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ENHANCEMENT OF FRACTURE TOUGHNESS IN HIGH STRENGTH STEEL BY MICROSTRUCTURAL CONTROL

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The development of new alloys with improved mechanical properties has been seriously hampered in the past by the inability of a metallurgist to relate quantitatively the variables of microstructure and fracture toughness. The emergence of a unified theory of fracture toughness in the past decade has done much to alleviate this difficulty. As a consequence of a recent interdisciplinary research effort involving both the disciplines of physical metallurgy and experimental fracture mechanics, we have been able to develop alloys with engineering properties superior to those of commercially available materials. This research has required the creation of new and unusual microstructures, utilizing a variety of thermal and thermomechanical processes. The quantitative relationships of mechanical properties (strength, ductility, work hardening, and fracture toughness) with composition and microstructure are discussed in detail for the newly developed TRIP steels. In the report of another development, it is shown how the fracture toughness of low alloy quenched and tempered steels with yield strengths over 200,000 psi can be improved by as much as 70% by microstructural control. Lastly, the initial results of research on alloys intended for cryogenic service are described. The composition, heat treatment, microstructure and properties of an alloy having more than three times the toughness of the presently used alloys are discussed.

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INTRODUCTION

The successful application of continuum mechanics theory to the problem of fracture toughness in metals is now well known and appreciated by engineers and metallurgists. Applied fracture mechanics is now a design parameter for many complex structures. The intrinsic fracture toughness of a complex alloy, expressed as either the energy release rate, G, or the stress intensity, K, is a macroscopic measure of the integrated effects of micro and macro factors which contribute to the fracture toughness. These include chemical composition, crystal structure, defect structure, and microstructure. The internal structural state of an alloy is of vital concern to the metallurgist.

In principle it should be possible to relate the chemical and microstructural features to strength and fracture toughness. By applying the principles of materials science that have evolved during the past two decades, it should be possible to design new alloys with superior properties. However, virtually no new alloys have been designed by this approach. The inefficient and costly trial-and-error method has been used almost exclusively for the development of commercial alloys.

Typical studies that have been made consist largely of correlations of fracture toughness with impurity content [1-5], particle spacing [6], and methods of manufacture [2,3,7]. In retrospect, the continued adherence to the older approach is perhaps not surprising for fracture mechanics itself is not very old, and many of the high resolution instruments needed to analyze composition on a microstructural scale (such as the microprobe) and to characterize elements of structure whose dimensions are of the order of a micron (such as the scanning electron microscope) are relatively new.

The authors of this paper believe that a quantitative and interdisciplinary form of alloy design will emerge in the next several years. This will render obsolete to a large extent the empirical methods of the past. They also believe that much of the research now devoted to studying why the "good" alloys are good will be supplanted by research directed toward either the creation of entirely new and superior alloys, or, alternatively, toward a large, rather than incremental, improvement of existing ones.

In the present paper, a discussion of results obtained with three alloys is used to illustrate the effectiveness of the newer approach to alloy design. In the first example the development of TRIP steels is summarized. By controlling a single parameter, the thermodynamic stability, metastable austenitic steels having a wide variety of useful combinations of mechanical properties can be produced. Some of the interrelationships between stability, properties, processing and testing conditions are discussed.

The second example shows how the fracture toughness of low alloy quenched and tempered steels (e.g. AISI 4340) can be significantly improved (without a sacrifice in yield strength) through control of microstructure. This control is dictated by the principles of both nucleation theory and the micromechanics of fracture.

The third example is a carbon free iron base alloy designed for use at cryogenic temperatures. This material has extraordinarily high toughness at liquid nitrogen temperatures. The principles involved in improving properties through metallurgical control are discussed.

TRIP STEELS

Tensile Properties: The mechanical properties of solids are known to depend on crystal structure, defect structure and microstrucure. Phase transformations play a major role in determining the nature of the defect structure and microstructure in an alloy. The thermodynamic driving force for most phase transformations is controlled by varying the temperature. In some alloy systems a phase transformation may be initiated by elastic or plastic deformation. Some commercial stainless steels (AISI Type 300 series) undergo such transformations when deformed. However, these commercially available steels have relatively low yield strengths. In a recent development, phase transformations have been utilized to create a new class of ultra high strength metastable austenitic steels, known as TRIP steels [8]. (The word TRIP is an acronym of transformation induced plasticity.) The alloy content of TRIP steels is adjusted so that the face centered cubic phase is stable at room temperature. When these alloys are deformed, they transform to the body centered martensitic phase. The fraction of the volume that transforms for each unit of strain depends upon the composition, the prior history, the temperature, and the strain rate [9-11]. The uniform elongation [8-14], rate of work hardening [9-13], yield strength [10-14], ultimate tensile strength [10,12-14], and the fracture toughness [15-19] are all strongly influenced by the stability of the austenite, as will be shown in detail in a later section.

In a recent study, Bhandarkar, et al [11], determined the influences of composition, processing conditions and test temperature on the stability of austenite. Parts of the following discussion are based upon the results of this study. The chemical composition of several of the steels involved are given in Table I. All of the alloys listed were deformed 70 percent at a temperature of 450°C, unless otherwise designated. The primary purpose of this <u>prior deformation</u> of the <u>austenite</u> (hereafter abbreviated as "PDA") was to raise the yield strength to 200,000 psi or more.

It is possible to obtain experimentally an austenite stability index for TRIP steels. With this index, predictions can be made of the effect of composition and processing conditions on mechanical properties. Gerberich, et al [9], have suggested that the volume fraction of martensite, V_α produced during a tensile test varies as

α alpha

$$V_\alpha = m\epsilon^{\frac{1}{2}}$$

ϵ epsilon

TABLE I. CHEMICAL COMPOSITIONS OF TRIP STEELS

Designations	Compositions, Wt Pct			
	C	Ni	Cr	Mn
CN8Cr	0.325	8.0	9.0	2.0
CN12Cr	0.290	12.0	9.0	2.0
CN16Cr	0.292	16.0	9.0	2.0
CN21Cr	0.287	21.4	9.0	2.0

where m is a constant for a given set of test conditions and ϵ is the conventional strain. The transformation coefficient, m, was found to be a useful index of austenite stability, with higher values of m indicating lower degrees of stability. The value of m is readily obtained by plotting V_α vs $\epsilon^{\frac{1}{2}}$ and fitting the best straight line to the plot. Typical experimental data for steel CN8Cr are shown in Fig. 1 for a test temperature of -78°C. Gerberich showed that for a wide variety of steels and processing conditions, the coefficient, m, was a useful index of austenite stability for TRIP steels. In these steels this coefficient can be varied from zero (completely stable) to about 3.5 (highly unstable). The value of m is zero when the test temperature is at or above the M_d temperature--the temperature above which plastic strain will not induce a transformation. Another phase transformation temperature of importance in these steels is the M_s, which is the temperature at which martensite starts to form in an unstressed steel during cooling. The M_s is always below the M_d. TRIP steels are designed to be used at service temperatures between the M_s and the M_d. In this temperature range, the austenite is thermally stable with respect to the service temperature, but it is unstable with respect to strain. At temperatures well below the M_d, but close to the M_s, the steel becomes so unstable that even an elastic deformation can induce a transformation [11,12,14,20-22].

The exceptionally high fracture toughness of TRIP steels is a consequence of the unusual microstructures produced by a stress or strain induced phase transformation. Deformation induced martensite in highly dislocated austenite is stronger than the parent phase. Martensite plates distributed throughout the austenitic matrix profoundly alter ductility, strain hardening rate, tensile strength, and fracture toughness. When martensite forms, necking in a tensile test is delayed until higher strains are reached. The uniform elongation and the tensile strength are increased because of the higher rate of strain hardening that martensite formation causes. The correlation between the stability coefficient, m, and the elongation to fracture for a large group of alloys of widely varying composition, processing histories and testing temperatures is shown in Fig. 2(a) [9]. The dilatational and shear components of the austenite-to-martensite transformation enhance elongation and increase the Lüders strain (i.e., the strain in the flat part of the stress-strain curve following initial yielding). The correlation between the stability coefficient, m, and the extent of the Lüders strain for the same group of alloys is shown in Fig. 2(b) [9]. Similar correlations exist for the rate of strain hardening and the tensile strength [9-14].

The shape of the engineering stress-strain curves of TRIP steels can be quantitatively predicted by the relations between the stability index, m, and the tensile properties [9]. The rule of mixtures is invoked to describe the mechanical behavior of austenite-martensite combinations. The austenite stability index is generally determined experimentally or it can be calculated from the relationships between stability, composition, and processing conditions [9]. The "predicted" and the observed room temperature stress-strain curves for three steels of varying carbon contents are shown in Fig. 3 [9]. In general, the agreement between the two sets of curves is reasonably good. The only serious discrepancy is between the predicted and measured elongation of the 0.34%C steel. The low ductility obtained experimentally in this case was associated with a premature failure, subsequently determined to be due to a defect in the test specimen. No necking was observed, and the tensile strength of this steel was less than that of the lower carbon (0.25%C) steel.

Bhandarkar, et al [11] showed the relationships that exist between stability and mechanical properties for steels with stabilities varied by changes in test temperature (22° to -196°C), composition (8, 12, 16 and 21 percents of Ni) and PDA temperature (25° to 450°C). Examples of their results are shown in Figs. 4 to 6.

The engineering stress-strain curves at 22°C and -78°C are shown in Fig. 4 for the CN8Cr steel (composition given in Table I) deformed 70 percent at 450°C. The m values are also shown in the figure. The stress-strain curve obtained at 22°C exhibited a well defined Lüders strain of about 6 percent, a low strain hardening rate, and an elongation of 20 percent; the m value was 1.85. The relatively low rate of strain hardening was a consequence of the comparatively low rate of formation of martensite with strain. At a test temperature of -78°C, the stress-strain curve was quite different. The yield strength was lower than that at 22°C by about 60,000 psi, the Lüders strain was smaller and less well defined, the rate of strain hardening was much higher, and the elongation was about one-half of the room temperature value. These features are a consequence of the change in stability produced by the temperature change (as reflected by the m value). Of particular interest are the low yield strength and the high rate of strain hardening. These features are characteristic of an alloy undergoing a stress induced phase transformation. At a strain of only 0.02, about half the austenite had already transformed to martensite in the -78°C test, as shown in Fig. 1. In the specimen tested at 22°C, less than 10 percent of the austenite had transformed at the same strain.

The stability can also be altered by changing the chemical composition or the PDA temperature. The stress-strain curves for three steels of different nickel contents, deformed 70 percent at 450°C and tested at -78°C, are shown in Fig. 5 [11]. Most of the key features in these curves have been previously discussed. However, the striking difference between the curves is worthy of note.

As Bressanelli and Moskowitz [23], Gerberich et al [9], and Tamura, et al [24] have observed, maximum elongation results when martensite is produced at an optimum rate with strain. Too little martensite forming per unit of strain results in early necking and too much causes premature failure. The relatively low m value for steel CN12Cr (estimated to be between 1.0 and 1.5) indicates that the criterion of an optimum m value for a large elongation has been met.

Changes in austenite stability can be induced by variations in the PDA temperature [9,11]. The room temperature engineering stress-strain curves of steel CN8Cr, deformed 70 percent at PDA temperatures of 200° and 450°C are shown in Fig. 6. The difference in the stability (as reflected by the m values) produced by varying the PDA temperature markedly influenced the shape of the stress-strain curves. The well defined Lüders strain and the high strain hardening rate of the specimen deformed at 450°C (as compared with the one deformed at 200°C) reflected the decreased stability produced by the higher deformation temperature. These features are consistent with the data shown in Fig. 2.

Fracture Toughness: Several investigators have suggested that a stress or strain induced phase transformation might enhance the absorption of energy and thereby increase the fracture toughness [15-19,25]. The fracture toughness of TRIP steels has been studied from both the theoretical and experimental viewpoints [15-19,25]. It is clear from these studies that the toughness is dependent upon the stability, the chemical composition of the strain induced martensite, and the strain rate. Gerberich, et al, have shown that, as a first approximation, the fracture toughness K_c, is proportional to $m^{\frac{1}{2}}$ [19]. Room temperature plane stress fracture toughness values of almost 500,000 psi-$in^{\frac{1}{2}}$ were reported for highly unstable (m=2) steels having yield strengths of 200,000 psi or higher; a summary of the data is shown in Fig. 7. The fracture toughness is decreased with increasing amounts of carbon plus nitrogen in an alloy. The effect of carbon content on the apparent K_{Ic} value at -196°C is shown in Fig. 8. Fractographic analysis showed that the martensite in the higher carbon steels (over 0.27 percent) had a tendency to fail by cleavage rather than by shear. The variation with carbon content of apparent K_{Ic} values at -196°C reflects this change of fracture mode.

