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Mark Jay Schanfein
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THE CRYOGENIC PROPERTIES OF Fe-Mn and Fe-Mn-Cr ALLOYS

Mark Jay Schanfein

Inorganic Materials Research Division, Lawrence Berkeley Laboratory and Department of Materials Science and Engineering, College of Engineering; University of California, Berkeley, California

ABSTRACT

A wide range of microstructures were obtained in Fe-Mn alloys by varying the manganese and chromium contents. When a bcc (α) structure was produced, increasing amounts of manganese were found to be detrimental to low temperature toughness. At manganese levels greater than 12% where appreciable amounts of ε and γ phases formed, the ductile-brittle transition temperature dropped rapidly. In terms of the (ε+γ) phases present, the ductile-brittle transition temperature decreased at a rate of 1.3°C/vol% (ε+γ). Increasing the (ε+γ) content to achieve good low temperature toughness, however, also caused a decrease in the yield strength. Increases in the yield strength were achieved without appreciable increase in the ductile-brittle transition temperature or decreases in the Charpy upper shelf energy and fracture toughness by greater manganese additions and by chromium additions. The exception was the 16%Mn-8%Cr alloy which did show appreciable decreases in the upper Charpy shelf energy and fracture toughness.
I. INTRODUCTION

Industrial demand for the storage and transportation of cryogenic liquids has brought about research efforts to develop an alloy which exhibits both high strength and toughness at low temperatures. Current alloys in use are either strong with mediocre toughness or tough with low strength. One research effort has been directed towards the development of a strong tough Fe-Ni-Ti alloy.\(^1\)-\(^4\) The principle features of this alloy are (1) a low interstitial content (through the use of high purity elements) and small additions of Ti for gettering the remaining interstitials, (2) small grain size through controlled heat treatments, (3) the presence of noncoherent precipitates \((\text{Ni}_3\text{Ti})\) for strengthening, and (4) high nickel contents. This alloy has shown excellent low temperature strength and toughness. However, in the Fe-Ni-Ti alloy the nickel content was approximately 12\%. In order to make a lower cost cryogenic alloy efforts have been made to reduce the nickel content by substituting manganese for some of the nickel.

A different approach has been to investigate the behavior and properties of non-nickel containing alloys, utilizing the knowledge achieved in the development of the Fe-Ni-Ti alloy. The present investigation was directed toward the use of the Fe-Mn-Ti-Al system as a potential cryogenic alloy.
The beneficial effect of nickel in lowering the ductile-brittle transition temperature (DBTT) of iron-carbon alloys has been known since the 1940's. The knowledge of this phenomenon has led to the development of an entire class of commercial cryogenic alloys. Recently it was shown that Ni lowers the DBTT of carbon free iron as well. The atomistic and microstructural mechanisms responsible for the improvement in notch toughness resulting from nickel additions are not well understood. It is usually assumed, however, that the crystal lattice (rather than the microstructure) is influenced by the nickel solute and that the effect of nickel is to raise the cleavage strength of iron relative to the flow stress. Manganese is another solute thought to improve the low temperature ductility of iron but apparently it behaves in a different way from Ni. Jolley has reported that manganese improves the notch impact properties of iron only when carbon is present. He found no improvement in the toughness of decarburized iron alloys, and concluded that the effectiveness of manganese was primarily through alteration of the morphology and distribution of carbides. Roberts, in a later study of Fe-Mn alloys containing up to 9%Mn, found that the impact toughness and DBTT were relatively insensitive to the manganese content. Instead, he suggested that manganese influenced the transformation substructure and grain size. The grain size dependence of the DBTT amounted to 6-7°C/mm-1/2 which is roughly the same grain size dependence Leslie and Sasaki et al. have found for Fe-Ni alloys.
In the Fe-Mn system, a variety of substructural changes occur in the bcc lattice, as shown by Roberts. Additionally, when the manganese content exceeds about 10%, austenitized and quenched alloys contain a hexagonal ε phase, and a mixed α' + ε microstructures result. When the manganese content is increased beyond 15% the face centered cubic γ phase is stabilized and is resistant to transformation even when cooled to liquid nitrogen temperatures; mixtures of α + ε + γ microstructures are obtained.

