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Fe-Mn ALLOYS FOR CRYOGENIC USE: A BRIEF SURVEY OF CURRENT RESEARCH

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ABSTRACT

The desire to minimize the nickel content of steels intended for service in cryogenic systems had led to research on the applicability of iron manganese alloys at low temperatures. In this paper the relevant physical metallurgy of the Fe-Mn system is reviewed and cryogenic alloy development activities are briefly discussed, drawing on recent and current research in the USSR, the US, and Japan. The Fe-Mn alloys of interest may be divided into three general classes depending on their Mn content. Alloys of 12%Mn or below have principally a ferritic structure with other phases (austenite or ε-martensite) occasionally present depending on the Mn concentration and the concentration of other alloying species. These alloys have good cryogenic strength but poor toughness due to the intrusion of catastrophic intergranular fracture. Current research emphasizes the use of metallurgical treatments to overcome this intergranular fracture. Alloys of intermediate Mn content (14~27%) have a mixed austenite-martensite structure. The residual austenite in these steels has low stability resulting in deformation-induced transformation at low temperature. As a consequence, the alloys usually exhibit good cryogenic strength but may be adapted for applications to which cryogenic strength is not critical. Alloys of 28~36%Mn have a stable austenitic structure; martensitic transformations occur only at high deformation levels or at very low temperatures. These stable austenitic Fe-Mn alloys are, however, highly alloyed, with the result that some of their advantage with respect to Fe-Ni cryogenic alloys is lost. When Mn content exceeds approximately 40%, manganese-rich phases intrude, causing a deterioration of low temperature toughness.

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I. INTRODUCTION

The steels now commonly specified for structural applications at LNG and lower temperatures, 9%Ni steel, austenitic stainless steels, and invar alloys, have in common a relatively high content of nickel. While the nickel alloy addition contributes significantly to the good low temperature properties of these alloys, it also adds substantially to the cost. There is, consequently, an incentive to develop techniques for reducing or eliminating the nickel content of cryogenic steels while retaining good cryogenic properties. 5-6%Ni steels were recently introduced in the U.S. (1) and in Japan (2) in response to this need. Further decreases in the acceptable nickel content would be desirable.

Of the common alloying elements in steel manganese is the most obviously attractive as a substitute for nickel in cryogenic alloys. Manganese is readily available, relatively inexpensive, and has an intriguing metallurgical similarity to nickel in its effect on the microstructures and phase relationships of iron-based alloys. A number of laboratories have conseqently become interested in the potential of Fe-Mn alloys for cryogenic use. While research on Fe-Mn alloys has not yet led to industrial application in cryogenic service the laboratory results are promising. They show that the Fe-Mn system can be used as a basis for alloys having useful mechanical properties to LNG temperature and below. With further development these alloys may offer an economically attractive alternative to the nickel alloy steels for structural use in cryogenic systems.

In the following, the physical metallurgy of the Fe-Mn system is briefly reviewed, with emphasis on those differences from the Fe-Ni system which impede the development of Fe-Mn cryogenic steels. Several recent and on-going alloy development projects are then briefly described, with examples taken from work in the Soviet Union, the United States, and Japan.

II. METALLURGICAL CONSIDERATION

A. Properties of Fe-Mn Binary Alloys:

The base mechanical properties of Fe-Mn binary alloys in the as-cooled condition are illustrated in Figures 1 (3) and 2 (4), and in Table I. (4) On the basis of phase content and properties Fe-Mn alloys containing up to 46% (by weight) of manganese can be divided into four ranges by composition:

1. Alloys containing less than 10%Mn show transformation behavior similar to that of the Fe-Ni alloys. (5-8) For Mn contents of 8-10% the as-cooled structure is a dislocated lath martensite. Both the yield and tensile strengths of the alloys increase with Mn concentration. The ductile-brittle transition temperature, however, is high with the consequence that these alloys are unsuited for cryogenic service in the as-cooled conditions. As in the case of the Fe-Ni alloys, a tempering treatment in the two-phase (α+γ) region causes a decrease in the ductile-brittle transition temperature of ferritic Fe-Mn alloys. (9-13) This beneficial tempering response has been used to improve the toughness of 4Mn alloys for use near toom temperature (13) but is not sufficient to eliminate brittleness at LNG or liquid nitrogen temperatures.

2. At approximately 10% manganese the hexagonal ε-martensite is found along with α-martensite in the as-cooled structures. (14-18) Alloys having 10-14%Mn are predominantly mixtures of α and ε-martensite after cooling to room temperature with some admixture of untransformed γ (austenite) for 12-14%Mn. The ε-martensite phase is metastable and

tranforms readily to α on deformation at room temperature or below. Alloy yield strength drops dramatically as the volume fraction of ε increases for Mn content >12% (Figure 1(b)). On the other hand, the tensile strength remains high, presumably reflecting the ε→α transformation. The behavior of alloys having 12%Mn or less is dominated by the α phase and low temperature toughness may be improved by tempering treatments. For Mn contents >12% the ε-phase begins to dominate alloy behavior, causing a rapid deterioration in yield strength at both room and cryogenic temperatures.

3. At about 14%Mn the martensite fraction in the as-cooled structure becomes small. Over the range 14-27%Mn the alloy is essentially a mixture of ε-martensite and untransformed austenite, with the austenite fraction and stability increasing as the Mn content becomes higher. The room temperature yield strength remains roughly constant over this composition range. Strength at 77°K increases slightly, presumably because of the increasing stability of the austenite phase. The ductile-brittle transition temperature decreases with increasing Mn (Figure 1(c), Figure 2).

The interpretation of the behavior of Fe-Mn binary alloys of 10-27%Mn requires an understanding of the behavior of the ε-martensite phase. Despite informative recent research, particularly by Lysak and co-workers,(19) the crystallography, formation kinetics, and mechanical behavior of the ε-martensite phase are not fully resolved. The appearance of ε-martensite is associated with a low stacking fault energy of the austenite. Recent measurements (20-21) (Figure 3) suggest that the stacking fault energy of austenitic Fe-Mn reaches a minimum at ~22%Mn. A rapid increase in austenite stability is experimentally observed at higher Mn contents. The ε phase is mechanically unstable with respect to deformation at low temperature and transforms to α martensite. The direct transformation path $\varepsilon \to \alpha$ is generally assumed,(18, 22-24) though at least one piece of recent research suggests the reversion path $\varepsilon \to \gamma \to \alpha$.(25) The ε-phase appears to have a uniformly deleterious effect on cryogenic toughness of austenitic alloys; both strength and toughness increase as the austenite is stabilized by increasing Mn content.