Both Gerberich, et al [16] and Antolovich and Singh [18] have derived analytical expressions which include the contribution of the phase transformation to the observed fracture toughness of these steels. Both groups of investigators have concluded from theoretical and experimental evidence that the phase transformation is a major source of the fracture toughness of TRIP steels. Antolovich and Singh [18] experimentally determined this contribution to be between two-thirds and three-fourths of the measured crack extension force, G_{Ic}. However, no previous attempt has been made (to the authors' knowledge) to estimate the change in stress state existing near the tip of a crack in a steel undergoing a stress or strain induced transformation. This is a very important factor. When austenite transforms to martensite, there is an increase in volume of about three percent. This corresponds to a linear increase of one percent in each of three perpendicular directions. This volumetric expansion effectively cancels a large part of the triaxial stress that exists near the root of a sharp deep crack or notch. The reduction in the triaxial component of stress has a marked effect on the behavior of thick specimens, where triaxiality is a major contributor to brittle behavior. As a consequence, TRIP steels exhibit an unusual variation in fracture toughness with specimen thickness. The fracture toughness of virtually all high strength alloys decreases sharply with increasing thickness. This does not appear to be the case with TRIP steels. As shown in Fig. 9, the critical stress intensity factors (K), at room temperature, of both the low alloy quenched and tempered steel and the precipitation hardening stainless steel decrease with thickness more severely than that of the TRIP steel [19].

It has been shown both analytically and experimentally that the rate of production of strain induced martensite decreases with increasing crack velocity because of adiabiatic heating [19,26]. The consequence is a lowering of the fracture toughness.

Interrelationships of Stability, Properties, Processing and Testing: The qualitative relationships between the stability of TRIP steels and the mechanical properties are shown in Table II. Vertical arrows indicate whether the property is increased (arrow up) or decreased (arrow down) by a corresponding change in a particular variable. In some cases a property may change in either direction, depending on particular circumstances. Where the effects of a variable are unknown, a question mark is shown. A brief discussion of one example taken from the table follows [11].

An increase in the alloy content of a steel (with the possible exception of Co) increases the stability. However, this is true only when the elements remain in solution in the austenite. An increase in stability can result in either an increase or a decrease in elongation. The elongation is high for values of m between about 0.5 and 1.0, as was shown in Fig. 2(a). Below 0.5 the martensite produced per unit strain is small and therefore the rate of work hardening is too low to prevent necking, and above about 1.0 the elongation decreases with decreasing stability because the large amount of martensite produced per unit strain leads to a loss in ductility.

TABLE II

Probable Relationships Between Stability, as Affected by Several Processing and Testing Variables, and Selected Mechanical Properties

Variable	Stability	Mechanical Property					
		Yield Strength	Lüders Strain	Elongation	Work Hardening Rate	Fracture Toughness	Resistance to Hydrogen Embrittlement
COMPOSITION (increasing)							
Substitutional solutes (exception of Co)	↑ [9,11,12]	↑ [11,12]	↑ [9,11,12]	↑↓[9,11,12]	↓ [11,12]	↓ [19]	↑ [29-31]
Interstitial solutes (C and N)	↑ [9,10, 12,14]	↑ [10,12, 14]	↑ [9,10 12,14]	↑↓[9,10, 12,14]	↓ [10,12, 14]	↓ [19]	↑↓[29-31]
PROCESSING (All variables increasing)							
Amount of deformation (PDA)	↓ [9]	↑ [9,12,14]	↓ [9,12,14]	↑↓[9,12-14]	↑ [12-14]	↑ [19]	↓ [29-31]
Temperature of deformation (PDA)	↓↑[11]	↑↓[11]	↓↑[11]	↑↓[11]	↑↓[11]	↑↓[19]	↓↑[29-31]
Time at temperature of deformation (PDA)	↓ [28]	↑ [28]	↓ [28]	↑↓[28]	↑ [28]	?	↓ [29-31]
TESTING							
Test Temperature (decreasing)	↓[9-11, 14]	↑↓[9-11, 14]	↑↓[9-11, 14]	↑↓[9-11, 14]	↑↓[9-11, 14]	↑↓[15,16 19]	↑↓[29-32]
Strain Rate (increasing)	↑ [23]	↑↓[23]	↑↓[23]	↑↓[23]	↑↓[23]	↑↓[15,16 19]	↑↓[29-32]

Note: [ ] refers to relevant paper in the bibliography.

In general, decreasing the stability by changes in composition results in higher levels of fracture toughness [19]. As mentioned earlier, a steel with an m value of about two exhibited a K_c of 450,000 psi-in$^{\frac{1}{2}}$. It is anticipated that further increase in stability would result in a lower fracture toughness because of the large amount of strain induced martensite produced at the crack front [27].

LOW ALLOY QUENCHED AND TEMPERED STEELS

The low alloy quenched and tempered steels currently in use were developed decades ago by trial and error methods. They all have the undesirable characteristic of low fracture toughness at high levels of strength. Yield strengths above 200,000 psi are obtained by quenching from the austenite temperature range and tempering at a low temperature (e.g. 200°C). It is generally assumed that the low fracture toughness resulting from this treatment is associated with the intrinsic brittleness of high strength martensite. To increase the toughness, it is common practice to temper the steels at higher temperatures. However, this procedure causes a marked lowering of strength.

An important result of recent research in our laboratory has been the discovery that the observed brittleness of untempered steels or those tempered at a low temperature, is not an intrinsic characteristic of martensite; it is a consequence of the presence of small amounts of isothermal austenite decomposition products, or of an unfavorable distribution of carbides that precipitate during tempering. We have been able to increase the fracture toughness of steels having yield strengths in excess of 200,000 psi by as much as 70 percent by using thermal treatments that preclude the formation of unfavorable microstructures [33].

The standard heat treatment for quenched and tempered low alloy steels involves heating to the lower end of the austenite temperature range(to minimize grain size), quenching fast enough to produce martensite, and tempering at a temperature that optimizes mechanical properties. The treatment that we used to improve properties differed in a significant way from commercial practice. The difference is discussed in detail below.

The tensile properties and the fracture toughness of two commercial steels (AISI 4130 and 4340), and an experimental secondary hardening steel (5%Mo-0.60% Mn-0.30%C), were determined as a function of austenitizing temperature. The influence of austenitizing temperature on the room temperature plane strain fracture toughness, K_{Ic}, is shown for the secondary hardening steel in Fig. 10. All specimens were quenched in iced brine and tested in the as-quenched condition. Austenitizing above about 1100°C increased the fracture toughness by a factor of two. Within the temperature range corresponding to the increase in fracture toughness, there was a concomitant increase in the austenite grain size. A large austenite grain size (long thought to be an undesirable microstructural feature for optimum strength and toughness) is actually beneficial to toughness. The reason for this is discussed later in this section.

The yield and the tensile strengths were relatively insensitive to wide variations in austenitizing temperature, as is shown in Table III, but the elongation and reduction in area decreased with increasing austenitizing temperature above about 1100°C. The loss in tensile ductility caused by drastic quenching from high austenitizing temperatures can be recovered in this steel, however, by a low tempering treatment (below 225°C) without a significant decrease in either strength or toughness.

A similar variation of fracture toughness with austenitizing temperature has been obtained with two commercial medium carbon low alloy steels. The influences of austenitizing temperature and quenching medium on AISI 4130 steel are shown in Table IV. The more drastic quench (iced brine vs oil) at the higher austenitizing temperature (1200°C) further enhanced the fracture toughness. (The range of austenitizing temperatures recommended in commercial practice is 835-915°C.)

The combined influence of austenitizing temperature and quenching rate on the fracture toughness was also investigated for a second widely used commercial steel (AISI 4340). Specimens were given an initial austenitizing treatment at a temperature of 1200°C and either directly quenched or, alternatively, cooled to 870°C and then quenched into one of three different media--iced brine, water, or oil. The resulting values of the room temperature plane strain fracture toughness are given in Table V, which also shows the fracture toughness obtained when the conventional austenitizing temperature of 870°C was employed (all tests conducted in the as-quenched condition).

TABLE III

AUSTENITIZING TEMPERATURE AND TENSILE PROPERTIES OF A 5%Mo-0.60%Mn-0.30%C STEEL†

Austenitizing Temperature, °C	Yield Strength 1000 psi	Tensile Strength 1000 psi	Elong., % in 1"	Red. of Area, %
1255	205	245	7	20
1225	214	261	8	26
1115	210	260	11	32
1060	212	251	12	48
1005	198	244	13	47
895	194	228	13	47

†Quenched in iced brine and tested at room temperature in the as-quenched condition.

TABLE IV

AUSTENITIZING TEMPERATURE, QUENCHING MEDIA AND FRACTURE TOUGHNESS OF A COMMERCIAL AISI 4130 STEEL†

Austenitizing Temperature, °C	Quenching Medium	Fracture Toughness, K_{Ic}, (Ksi-in$^{1/2}$)
1200	iced brine	98.5
1200	oil	83.5
870	oil	58.0

†All tests conducted at room temperature in the as-quenched condition.

TABLE V

AUSTENITIZING TREATMENTS, QUENCHING MEDIA AND FRACTURE TOUGHNESS OF A COMMERCIAL AISI 4340 STEEL†

Austenitizing Temperature, °C (and quenching procedure)	Quenching Medium	Fracture Toughness, K_{Ic}, (Ksi-in$^{\frac{1}{2}}$)
1200, direct quench	iced brine	cracked on quenching
	water	cracked on quenching
	oil	67.3
1200 to 870 and quench	iced brine	62.3
	water	61.1
	oil	63.8
870, direct quench	iced brine	cracked on quenching
	water	cracked on quenching
	oil	40.0

†All tests conducted at room temperature in the as-quenched condition.

Several conclusions can be drawn from these experiments. It appears that to achieve high fracture toughness it is neither necessary nor desirable to quench directly from a high austenitizing temperature. Reducing the temperature from 1200 to 870°C before quenching minimizes the danger of cracking and results in about the same fracture toughness as does quenching directly from the highest austenitizing temperature (Table V). The higher carbon content of the 4340 steel (compared to the 4130) led to cracking when the steel was quenched directly from 1200°C into iced brine or water. Seoondly, it appears that for the more highly alloyed 4340 steel the austenitizing temperature is more important than quenching rate for optimizing toughness. This is indicated by the results of the two step treatment reported in Table V. Thirdly, it is apparent that with the low austenitizing temperature employed in commercial practice (870°C), the fracture toughness is substantially lower than that attainable with a high austenitizing temperature followed by a quench from 870°C. Finally, it is significant that quenching into media that produce faster cooling than oil results in quench cracking to a much greater extent when the 870°C austenitizing temperature is used than when the higher austenitizing treatment is employed. The quench cracks followed the prior austenite grain boundaries.

Metallurgists generally assume that martensite in medium carbon as-quenched steels is intrinsically brittle. Many theories have been advanced to explain this brittleness. However, experiments of the type described above show that untempered medium carbon martensite can be extraordinarily tough as well as strong and hard. Indeed, the strength and toughness of drastically quenched and untempered low alloy steels (less than 5 percent of alloying elements) are equaled only by those of the high alloy (about 30 percent total alloying elements) and more costly maraging steels.

The initial effort in our laboratory was directed toward determining the cause of the low fracture toughness of conventionally quenched and tempered low alloy steels. Although many factors could contribute to the observed brittleness, our experiments suggested that reactions occur during quenching and tempering that lead to transformation products and microstructures that decrease toughness. Mixed microstructures are known to be deleterious to toughness, especially when a minor phase is present as a network at the prior austenite grain boundaries. The effectiveness of a high austenitizing temperature in reducing brittleness is attributed to the fact that the average grain boundary energy is lower after grain growth, and this in turn delays the nucleation of a decomposition product at the grain boundaries. In effect, this increases the bainite hardenability.

Our continuing research program is directed toward developing a new class of ultra high strength steels having markedly high fracture toughnesses than existing steels. To accomplish this objective we plan to apply the basic principles of materials science to the design of multiphase high strength materials.

