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THE APPLICATION OF MATERIALS SCIENCE TO THE DESIGN OF ENGINEERING ALLOYS

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ABSTRACT

The functional roles of materials science and materials engineering in modern alloy design are discussed. It is suggested that following the end of World War II evolutionary trends in both methodology and language have tended to divide, rather than unite, these two branches of knowledge. Over a period of several decades the net effect has been, on the one hand, to isolate materials scientists from materials engineers, and thereby prevent them from making greater contributions to technology and, on the other, to deny to materials engineers the potential benefits of the vast understanding achieved by the materials scientists. Several new programs in the materials education field are described which have as their aim a partial redress of this undesirable situation. The current state of development of alloy design, as seen from an academic viewpoint, is described with the aid of four illustrative examples taken from current student theses research. These examples are:

(1) a bcc iron alloy having unusual toughness at temperatures of liquid nitrogen and below; (2) a series of medium alloy steels whose strength and toughness are equivalent to those of the high alloy maraging steels; (3) an ultra high strength Fe-Cr-C steel of great toughness which was

developed from fundamental considerations of microstructure and substructure; and (4) bcc iron alloys which utilize intermetallic compounds, rather than alloy carbides, for elevated temperature dispersion hardening.

1. INTRODUCTION

Materials science and materials engineering are generally considered to be separate, though related, fields of endeavor. To the designer of alloys, a knowledge of both fields is essential. The design of alloys involves coupling basic knowledge with technological needs. One objective of this paper is to review some of the attempts that have been made to strengthen the coupling between the two fields and enhance alloy design. A second objective is to describe the interdisciplinary educational programs which have been instituted at the University of California at Berkeley, at both the undergraduate and graduate levels, to prepare students for careers that require competence in both materials science and materials engineering.

Materials science is a young relative of the physical sciences, but it has evolved rapidly to a highly sophisticated state. Viewed from the limited perspective of the alloy designer, there is no question that, in spite of its relative youth, the impact of materials science upon physical metallurgy has been truly profound. In essence it may be said that from materials science have come the basic laws that govern the design of alloys to meet specific property requirements. The modern sophisticated analytical and experimental tools of materials science especially those using electron optical, diffraction and spectroscopic methods have enabled the metallurgist to identify the details of defect- and microstructures, to ascertain their roles in plastic behavior and fracture of solids and, lastly, to produce those elements of internal structure that endow alloys with superior properties. Desirable configurations of

structure can be produced in new alloys by controlling chemical composition, mechanical processing, and the phase transformation characteristics.

In principle, the metallurgist should be able to create alloys having mechanical properties that satisfy the design requirements of any existing engineering structure by controlling the chemical composition and the thermal, mechanical, thermomechanical and complex processing and fabrication treatments of the alloys. Although this is theoretically possible, it is not often accomplished in practice. The difficulty is, of course, that not enough is yet known about the complex relationships between internal structure and the engineering properties. Conversely, engineers cannot define the properties of structural alloys in terms of microstructural requirements. Consequently, designers must rely upon experience and use "factors of safety" to guard against certain kinds of service failures, particularly fracture.

Materials engineering is much older than materials science. It has evolved slowly over the centuries. Materials engineering is part art and part science--the proportion of each continually but subtly changing with time. As an example, Cahn, in a recent review paper, has described the roles of materials science, materials engineering and empiricism in the development of modern superalloys for elevated temperature service.[1]

In the design of alloys for room and cryogenic service there is a highly significant development worthy of special mention. This is the development of a branch of continuum mechanics dealing with fracture. Utilization of the recent advances in this field greatly enhances the coupling between material science and materials engineering.

The resistance to sudden and catastrophic failure is one of the most important design requirements of engineering structures. Until the advent of modern fracture mechanics, there was no known quantitative way to associate the microstructure of an alloy with its tendency to fail catastrophically. Earlier tests, such as the Charpy, do not provide quantitative design data. The pioneering work of Irwin, et al. in the late fifties has resulted in a totally new approach to the problems of fracture of complex engineering structures. As is now well known, the intrinsic fracture toughness of certain alloys can be quantitatively evaluated in terms of the stress intensity factor, K, with K_C being the stress intensity required for the catastrophic propagation of a crack in a sheet of material subjected to plane stress, and K_{IC} being the parameter for the plane strain state. Of particular interest to the materials scientist is the observation that both K_C and K_{IC} are highly dependent on internal structure. The development of a quantitative approach to fracture has provided a vital link between materials science and materials engineering. The materials scientist can hence use his knowledge of defect structure and microstructure to produce alloys with enhanced resistance to fracture. The mathematical formalism of modern fracture mechanics has also been applied to the problem of fracture by fatigue, but the quantitative correlation between fatigue failure and microstructure is not as well understood, as is catastrophic fracture.

Modern fracture mechanics also provides an important link between process metallurgists and their professional counterparts, the materials engineers and the materials scientists. Numerous investigations have shown that processing variables, especially those influencing the kind,

amount, and distribution of impurities, can have a profound effect on fracture toughness and fatigue strength. Thus, opportunities now exist for an integrated approach to alloy design encompassing alloy chemistry, mechanical processing, materials science and materials engineering.

Some examples are cited in the paper of alloy design for cryogenic, room, and elevated temperature service. The examples chosen represent attempts to either improve the combination of the ambient temperature strength and toughness of commercially available alloys, or to create new alloys with properties similar to or better than those existing for cryogenic and elevated temperature use. The emphasis is upon the integrated materials science-materials engineering approach. The examples used as illustrations are intended to suggest some of the present capabilities of the materials scientist and the materials engineer that can be applied to the design of new alloys. The commercial exploitation of wholly new materials designed to meet in some way the needs of present and future technology, must await the establishment of cooperative programs involving the talents of industry, government and universities.

There is another area where educational institutions can make a significant contribution, namely, that of training students in the combined fields of materials science and engineering design. In general, design engineers have a very limited knowledge about the relationship between microstructure and mechanical properties. The language of the metallurgist and materials scientist is largely foreign to design engineers. Conversely, the language and analytical methods of the designer are equally mysterious to most metallurgists. An intimate

knowledge of both fields is required for the design of structures that will never be in danger of failing catastrophically in service. The need for engineering students trained in both fields is evident to employers as well as faculty members. Consequently, there is a movement in curricula planning in engineering colleges toward broadening the training base to permit students to acquire competence in two engineering fields. These are called "double major" programs. An example of one such program will be discussed in the next section.

2. EXAMPLES OF MATERIALS SCIENCE PRINCIPLES APPLIED TO ALLOY DESIGN

The concept of materials scientists designing new alloys is an optimistic one. Even though research workers in the materials field are cognizant of the basic principles of alloy design and are capable of developing new alloys with interesting properties, there are certain critical questions that they are rarely in a position to answer. One such question is, "Who needs the new alloys?". Another is, "Are the new materials economic and timely?". Market analyses and current awareness of advanced technological needs are not the forte of professors of materials science. Yet the need for new and better materials is obvious even to laymen. What then, can people in universities contribute to the field of useful materials? The position is by no means hopeless. Educational institutions have the responsibility for training the future generation of materials scientists and engineers, and they can do a more effective job than has been common in the past. Furthermore, the faculty themselves can become more aware of the needs of technology by regular interaction with their colleagues in industry.

During the past two decades, materials science has grown farther and farther away from the technologically related fields of materials

engineering and engineering design. In many ways the concentration of efforts on science oriented subjects has been highly useful. It has resulted in a far better understanding by metallurgists of the basic principles of materials science that must be thoroughly mastered before new and better alloys can be designed. However, it is now time for materials scientists to apply these principles to the solution of important technological problems. Students trained narrowly in a specialized field, such as materials science, will not be able to do this effectively after they graduate. A broader educational base is now required, consisting of core courses in materials science, materials engineering, and engineering design. New programs of study must be made available to students. They must contain the core courses required of majors in at least two of the conventional departments, for example, Mechanical Engineering/Materials Science, or Civil Engineering/Materials Science, or Electrical Engineering/Materials Science. Double Major programs in these combined fields have been made available to students in the College of Engineering, University of California, Berkeley since September 1971. The number of students selecting these programs has doubled each year, which shows the growing attention the programs are receiving. Equally favorable responses have come from employers who are learning about the programs and appreciate their merits.

At the graduate level, the same philosophy has prevailed, but in a much less formalized manner. Many of the students prefer the traditional science orientation, and their programs of study are so oriented. However, a smaller, but increasing number, are combining their basic training in materials science with engineering design and business administration

minors. Those who elect this educational route beyond the Masters degree, often prefer to receive the Doctor of Engineering degree, instead of the Ph. D., at the end of their program. The theses topics of such students are generally of the "alloy design" type. Four examples of the products of such research are cited below to illustrate what can be accomplished in educational institutions as a result of teaching students how to design new alloys or enhance the properties of existing ones. Students who have worked in this area carry the new concepts and methods to industrial laboratories where they will learn how to combine their science based knowledge and experimental skills with practical considerations, such as technological needs, economic considerations, and feasibility of production. Hopefully, the long range results will be the commercial production of new and better alloys.

2.1. Enhancement of Cryogenic Notch Bar Properties of an Iron Based BCC Alloy

Metals or alloys with the bcc structure can fail either by shear, as in fcc metals, or by cleavage. It is now well established that the propensity of bcc metals to fail by cleavage is influenced by a host of compositional and structural factors. Some of the compositional and microstructural features that are desirable in a bcc cryogenic alloy are: (1) the lowest possible level of interstitials in solid solution (interstials raise the critical resolved shear stress of bcc metals to the cleavage stress as the temperature is lowered), (2) the maximum amounts of either nickel or manganese in solid solution commensurate with other metallurgical considerations, and (3) the finest possible grain size. This listing of desirable features is, of course, not complete and is intended only as a guide for the selection of an alloy system for study. Iron-rich alloys in the Fe-Ni-Ti system meet the requirements cited above. The titanium and nickel contents

in these alloys can be varied to meet the requirements (1) and (2) above. Fine grain sizes can be obtained by thermal or thermal-mechanical means. In the study discussed below, the requirements (1) and (2) were met by the alloys Fe-12 pct Ni-0.5 pct Ti and Fe-12 pct Ni-0.25 pct Ti.* After initial processing and forming into desired shape, the alloys were solution annealed for 2 hr , air cooled to room temperature, then reheated to a designated temperature between 650°C and 800°C, and ice brine quenched.

The results of grain size and impact studies are shown graphically in Fig. 1. There was not a simple relationship between the phase diagram and the heat treatments required for grain refinement. Current studies suggest that the refinement is produced by an increase in the nucleation rate of the prior austenite grains above the two phase ($\alpha+\gamma$) region. Apparently the residual defect structure remaining from the duplex ($\alpha+\gamma$) microstructure provides the additional nuclei. The temperatures for effective grain refinement were between about 675°C and 775°C, as can be seen in Fig. 1(a).

The -196°C Charpy V-notch impact energies of both alloys reheated for 2 hr at temperatures indicated are shown in Fig. 1(b). The impact energies were uniformly high for reheating temperatures between 675°C and 775°C, and low for temperatures on either side of this range. It was noted that fine grain size and high impact energies were both obtained for the same reheat temperatures. Correlations such as these were not possible for room temperature tests because of the limited capacity of the testing machine (225 ft lbs).

*All chemical compositions are in wt pct except where otherwise indicated.

Macrographs of the broken Charpy V-notch bars, optical photomicrographs of the microstructure showing the grain size, and scanning electron micrographs of the fracture surface of the Fe-12Ni-0.5Ti alloy are shown in Fig. 2 for several reheat temperatures. It is readily apparent that there was a correlation between the grain size and the macro- and micro-fracture appearance. Specimens having a fine grain size showed gross amounts of plasticity on the fracture surfaces of the broken Charpy bars. Their fracture surfaces at high magnification exhibited dimpled rupture that is characteristic of shear (high energy) failure. Conversely, specimens having a large grain size showed little plasticity on the fracture surface of the Charpy bars and their fracture surfaces at high magnification were characterized by flat facets indicative of cleavage (low energy) failure.

The effect of the time of reheat on toughness is shown in Fig. 3 for temperatures between 650°C and 800°C. The energy absorbed at -196°C was clearly a function of both temperature and time. The grain size in microns is shown in parentheses on the curves in Fig. 3. At the reheat temperature of 650°C, the energy absorbed did not appreciably vary from its initial low value for times up to 30 hr. At the highest reheat temperature of 800°C the energy absorbed reached a low peak in about 1/2 hr and then decreased with time to a relatively low value. At intermediate temperatures, viz , 700°C and 750°C, the impact energy rose rapidly as a function of time to high values and did not change for times up to 6 hr. As shown in Fig. 3, there was a corresponding reduction in grain size with time for specimens treated in the intermediate

temperature region. This time-temperature-grain size relationship was especially evident for those specimens reheated at 700°C.

The -196°C impact toughness and yield strength of the Fe-12Ni-0.5Ti alloy are compared in Fig. 4 with those reported in the literature for two commonly used commercial cryogenic steels.[2] The superior combination of strength and toughness of the new alloy is clearly evident. The Charpy V-notch impact properties at liquid helium temperatures were in excess of 100 ft-lbs.

The Charpy V-notch impact test has the well known advantages of being both inexpensive and convenient in exploratory alloy design studies such as this one. However, the information this test provides has neither fundamental significance nor is it of value in quantitative engineering design. Accordingly, attempts are being made to correlate structure with more meaningful quantities such as the plane strain fracture toughness and the fatigue crack growth rate. The relatively low yield strength (between 140 and 150 ksi) and the high toughness at liquid nitrogen temperatures precluded the determination of valid plain strain fracture toughness in 5/8 inch thick specimens because excessive plastic flow occurred.

Some typical tensile results are shown in Table 1 for test temperatures of 23°C and -196°C. The tensile properties were quite insensitive to the reheat temperature. It is apparent that those microstructural features which influence the impact toughness did not appreciably affect the tensile properties. In tests at near liquid helium temperatures (-266°C) higher yield and tensile strengths were observed with no

appreciable decrease in ductility. The tensile properties of the Fe-12Ni-0.25Ti alloy were similar to those of the Fe-12Ni-0.5Ti alloy, although the strength was somewhat lower (about 5 pct) at all test temperatures. (For additional details see references 3-8).