4. Fe-Mn alloys of 28-46%Mn are wholly austenitic. Steels containing 26-36%Mn retain good toughness at cryogenic temperatures to at least 20°K.(4) They do, however, have the relatively low strength characteristic of austenitic steels. For higher Mn contents the cryogenic toughness deteriorates (Figure 2) while the strength remains roughly constant. The metallurgical source of this behavior is not entirely clear.

B. Alloying Effects:

The principal alloy additions considered for use in Fe-Mn cryogenic alloys are Ni, Cr, C, and N.

Nickel alloy additions have a beneficial effect on both the strength and toughness of Fe-Mn austenitic alloys. The improvement appears to be associated with the stabilization of austenite (Figure 4) (26-27) with respect to formation of the ε-phase. In practice, of course, the Ni alloy addition must be small or the economic incentive to shift to Fe-Mn base alloys is lost.

Chromium is typically added to Fe-Mn austenitic alloys to improve corrosion resistance. A recent survey by Kato, et al (28) suggest that its influence on the phases appearing in the as-cooled Fe-Mn structure is small for Cr contents <13%, while for higher chromium contents δ-ferrite is retained in the as-cooled structure (Figure 5). The σ-phase is also found at high Mn, Cr levels. Chromium is found to have a beneficial effect on cryogenic strength and toughness, particularly in alloys containing carbon.(29) This improvement is attributed to an increase in the stability of austenite, reflected in a decrease in the fraction of ε-phase in the as-cooled structure, though the interpretation is tentative.

Carbon is a γ-stabilizer. At low concentration (<0.7%) carbon stabilizes the γ structure with respect to formation of the ε-phase. At concentrations >0.7% the formation of ε is suppressed(30) (Figure 6). Interestingly carbon promotes the retention of austenite in predominantly martensitic Fe-Mn alloys, but does not significantly affect the Mn concentration (~10%) at which the ε-phase intrudes. A moderately high yield strength can be obtained in a medium carbon, high manganese ternary austenite. However, these alloys encounter phase stability problems at cryogenic temperatures. Austenitic Fe-Mn-C alloys do not retain satisfactory impact resistance at cryogenic temperatures unless the austenite stability is increased by adding Cr or Ni as alloying elements.(29)

Nitrogen adds significantly to the strength of Fe-Mn-Cr-Ni austenitic alloys (30) but has a less significant influence on the cryogenic strength of Fe-25~36Mn austenites. (4) The nitrogen appears to increase austenitic stability but causes a deterioration in low temperature toughness. The embrittling effect may be due to nitrogen segregation to grain boundaries.

Other alloying elements which have received research attention include silicon, niobium, titanium, and vanadium. Yushchenko and co-workers(4) found apparently significant strengthening on introduction of Si into Fe-25~42%Mn with no evident loss of tensile ductility at cryogenic temperatures. Yoshmura, et al (32) studied the influence of Nb, Ti, and V on the strength and toughness of Fe-25Mn-5Cr-1Ni. Each of these alloy additions increases strength and reduces impact toughness properties. The best result was obtained with addition of ~0.11Nb (Table IV). The strengthening effect of Nb seemed to be principally due to its influence on austenite grain size. Chang (33) studied the aging response of austenitic Fe-20~28Mn-Ti alloys. He found that these alloys age-harden significantly through formation of $Fe_2(Ti, Mn)$ precipitates. However, the precipitation is strongly catalyzed at grain boundaries with the consequence that the aged alloys are brittle unless prior deformation is used to provide intragranular nucleation sites.

III. RECENT AND CURRENT RESEARCH ON Fe-Mn ALLOYS FOR CRYOGENIC USE

Several of the key problems facing the development of industrially practical Fe-Mn alloys for cryogenic use are evident from the physical metallurgy of the Fe-Mn system. The ferritic alloys (≲12%Mn) have excellent cryogenic strength but poor toughness. While the ductile-brittle transition temperature may be lowered by two-phase annealing treatments similar to those used for ferritic Fe-Ni cryogenic steels the transition temperature remains well above 77°K. Further progress requires the development of specific heat treatments or compositional modification to improve low temperature toughness. In the case of austenitic Fe-Mn alloys good low temperature properties seem to require a reasonably stable austenite (γ) phase and a relatively low interstitial content. To stabilize austenite the Mn content should be high which presents problems in steelmaking practice due to the volatility of Mn. Since the cryogenic strength of high Mn alloys is relatively low, strengthening techniques which preserve alloy toughness are desirable.

Recent and current activities in the development of cryogenic Fe-Mn alloys address these problems. Research on ferritic alloys concentrates on eliminating low temperature brittleness while research on austenitic alloys has largely focused on demonstrating the good inherent toughness of these alloys while minimizing Mn content and maximizing strength.

A. Ferritic Fe-Mn Alloys:

Research on ferritic Fe-Mn alloys for cryogenic use is underway at the Lawrence Berkeley Laboratory of the University of California. (9-10) This project addresses the sources of low temperature embrittlement of Fe-(8-12)Mn alloys and seeks metallurgical remedies. The results show a change in the brittle fracture mode as the Mn content increases from 8% to 12%, with intrusion of the ε-phase, from transgranular fracture to intergranular fracture. While these alloys respond favorably to thermal treatments analagous to those used for Fe-Ni cryogenic steels, toughness at 77°K remains low. Fe-12Mn alloys can, however, be made tough at 77°K by a cold work plus tempering treatment which suppresses intergranular fracture (Table II and Figure 7). Recently it has been shown (34) that the intergranular fracture of Fe-12Mn can also be eliminated by controlled cooling through the martensite transformation yielding an alloy with reasonable toughness at 77°K in the as-cooled condition.