A HIGH TOUGHNESS CRYOGENIC STEEL

The design requirements of structures intended for cryogenic service are demanding and complex. Among the most important of these is safety. Consequently, high fracture toughness is mandatory. Another requirement is that the yield strength should be as high as possible to minimize section thickness.

Steels generally used for cryogenic service are limited in number. A commonly used austenitic steel has a high toughness, but a low strength (about 40,000 psi yield strength). Another common steel contains low carbon and 9 percent nickel (about 120,000 psi yield strength); this steel has a relatively low toughness [34,35]. Ideally, a cryogenic structural steel should possess both high strength and high toughness.

We have attempted to design a superior cryogenic alloy by using the basic principles of materials science. In terms of defect theory, strength is enhanced by immobilizing or pinning dislocations. Toughness is increased, at a given strength level, by providing a high density of mobile or unpinned dislocations. The latter are necessary for the degree of plasticity that is required for high fracture toughness. This desirable combination is generally absent in the cryogenic alloys currently in use.

In our work, we attempted to resolve this problem by utilizing a thermally induced phase transformation which would allow selective decoration of some, but not all, dislocations. An example of such a transformation is found in iron-rich alloys of the Fe-Ni-Ti system.

(These alloys are not steels in the conventional sense, but are often classed as such.)

Although the temperature vs composition of the α (BCC) and γ (FCC) phase boundaries in this system are not precisely known, for small concentrations of titanium it was found by dilatometric experiments that they were similar to, but slightly higher than, those of the Fe-Ni system. From dilatometric and metallographic studies of the steel chosen for study (12 percent Ni, 0.5 percent Ti), it is clear that the iron-rich Fe-Ni-Ti system consists of a two phase region at low temperatures ($\alpha+\mathrm{Ni}_x\mathrm{Ti}_y$), three phases at an intermediate temperature ($\alpha+\gamma+\mathrm{Ni}_x\mathrm{Ti}_y$), and a single phase at the highest temperature (γ). The nickel-titanium phase is believed to be hexagonal $\mathrm{Ni}_3\mathrm{Ti}$. Based on preliminary studies, a tentative phase diagram was constructed, as shown in Fig. 11.

The existence of the three phase region at intermediate temperatures suggested the possibility of selectively decorating some dislocations, with the opportunity to allow those generated by an $\gamma\rightarrow\alpha$ transformation during fast cooling to remain free. The precipitation of $\mathrm{Ni}_3\mathrm{Ti}$ occurs only on dislocations in the BCC phase, and only at elevated temperatures. Hence, the ratio of the densities of pinned and unpinned dislocations can be controlled by regulating the ratio of the volume fractions of BCC (α) and FCC (γ) phases during heat treatment in the three phase field. The latter ratio is uniquely determined by the heat treating temperature. Upon quenching to room temperature, the FCC phase transforms to BCC by a martensitic reaction, producing a large number of unpinned dislocations. Thus, in principle, it should be possible to produce alloys with different combinations of strength and toughness. Our experiments are in their early stages but they show a significant and effective trend.

The influence of the heat treating temperature on the toughness (as measured by the Charpy V-notch value at -196°C) and the hardness at room temperature are shown in Fig. 12. The heavy bar shown on the abscissa of the graph indicates the approximate temperature range of the experimentally determined three phase region. For heat treating temperatures associated with the single phase (γ) region, both the hardness and the toughness were low. For temperatures corresponding roughly to equal proportions of gamma and alpha, with a small volume fraction of Ni_3Ti, the toughness was approximately eight times greater than that resulting from quenching from the single phase (γ) region. For decreasing temperatures between about 720°C and 650°C, corresponding to increasing volume fractions of α relative to γ, the expected increase in hardness was observed. The fracture appearances of two Charpy bars, given solution treatments of 800° and 700°C, and broken at -196°C, are shown in Fig. 13. The high degree of plasticity associated with the fracture of the bar treated at 700°C is evident.

Lastly, the toughness and yield strength at -196°C for the two commercial steels mentioned earlier are contrasted with those of the new steel in Fig. 14. The superior combination of strength and toughness of the new steel is clearly evident.

The preliminary results reported herein have encouraged us to believe that substantial progress can be made toward developing new and useful alloys by applying the basic principles of materials science to complex multiphase alloys.

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REFERENCES

[1] S. J. Matas, Influence of impurities and related effects on strength and toughness of high strength steels, Metals Engng. Q., 4, 48-57 (1964).

[2] G. E. Gazza and F. R. Larson, Impact properties of air and vacuum melted AISI 4340 steel, Trans. Am. Soc. Metals, 58, 183-194 (1965).

[3] R. P. Wei, Fracture toughness testing in alloy development, Symposium on Fracture Toughness Testing and Its Applications, ASTM STP 381, Am. Soc. Test. Mater., Philadelphia, 279-289 (1965).

[4] A. J. Birkle, D. S. Dabkowski, J. P. Paulina and L. F. Porter, A metallographic investigation of the factors affecting the notch toughness of maraging steels, Trans. Am. Soc. Metals, 58, 285-301 (1965).

[5] C. L. M. Cottrell, Effect of impurities on fracture toughness of high strength steel, Fracture Toughness of High Strength Materials: Theory and Practice, ISI Publication 120, The Iron and Steel Inst., London, 112-115 (1970).

[6] A. S. Tetelman and A. J. McEvily, Jr., Fracture of high strength materials, Fracture, An Advanced Treatise, Vol. VI, ed. H. Liebowitz, Academic Press, Inc., New York and London, 137-180 (1969).

[7] A. T. English and W. A. Backofen, Metal processing and fracture, Fracture, An Advanced Treatise, Vol. VI, ed. H. Liebowitz, Academic Press, Inc., New York and London, 83-136 (1969).

[8] V. F. Zackay, E. R. Parker, D. Fahr and R. Busch, The enhancement of ductility in high strength steels, Trans. Am. Soc. Metals, 60, 252-259 (1967).

[9] W. W. Gerberich, G. Thomas, E. R. Parker and V. F. Zackay, Metastable austenites: decomposition and strength, Proc. Second Int. Conf. on the Strength of Metals and Alloys, Asilomar, California, August 1970, 894-899.

[10] G. R. Chanani, V. F. Zackay and E. R. Parker, Tensile properties of 0.05 to 0.20 pct C TRIP steels, Met Trans., 2, 133-139 (1971).

[11] D. Bhandarkar, V. F. Zackay and E. R. Parker, Stability and mechanical properties of metastable austenitic steels, submitted to Met. Trans. (1972).

[12] D. Fahr, Enhancement of ductility in high strength steels, Ph. D. Thesis, UCRL Report 19060, Univ. of California, Berkeley, California, September 1969.

[13] J. A. Hall, V. F. Zackay and E. R. Parker, Structural observations in a metastable austenitic steel, Trans. Am. Soc. Metals, 62, 965-976 (1969).

[14] D. Fahr, Stress- and strain-induced formation of martensite and its effects on strength and ductility of metastable austenitic stainless steels, Met. Trans. 2, 1883-1892 (1971).

[15] W. W. Gerberich, P. L. Hemmings, M. D. Merz and V. F. Zackay, Preliminary toughness results on TRIP steel, Trans. Am. Soc. Metals, 61, 843-847 (1968).

[16] W. W. Gerberich, P. L. Hemmings, V. F. Zackay and E. R. Parker, Interactions between crack growth and strain induced transformations, Fracture 1969, ed. P. L. Pratt, Chapman and Hall Ltd., London, 288-305 (1969).

[17] W. W. Gerberich and J. P. Birat, A metastable austenite with plane stress fracture toughness near 500,000 psi-in$^{\frac{1}{2}}$, Int. J. Fract. Mech., 7, 108-110 (1971).

[18] S. D. Antolovich and B. Singh, On the toughness increment associated with the austenite to martensite phase transformation in TRIP steels, Met. Trans., 2, 2135-2141 (1971).

[19] W. W. Gerberich, P. L. Hemmings and V. F. Zackay, Fracture and fractography of metastable austenites, Met. Trans., 2, 2243-2253 (1971).

[20] E. Scheil, Uber die umwandlung des austenits in martensite in eisen-nickel-legierungen unter belastung, Z. Anorg. Allg. Chem. 207, 21-40 (1932).

[21] S. A. Kulin, M. Cohen and B. L. Averbach, Effect of applied stress on the martensitic transformation, Trans. AIME, 194, 661-668 (1952).

[22] T. Angel, Formation of martensite in austenitic stainless steels: effects of deformation, temperature and composition, J. Iron Steel Inst., 177, 165-174 (1954).

[23] J. P. Bressanelli and A. Moskowitz, Effects of strain rate, temperature and composition on tensile properties of metastable

austenitic stainless steels, Trans. Am. Soc. Metals, 59, 223-239 (1966).

[24] I. Tamura, T. Maki, H. Hato, Y. Tomota and M. Okada, Strength and ductility of austenitic iron alloys accompanying strain induced martensitic transformation, Proc. Second Int. Conf. on the Strength of Metals and Alloys, Asilomar, California, August 1970, 900-904.

[25] S. D. Antolovich, Fracture toughness and strain induced phase transformations, Trans. Metall. Soc. AIME, 242, 2371-2373 (1968).

[26] C. Dokko, TRIP phenomena in impact tests, M. S. Thesis, UCRL Report 19068, Univ. of California, Berkeley, California, September 1969.

[27] W. W. Gerberich, Univ. of Minnesota, Dep. of Chemical Engng. and Mater. Sci., Minneapolis, Private communication.

[28] M. E. Sauby, The investigation of high strength in high carbon stainless steel, M. S. Thesis, UCRL Report 19678, Univ. of California, Berkeley, California, September 1970.

[29] R. A. McCoy, W. W. Gerberich and V. F. Zackay, On the resistance of TRIP steel to hydrogen embrittlement, Met. Trans., 1, 2031-2034 (1970).

[30] R. A. McCoy, The resistance of TRIP steels to hydrogen embrittlement, D. Eng. Thesis, LBL Report 135, Univ. of California, Berkeley, California, September 1971.

[31] V. F. Zackay, W. W. Gerberich and S. F. Ravitz, Mechanical properties and corrosion resistance of TRIP steels, Western

Technical Conf., Los Angeles, California, March 1971.

[32] E. Gold and T. Koppenaal, Anomalous ductility of TRIP steel, Trans. Am. Soc. Metals, 62, 607-610 (1969).

[33] V. F. Zackay, E. R. Parker, R. D. Goolsby and W. E. Wood, Untempered ultra high strength steels of high fracture toughness, to be published in Nature.

[34] F. R. Schwartzberg, S. H. Osgood, R. D. Keys and T. I. Kiefer, Cryogenic Materials Data Handbook, Technical Documentary Report No. ML-TDR-64-280, Martin Company, Denver, Colorado (1964).

[35] C. W. Marschall, R. F. Hehemann and A. R. Troiano, The Characteristics of 9% Nickel Low Carbon Steel, Trans. Am. Soc. Metals, 55, 135-148 (1962).

FIGURES

1. The relation between engineering strain and the volume fraction of martensite that is produced in steel CN8Cr, deformed 70 percent at 450°C, when tested at a temperature of -78°C [11].
2. (a) The correlation between the stability coefficient, m, and the elongation to fracture for a large group of alloys of widely varying composition, processing histories, and testing temperatures [9]. (b) The correlation between the stability coefficient, m, and the Lüders strain for a large group of alloys of widely varying composition, processing histories, and testing temperatures [9].
3. Experimental and calculated room temperature stress-strain curves for three TRIP steels of different stabilities [9].
4. The engineering stress-strain curves of steel CN8Cr, deformed 70 percent at 450°C, at 22°C and -78°C. The values of the stability coefficients, m, are shown [11].
5. The engineering stress-strain curves for steels containing 8, 12, and 16 percent nickel, deformed 70 percent at 450°C and tested at -78°C. The values of the stability coefficients, m, are shown [11].
6. The room temperature engineering stress-strain curves of steel CN8Cr, deformed 70 percent at PDA temperatures 200°C and 450°C. The values of the stability coefficients, m, are shown [11].
7. Influence of austenite stability (m value) on the plane stress fracture toughness of high strength metastable austenites [19].
8. Effect of carbon content on cleavage of martensite and, hence, on apparent K_{Ic} at -196°C [19].