2.2. Ultra High Strength Steels--Role of Microstructure

As mentioned earlier, the evolution of a unified theory of fracture mechanics and the development of reliable test methods for obtaining an accurate value for the plane strain fracture toughness (K_{IC}) have provided the metallurgist with a quantitative method for evaluating the effects of microstructural details on the tendency of an alloy to fracture in a brittle manner. Studies of the relationship between the microstructure and the fracture toughness of ultra high strength steels have been especially rewarding. For example, the deleterious effects of sulfur and other trace impurities have been quantitatively determined.[9,10] Also, the relative merits of steels with bainitic, martensitic, or tempered martensitic microstructures have been well documented with respect to their strength and fracture toughness.[11-13] Steels are often rated according to their relative positions on master plots of plane strain fracture toughness vs yield strength.[14]

The assumption is generally made that the high hardenability of commercial quenched and tempered steels leads to uniform microstructures throughout the thickness of fracture toughness specimens, because the hardness is nearly constant throughout the thickness. Elementary considerations of micromechanics of fracture lead to the conclusion that the fracture toughness of ultra high strength steels should be

highly dependent on relatively small amounts of embrittling microconstituents, whereas such microconstituents have little effect on the hardness. The detection of minor amounts of austenite decomposition products by either optical or electron microscopic techniques is deceptive in its apparent simplicity. The detection by optical microscopy, for example, of small amounts (of the order of 1 pct) of upper bainite in a 5/8 inch thick specimen consisting largely of lower bainite or autotempered martensite demands the utmost care and patience in all stages of specimen preparation. It is now established that high resolution electron microscopy is needed to distinguish between lower bainite and autotempered martensite. Unfortunately, the advantages of electron microscopy are offset by the disadvantage that large areas cannot be scanned readily. A combination of metallographic techniques is generally the most effective way to study the structures of heat treated steels. When metallographic techniques reveal the presence of embrittling structures in conventionally heat treated steels, alternate heat treatments should be devised to either eliminate, or at least minimize, such structures.

Recent studies on a secondary hardening 5 Mo-0.3 C steel have suggested one way in which improvements in microstructural uniformity can be achieved.[15-17] Increases in fracture toughness of more than 50 pct were obtained in as quenched specimens by the use of high austenitizing temperatures. In this case, the improvement was attributed to the reduction of undissolved alloy carbides. In the present investigation several commercial steels were heat treated at

different austenitizing and tempering temperatures and their strength and fracture toughness determined. The results obtained are reported herein.

The compositions and properties of the steels used in the present investigation are shown in Table 2. Specimens for optical and electron microscopy were taken from the fracture toughness specimens which had been designed and tested in accordance with ASTM recommended practices.[18] Sufficient sampling was done to ensure that representative microstructures were obtained.

The effect of austenitizing temperature on the fracture toughness of as quenched specimens of the 5 Mo-0.3 C steel is shown in Fig. 5. The abrupt increase in fracture toughness at a critical austenitizing temperature was associated with an increase in grain size (ASTM 7-8 to 1); the grain size change was concomitant with complete solution of the alloy carbides.

Several different microstructural features were responsible for the lower fracture toughnesses of the three steels when they were austenitized at the conventionally used temperature (870°C). The optical micrographs of AISI 4130 steel, oil quenched from 870°C, clearly showed grains of ferrite and many regions of upper bainite.[19] The remainder of the structure was identified by transmission electron microscopy as being lower bainite and autotempered martensite. When the austenite grain size was increased by first heating the steel to 1200°C (then furnace cooled to 870°C before oil quenching), there were no ferrite grains visible in the optical micrograph, and the amount of

upper bainite was substantially lower than that formed during the direct quench from 870°C. The fracture toughness was increased about 30 pct by the 1200°C treatment.[17]

Free ferrite grains and ferrite plates in upper bainite are regions that are mechanically weak. They can fail readily by either plastic flow or by cleavage, and thus they tend to initiate microcracks at relatively low levels of plastic strain. This results in low values of fracture toughness, even when dimpled rupture occurs, as scanning electron microscopy revealed was the case for this steel. In additional experiments, fracture toughness specimens were quenched into iced brine directly from 1200°C. The cooling rate in this case was fast enough to suppress the formation of upper bainite, although small amounts of a bainitic product were visible in isolated regions in optical micrographs. The remainder of the microstructure appeared to be autotempered martensite. Specimens given the iced brine quench had exceptionally high fracture toughness (100 ksi-in.$^{1/2}$).

Another microstructural feature that can have a marked influence on fracture toughness is retained austenite. The presence of austenite films has been observed by other investigators, and there has been some speculation about its influence on toughness.[20-22] However in martensitic steels it is generally assumed that no retained austenite exists if no evidence for fcc reflections can be obtained by x-ray diffraction. Since standard x-ray methods have a resolution limit of approximately 1 pct, and since the morphological distribution of austenite needs to be characterized it is necessary to utilize transmission electron microscopy and diffraction. Proof of the existence of very thin films (approximately 200 Å thick)

of austenite at martensite lath boundaries was first obtained in the studies of Fe-Cr-C steels discussed in Section 2.3 (see ref. 23). In the case of AISI 4340 steel, the presence of retained austenite in oil quenched specimens with the larger austenite grains appeared to markedly enhance the fracture toughness.[17] Optical micrographs did not reveal the structural nature of this steel in any of the conditions investigated. Transmission electron microscopy and diffraction showed that the structure was primarily autotempered martensite with small amounts of lower bainite and untempered martensite for both the 870°C, and the 1200° to 870°C austenitizing conditions. The only significant difference observable in the two steels was that austenite films, 100 to 200 Å thick, surrounded a majority of the martensite laths in the specimens that had been heated to 1200°C, as can be seen in the dark field electron micrograph of Fig. 6, whereas there was only a trace of retained austenite visible in the specimens heated to 870°C. Additional diffraction and dark field microscopy showed that the retained austenite did not transform when specimens were cooled to liquid nitrogen temperature.

Austenite is not sensitive to high local stress concentrations and does not fail by cleavage, as does ferrite. Consequently, it seemed reasonable to conclude that in this case, the presence of another phase, properly dispersed, can actually enhance the fracture toughness.

As is predictable from basic principles of materials science, low fracture toughness of a quenched and tempered steel is associated with the presence of certain types of microstructural features. Sulfide inclusions act as microcrack nuclei and therefore induce macrofracture at relatively low strains in fracture toughness specimens. Similarly,

carbide particles undissolved during austenitizing lower toughness. Free ferrite, whether present as separate grains or as platelets in upper bainite in ultra high strength steels, lowers fracture toughness by a substantial amount.

Autotempered martensite (with no interlath carbides) formed during the quenching operation is tough. Lower bainite, and tempered martensite free from lath boundary films of carbides, are also tough and fracture resistant microstructural constituents. The presence of retained austenite films around autotempered laths of martensite adds substantially to the inherent toughness of the autotempered martensitic structure. The substructure of martensite itself is also important, e.g. transformation twinning in carbon steels lowers toughness.[11]

Figure 7 summarizes the results of tests on steels that had been given the 1200°C austenitizing treatment. The fracture toughness, K_{IC} is plotted against the yield strength. In this figure there are two bands which show the ranges of yield strength and fracture toughness values reported in the literature for commercial AISI 4340 steel and the 18 Ni maraging alloy. The maraging steels are usually considered to have the highest value of plane strain fracture toughness obtainable at a given yield strength. The circles are test points for steels given special heat treatments described herein. The toughness values have been moved out of the lower and into the upper band. Furthermore, there are theoretical reasons to believe that fracture toughnesses well above the maraging steel band can be obtained with quenched and tempered steels through modifications of chemical composition and thermal treatments.

Figure 8 shows the approximate range of results obtained with TRIP

steels[24-27] and how the fracture toughness values compare with those of quenched and tempered, and maraging steels. The TRIP steels are metastable austenitic ultra high strength steels that transform martensitically when plastically deformed. These steels contain 0.3 pct carbon or more, and the strain-induced martensite formation provides an additional strengthening mechanism. A volume increase of approximately 3 pct (corresponding to a linear change of 1 pct) is associated with the austenite to martensite transformation. The transformation strains augment the ductility and add to the fracture toughness. The volumetric expansion tends to reduce the three dimensional tensile stresses that are developed during plastic straining near the apex of a notch. This changes the stress state toward a condition which favors a more ductile performance of a fracture toughness specimen.

At least in theory, steels with lower alloy content than TRIP steels, but with some TRIP characteristics, can be designed. The fracture toughness values for such steels should fall in the region between the maraging and the TRIP steel bands. (For additional details see references 15-17, 19, 28 and 29).

2.3. Development of Tough, Ultra-High Strength Fe-Cr-C Steels

The objective of this study was to select a low alloy hardenable steel having transformation characteristics that would permit control of microconstituents that are deleterious to fracture toughness. In this case, the purpose was to devise ways of avoiding the formation of twinned martensite, which is known to reduce fracture toughness.[30] Iron-chromium-carbon steels were selected for the investigation. Chromium is not an effective solid solution strengthener in ferritic alloy steels,[31] and so

substructural changes due to alloy additions can be readily correlated with changes in mechanical properties. Furthermore, chromium is relatively inexpensive, and it is effective in increasing hardenability. The compositions of the steels are given in Table 3. All steels were austenitized at 1100°C and quenched in agitated oil.

A mixed morphology of dislocated and twinned martensites was found in all the as-quenched steels. Increases in either carbon or chromium content raised the percentage of transformation twins (Figs. 9 and 10). The 0.17 pct C steels were only slightly twinned, but there was a noticeable increase in twinning as chromium level increased. Although all three 0.17 pct C steels exhibited autotempering, it was more prevalent at the lower chromium levels (with correspondingly higher M_s temperatures).

The 0.35 pct C steels showed the maximum difference in relative amounts of twinned martensite. The twin density increased by a factor of approximately two when the chromium content was raised from 4 to 12 pct. These results demonstrated conclusively that at a given carbon level, chromium enhances transformation twinning in martensite. (Some autotempering was observed in isolated areas of steel 0435).

The mechanical properties of as-quenched martensites are summarized in Fig. 10. The low carbon steels showed little change in strength or toughness with increased chromium. This corresponds with only a minor increase in the amount of twinning, and confirms that chromium is not a solid solution strengthener in steel. However, the high carbon steels had a major increase in strength and drop in toughness as the twin density increased with chromium additions. The brittleness of steel 1235 was also indicated by quench cracks along prior austenite grain boundaries.

Quench cracking was not encountered in steel 0435. The quench cracks did not compromise the K_{IC} results, since scanning electron microscopy showed that crack propagation was transgranular.

One of the most interesting results was the resolution of thin sheets of highly deformed retained austenite surrounding some of the martensite laths in all the as-quenched alloys (identified as austenite by selected area diffraction and dark field imaging of fcc spots, Fig. 11). On tempering at 200°C, the interlath austenite was observed less frequently. After tempering at 400°C, none was seen, indicating that tempering caused the retained austenite to transform to ferrite, followed by precipitation of interlath carbides. This replacement of the austenite by interlath carbides was accompanied by a drop in toughness. The variation in properties with tempering is shown in Fig. 12(a) and (b).

Several authors[30,32] have proposed that chemical composition determines martensitic substructure through its effects on M_s temperature and strength of martensite. Since most alloying elements act as solid solution strengtheners in austenite, they increase the thermodynamic driving force needed to initiate the martensite shear transformation, and hence lower the M_s temperature. The strength of the martensite over the temperature ranges of transformation then becomes the controlling factor, and slip or twinning or both will depend on the relative values of the critical resolved shear stress over the M_s-M_f temperature range.[30] In the present work, the steel 0417 had the lowest alloy content, the highest M_s temperature, and was mainly dislocated. Conversely, the steel 1235 with the highest alloy content had the lowest M_s temperature and the highest twin density.

The observations of retained (stabilized) austenite between martensite laths confirmed suggestions of Seal and Honeycombe,[20] that the absence of interlath carbides in Fe-Cr-C steels at tempering temperatures below 400°C is related to the presence of austenite films at lath boundaries. Such films of retained austenite are thus important in stabilizing the microstructure and properties up to tempering temperatures of 400°C. In the present study, austenite films disappeared after tempering at 400°C, and were replaced by interlath carbides.

This work showed the important role of substructure on the mechanical properties when composition is varied with one component held constant. The difference in as-quenched yield strength in the steels studied can be explained by the relative contributions to solid solution strengthening, especially that from carbon, and transformation substructure as discussed in detail elsewhere.[33-35]

The effect of chromium as a solid solution strengthener in martensite is shown to be negligible by the results obtained for the low carbon alloys (Fig. 10). Hence, for a given carbon level, any increase in strength associated with a higher chromium content must be caused by the increased twin density, not by the chromium addition directly. Kelly and Nutting[33] have drawn attention to the crystallographic restrictions on slip due to transformation twinning. Recent work has shown that twins can themselves be twinned which imposes further restrictions on the continuity of dislocation motion.[36] Carbon atom pinning of dislocations is also important. Twinning has also been observed to increase the hardness of fcc metals.[37] If slip is difficult, mechanical twinning is very deleterious, since twins are known to be effective as crack nuclei in bcc metals.[34]

Furthermore twinning dislocations in the bcc structure cannot cross slip and are not as effective as total dislocations in blunting cracks by plastic flow. In untempered martensites especially, twinning must be avoided if high toughness is to be achieved.[38] These conclusions are well demonstrated in Fig. 10 where for the 0.17 pct carbon steels. Although yield strength did increase as chromium content was increased from 4 to 12 pct, the toughness dropped as the twin density increased (8 pct Cr). The property changes were more dramatic for the 0.35 pct C steels where for a three-fold increase in chromium content, which caused a two-fold increase in twin density, the strength of the as-quenched martensites increased from 240 to 300 ksi, while the toughness dropped from 70 to 20 ksi-inch$^{1/2}$. It is also evident in Fig. 10 that a higher yield strength does not necessarily correspond to lower toughness in martensite. The as-quenched yield strength of steel 0435 was 84 ksi higher than that of steel 1217, yet both had the same fracture toughness. The increased carbon content of steel 0435 raised its yield strength over that of steel 1217, while the simultaneous reduction in chromium prevented a large increase in twinning and a corresponding drop in toughness. Hence, by adjusting carbon and alloying element contents in order to control the substructure, the strength of martensitic steels can be improved without suffering a loss of toughness. This suggests a simple three-step process for alloy steel design:

1) add that amount of carbon which will produce the desired strength;
2) add other alloying elements to produce the required hardenability and whatever other properties are desired; and 3) the total solute content must be restricted to avoid extensive twinning and thereby retain desired toughness (e.g. C < 0.4 pct).