B. Austenitic Fe-Mn Alloys:

Extensive research on austenitic Fe-Mn alloys for cryogenic service has been conducted in the Soviet Union. Active current programs exist at the E. O. Paton Institute for Electrowelding in Kiev and at the Metallurgical Institute of the Georgian Academy of Sciences in Tblisi, with supporting fundamental research projects underway in several laboratories. While a variety of alloy compositions are under investigation (for example, Table I) alloys of major current interest include 03X13AI19, of composition 19Mn-13Cr-0.03C-0.02N, for use at temperatures of 111°K and higher, and a nickel-modified version, 03X13H5AI19, containing 5%Ni, intended for use to 20°K. (4) The former alloy was included in the US-USSR exchange program on the properties of materials for LNG, and is hence one of the most thoroughly characterized (35-39) cryogenic alloys insofar as its mechanical properties are concerned. Typical tensile properties are presented in Table III, and fracture toughness data is plotted in Figure 8 in comparison to that of commercially processed 5% and 9%Ni steels. (39) The toughness of this alloy is competitive with that of the Fe-Ni alloys over the full range of test temperatures and is superior at 4°K. The yield strength is, however, below that of the ferritic alloys.

Austenitic Fe-Mn alloys have also attracted recent interest in Japan. An extensive alloy development project was recently completed at the Process Technology Laboratories of the Nippon Steel Corporation with the objective of combining good cryogenic mechanical properties with a low coefficient of thermal expansion. After a survey of Mn-Cr compositions Yoshimura, et al (32) selected the stable austenitic composition Fe-25Mn-5Cr-1Ni. This composition yielded good notch toughness at 77°K (Table IV) together with a low thermal expansion coefficient ($6.7\text{-}7.2\times10^{-6}/°K$). In an attempt to increase cryogenic strength without sacrificing toughness they studied the influence of small additions of niobium, vanadium, and titanium. Alloying with niobium (0.11%) appeared particularly promising in that cryogenic strength increases substantially (Table IV) while reasonable toughness is retained. The authors concluded that the principal source of the Nb effect was its influence in refining the grain size of the austenite.

Austenitic Fe-Mn alloys are also under study at the Central Research Laboratories of the Daido Steel Company in a project whose declared objective is the development of castable alloys for cryogenic valves. (40) Basic research portions of this project have been published. (28) The results published to date suggest that Mn content should be high to ensure low temperature toughness, and that the Cr content should be high to stabilize austenite and improved ancillary properties, but not so high (~13%Cr) that δ-ferrite is retained. Alloys of composition near 25Mn-10Cr are currently under investigation.

C. Mn-Modified Weld Wire for Fe-Ni Cryogenic Steels:

A final area of active research interest in the substitution of Mn for Ni in cryogenic steels concerns the development of austenitic weld wire for 9%Ni steel. In current practice "9Ni" steel is welded with high nickel (70-80%) weld wire, with a significant resulting increase in the cost of welded structures. While some recent success has been obtained through research on low-Ni ferritic weld compositions, particularly for use in TIG welding,(39, 41-42) low nickel, chrome-manganese stabilized austenitic filler metals are under active development. Relevant recent research in the Soviet Union includes studies of the properties of 5% and 9%Ni steels welded with austenitic Cr-Ni-Mn-N wire (19Cr-15Ni-6Mn) reported by Yushchenko and co-workers elsewhere in this conference.(36, 38-39) Similar studies are underway in Japan, particularly at the Nippon Steel Corporation,(43) where laboratory success has been obtained with Ni-Cr-Mn-Mo austenitic wires of various compositions, (for example, 13Ni-12Mn-7Cr-2.5Mo) in welding 9%Ni steels by the MIG process.

IV. CONCLUSION

Both the fundamental and applied research results cited above suggest that alloys based on the Fe-Mn system have significant potential for use as structural alloys in cryogenic systems. These alloys are, however, only in the initial stages of development. There are clear opportunities for valuable research in at least four areas: (1) the prevention of low-temperature brittleness in ferritic alloys; (2) the stabilization of austenite to insure good cryogenic toughness in austenitic alloys of lower Mn content; (3) the strengthening of austenitic Fe-Mn alloys; and (4) the development of austenitic Fe-Mn alloys for structural use in liquid helium as an alternative to the non-magnetic high nickel alloys now under consideration. The successful completion of these research projects will almost certainly require a more thorough fundamental understanding of the cryogenic mechanical behavior of Fe-Mn alloys and particularly of the physical and mechanical behavior of the ε-martensite phase which appears to play an important role at intermediate Mn contents. The rate at which these new Fe-Mn cryogenic alloys are developed and exploited industrially will almost certainly depend on the extent to which the development of cryogenic systems creates a demand for structural alloys and on the magnitude of the incentive provided by the relative cost of nickel.

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TABLE I. MECHANICAL PROPERTIES OF Fe-Mn ALLOY STEELS (4)

GRADE	CHEMICAL COMPOSITION, %					TEST temp. (°C)	MECHANICAL PROPERTIES			
	C	Mn	Si	S	P		$\sigma_{0.2}$ (MPa)	σ_b (MPa)	Elong. (%)	R.A. (%)
05G4	0.05	4.1	-	-	-	+22	564	598	11	73
						-196	897	957	15	33
05G8	0.05	8.1	-	-	-	+22	633	663	10	68
						-196	958	998	1	1
05G12	0.05	12.1	-	-	-	+22	605	873	17	63
						-196	1067	1354	19	45
06G21	0.056	20.8	0.33	0.011	0.016	+20	453	852	28.5	20.7
						-196	670	1221	29.3	24.9
08G25	0.082	25.6	0.27	0.008	0.017	+20	252	724	43.4	41.1
						-196	412	1190	47.7	35.1
06G32	0.057	32.0	0.62	0.007	0.021	+20	210	524	60.7	74.4
						-196	394	898	41.1	39.2
06G35	0.060	34.7	trace	0.004	0.014	+20	220	496	48.3	70.4
						-196	384	826	52.7	71.0
07G42	0.067	42.0	0.3	0.008	0.019	+20	238	507	46.3	71.0
						-196	431	878	53.0	55.4
06G46	0.060	46.0	-	0.005	0.021	+20	251	530	49.4	74.4
						-196	407	805	21.6	19.0
05AG25	0.045	24.3 ~25.6	0.10 ~0.14	0.006	0.013	+20	245	755	43.6	41.2
						-196	554	923	10.4	16.0
05AG30	0.054	30.0	0.16	0.005	0.013	+20	272	580	66.8	75.0
						-196	739	1116	25.0	24.9
08AG34	0.078	33.5	0.17	0.007	0.015	+20	270	569	53.0	70.7
						-196	679	791	6.0	5.3