9. Effect of thickness on the critical stress intensity factors at room temperature for TRIP steel and two commercial steels [19].

10. Influence of austenitizing temperature on the room temperature plane strain fracture toughness, K_{Ic}, of an as-quenched experimental secondary hardening steel (5% Mo-0.60% Mn-0.30%C) [33].

11. Portion of a tentative phase diagram of the Fe-Ni-Ti system showing the transformation temperatures for the steel 12Ni-0.5Ti.

12. The influence of the heat treating temperature, for the 12Ni-0.5Ti steel, on (a) the room temperature hardness and (b) the Charpy-V notch value at -196°C. The heavy bar shown on the abscissa indicates the approximate extent of the three phase region ($\alpha+\gamma+Ni_3Ti$).

α alpha
γ gamma

13. Fracture surfaces of the Charpy bars of the 12Ni-0.5Ti steel heat treated at (a) 800°C (b) 700°C, and broken at -196°C. The specimen heat treated in the three phase region ($\alpha+\gamma+Ni_3Ti$) exhibits a higher degree of plastic deformation and a much greater toughness (140 ft. lbs.) as opposed to the one heat treated in the single phase (γ) region (30 ft. lbs.).

α alpha
γ gamma

γ gamma

14. A comparative plot of the yield strength and the toughness, both measured at -196°C, for two commercial cryogenic steels (data from literature [34]) and the experimental 12Ni-0.5Ti steel.

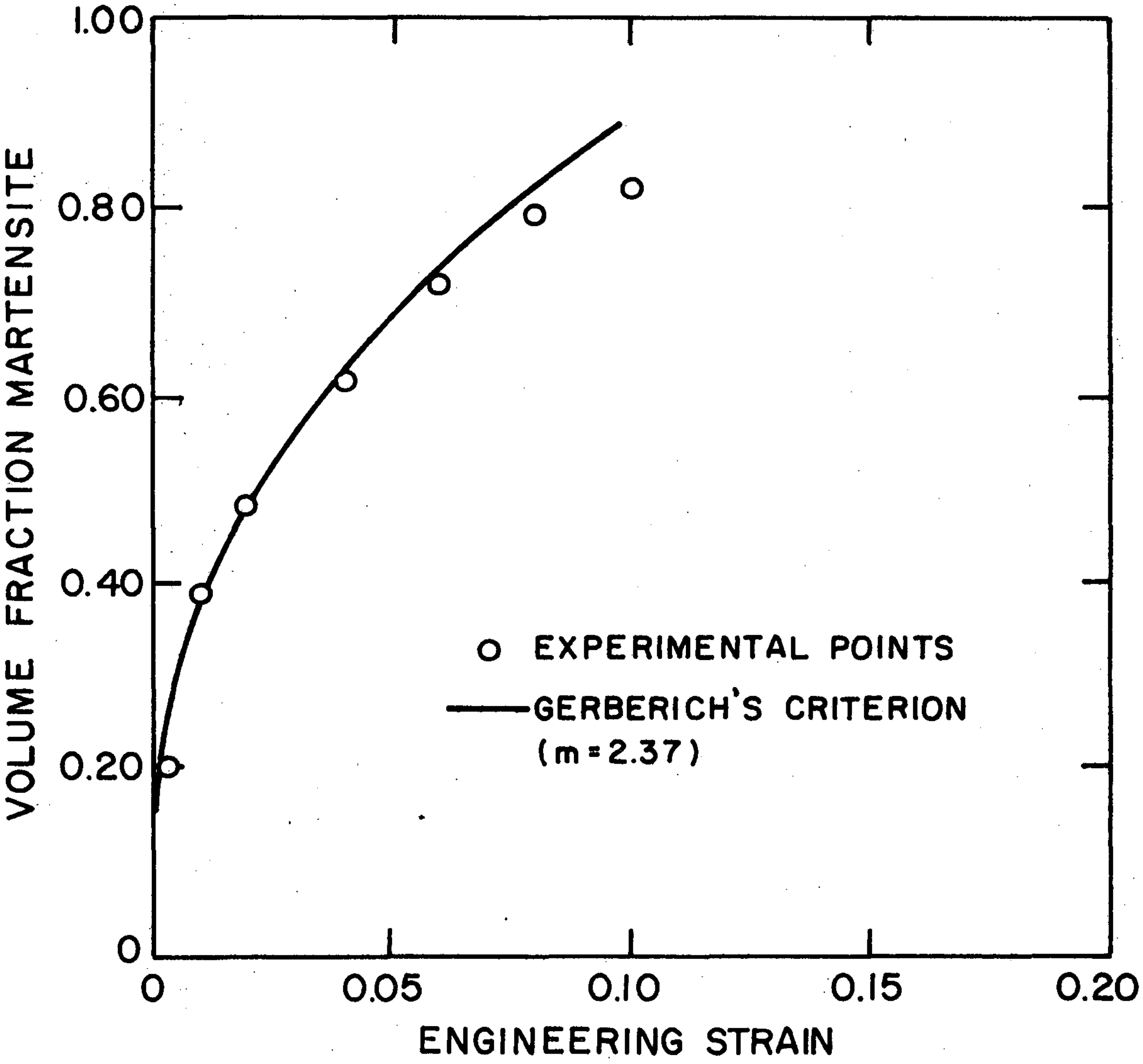

1.00
0.80
0.60
0.40
0.20
0
0
0.05
0.10
0.15
0.20
VOLUME FRACTION MARTENSITE
ENGINEERING STRAIN
○ EXPERIMENTAL POINTS
GERBERICH'S CRITERION
(m = 2.37)

Fig. 1

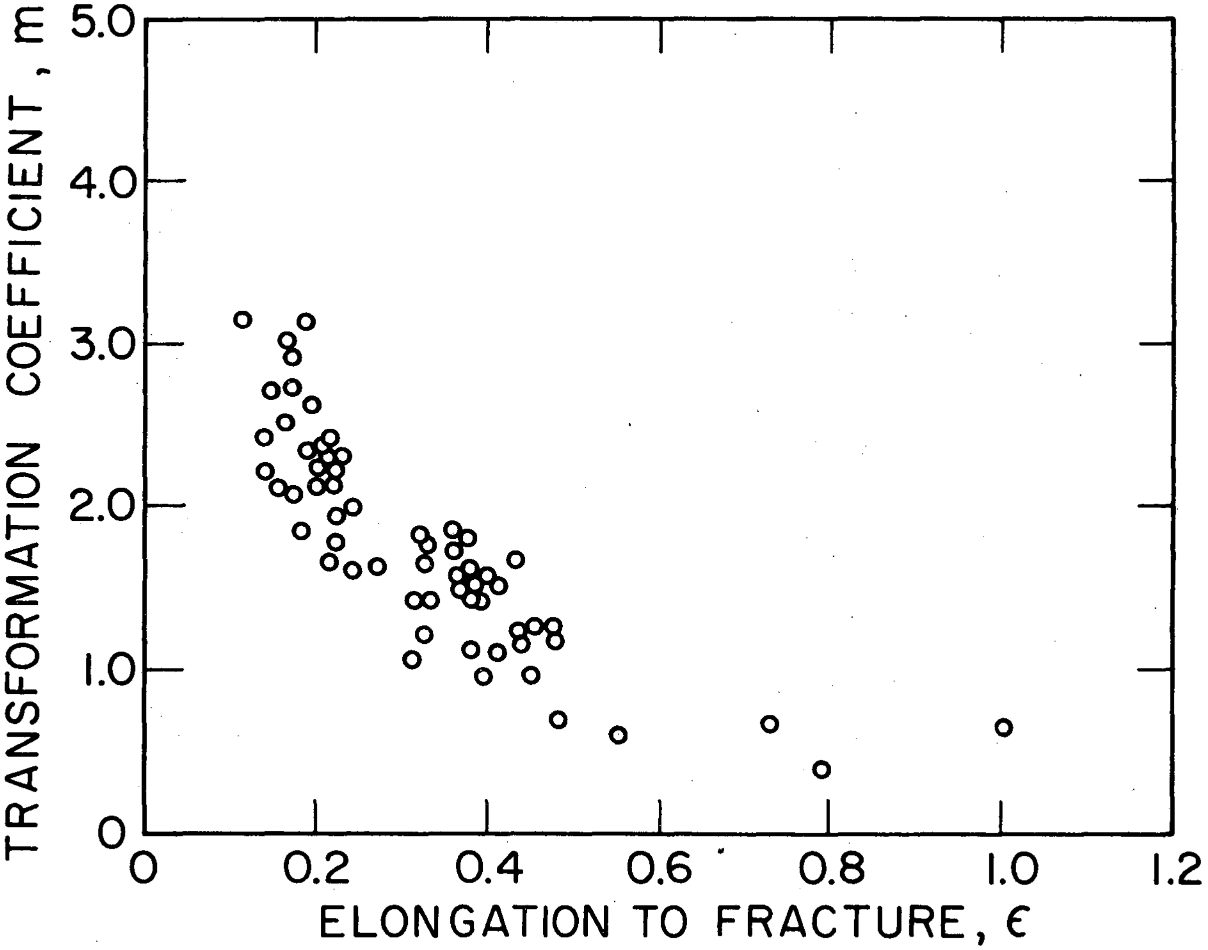
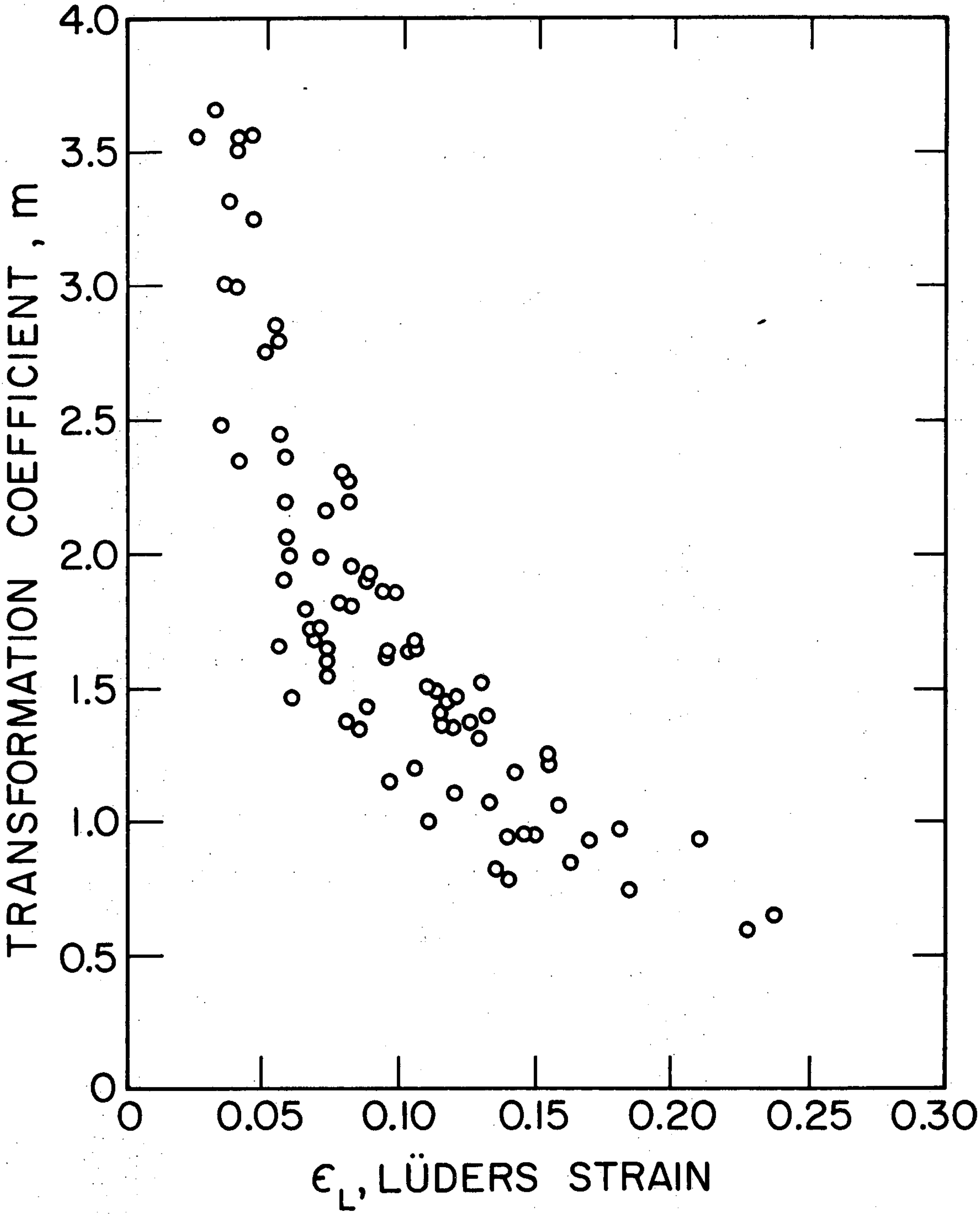