An important result of this research was further experimental evidence that as-quenched martensites are not necessarily brittle (e.g., none of the 0.17 pct C martensites were brittle, as-quenched). While it has often been assumed that martensite must always be tempered after quenching to raise toughness to a usable level, so that the consequent reduction of strength on tempering is unavoidable, it is emphasized that steel 0435, with a plane strain fracture toughness of 70 ksi-in.$^{1/2}$ and yield strength of 240 ksi, qualifies as an ultra high strength, high toughness steel in the as-quenched condition. Its toughness is improved without a serious loss in strength when tempered at 200°C. Similar high strength and toughness in as-quenched, autotempered, Fe-5 Mo-0.3 C martensite have been reported.[15] Figure 13 compares the ultimate tensile strength and fracture toughness of steel 0435 with several commercial ultra high strength steels.[39] It shows that the steel 0435 matches or exceeds the properties of 18 Ni maraging steels. (For additional details, please see reference 23).

Some further examples of the utilization of phase transformations include current programs on dispersion strengthening of martensite. This program involves application of the principles of dispersion strengthening by producing precipitation in austenite prior to the phase transformation to martensite--controlling conditions so as to obtain dislocated martensite. The dislocations generated during the transformation multiply at the particles and simulate the structures otherwise obtainable by thermal-mechanical processing such as ausforming.[34,40] Initial results utilizing ausaging[41] and spinodal transformations[42] to produce dispersions in austenite are very encouraging.

2.4. A BCC Iron Based Alloy for Elevated Temperature Service

Most commercial creep resistant materials derive their strength from a fine dispersion of a second phase which hinders the movement of dislocations. In creep resistant ferritic steels, the fine dispersion almost invariably consists of carbides, whereas in high nickel content superalloys, the dispersed phase may be an intermetallic compound such as $Ni_3(Al,Ti)$. The particle size, morphology, distribution and volume fraction of the second phase, the degree of its coherency with the matrix, and the mechanical properties of the matrix all affect the creep resistance of dispersion strengthened alloys. The thermal stability of the second phase particles is also an important factor.[43]

The morphology and composition of carbide particles tend to change slowly at elevated temperatures and this often results in an increase in creep rate. An example of such an effect is the loss in creep strength of 0.1C-1.0Cr-0.5 Mo steel after long time exposure at service because of the formation of an M_6C type of carbide.[44]

The concept of using intermetallic compounds, rather than carbides, for increasing the high temperature strength of alloys is decades old, but progress toward a realization of this concept has been slow. The effects of intermetallic compounds such as the sigma phase and $Ni_3(Al,Ti)$ have been investigated by Mihalisin, et al[45] and Decker.[46] Mihalisin et al[45] found that the presence of sigma lowered both stress-rupture and room temperature strengths. Decker[46] also summarized the status of knowledge about the effects of the Laves phase particles on strength. He concluded that in general, when the Laves phase is present in significant amounts it "...can degrade room temperature ductility with little

effect on creep properties". The results from the present investigation showed, however, that with microstructural control, satisfactory values of room temperature strength and ductility, as well as good stress-rupture life, can be attained in very low carbon alloys strengthened with the Laves phase Fe_2Ta.

Recently it was demonstrated by Jones, et al[47] that in bcc Fe-Ta alloys, the brittleness, which was due to the presence of a continuous grain boundary network of the Laves phase Fe_2Ta, could be overcome by a simple heat treatment which spheroidized the precipitate. Spheroidization of the Laves phase resulted in considerable enhancement of room temperature ductility and elevated temperature strength of Fe-Ta alloys. Two major limitations of Fe-Ta alloys with respect to their possible practical utilization for elevated temperature service were: (1) the high $\alpha \rightarrow \gamma$ and $\gamma \rightarrow \delta$ phase transformation temperatures that necessitated high temperature heat treatments, and (2) inadequate oxidation resistance. An examination of the binary Fe-Cr phase diagram[48] indicated that increasing chromium additions to iron continuously lowered the $\gamma \rightarrow \delta$ phase transformation temperatures. The $\alpha \rightarrow \gamma$ transformation temperature, on the other hand, was lowered by chromium additions of up to 7 pct, but was raised by further additions of chromium. It seemed reasonable to expect that qualitatively, a similar trend would be observed on addition of chromium to Fe-Ta alloys. Also, it is well known that improvements in oxidation resistance are not obtained in ferritic alloys until about 7 pct chromium is present.[49,50]

The above consideration led to the development of a ternary Fe-1 at pct Ta-7 at pct Cr alloy (hereafter referred to as alloy Ta7Cr).

The Fe-rich portion of the equilibrium phase diagram[51] of the Fe-Ta system is shown in Fig. 14. There are three solid solution regions, α (bcc), γ (fcc) and δ (bcc). The intermetallic compound, Fe_2Ta, is a Laves phase, and it has an hexagonal $MgZn_2$ structure. The δ phase decomposes eutectoidally at 1239°C in accordance with the following reaction:

$$\delta \text{ (1.1 at pct Ta)} \rightarrow \gamma \text{ (0.5 at pct Ta)} + Fe_2Ta$$

The γ phase reacts peritectoidally with Fe_2Ta at 972°C to produce the α solid solution:

$$\gamma \text{ (0.3 at pct Ta)} + Fe_2Ta \rightarrow \alpha \text{ (0.6 at pct Ta)}.$$

Chromium additions change the compositions and temperatures of these transformations,[52] as shown in Table 4. The $\alpha \rightarrow \gamma$ and $\gamma \rightarrow \delta$ transformation temperatures listed in Table 4 were obtained by dilatometry, and the $(\gamma + \delta) \rightarrow \delta$ and $(Fe_2Ta + \delta) \rightarrow \delta$ temperatures were determined by metallography.

The structure of the alloy Ta7Cr in the solution treated (for 1 hr at 1320°C) and water quenched condition was characterized by large grains (approximately 1 mm in average diameter) of the retained δ phase, and by an almost continuous grain boundary network of precipitate. When the alloy was aged at 700°C, additional precipitate formed within the grains, but there was no noticeable change in the grain boundary network. Microhardness measurements on sampels aged for several time intervals at 700°C showed that the hardness first increased with increasing aging time, reached a maximum after 40 minutes of aging, and then decreased. The microstructure of the alloy in the peak hardness condition is shown in Fig. 15(a). When the aged alloy was heated to 1100°C, the matrix

transformed to the face centered cubic solid solution, and the grain boundary precipitate spheroidized. On cooling from the γ phase field, the matrix again transformed to α, with a consequent refinement in grain size. The structure after this treatment consisted of spheroidized precipitate particles in a matrix of α which was characterized by irregular grain boundaries, as shown in Fig. 15(b). In Fig. 16 is shown a transmission electron micrograph of a carbon extraction replica showing a uniform distribution of almost spherical particles of the Laves phase in the spheroidized alloy.[53]

The tensile properties of the Ta7Cr alloy, after aging at 700°C for 40 minutes were characterized by yield and ultimate tensile strengths which decreased very gradually with increase in test temperature up to about 600°C; at this temperature the 0.2 pct offset yield strength was about 70 pct of the room temperature value. The room temperature fracture was brittle as indicated by the scanning electron fractograph shown in Fig. 17(a). The room temperature fracture behavior was changed from brittle to ductile by the spheroidizing heat treatment that followed aging. The fracture surface of the tensile specimen tested at room temperature after spheroidizing at 1100°C for 10 minutes is shown in Fig. 17(b). In this figure, there is evidence of a significant amount of plastic deformation, as indicated by a dimpled appearance. Fracture occurred by void nucleation and growth at the interfaces between Fe_2Ta particles and the matrix. The yield strength,* tensile strength, and fracture elongation are plotted as a function of the test temperature in Fig. 18 for the alloy in spheroidized condition. The results were similar to those of the alloy in the aged condition except that all the mechanical

*0.2 pct offset.

properties were increased in the spheroidized alloy. The improvements were attributed to the combined effects of spheroidization of the grain boundary precipitate, reduction in grain size, and an increase in dislocation density.

Short-time tensile properties do not provide adequate information about high temperature strength, and so stress-rupture tests were made on the Ta7Cr alloy. The temperatures selected for evaluation were 1000°F (538°C) and 1100°F (593°C). Stress vs time to rupture data for 1100°F (593°C) are plotted in Fig. 19. Also shown in the figure are the stress-rupture properties reported in the literature for 0.3C-1 Cr-1Mo-0.25V steel, 0.15C-9Cr-1Mo steel, Greek Ascoloy and 403, 410 and 422 stainless steels.[54] At 1100°F, the Ta7Cr alloy results were above those reported for the 0.15C-9Cr-1Mo steel, and 403 and 410 stainless steels, but below those for the Greek Ascoloy, 422 stainless steel and the 0.3C-1Cr-1Mo-0.25V steel.

The strengthening effect of molybdenum in solid solution in ferrite is well known[55-57] so 0.5 at pct of molybdenum was added to the Ta7Cr alloy, and the alloy Ta7CrMo was developed. The expected strengthening was found when stress-rupture tests were performed at 1100°F (593°C) on the Ta7CrMo alloy. The results are shown in Fig. 19. Additional detailed information on this study is reported elsewhere.[53,58-60]

3. SUMMARY

The functional roles of materials science and materials engineering have been broadly discussed. It has been suggested that following the end of World War II evolutionary trends in both methodology and language have tended to divide, rather than unite, these two branches of knowledge. Over a period of several decades the net effect has been, on the one hand, to isolate materials scientists from materials engineers, and thereby prevent them from making greater contributions to technology and, on the other, to deny to materials engineers the potential benefits of the vast understanding achieved by the materials scientists. Several new programs in the materials education field are described which have as their aim a partial redress of this undesirable situation.

The current state of development of alloy design, as seen from an academic viewpoint, has been described with the aid of four illustrative examples taken from current student theses research. The examples used for illustration are by no means ready for immediate engineering applications. The graduate students involved in these programs have acquired a breadth of experimental skill as well as basic knowledge in both materials science and materials engineering. When these students leave the university to go into industrial and government laboratories they will be in strong positions to combine their research experience and fundamental knowledge with the practical considerations of commerce. Armed with this knowledge and experience they should be in a favorable position to attack the unsolved technological problems of modern society.

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TABLE 1. Tensile Properties of the Fe-12Ni-0.5Ti Alloy

	2 Hour Reheat at 750°C				2 Hour Reheat at 850°C			
Test Temp., °C	Yield Strength, ksi	Ultimate Tensile Strength, ksi	Elong., pct	Red. in Area, pct	Yield Strength* ksi	Ultimate Tensile Strength, ksi	Elong., pct	Red. in Area, pct
23	90.3	98.6	21	84	90.8	98.3	21	86
-196	139.5	153.0	18	75	140.0	150.5	21	74

*0.2 pct offset.

TABLE 2. Austenitizing and Tempering Temperatures, Fracture Toughness and Yield Strength of 5 Mo-0.3C, AISI 4130 and AISI 4340 Steels

Steel (Composition)	Austenitizing Temperature, °C	Quenching Medium	Tempering Temperature, °C	0.2% Yield Strength ksi	Fracture Toughness, ksi-in$^{1/2}$
5Mo-0.60Mn-0.30C	870	Iced Brine	As Quenched	194	52
(Laboratory heat)	1200	Iced Brine	As Quenched	214	100
	1200	Iced Brine	150	210	104*
	1200	Iced Brine	225	196	109*
AISI 4130	870	Oil	As Quenched	201	57
(0.30C-0.85Cr-	1200→870**	Oil	As Quenched	210	73
0.46Mn-0.20Mo)	1200	Iced Brine	As Quenched	214	100
(Commercial steel)	1200	Iced Brine	200	210	110
AISI 4340	870	Oil	As Quenched	231	35
(0.40C-0.72Cr.	1200→870**	Oil	As Quenched	231	63
1.73Ni-0.80Mn-	870	Oil	200	235	62
0.24Mo)	1200→870**	Oil	200	230	75
(Commercial steel)					

*K_Q, rather than K_{IC}, values.

**Specimens cooled slowly from 1200°C to 870°C before quenching to maintain quenching conditions identical with 870°C austenitized specimens.

TABLE 3. Chemical Compositions of Fe-Cr-C Steels

Alloy Number	Composition, wt pct				M_s Temp., °C
	C	Cr	Si	O	
0417	0.18	4.2	0.14	0.001	408
0817	0.16	8.4	0.15	0.002	358
1217	0.17	12.0	0.15	0.002	286
0435	0.35	4.0	0.06	0.003	320
1235	0.34	12.2	0.10	0.002	182

TABLE 4. Phase Transformation Temperatures for Fe-Ta-Cr Alloys

Alloy Composition	Phase Transformations and Temperatures (°C)		
	$\alpha \rightarrow \gamma$	$\gamma \rightarrow \delta$	$\gamma + \delta \rightarrow \delta$ or $Fe_2Ta + \delta \rightarrow \delta$
Fe-1 at pct Ta	974	1238	1340
Fe-1 at pct Ta-3 at pct Cr	890	1230	1300
Fe-1 at pct Ta-5 at pct Cr	880	1210	1280
Fe-1 at pct Ta-7 at pct Cr	870	1160	1250

FIGURE CAPTIONS

Fig. 1. The effect of reheat temperature on (a) the grain size of the Fe-12Ni-0.5Ti alloy, and (b) the Charpy V-notch impact energy at -196°C for the Fe-12Ni-0.5Ti and Fe-12Ni-0.25Ti alloys. The time at reheat temperature was 2 hr in each case.

Fig. 2. Macrographs of broken Charpy bars, optical micrographs of microstructure, and scanning electron fractographs of Charpy bars of the Fe-12-Ni-0.5Ti alloy reheated for 2 hr at the indicated temperatures, tested at -196°C.

Fig. 3. The effect of time of reheat at indicated temperatures on the Charpy V-notch impact energy at -196°C for the Fe-12Ni-0.5Ti alloy. The grain size in microns is shown in parentheses on the curves.

Fig. 4. A comparative plot of the yield strength and Charpy V-notch impact toughness, both measured at -196°C, for two commercial cryogenic steels[2] and the Fe-12Ni-0.5Ti alloy.