$\sigma_{0.2}$: 0.2% Off-set engineering yield strength

σ_b : Breaking strength

Elong.: Elongation

R.A.: Reduction in area

TABLE II: CRYOGENIC MECHANICAL PROPERTIES OF PROCESSED Fe-Mn ALLOY STEELS (9-10)

test temp.: -196°C

Nominal Composition	Treatment	$\sigma_{0.2}$ (MPa)	σ_{uts} (MPa)	Elong. (%)	R.A. (%)	K_{1C} (MPa$\sqrt{m}$)
Fe-12Mn-0.2Ti	Solution-Annealed	883	1351	25	54	59
	Tempered(500°C/10h)	952	1358	33	62	77
	cold-work (50%) plus tempered (600°C/4h)	1179	1503	38	66	100
Fe-8Mn-0.2Ti	Solution-Annealed	965	1044	4	6	-
	Thermal-Cycled*	967	1054	26	70	-

σ_{uts}: Ultimate tensile strength

*: (750°C/2h + 650°C/2h)/2 cycles

TABLE III. CHEMICAL ANALYSIS AND TENSILE PROPERTIES OF 03K13AI19 STAINLESS STEEL (38)

CHEMICAL COMPOSITION (wt %)

Fe	C	Mn	P	S	Si	Cr	Ni	Cu	Mo	V	Ti	N	B
bal.	0.055	19.0	0.020	0.011	0.38	13.40	0.82	0.07	0.01	0.10	<0.01	0.16	0.0005

Tensile Properties (transverse orientation)

Test temp. (°C)	$\sigma_{0.2}$ (MPa)	σ_{uts} (MPa)	Elong. (%)	R.A. (%)
27	319	666	60	74
-196	711	1114	-	23

TABLE IV. MECHANICAL PROPERTIES OF Fe-Mn-Cr-Ni (32)

Nominal Composition	Test temp. (°C)	$\sigma_{0.2}$ (MPa)	σ_{uts} (MPa)	Elong. (%)	C_V (Joules)
-0.15-0.1N Fe-25Mn-5Cr-1Ni	25	210	630	60	-
	-196	540	1200	40	146
Above + 0.11Nb	25	340	730	60	-
	-196	730	1250	25	80

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FIGURE CAPTIONS

Fig. 1. The effect of Mn content in Fe-Mn alloys on

(a) Phase transformations during continuous heating and cooling,

(b) tensile properties and

(c) ductile-brittle transition temperatures.(3)

Fig. 2. The impact toughness of Fe-Mn steels of high Mn concentration.(4)

Fig. 3. The effect of Mn on the stacking fault energy of steels.(20-21)

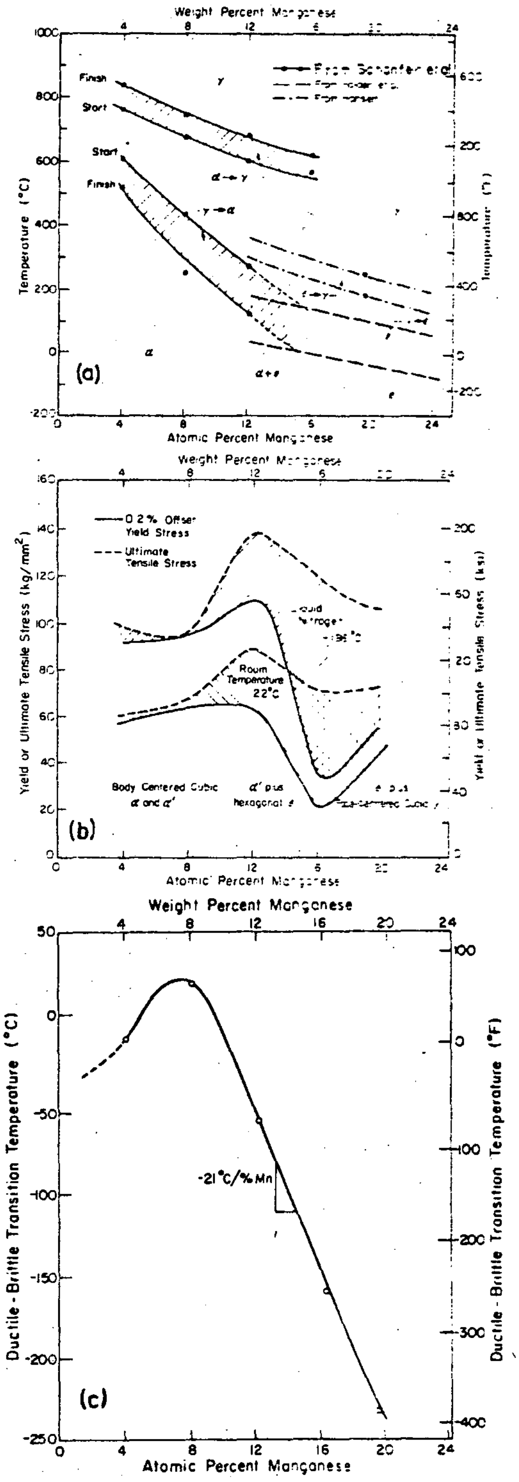
Fig. 4. The effect of Ni on the phase composition of Fe-Mn alloys quenched to room temperature.(26-27)

Fig. 5. Structural diagram of Fe-Mn-Cr system at 27°C.(28)

Fig. 6. Structural diagram of Fe-Mn-C system.(30)

