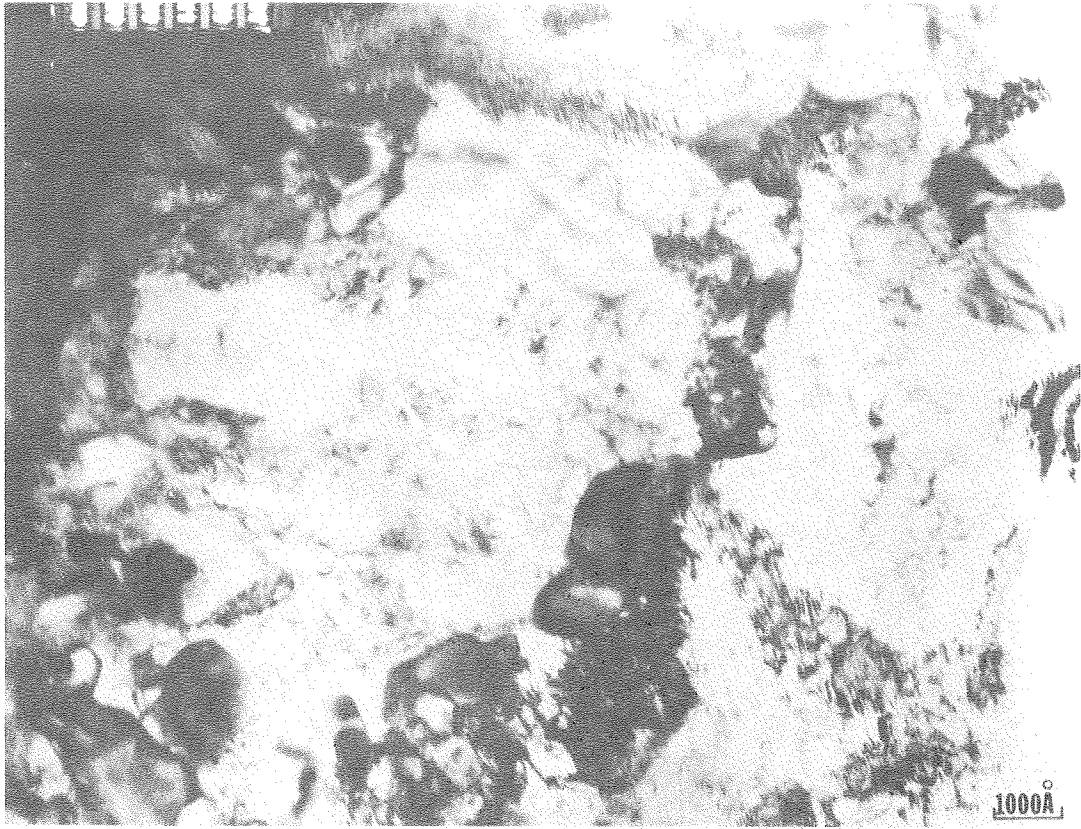


Figure 17

XBB802-2119

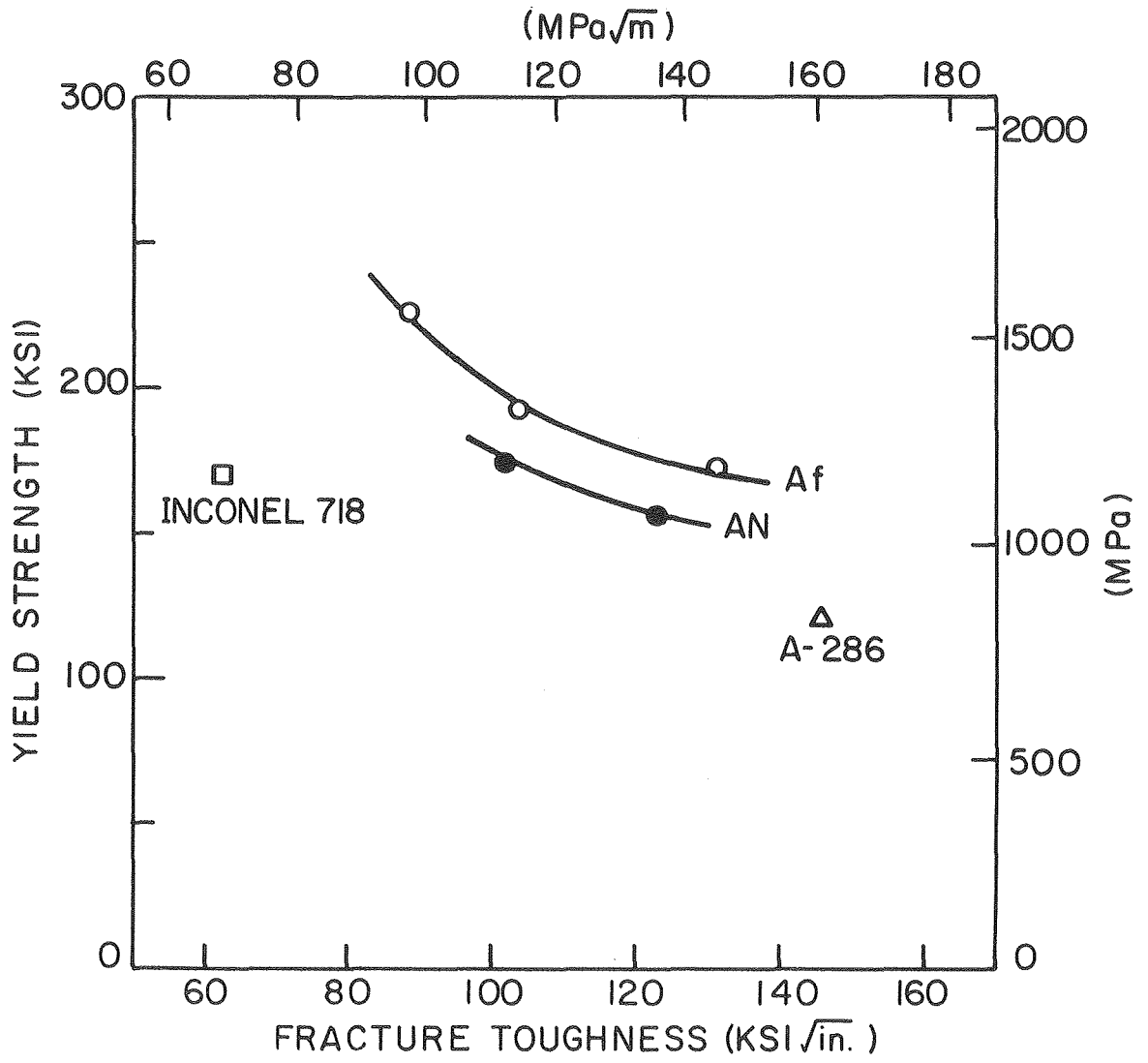


Figure 18

XBL796-6406