The DBTT phenomenon is normally associated with alloys having a bcc or bct crystal structure. Alloys with these crystal structures usually have adequate strength but are limited by a tendency toward catastrophic failure at low temperatures. Metals having face centered cubic and some with hexagonal structure, do not possess a DBTT (except in certain cases where solute or precipitate segregation at grain boundaries occurs). The strengths of alloys with the fcc crystal structure is generally lower than those with the bcc structure, although the low temperature ductility associated with these structures is generally better than those of bcc alloys. A primary objective of the present investigation was to explore the feasibility of designing Fe-Mn alloys with mixed microstructures for attaining combinations of strength and toughness which approach those of the cryogenic Fe-Ni-Ti alloys.

Recent studies in this laboratory and elsewhere have shown that strain induced transformations can significantly raise the ductility and fracture toughness of alloys having metastable matrices. In the Fe-Mn system, the possible strain induced transformations are
those of the hexagonal $\epsilon$ phase transforming during deformation to bcc $\alpha$ and of the retained austenite transforming to either $\epsilon$ or $\alpha$, or both. Troiano and McGuire, White and Honeycombe, and more recently, Holden, et al. found such phase transformations occurring during cold working of Fe-Mn alloys whose compositions were similar to those used in the present investigation.
II. EXPERIMENTAL PROCEDURES

A. Material Preparation

The alloys used for this investigation were prepared by induction melting high purity (99.9+) iron, manganese, chromium, aluminum, titanium and carbon in zirconium oxide crucibles under first a vacuum at $10^{-3}$ mm Hg and then an argon atmosphere. From this melt three inch diameter ingots were cast in copper chill molds and cooled in an argon atmosphere. The ingots were next homogenized for twenty-four hours at 1200°C in a vacuum and then furnace cooled. After being reheated to 1200°C they were then upset forged to bars of dimension 2-3/4 in. wide by 1 1/4 in. thick and air cooled. Material was cut from these bars to prepare one inch thick fracture toughness specimens ($K_{IC}$). The remaining material was then rolled down to one half inch thickness at 1200°C and air cooled. Material was removed again for Charpy V-notch specimens and again the remaining material was rolled down to one quarter inch thickness at 1200°C and air cooled. From this remaining material, flat tensile specimens were prepared. All machining was performed with a cooling medium directed on the machined surface to keep heating to a minimum.

The compositions and designations of the alloys are listed in Table I. All alloys contained 0.10% titanium and 0.05% aluminum, which were added to inactivate the interstitials, carbon and nitrogen.
The C + N content of the alloys was about 0.02%. The exception is the 16%Mn-0.05%C alloy where titanium was not added but carbon was.

B. Heat Treatment

All of the material in rough cut test specimen dimensions was initially austenitized at 900°C for two hours in an air furnace (±5°C) and first ice brine quenched (10% salt solution) and then liquid nitrogen quenched. The sections were then machined into standard Charpy V-notch specimens (Fig. 1), 0.15 in. thick flat tensile specimens (Fig. 2), and 1 in. thick fracture toughness specimens (KIC) (Fig. 3).

Grain refinement heat treatment techniques were evaluated on 1/2 in. square by 1/10 in. thick specimens by heating in a salt bath at the desired temperatures (±2°C) for one half to one hour and then ice brine quenching (10% salt solution). Grain refining treatments were performed only on Charpy V-notch specimens.

C. Mechanical Tests

1. Hardness Tests

Hardness measurements were performed with a Wilson Rockwell Hardness tester. The Rockwell "C" scale (150 Kg major load and diamond Braile indentor) was used. A minimum of four hardness impressions were made on Charpy V-notch specimens and the results averaged (see Table II).