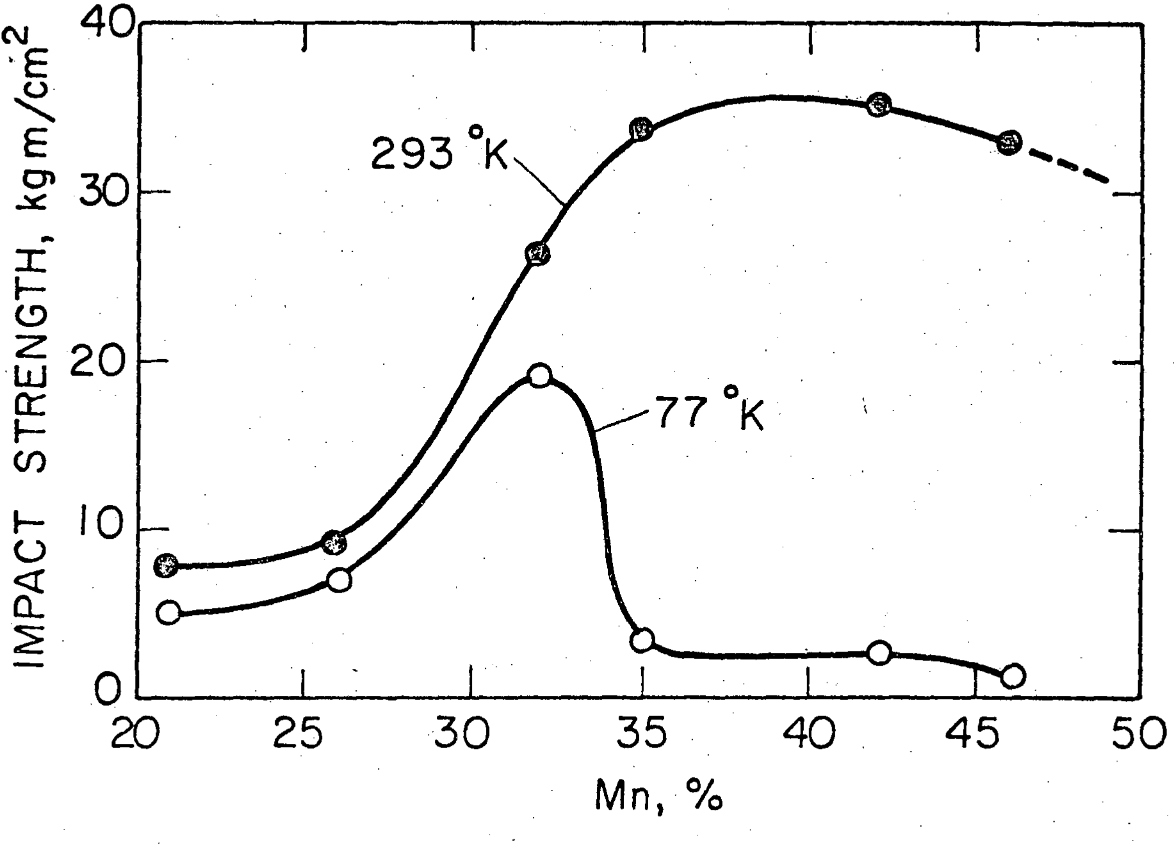
Fig. 7. The effect of processing techniques on the suppression of ductile-brittle transition temperature of an Fe-12Mn-0.2Ti alloy steel.(9-10)

Fig. 8. Temperature dependence of fracture toughness for three steels proposed for LNG tankage construction.(38)