TRANSFORMATION COEFFICIENT, m
5.0
4.0
3.0
2.0
1.0
0
0
0.2
0.4
0.6
0.8
1.0
1.2
ELONGATION TO FRACTURE, ϵ

Fig. 2(a)



TRANSFORMATION COEFFICIENT, m
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
0
0.05
0.10
0.15
0.20
0.25
0.30
ϵ_L, LÜDERS STRAIN

Fig. 2(b)

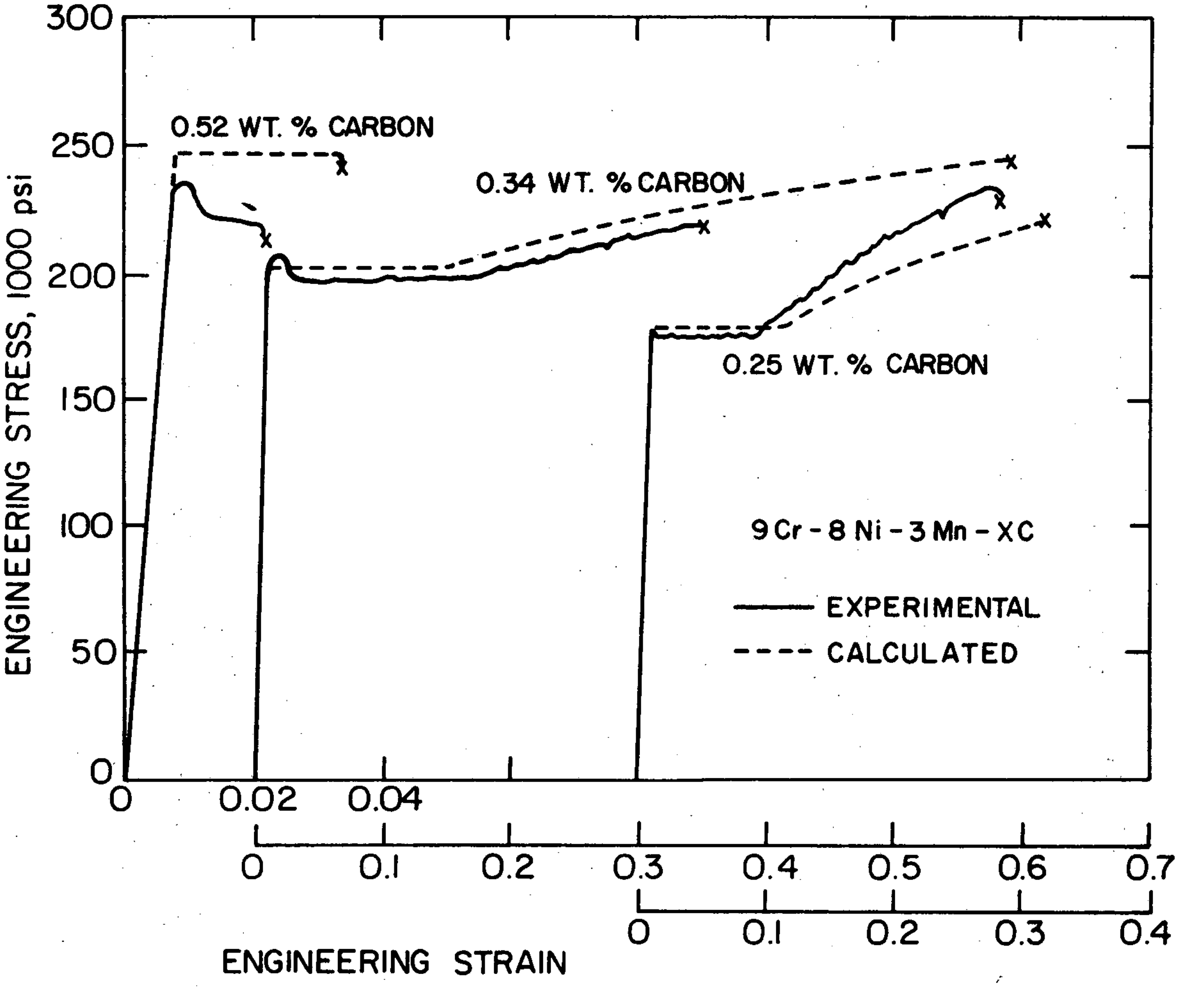

300
250
200
150
100
50
0
ENGINEERING STRESS, 1000 psi
0.52 WT. % CARBON
0.34 WT. % CARBON
0.25 WT. % CARBON
9 Cr - 8 Ni - 3 Mn - XC
EXPERIMENTAL
CALCULATED
0
0.02
0.04
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0
0.1
0.2
0.3
0.4
ENGINEERING STRAIN

Fig. 3

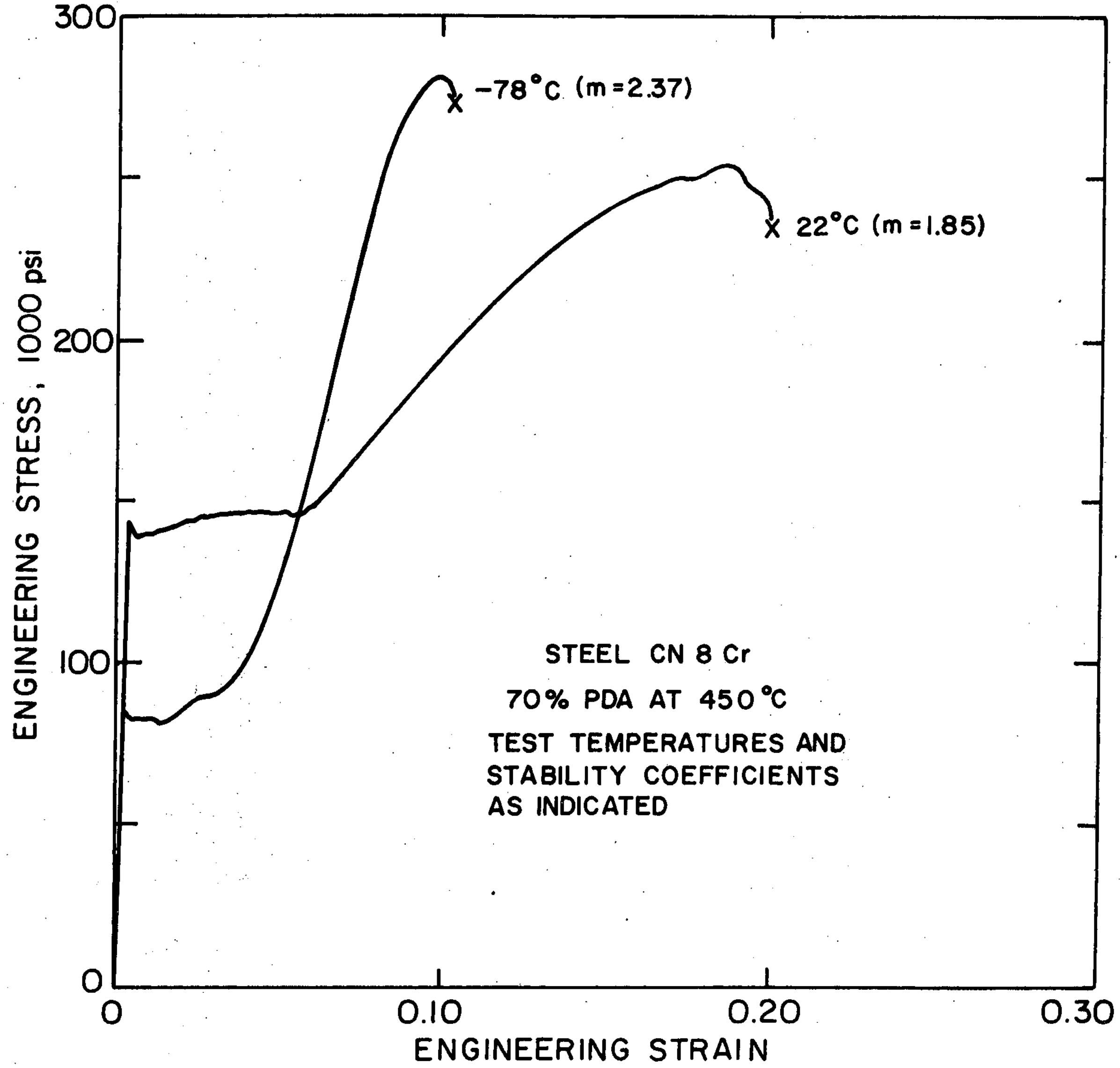

300
200
100
0
0
0.10
0.20
0.30
ENGINEERING STRESS, 1000 psi
ENGINEERING STRAIN
−78°C (m=2.37)
22°C (m=1.85)
STEEL CN 8 Cr
70% PDA AT 450°C
TEST TEMPERATURES AND
STABILITY COEFFICIENTS
AS INDICATED

Fig. 4

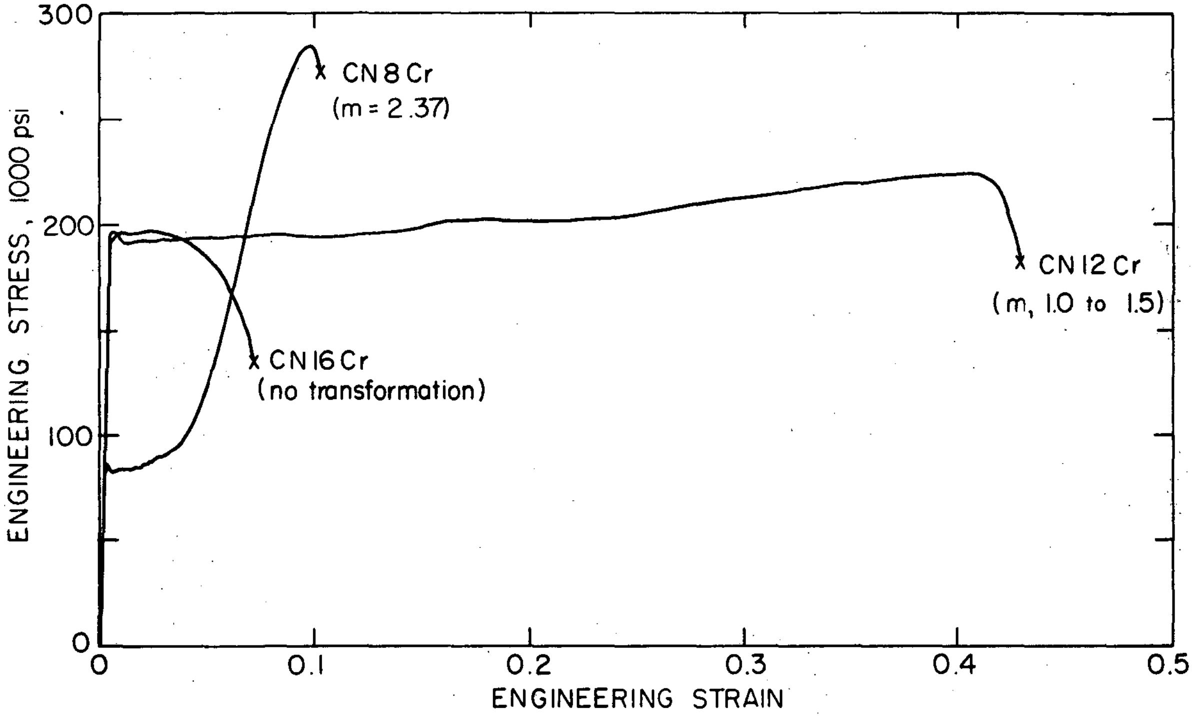

300
200
100
0
0
0.1
0.2
0.3
0.4
0.5
ENGINEERING STRESS, 1000 psi
ENGINEERING STRAIN
CN 8 Cr
(m = 2.37)
CN 12 Cr
(m, 1.0 to 1.5)
CN 16 Cr
(no transformation)