Fig. 5. Influence of austenitizing temperature on the room temperature plane strain fracture toughness, K_{IC}, of as-quenched 5Mo-0.3C steel.

Fig. 6. Transmission electron micrographs of AISI 4340 steel heated to 1200°C before oil quenching, (a) bright field image, (b) dark field image, showing reversal of contrast at austenite films between martensite laths.

Fig. 7. Plots of fracture toughness, K_{IC}, vs yield strength. The two shaded bands represent the range of values found in the literature for AISI 4340 and maraging steels. The circles are data points from the present investigation.

Fig. 8. Plots of fracture toughness vs yield strength. Bands for commercial steels (K_{IC}) and metastable austenitic TRIP steels (K_C).

Fig. 9. Transmission electron micrographs of as-quenched (a) dislocated (Steel 0435), and (b) twinned (Steel 1235) martensites.

Fig. 10. Room temperature mechanical properties and twin densities of as-quenched martensitic steels.

Fig. 11. Transmission electron micrographs showing retained austenite at martensite lath boundaries in steel 0817, (a) bright field image, and (b) dark field image.

Fig. 12. Room temperature mechanical properties of as-quenched and tempered (a) 0.17 pct C steels, and (b) 0.35 pct C steels. Drop in toughness at the 400°C tempering temperature is due to precipitation of interlaths carbide films.

Fig. 13. Tensile strength and fracture toughness of steel 0435 compared to some commercial alloy steels.[23,39]

Fig. 14. Iron-rich portion of the equilibrium phase diagram of the Fe-Ta system.

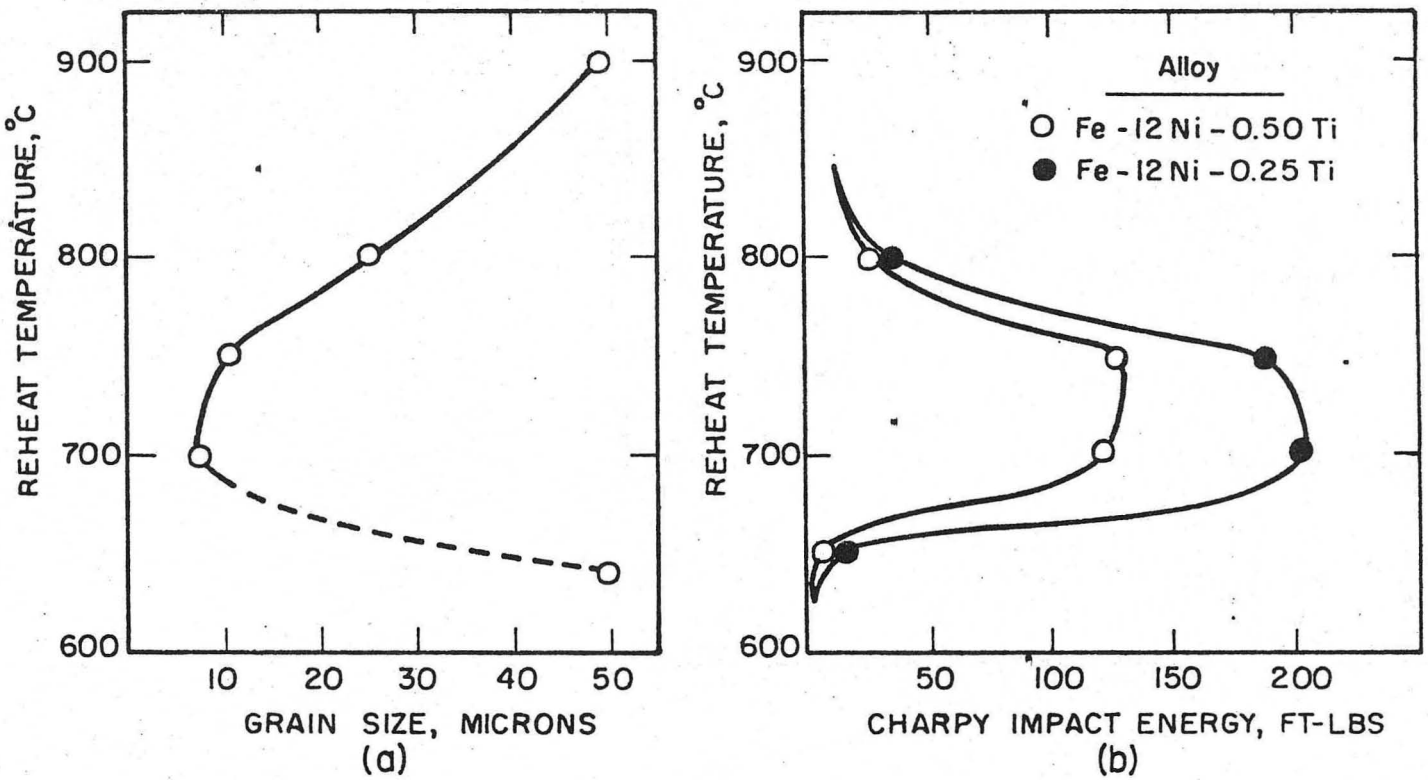
Fig. 15. Optical micrographs showing the microstructure of alloy Ta7Cr, (a) aged at 700°C for 40 min, and (b) aged at 700°C, and spheroidized at 1100°C for 10 min.

Fig. 16. Transmission electron micrograph of a carbon replica showing the Laves phase particles extracted from alloy Ta7Cr spheroidized at 1100°C for 10 min.

Fig. 17. Scanning electron fractographs of alloy Ta7Cr tested in tensile tests at 22°C following (a) aging at 700°C for 40 min, and (b) aging at 700°C, and spheroidizing at 1100°C for 10 min.

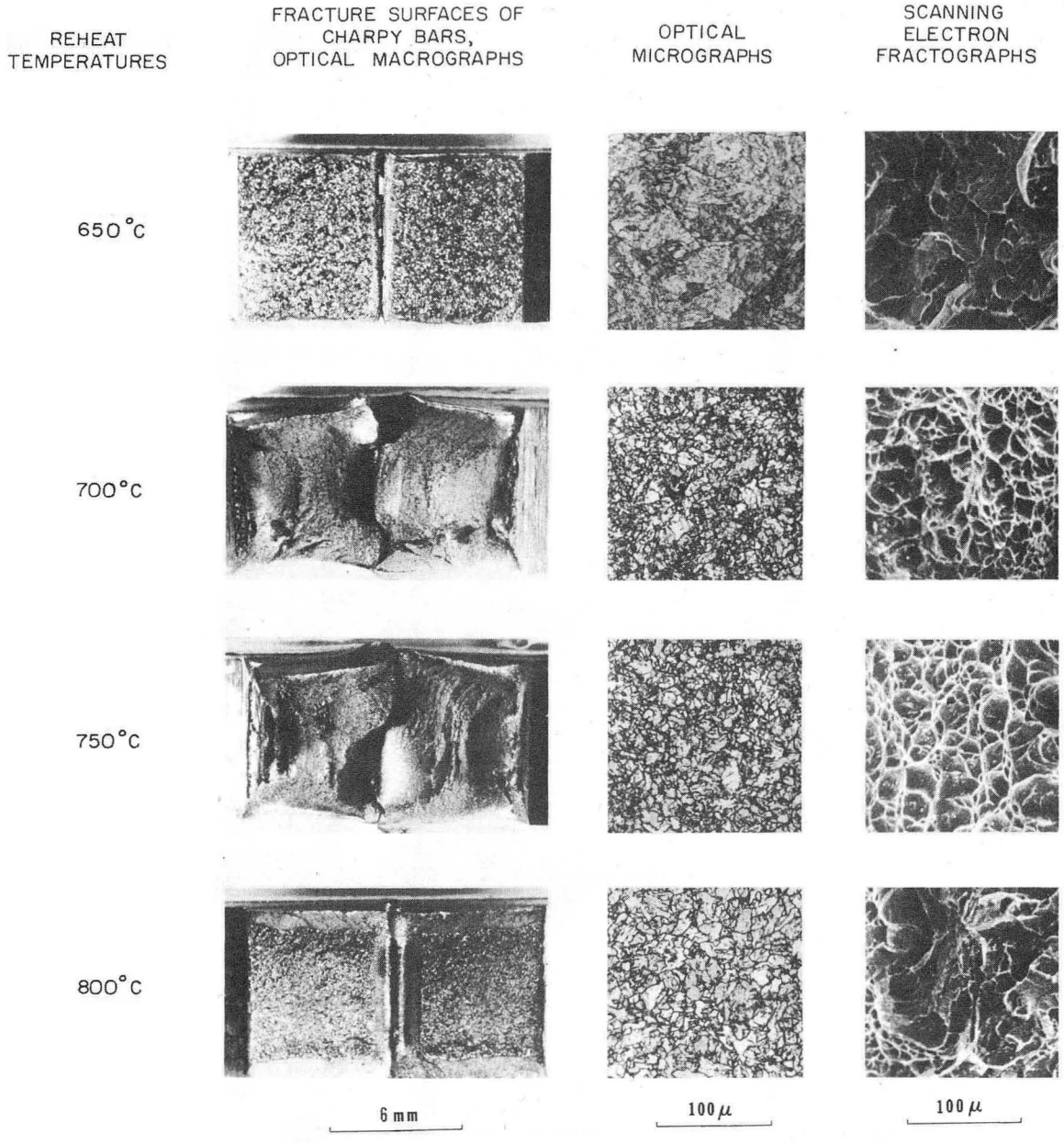
Fig. 18. Effect of test temperature on the short-time yield strength, ultimate tensile strength, and fracture elongation of spheroidized alloy Ta7Cr.

Fig. 19. Stress vs rupture time at 1100°F (593°C) for alloys Ta7Cr and Ta7CrMo. Also shown are the results reported in the literature[54] for 0.3C-1Cr-1Mo-0.25V steel, 0.15C-9Cr-1Mo steel, Greek Ascoloy, and the AISI types 403, 410 and 422 stainless steels.



XBL 733-5837

Fig. 1



XBB 733-1776

Fig. 2

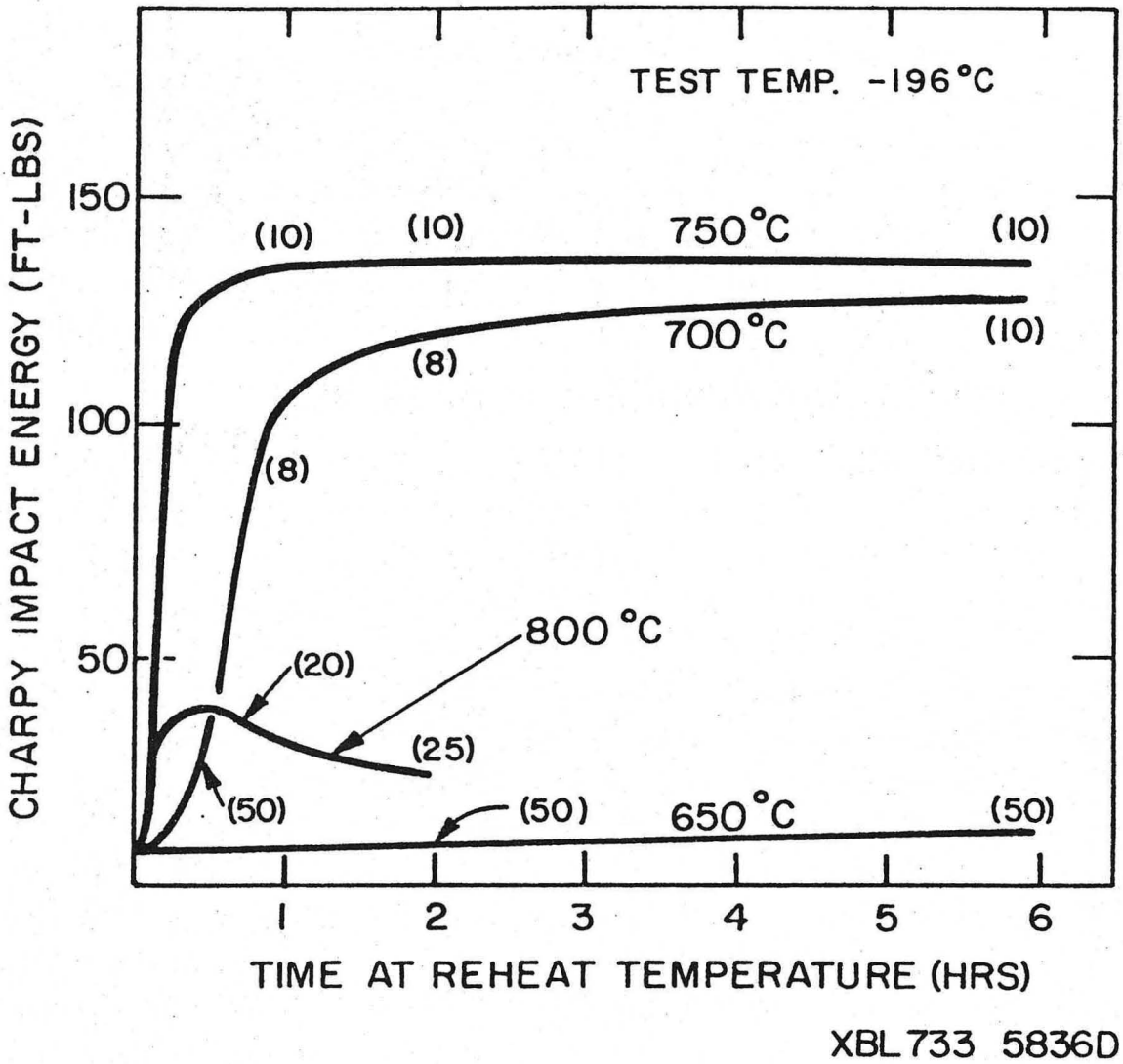
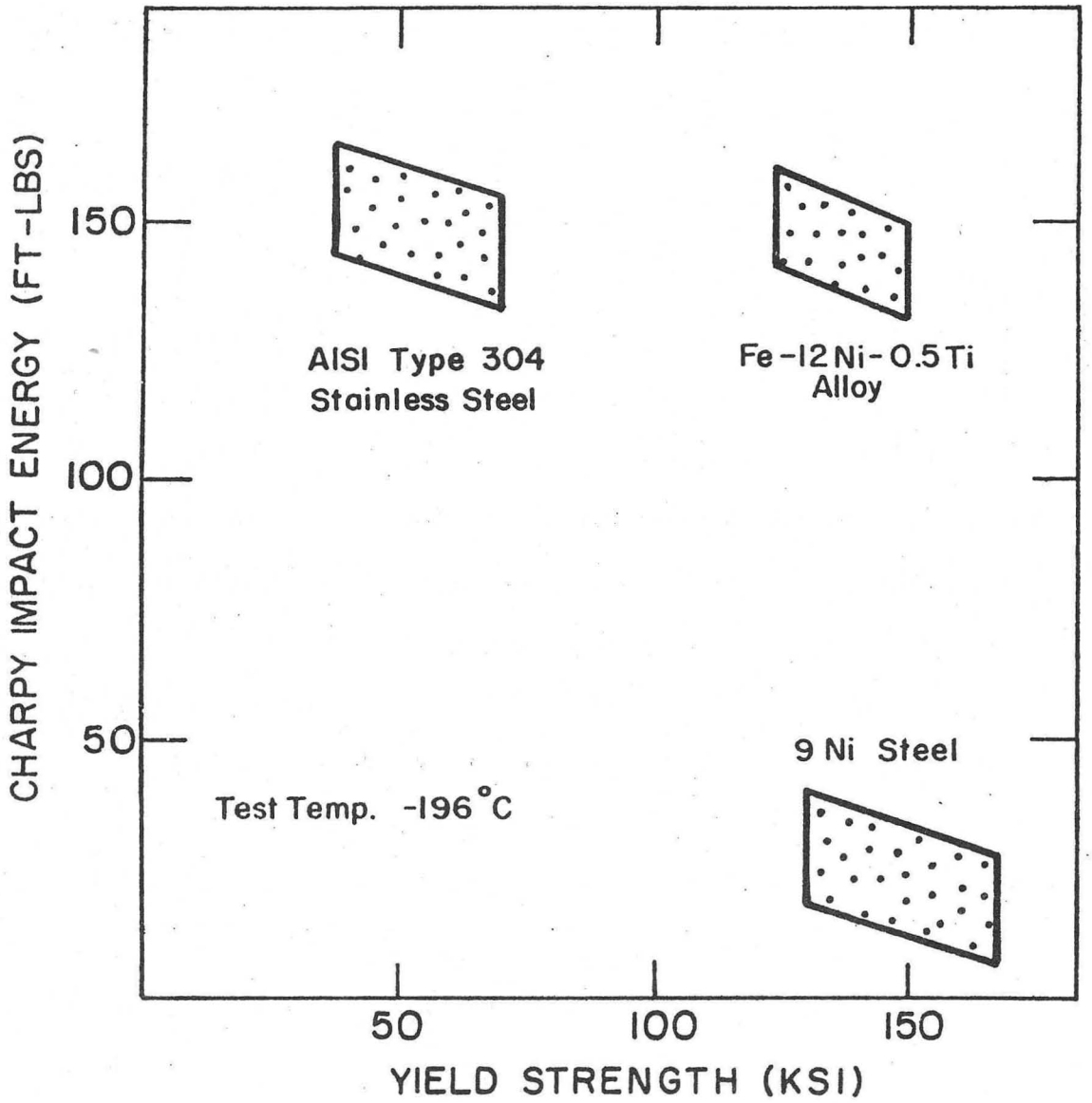