2. Charpy Impact Tests

Impact tests were carried out on a 225 ft-lb capacity impact testing machine as specified by ASTM procedure E-23-64. The tests were run at various testing temperatures with the use of liquid helium,
nitrogen and isopentane, methyl alcohol and dry ice, and distilled water and ice.

3. Tensile Tests

Tensile properties were determined on an 11,000 lb capacity Instron Testing Machine at a crosshead speed of 0.1 cm/min. Tests were carried out at room temperature, -78°C (dry ice and methyl alcohol), and -196°C (liquid nitrogen).

4. Fracture Toughness

Plane strain fracture toughness testing was performed following ASTM standard E399-72. The tests were carried out on a 300,000 lb capacity MTS machine using the 2-1/2 in. square, one inch thick crack-line loaded specimens shown in Fig. 3. From bars which were rolled to 1-1/4 in. thickness transverse sections were cut to prepare fracture toughness specimens. These specimens were machined oversize and then austenitized at 900°C for two hours and then immediately ice brine and liquid nitrogen quenched. They were then machined to final dimensions. Next the specimens were fatigue precracked using the MTS at a cycling rate of 6 cycles/sec and at a load sufficient to obtain a crack of at least 0.10 inches in approximately 20,000 cycles but not so high as to exceed ASTM specifications. All specimens were pulled at -196°C (liquid nitrogen bath). To monitor displacement a crack opening displacement gauge similar to that described in the ASTM standards was used (See Fig. 4). Both loading and displacement were recorded on an X-Y recorder (See Appendix C).
D. **Dilatometry**

Dilatometry tests were performed to study the kinetics of the phase transformations during both heating and cooling. The specimens were one inch long by 1/4 inch in diameter with a 3/32 inch center hole to facilitate uniform heating. Dilatometric curves were made by heating and cooling the specimens at 630°C/hour in an air atmosphere tube furnace and simultaneously recording the specimen expansion and contraction and its temperature. The expansion was monitored by a transducer and the temperature by a chromel-alumel thermocouple spot welded directly on the specimen. This information was directly recorded on an X-Y recorder. (See Appendix A)

E. **X-Ray Diffraction**

Quantitative measurements of the amounts and types of phases present were conducted using a Picker X-ray diffractometer. The percentages of the phases present were estimated by comparing the integrated diffraction intensities of the (200) and (211) alpha peaks, (220) gamma peak, and (012) and (013) epsilon peaks from a copper K\(_\alpha\) source with a LiF monochromator between the diffracted beam and detector. A check was made to determine if a high degree of preferred orientation existed in the X-ray specimens by taking Laue back reflection pictures using a Norelco vertical tube unit with a Mo source (see Appendix B). The results were negative, but the specimens were run twice with the sample rotated 90° for the second run to minimize the effects of small amounts of preferred orientation. From the two runs, the integrated intensities were averaged for every peak. Hot stage X-ray work was also performed using the Picker X-ray
diffractometer to correlate the expansion during transformation, from the dilatometric graphs, to the epsilon to gamma transformation. The surfaces of the X-ray specimens were carefully prepared following the same procedure as that for the optical microscopic specimens up to but not including the etching (see Appendix B).