Fig. 5

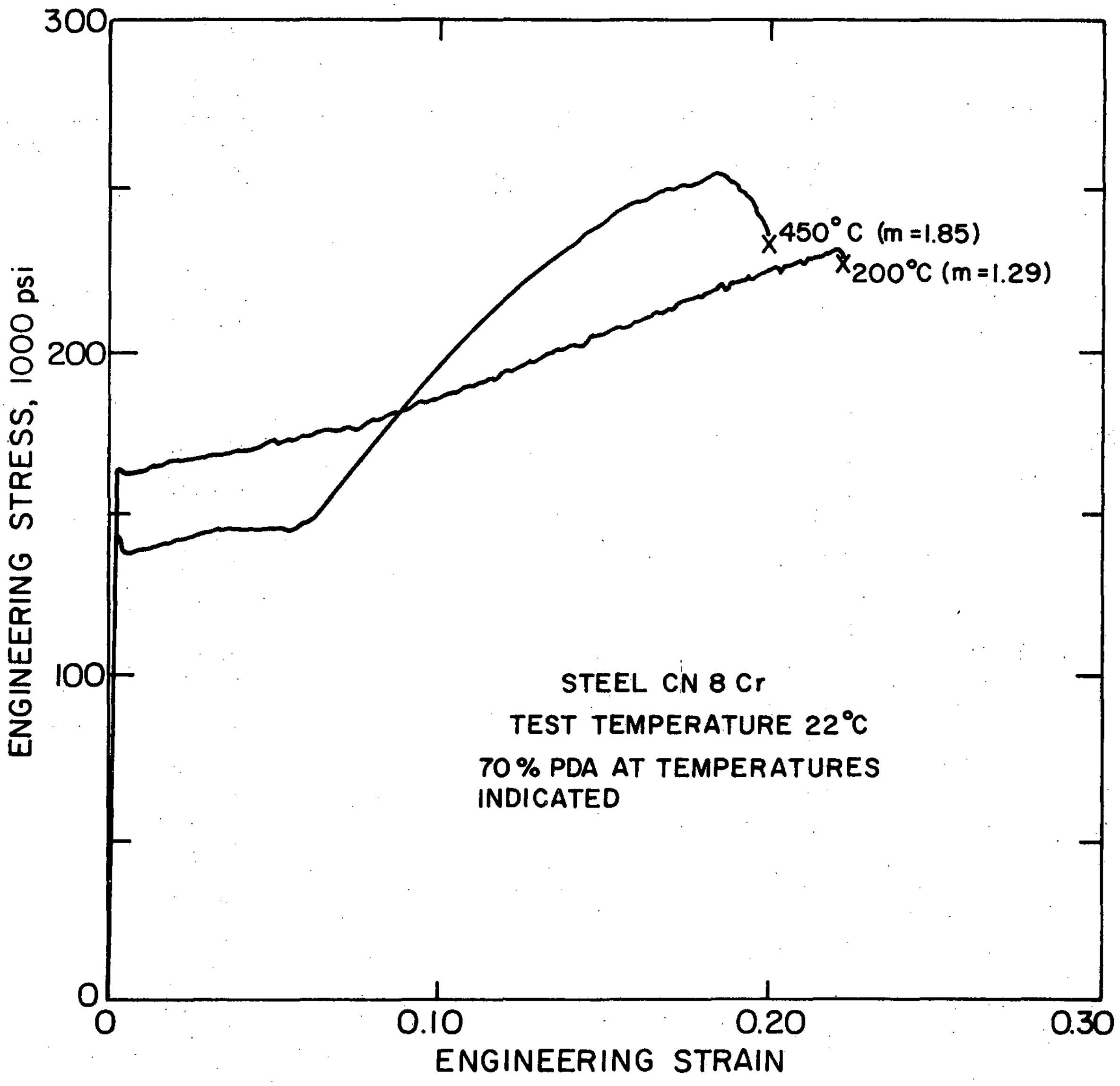
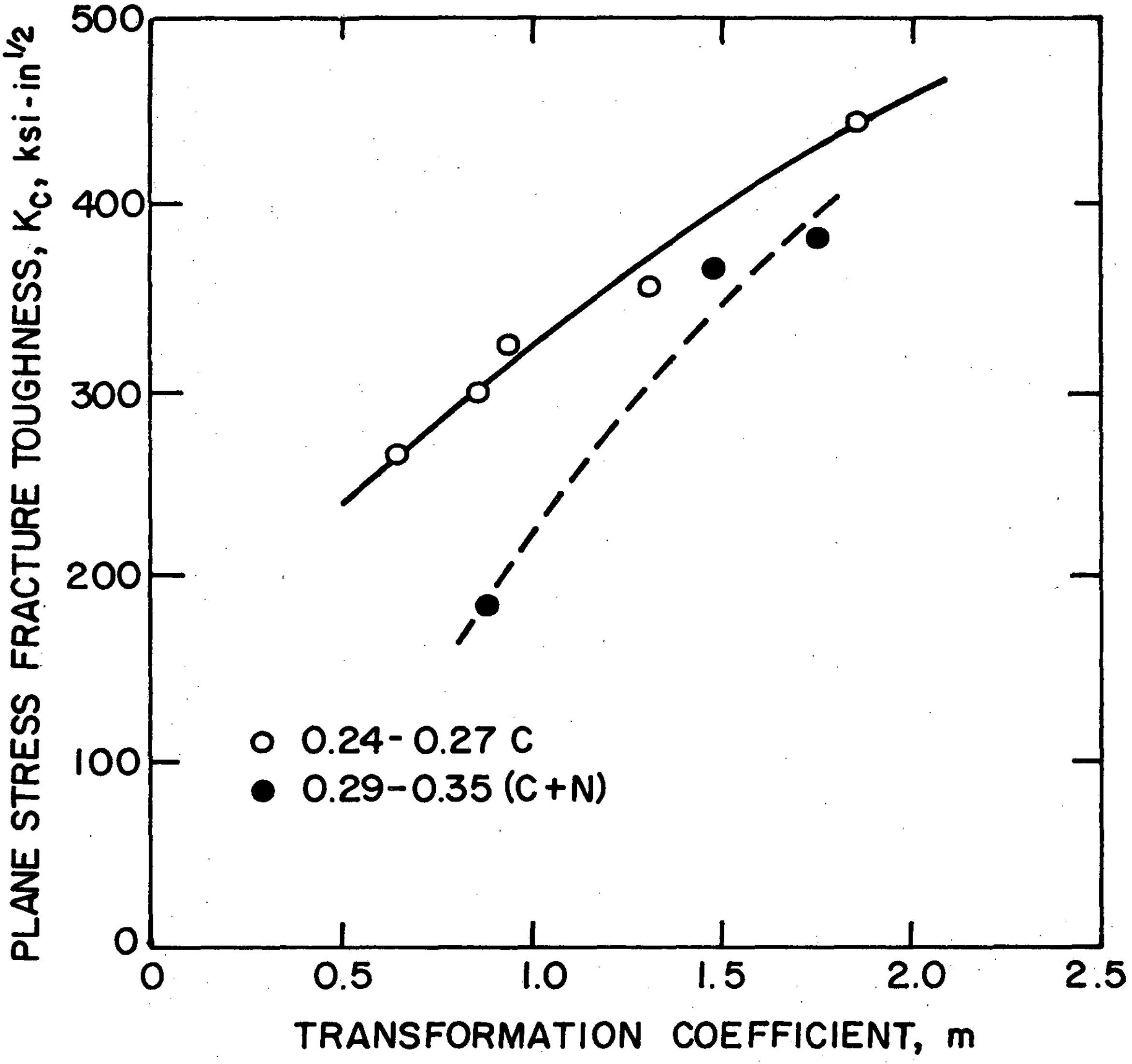

300
200
100
0
0
0.10
0.20
0.30
ENGINEERING STRESS, 1000 psi
ENGINEERING STRAIN
450° C (m = 1.85)
200°C (m = 1.29)
STEEL CN 8 Cr
TEST TEMPERATURE 22°C
70% PDA AT TEMPERATURES INDICATED

Fig. 6



PLANE STRESS FRACTURE TOUGHNESS, K_c, ksi-in$^{1/2}$
500
400
300
200
100
0
0
0.5
1.0
1.5
2.0
2.5
TRANSFORMATION COEFFICIENT, m
○ 0.24-0.27 C
● 0.29-0.35 (C+N)

Fig. 7

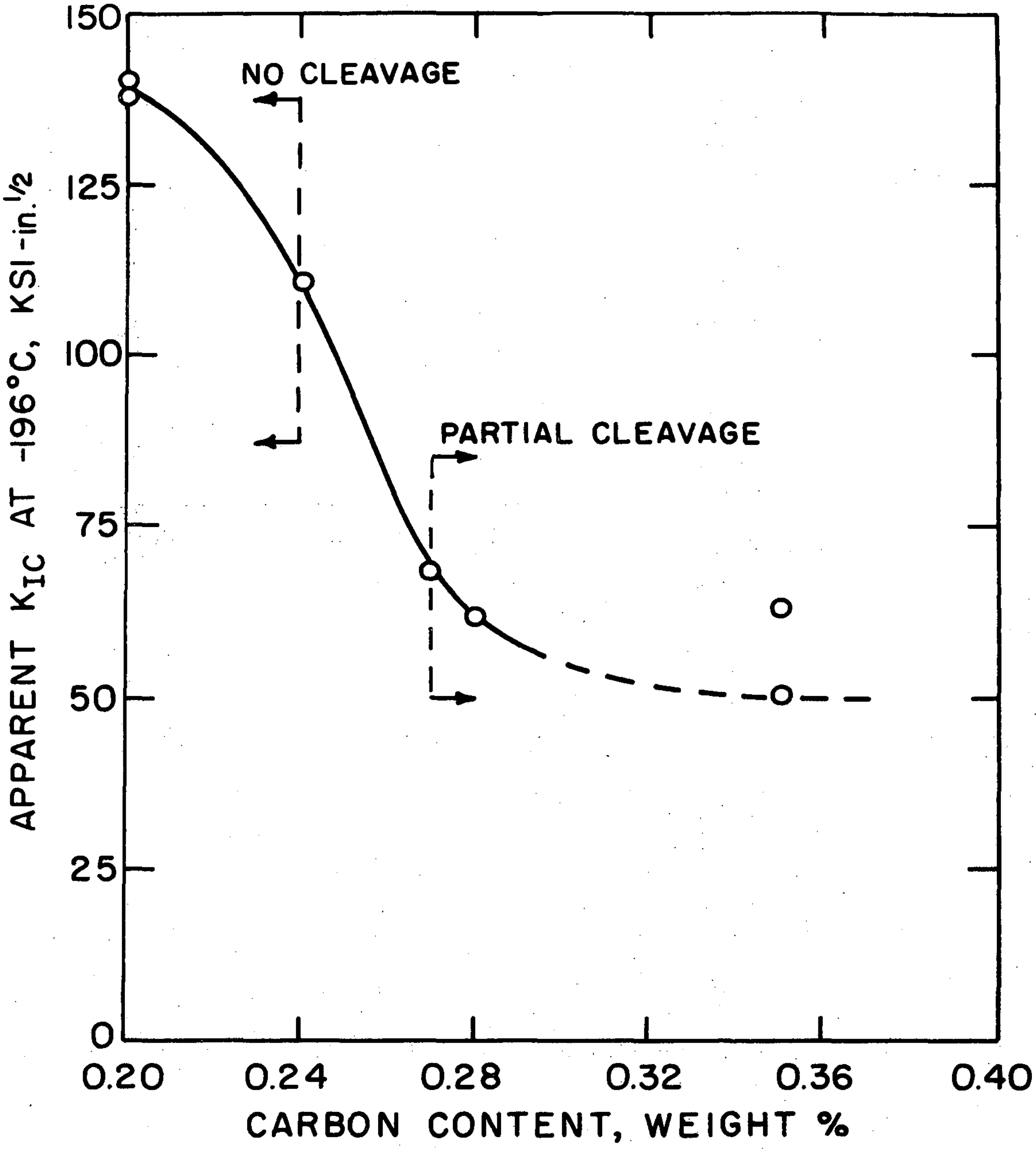

APPARENT K_{IC} AT -196°C, KSI-in.$^{1/2}$
150
125
100
75
50
25
0
NO CLEAVAGE
PARTIAL CLEAVAGE
0.20
0.24
0.28
0.32
0.36
0.40
CARBON CONTENT, WEIGHT %