TEST TEMP. -196°C
CHARPY IMPACT ENERGY (FT-LBS)
150
100
50
(10)
(10)
750°C
(10)
700°C
(10)
(8)
(8)
800 °C
(20)
(25)
(50)
(50)
650°C
(50)
1
2
3
4
5
6
TIME AT REHEAT TEMPERATURE (HRS)
XBL 733 5836D

Fig. 3



XBL 733-5836C

Fig. 4

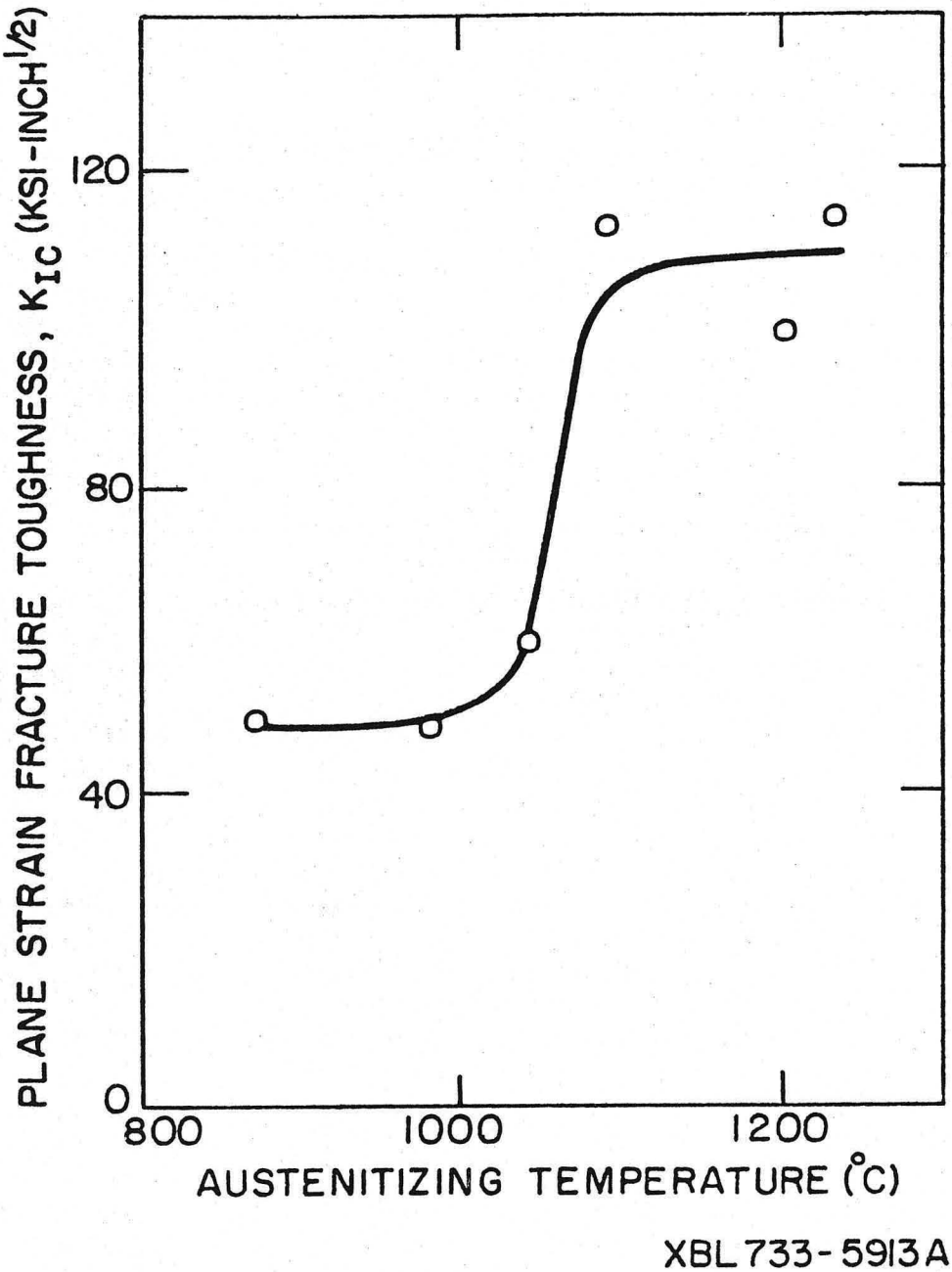
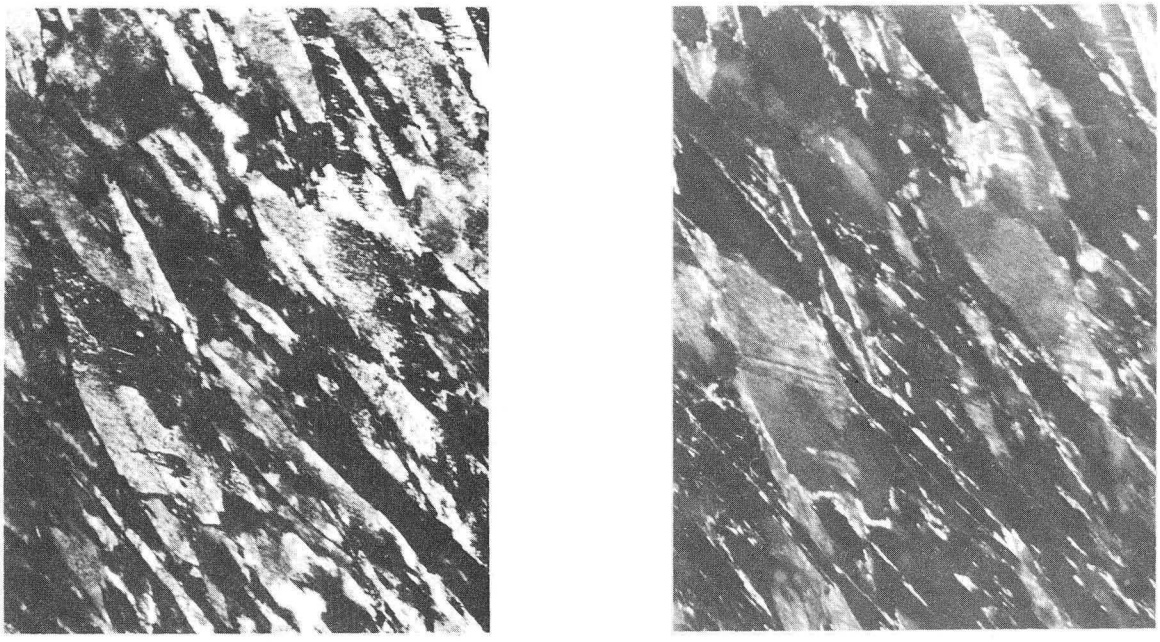

PLANE STRAIN FRACTURE TOUGHNESS, K_{IC} (KSI-INCH$^{1/2}$)
120
80
40
0
800
1000
1200
AUSTENITIZING TEMPERATURE (°C)
XBL 733-5913A

Fig. 5



(a) 1μ (b)

XBB 735-3368

Fig. 6

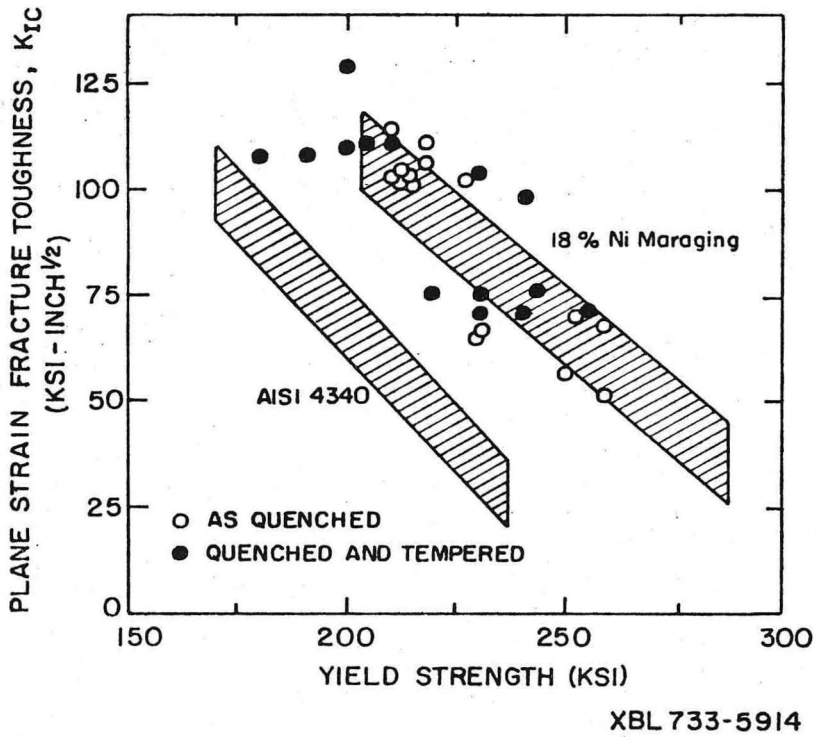

PLANE STRAIN FRACTURE TOUGHNESS, K_{IC}
(KSI - INCH$^{1/2}$)
125
100
75
50
25
0
150
200
250
300
YIELD STRENGTH (KSI)
18 % Ni Maraging
AISI 4340
O AS QUENCHED
● QUENCHED AND TEMPERED
XBL 733-5914

Fig. 7

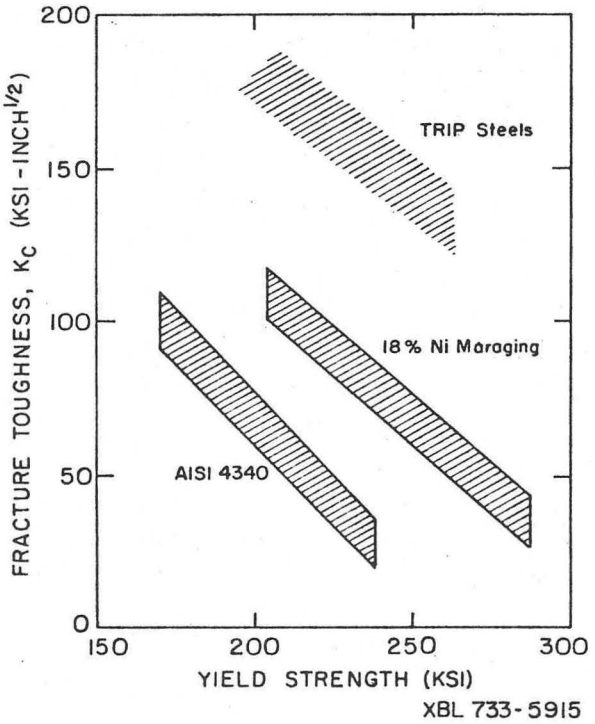
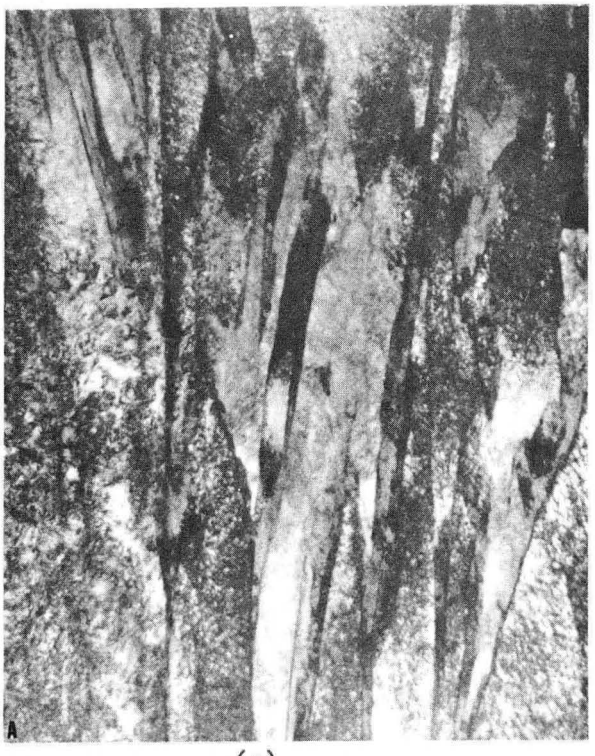