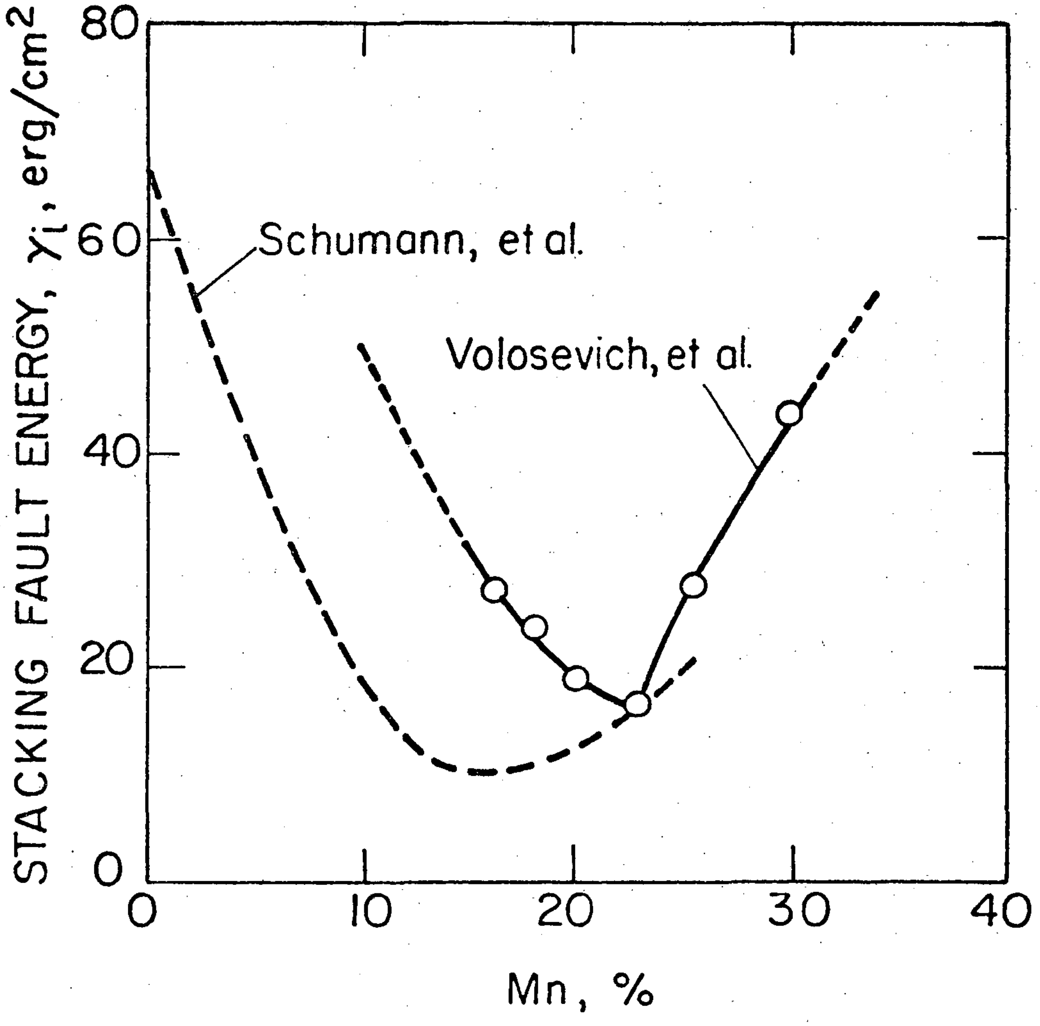
XBL 778-2815

FIG. 1



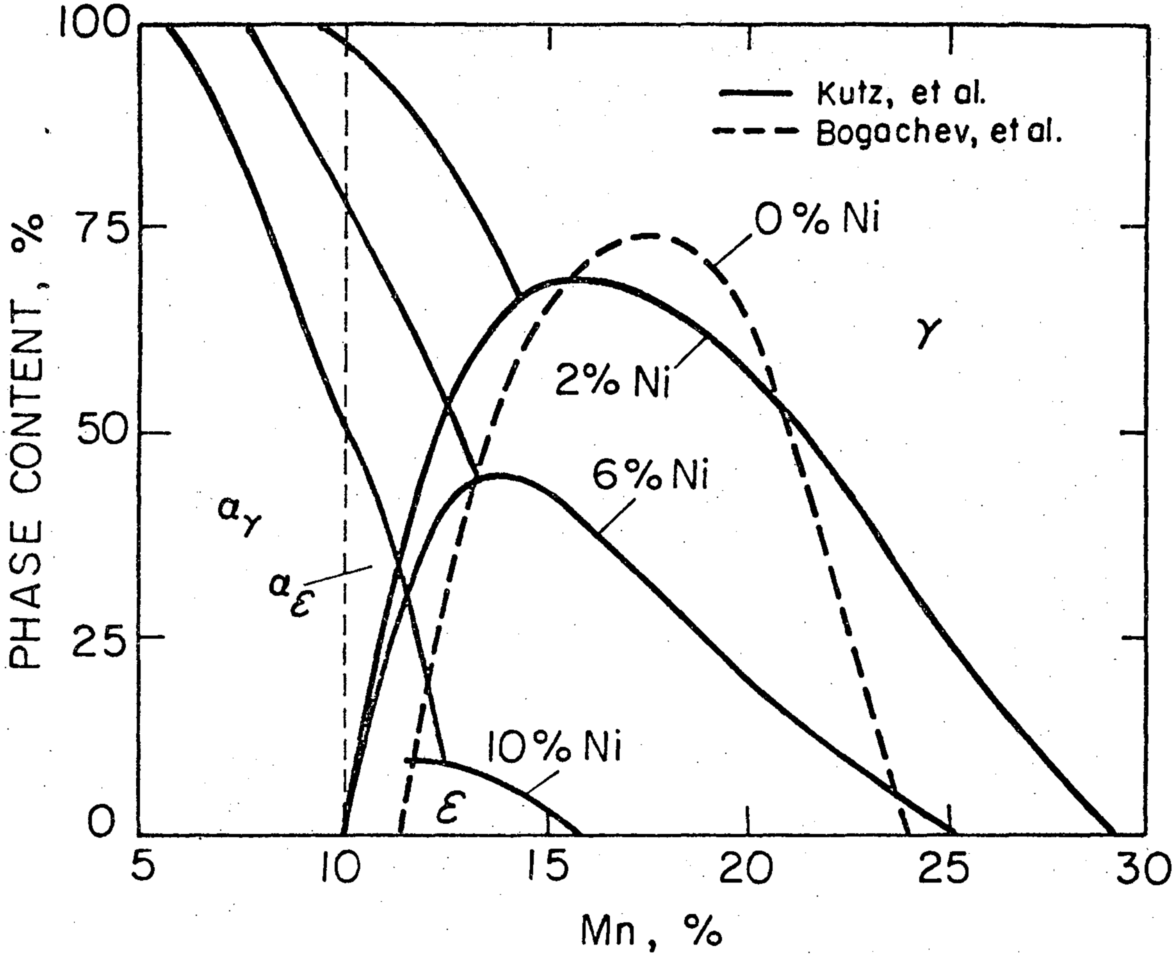
XBL 778-6016

FIG. 2



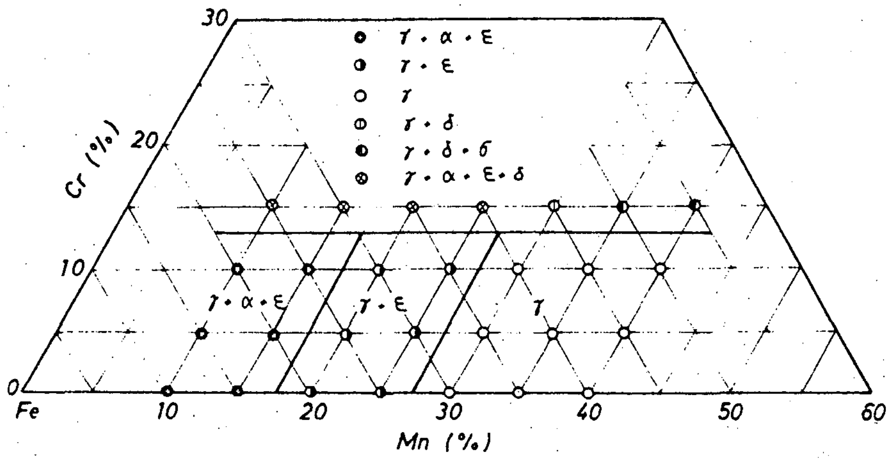
XBL 778-6015

FIG. 3



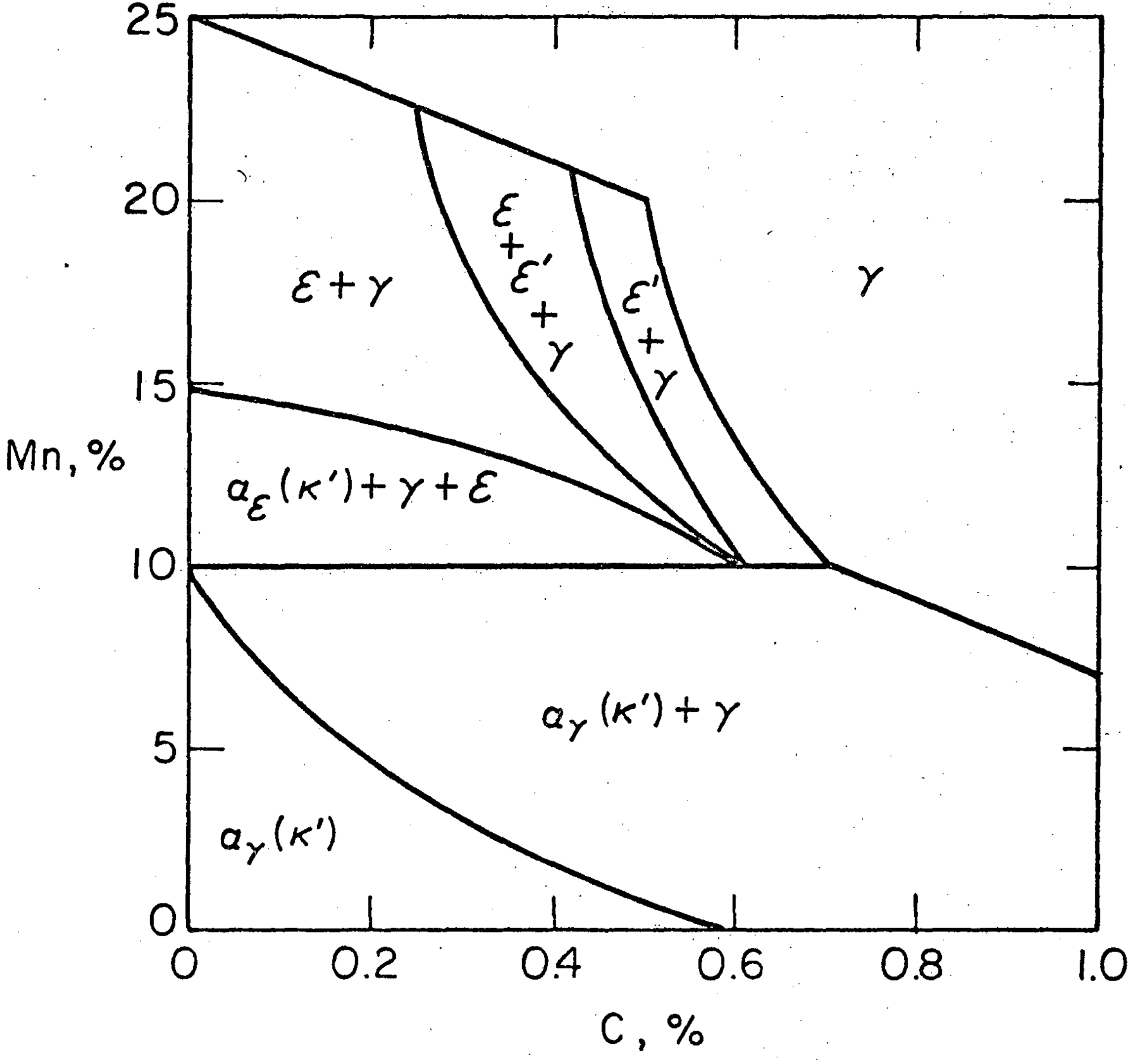