Fig. 8

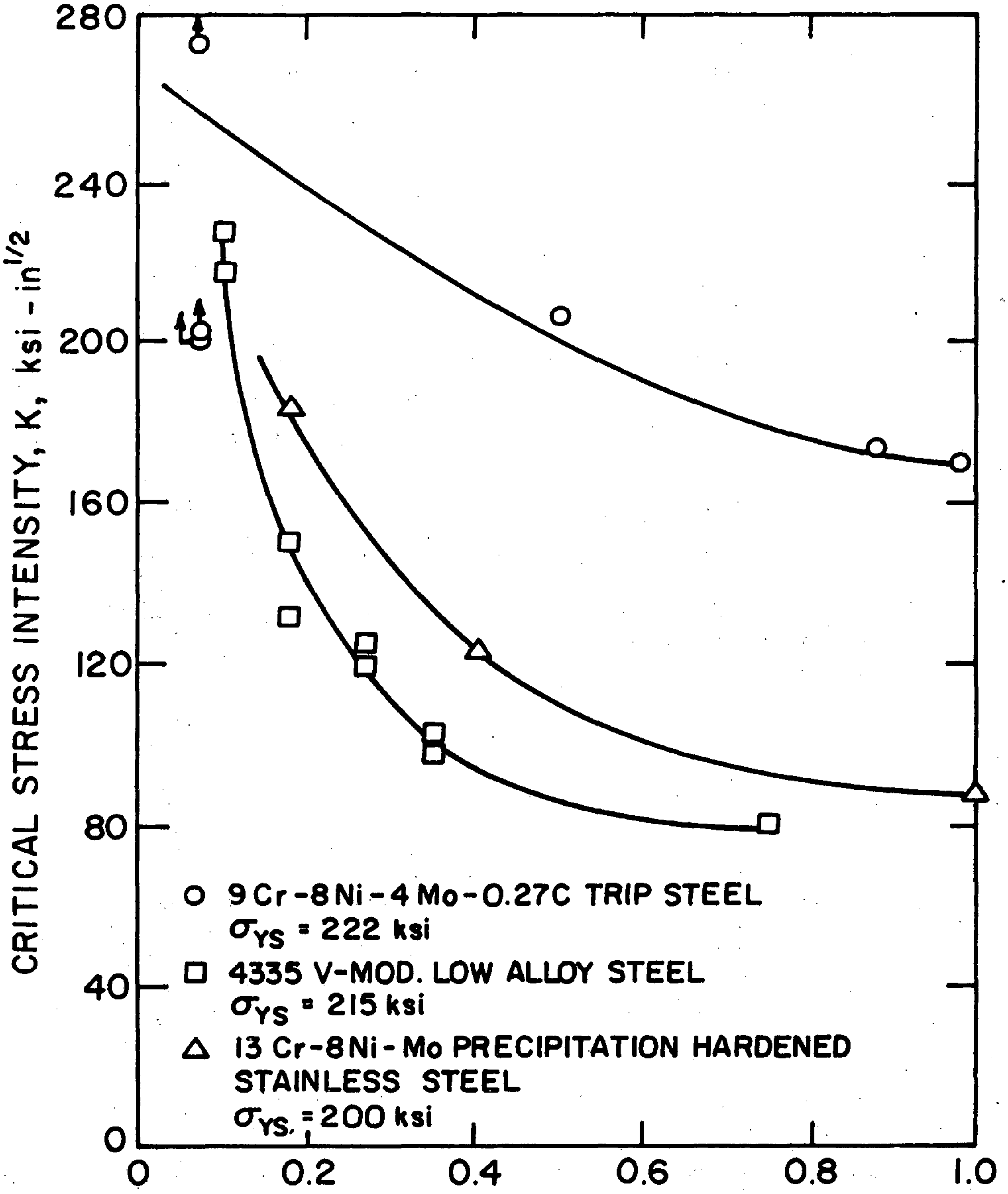

280
240
200
160
120
80
40
0
CRITICAL STRESS INTENSITY, K, ksi - in$^{1/2}$
0
0.2
0.4
0.6
0.8
1.0
THICKNESS, inches
○ 9 Cr-8 Ni-4 Mo-0.27C TRIP STEEL
σ_{YS} = 222 ksi
□ 4335 V-MOD. LOW ALLOY STEEL
σ_{YS} = 215 ksi
△ 13 Cr-8 Ni-Mo PRECIPITATION HARDENED STAINLESS STEEL
σ_{YS} = 200 ksi

Fig. 9

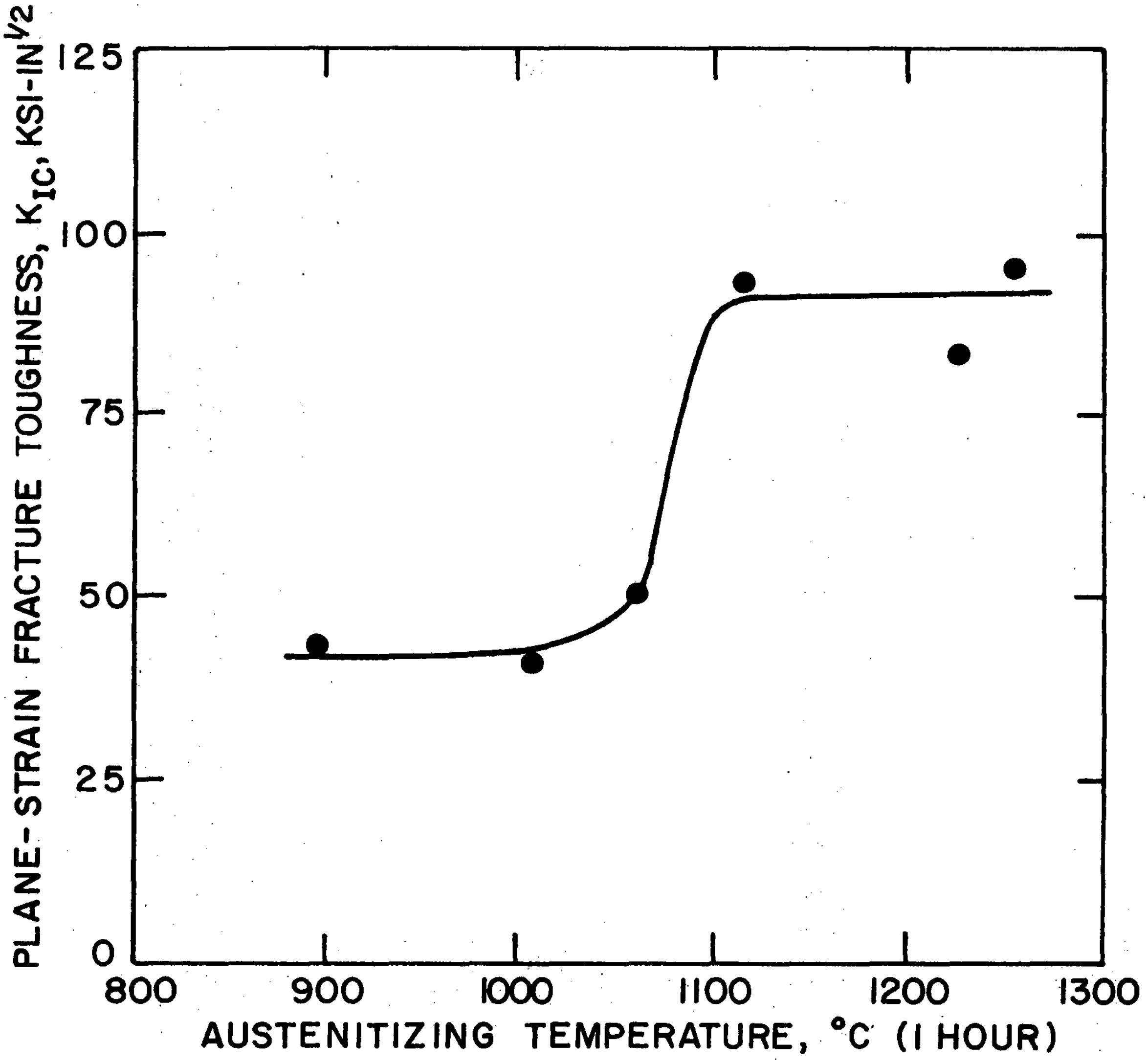

PLANE-STRAIN FRACTURE TOUGHNESS, K_{IC}, KSI-IN$^{1/2}$
125
100
75
50
25
0
800
900
1000
1100
1200
1300
AUSTENITIZING TEMPERATURE, °C (1 HOUR)

Fig. 10

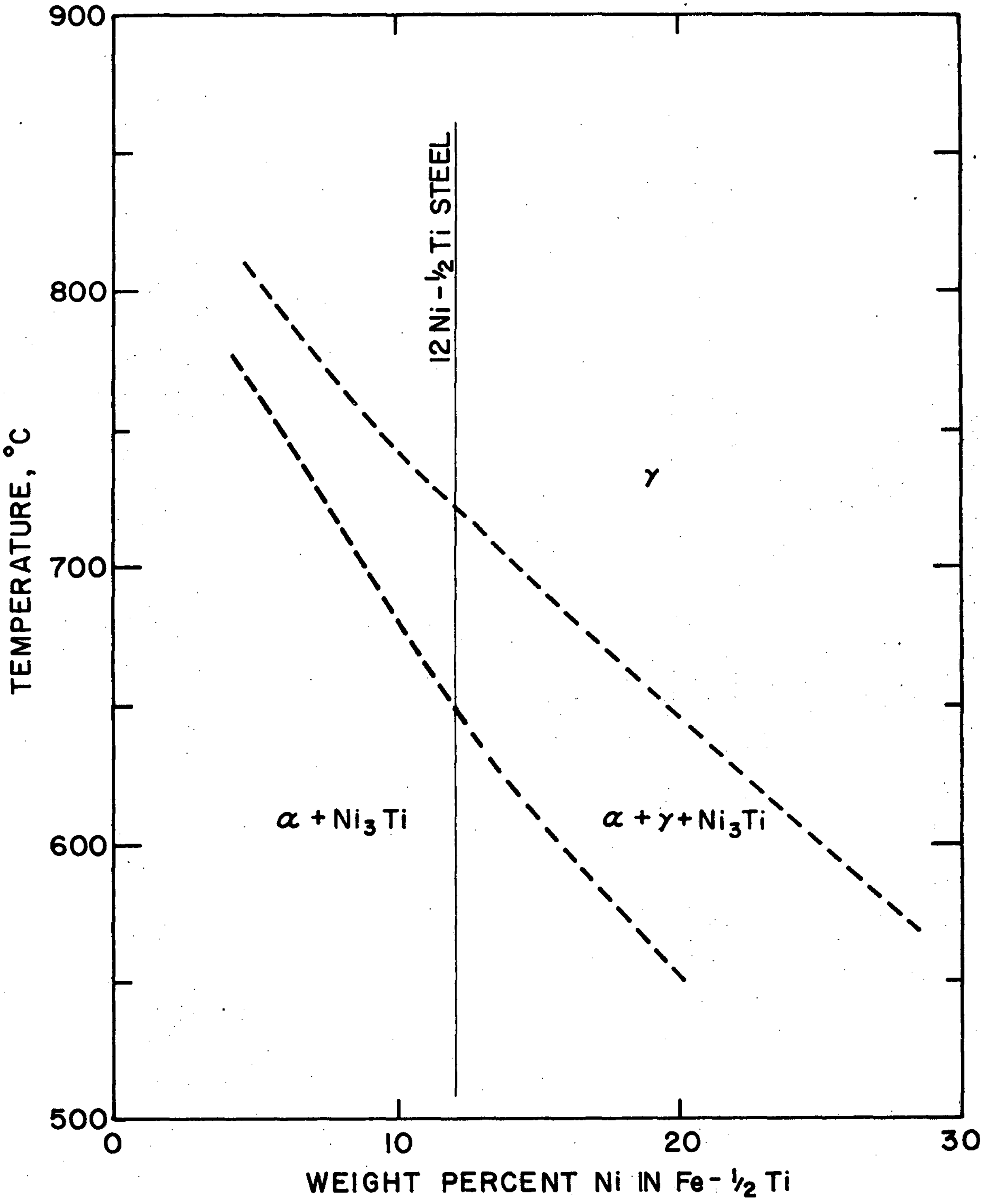

900
800
700
600
500
TEMPERATURE, °C
0
10
20
30
WEIGHT PERCENT Ni IN Fe-½ Ti
12 Ni-½ Ti STEEL
γ
α + Ni₃Ti
α + γ + Ni₃Ti