200
150
100
50
0
150
200
250
300
FRACTURE TOUGHNESS, K_C (KSI-INCH$^{1/2}$)
YIELD STRENGTH (KSI)
TRIP Steels
18% Ni Maraging
AISI 4340
XBL 733-5915

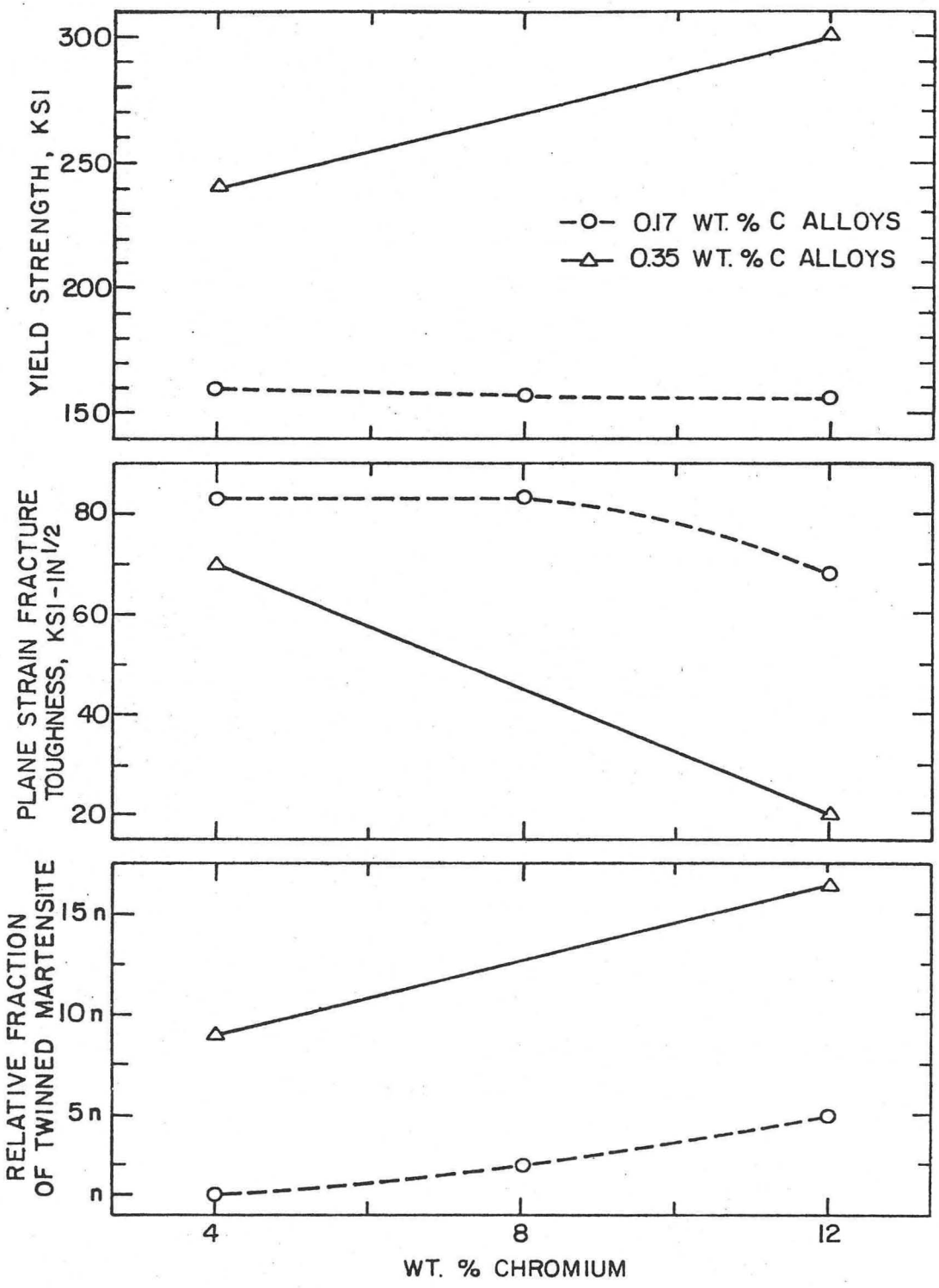
Fig. 8



(a)

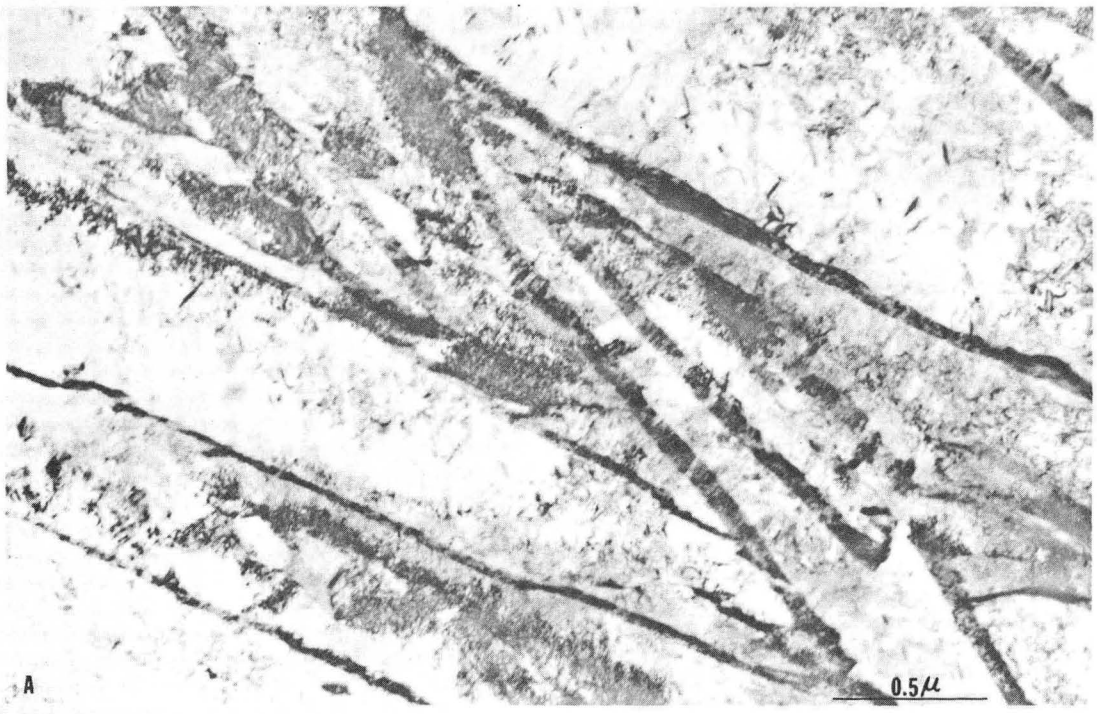
(b) XBB 7211-5622 (a&c only)