XBL 778-6014

FIG. 4



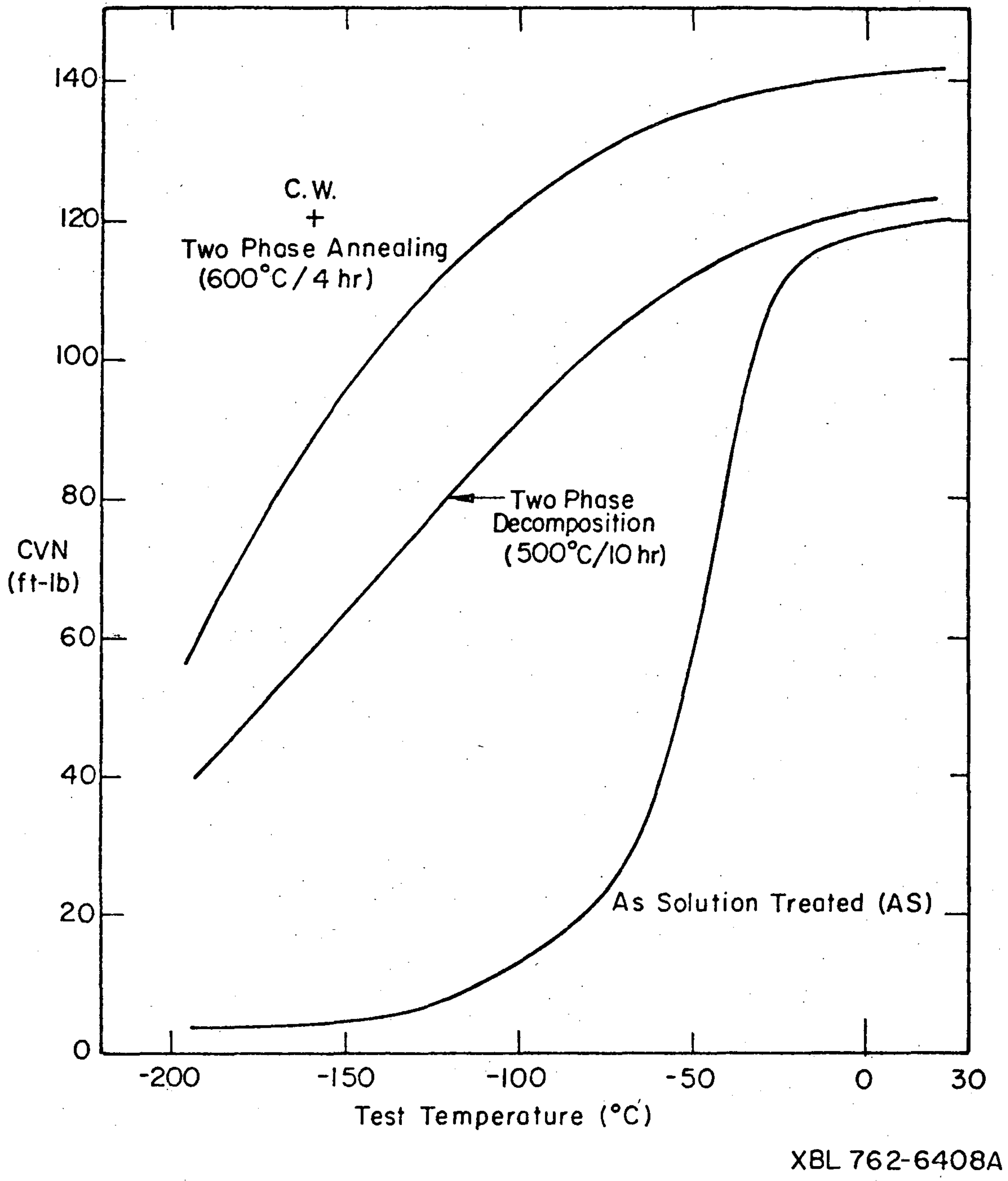
XBL 778-2816

FIG. 5



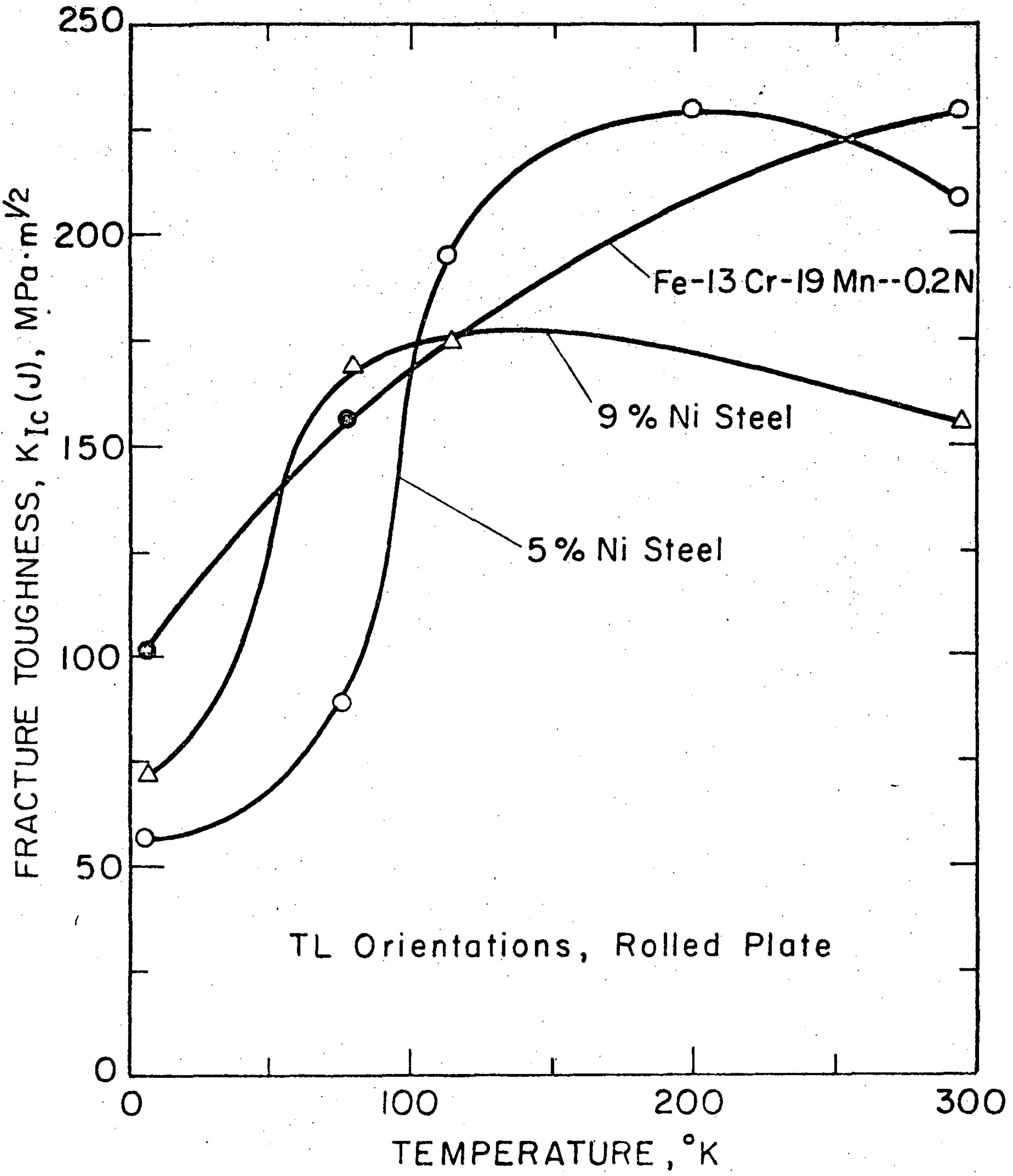
XBL 778-6017

FIG. 6



140
120
100
80
60
40
20
0
CVN
(ft-lb)
-200
-150
-100
-50
0
30
Test Temperature (°C)
C.W.
+
Two Phase Annealing
(600°C / 4 hr)
Two Phase
Decomposition
(500°C/10 hr)
As Solution Treated (AS)
XBL 762-6408A

FIG. 7



XBL778-6013

FIG. 8

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