Fig. 11

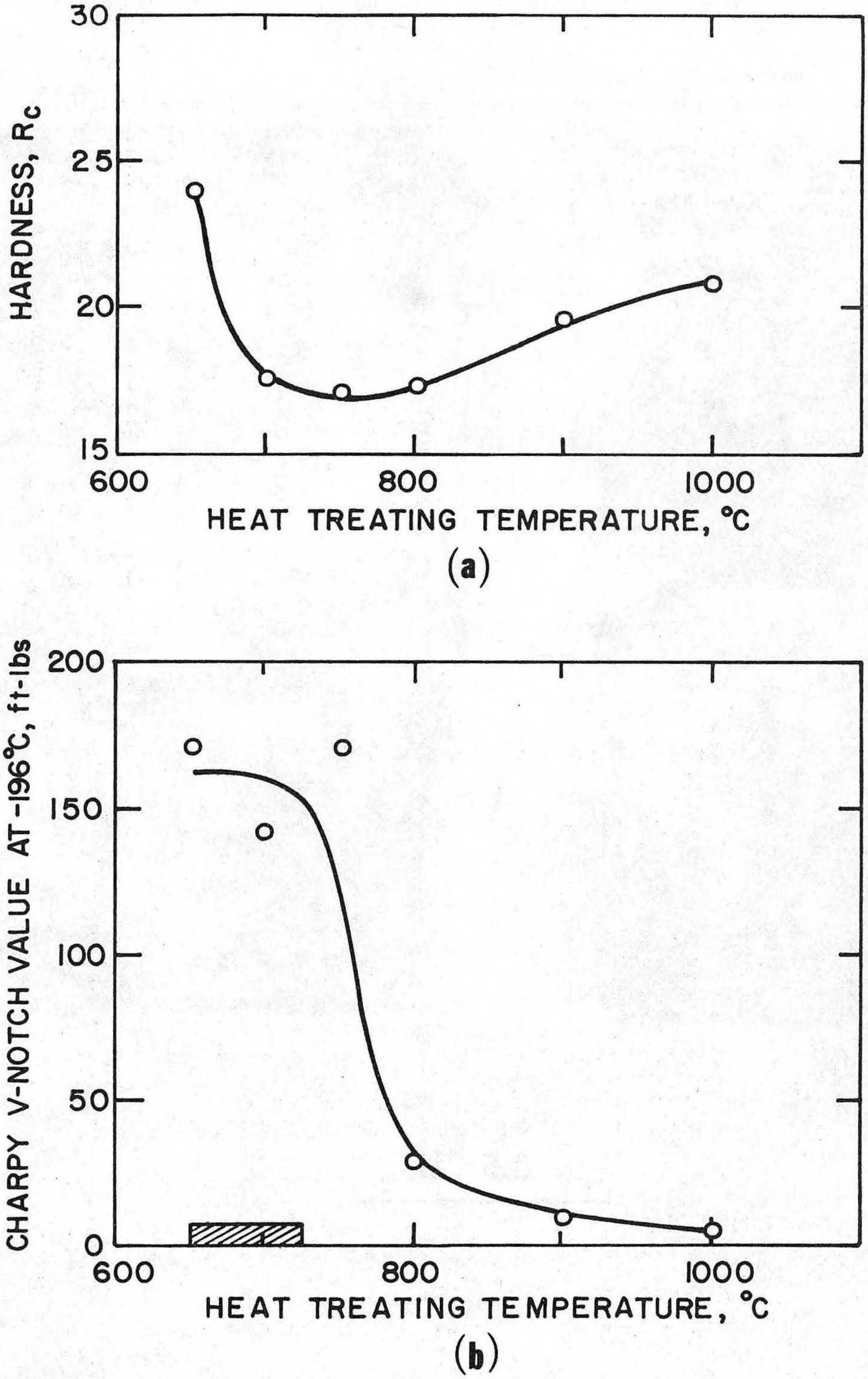

30
25
20
15
HARDNESS, R_c
600
800
1000
HEAT TREATING TEMPERATURE, °C
(a)
200
150
100
50
0
CHARPY V-NOTCH VALUE AT -196°C, ft-lbs
600
800
1000
HEAT TREATING TEMPERATURE, °C
(b)

Fig. 12

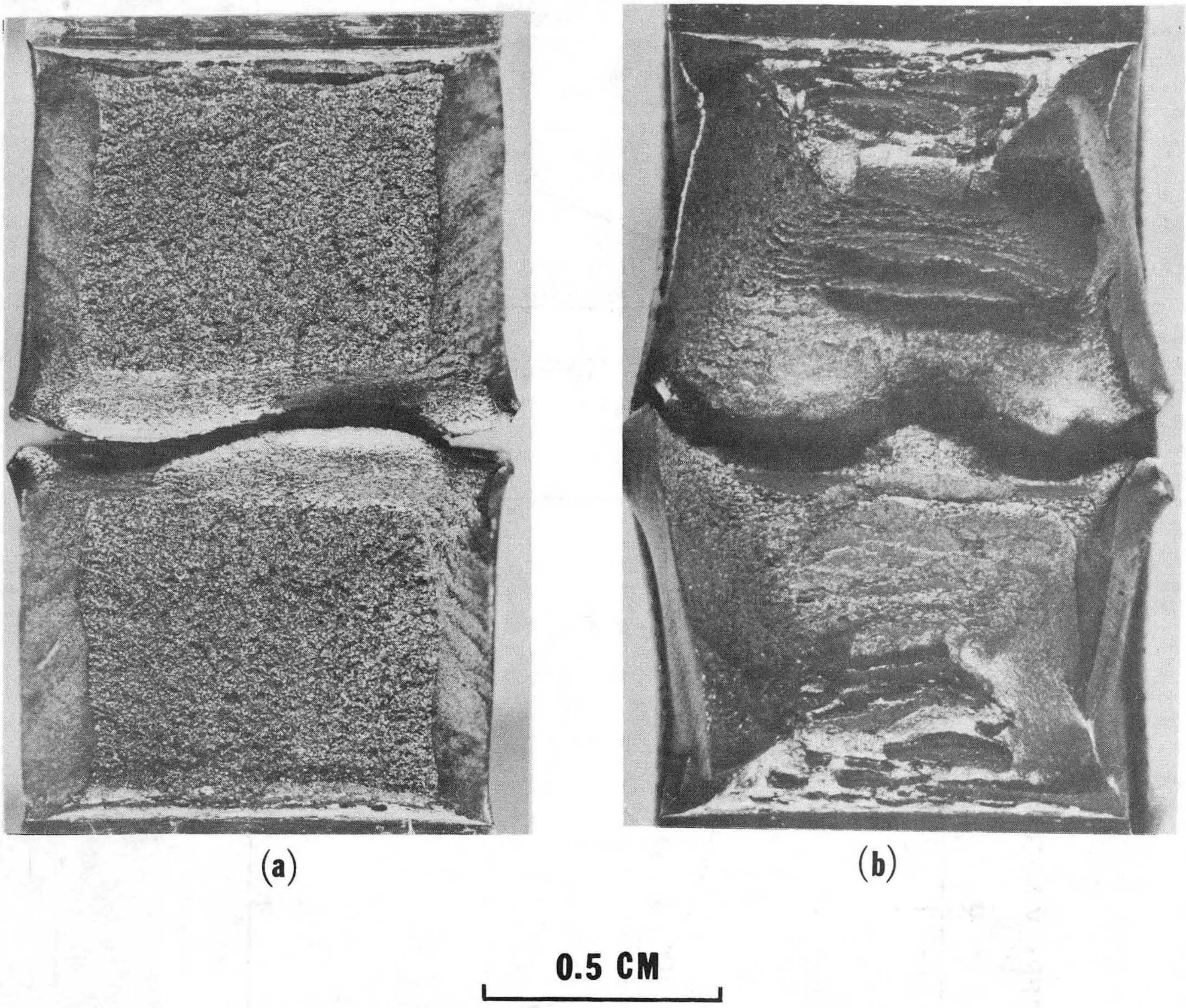


Fig. 13

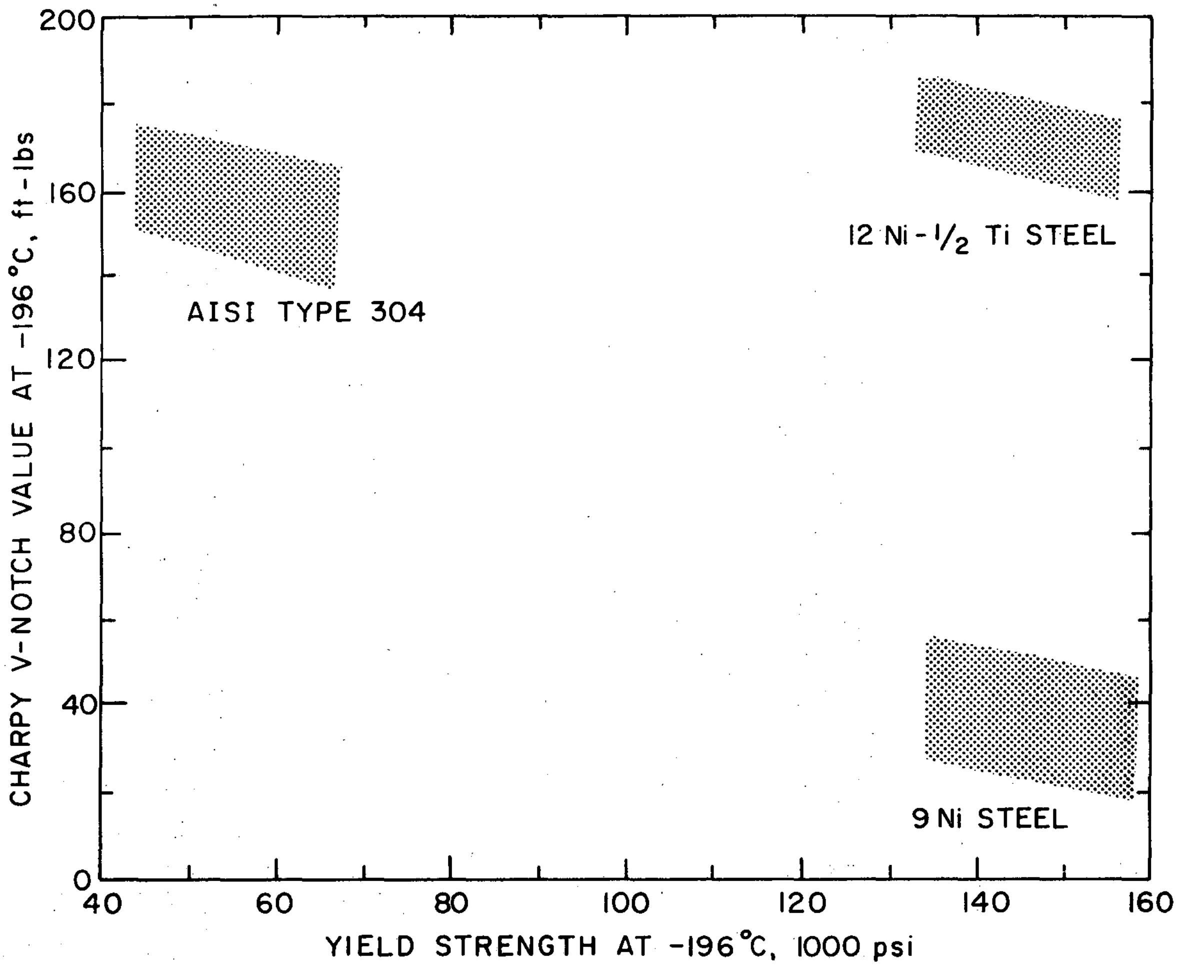

200
160
120
80
40
0
40
60
80
100
120
140
160
CHARPY V-NOTCH VALUE AT -196°C, ft-lbs
YIELD STRENGTH AT -196°C, 1000 psi
AISI TYPE 304
12 Ni-1/2 Ti STEEL
9 Ni STEEL

Fig. 14

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