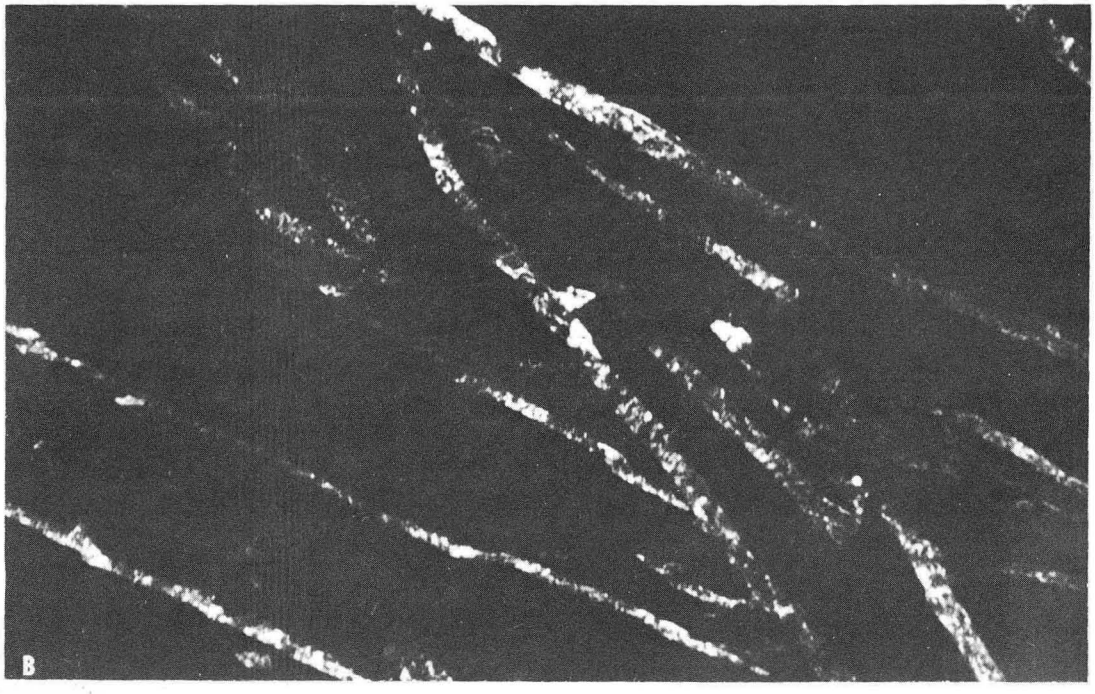
Fig. 9



XBL 7211-7156

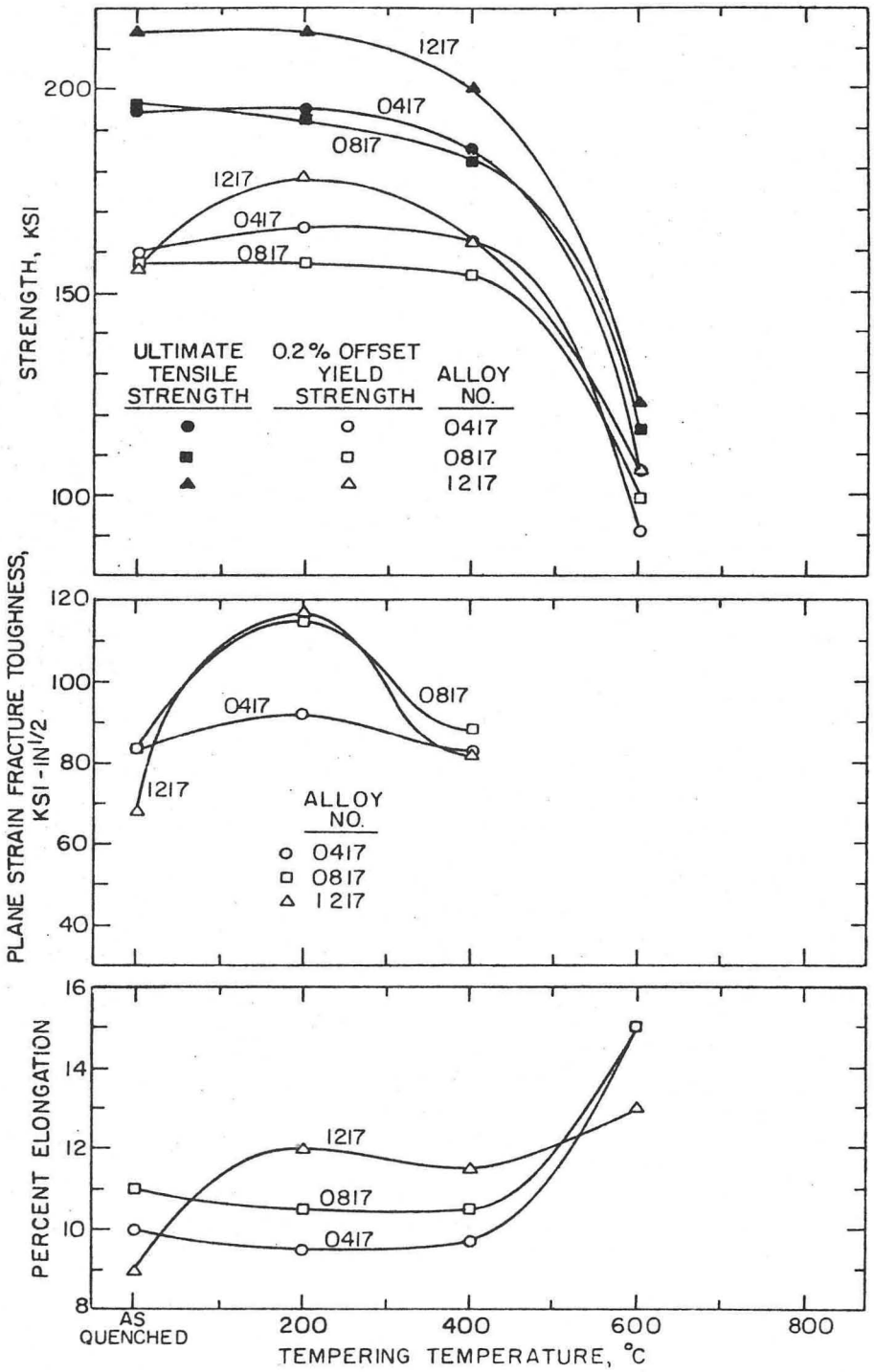
Fig. 10





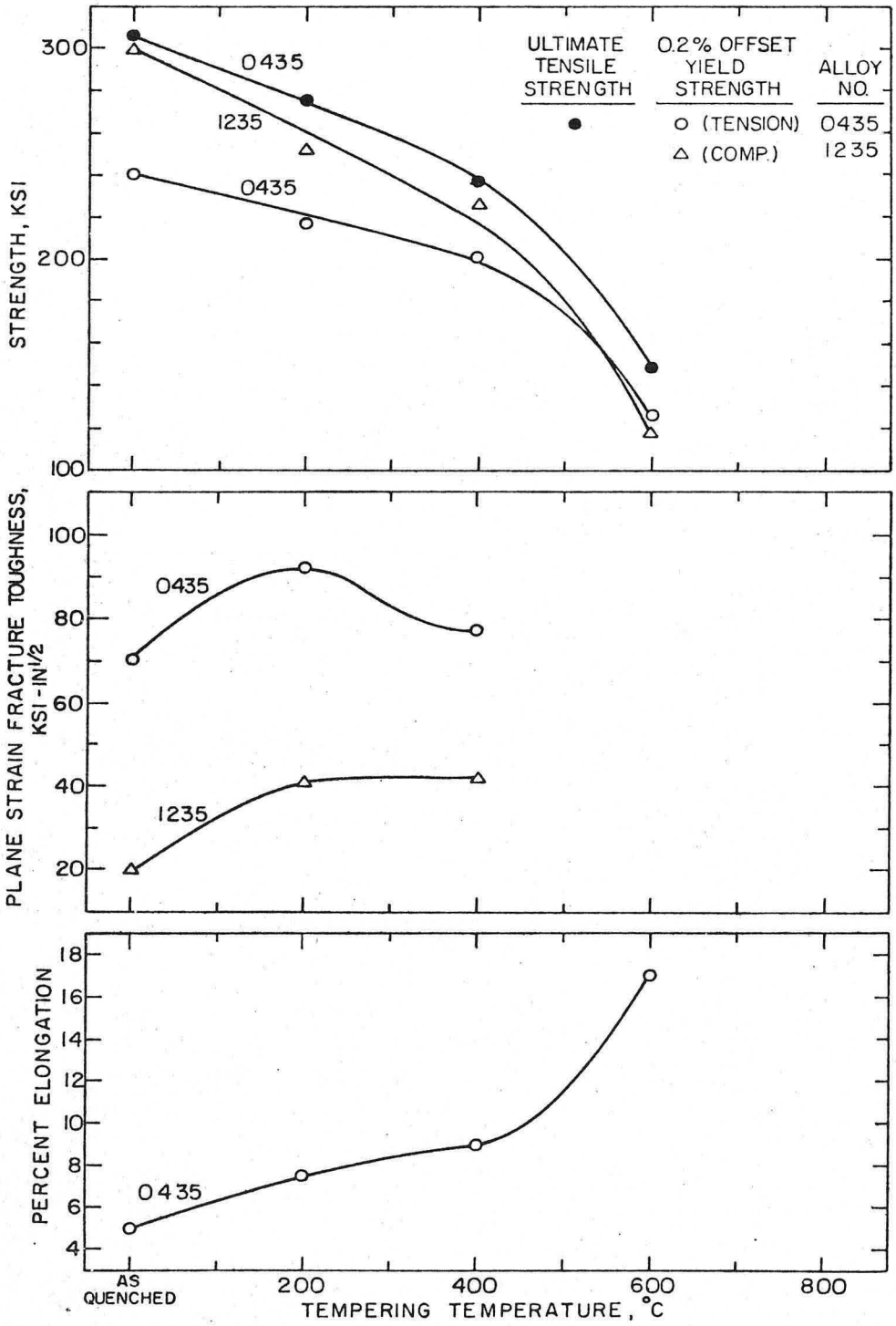
XBB 7211-5624

Fig. 11



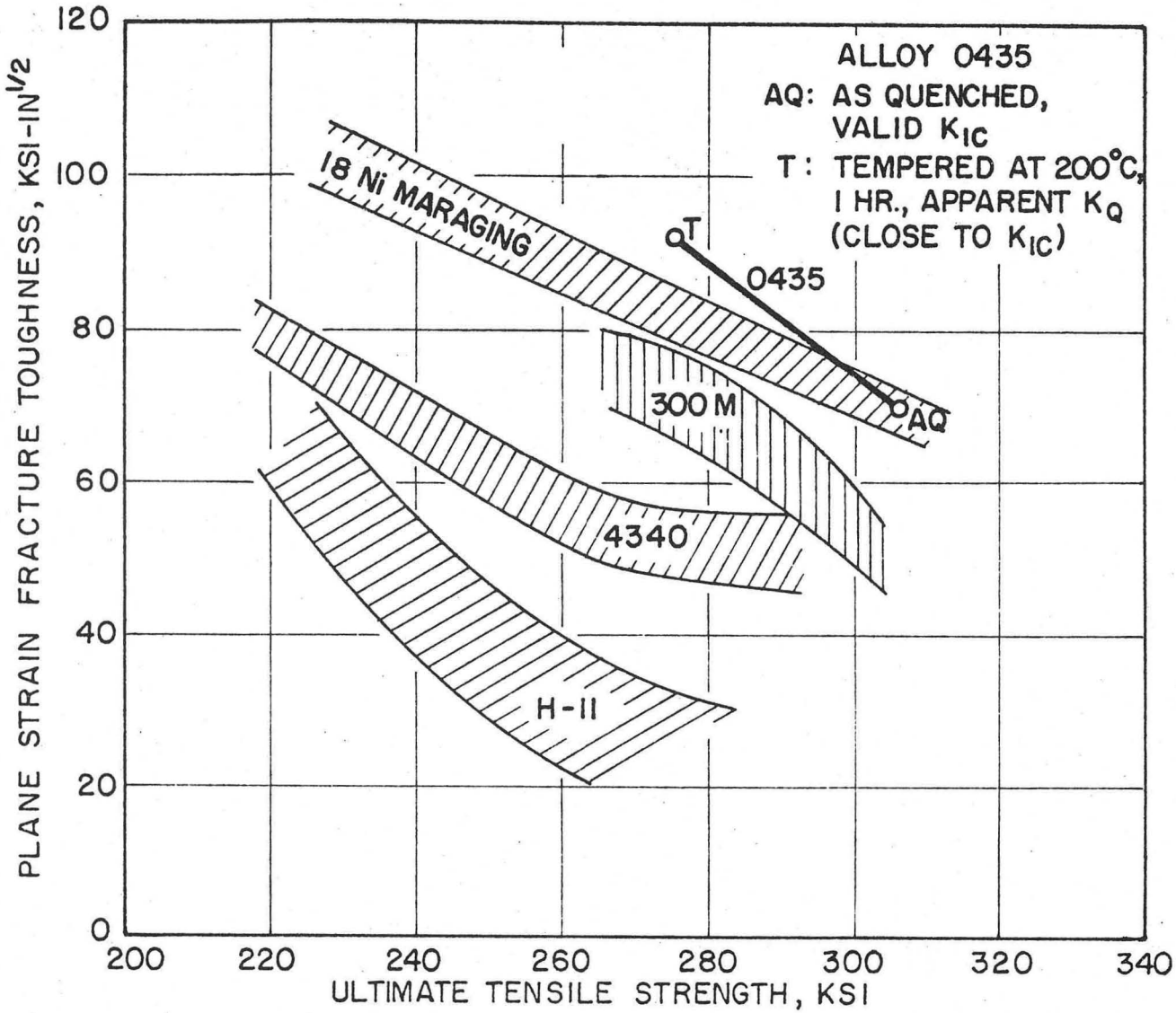
XBL 7310-1949

Fig. 12(a)



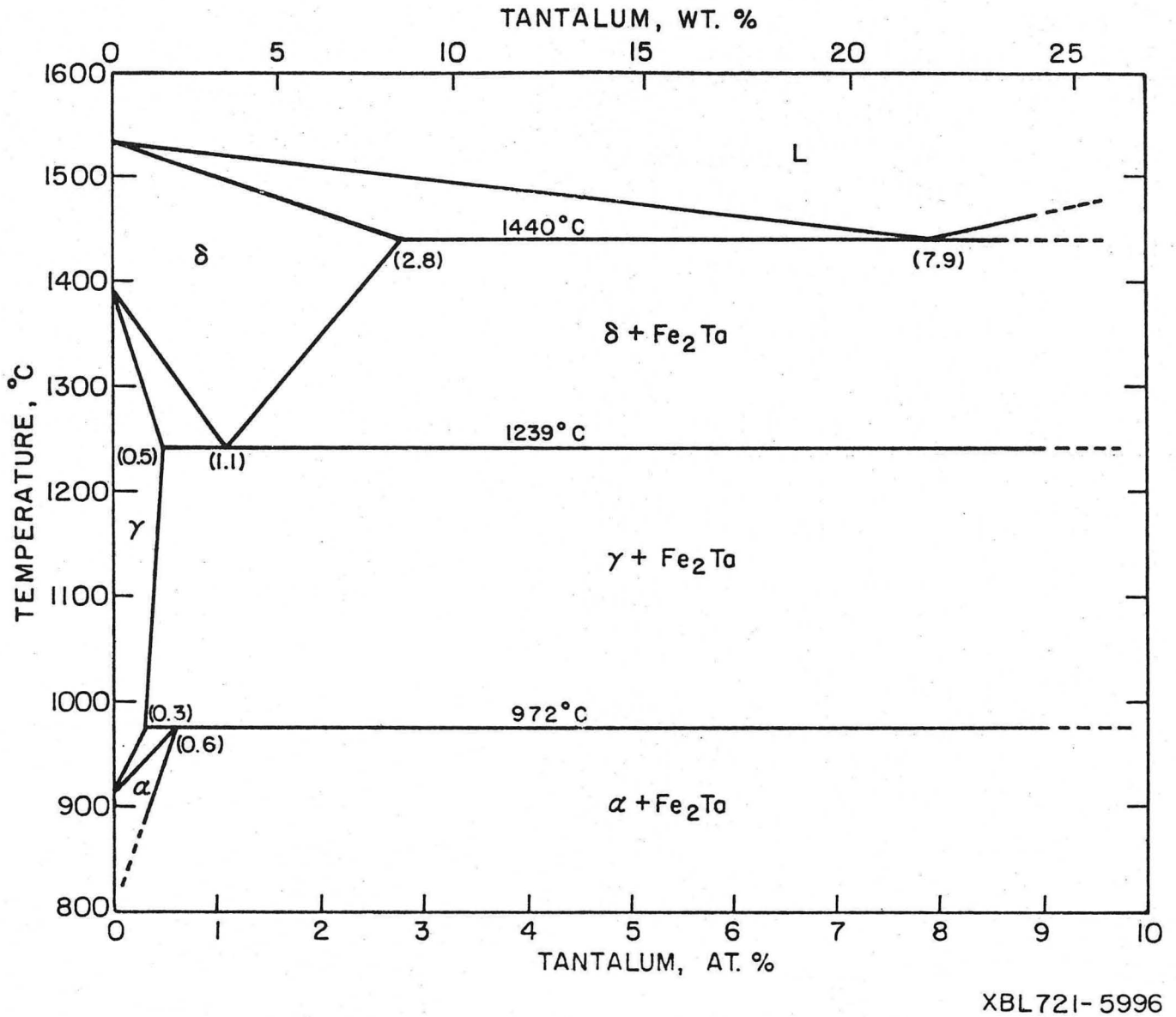
XBL 7310-1951

Fig. 12(b)



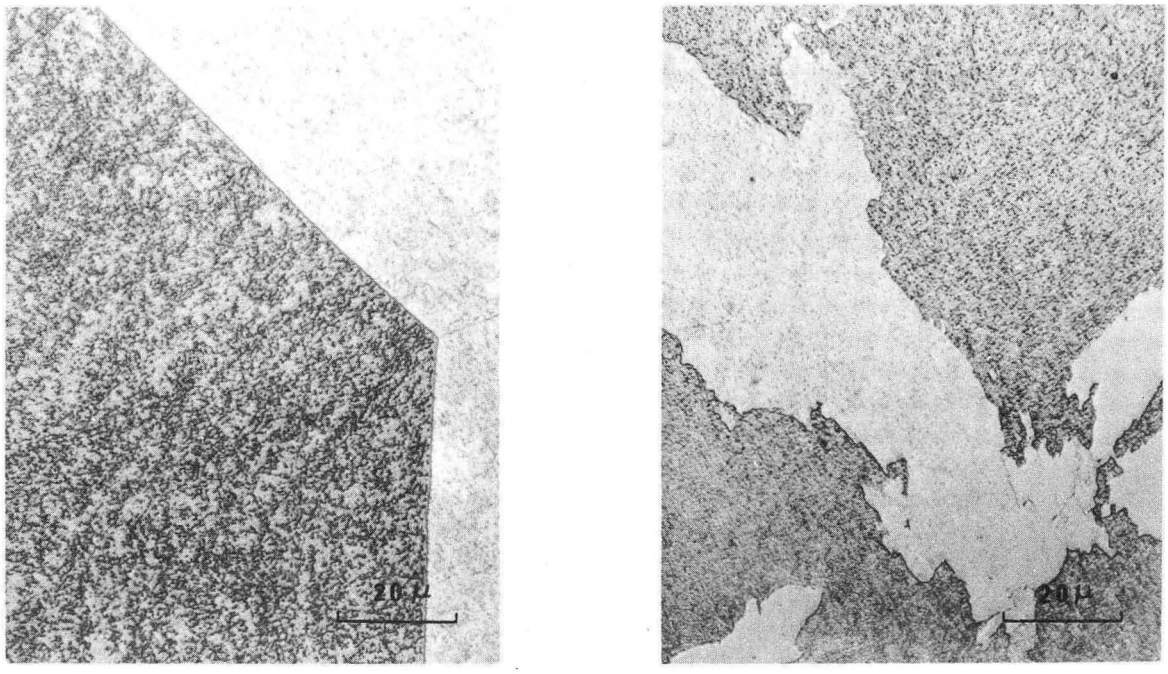
XBL 7310-1950

Fig. 13



XBL721-5996

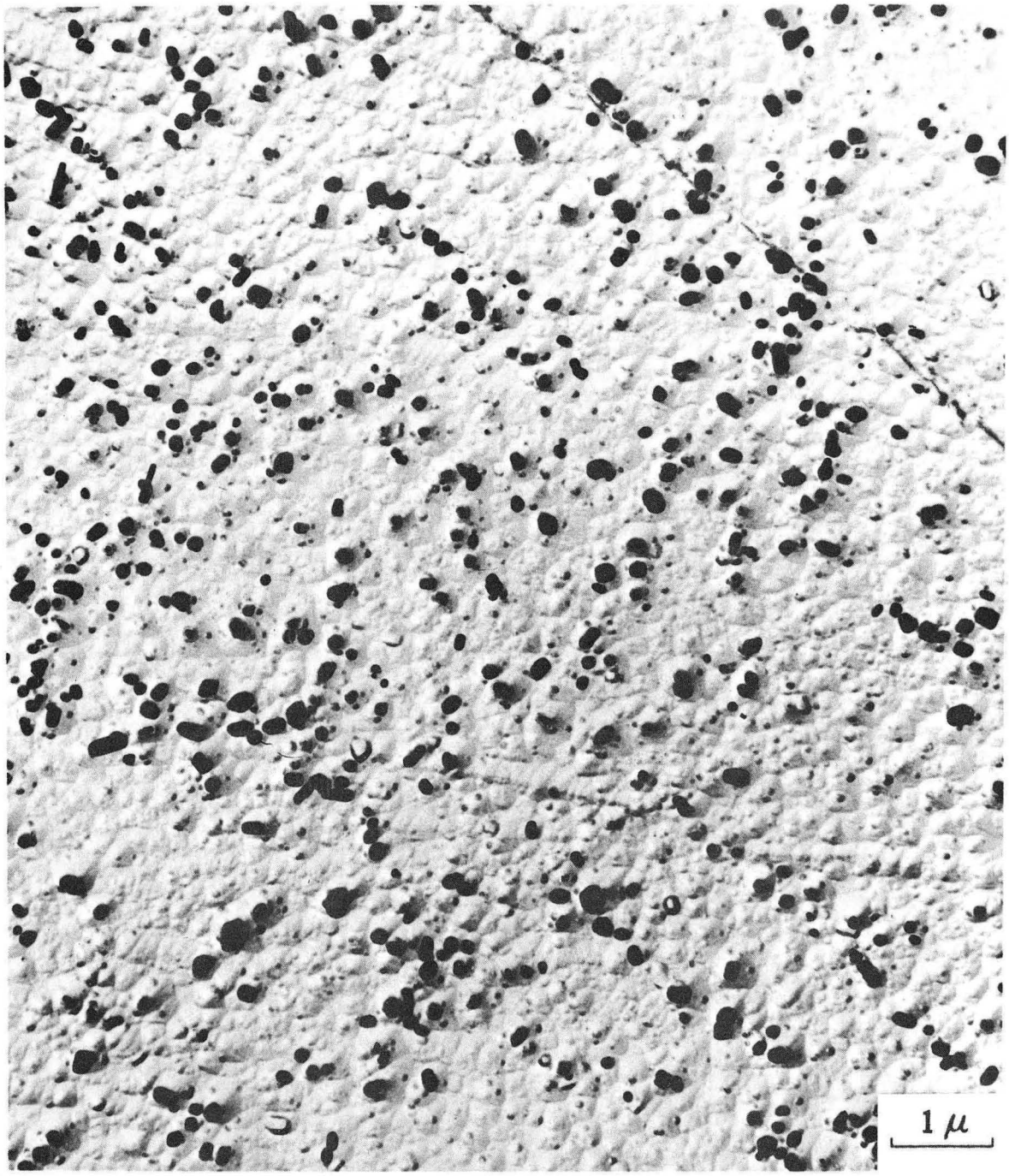
Fig. 14



(a) (b)

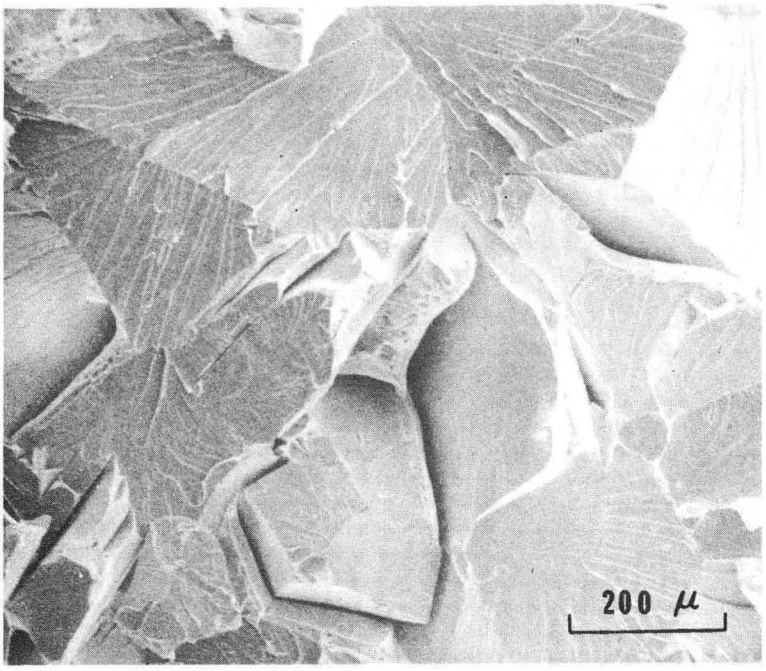
XBB 7210-4984

Fig. 15

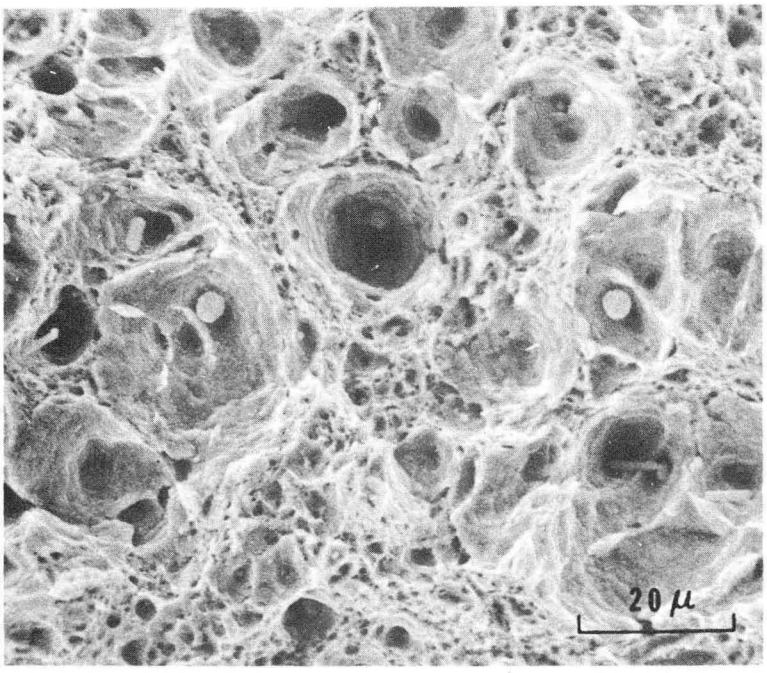


XBB 738-5043

Fig. 16



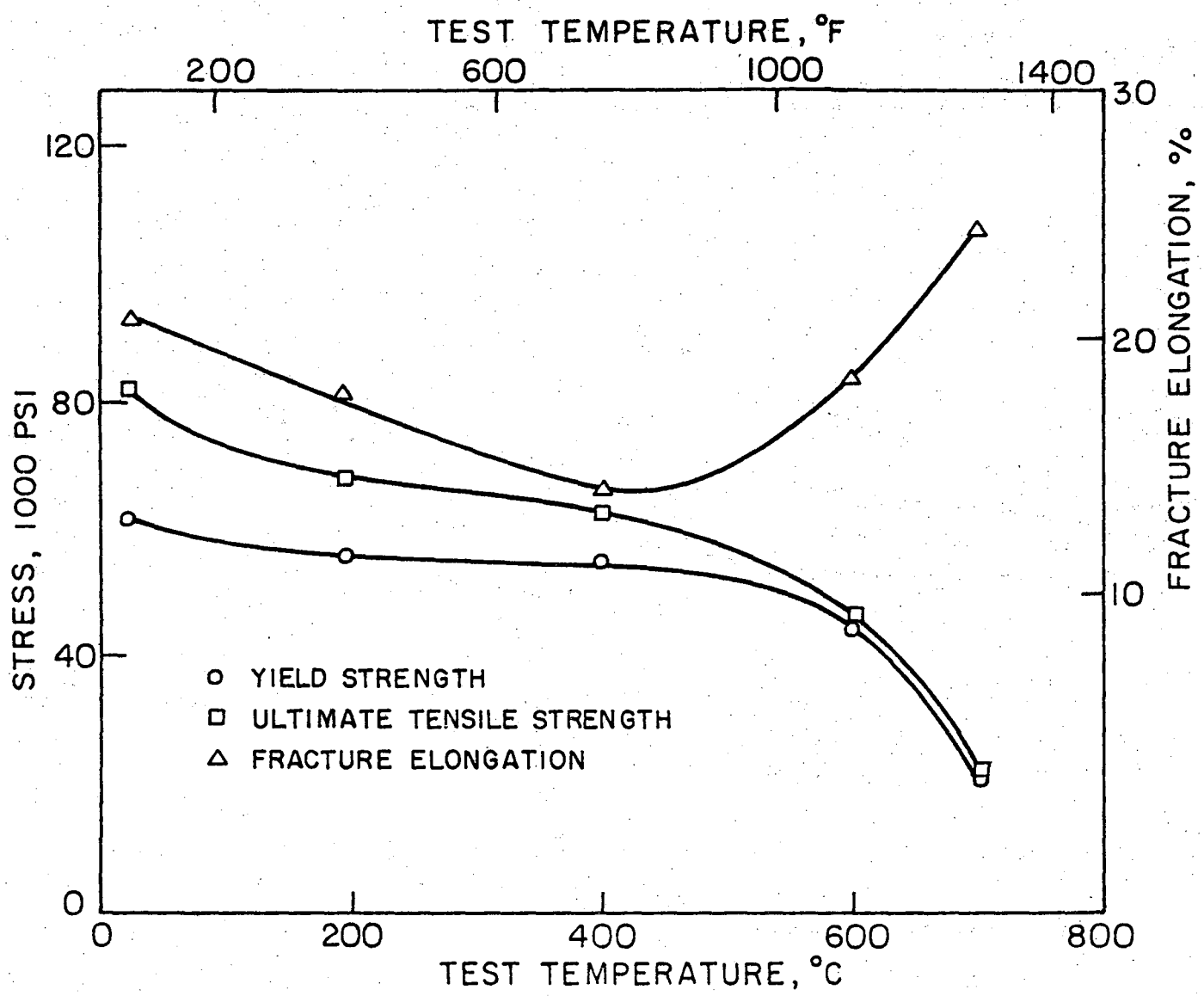
(a)



(b)

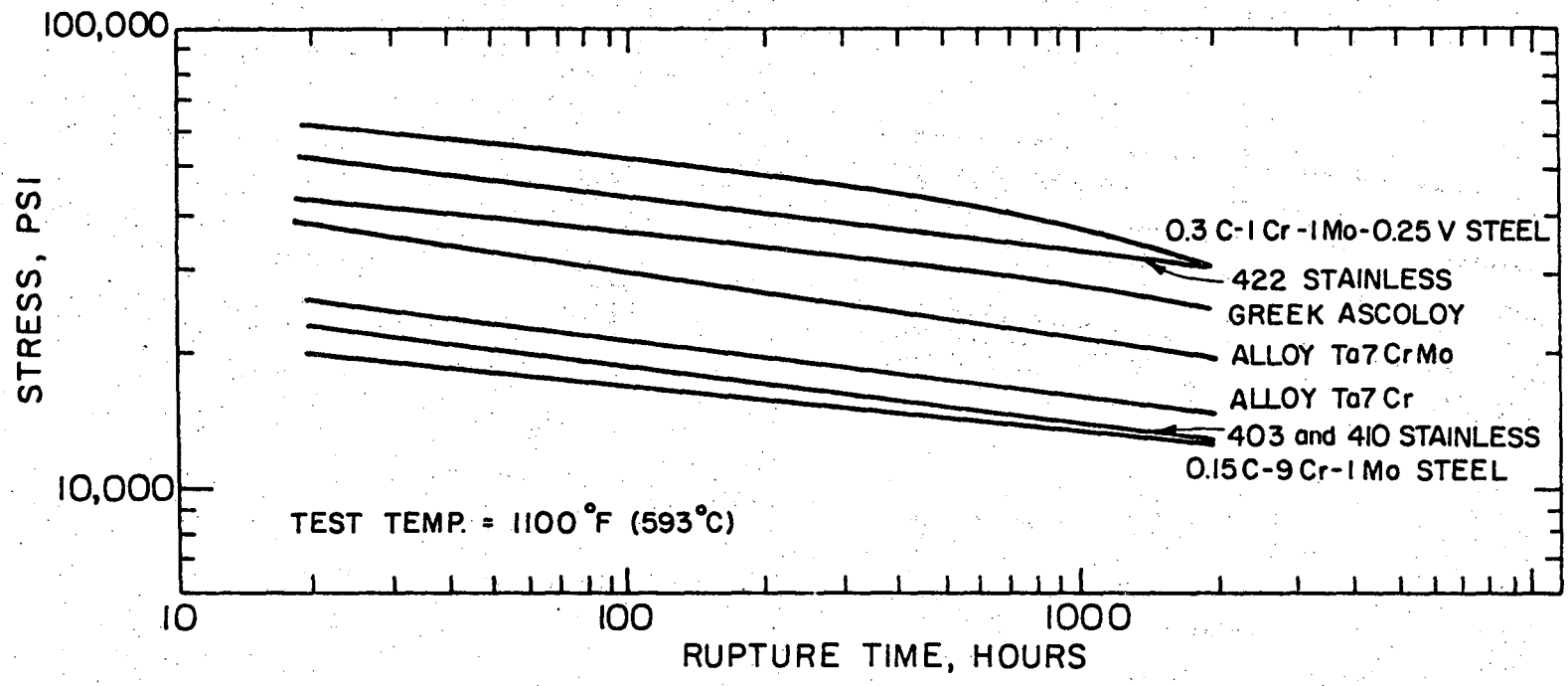
XBB 738-5037A

Fig. 17



XBL737-6449

Fig. 18



XBL738-1773

Fig. 19

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