F. Microscopy

1. Optical Microscopy

Specimens used for optical metallographic observation were obtained from transverse sections of broken Charpy bars. These specimens were mounted in Koldmount, ground on Al₂O₃ emery paper to 000 grit, polished on a 6µ and then a 1µ diamond wheel and final polished using a 0.05µ Al₂O₃ solution in a syntron. Before changing to a finer medium for polishing the specimens were ultrasonically cleaned to remove any remaining particles from the previous polish. Electropolishing was then performed on each specimen in a chromic acetic solution (400 ml acetic acid, 20 ml distilled water, 75 gm chromic trioxide) at 10°C until all surface scratches were removed. For those alloys without chromium a mixed acid reagent (20 ml HCl, 10 ml HNO₃, 10 ml H₂O₂, 20 ml glycerol) was applied for between 15 to 30 seconds. The electropolishing and application of mixed reagent were repeated several times to remove the deformed surface layer. Finally Klemm's reagent (100 ml cold saturated Na₂S₂O₃ (in H₂O) + 5 gm K₂S₂O₃) was applied for one minute. The chromium containing alloys presented a problem since this procedure did not reveal the microstructure. Instead, following the electropolish, ferric chloride (saturated solution of FeCl₃ in 20 ml HCl, 10 ml HNO₃,
20 ml glycerol) was applied for a maximum of 1 min. Repeated treatments were made using this new procedure to remove the deformed surface layer.

2. **Scanning Electron Microscopy**

   The fracture surfaces of broken Charpy specimens were examined with a Jeolco JSM-U3 scanning electron microscope (SEM) operated at 25 kV via secondary electron emission.
III. EXPERIMENTAL RESULTS AND DISCUSSION

A. Dilatometry Data

The phase transformation temperatures of Fe-Mn alloys as determined by dilatometry are shown in Fig. 5. Also shown in Fig. 5 are the results of other investigators. When these alloys were cooled from the austenite phase a variety of complex microstructures resulted. The as-quenched structure of low manganese alloys which were cooled to LN (-196°C) was entirely bcc. However, it has been reported that the substructure morphology changes from equiaxed ferrite (α) to lath and plate martensites (α') as the manganese content is increased to about 12%. In Figs. 6 and 7 are shown micrographs illustrating the structure of the Fe-Mn alloys of the present study. An example of the typical "massive" lath martensite microstructure of the 4, 8, and 12% Mn alloys are shown in Figs. 6(a), (b) and 7(a). In alloys with manganese contents greater than 12% increasing amounts of the hexagonal ε phase formed. It has been reported that in alloys with approximately 30% Mn the austenite (γ) phase is completely retained even on cooling to -196°C. Within the 12%-20% Mn composition range various complex microstructures, consisting of mixtures of the α', ε and γ phases, were obtained. Examples of these mixed microstructures are shown in Figs. 7(c,e). The microstructure of the 16%Mn alloy (Fig. 7(c)) was predominantly α' + ε while that of the 20%Mn alloy was entirely ε + γ (Fig. 7(e)).

The sequence of structural changes in Fe-Mn alloys was similar to that in the more familiar Fe-Ni system, except for the occurrence, in Fe-Mn alloys, of the hexagonal ε phase. This phase has been found only
in those alloy systems where solute additions decrease the stacking fault energy of the austenite to values approaching zero. At these very low stacking fault energies the driving force necessary for the \( \gamma \rightarrow \varepsilon \) transformation is reduced below that of the \( \gamma \rightarrow \alpha' \) transformation and a metastable \( \varepsilon \) phase forms.

B. Grain Refinement versus the Ductile to Brittle Transition Temperature

A mentioned previously grain refinement has been used in the Fe-Ni-Ti system to improve its toughness and lower the ductile to brittle transition temperature. Grain refinement was also applied to the 8\%Mn alloy in this investigation to see if the effects would be sufficient to make it ductile at cryogenic temperatures.

To refine the grain size a two step heat treatment, a modification of that used in the Fe-Ni-Ti system, was used.\(^4,21\) The procedure was to first bring the specimen up to a temperature just below the austenite start temperature and hold for 1/2 hr allowing extensive nucleation to occur. Then the specimen was heated up to the duplex region between the austenite start and finish temperature and held for 1/2 hr before quenching in an ice brine solution. This procedure was then repeated until the optimum refinement was obtained. Two different treatments were performed to compare against the as austenitized properties. The first refined the grain size down to 8 \( \mu \)m and required the following procedure:

550°C(HOLD FOR 1/2 HOUR) to 730°C(HOLD FOR 1/2 HOUR) ICE BRINE QUENCH

repeated four times. The second refined the grain size down to 4 \( \mu \)m and required the following procedure:

550°C(HOLD FOR 1/2 HOUR) to 700°C(HOLD FOR 1/2 HOUR) ICE BRINE QUENCH
repeated four times. The optical micrographs of these structures are shown in Fig. 8. The first micrograph in Fig. 8a is that of the as austenitized material. Figure 8b is the optical micrograph of the material refined down to 8 μm grain size and Fig. 8c shows the 4 μm material at two different magnifications. Charpy bars of this material were then broken and the energies compared as can be seen in Fig. 9. The refinement procedure did decrease the transition temperature a total of 56°C following the well known Hall-Petch relation.

Plotting the ductile to brittle transition temperature versus (grain size)$^{-1/2}$ showed the rate of decrease to be 6.5°C per mm$^{-1/2}$ as shown in Fig. 10. This rate is not sufficient to make it a good cryogenic alloy through grain refinement. In addition, reduction in grain size leads to serious limitations when the material must be welded.

C. Effects of Manganese

The Charpy impact toughness for the various as austenitized Fe-Mn alloys are plotted as a function of temperature in Fig. 11. The fracture surfaces of the Charpy bars broken at both room (22°C) and LN(-196°C) temperatures are shown in Figs. 12 and 13, and correspond to the microstructures illustrated in Figs. 6 and 7. The room temperature Charpy fractures of the 4%Mn, 8%Mn, 12%Mn, 16%Mn, and 20%Mn alloys revealed the dimpled rupture characteristics of ductile behavior. In the liquid nitrogen (-196°C) tests the 4%Mn and 8%Mn alloys exhibited features of cleavage failure whereas the 12%Mn alloy showed intergranular failure with dispersed areas of dimple rupture. These areas presumably correspond to the small amounts of epsilon and gamma as revealed
by X-ray analysis. Since they would preferentially appear at grain boundaries and are substantially weaker than the alpha phase it is reasonable to assume that these areas are responsible for the intergranular failure. The 16%Mn and 20%Mn alloys showed predominantly dimpled rupture with some quasicleavage.

In Fig. 14 is shown a plot relating the DBTT to manganese content. It is evident from Fig. 14 that as the manganese content increased the DBTT first increased up to approximately 8%Mn and then decreased rapidly at a rate of 21°C/at.%Mn (see Appendix D).

X-ray analysis was performed to determine the quantities of the phases present. To do this it was necessary to use integrated diffraction intensities (See Appendix B).

It was established from this X-ray phase analysis, Table III, that the 4%Mn and 8%Mn alloys were completely bcc while the 12%Mn, 16%Mn and 20%Mn alloys contained increasing amounts of the ε and γ phases. The 16%Mn alloy had an α+ε+γ mixed microstructure while the 20%Mn alloy had an ε+γ mixed microstructure. Thus the results of the X-ray analysis and DBTT determinations suggested that the ε or γ or both phases were responsible for the improved low temperature toughness of the higher manganese alloys (12 to 20%Mn). In terms of the (ε+γ) phases present, the DBTT decreased at a rate of 1.3°C/vol% (ε+γ).

It was evident that the relation between microstructure and cryogenic mechanical properties for the Fe-Mn alloys of the present study was quite different from that for the interstitial-free Fe-Ni alloys. The
Fe-Ni alloys exhibited a decrease in the DBTT with increasing nickel content in a single phase bcc structure. In the Fe-Ni system, as previously mentioned, the lowering of the DBTT by nickel is probably due to solute-lattice interaction rather than due to a microstructural effect, while in Fe-Mn alloys the decrease in DBTT was accompanied by microstructural changes involving variations in the relative amounts of the α, ε, and γ phases.

D. Ductile to Brittle Transition Temperature versus Yield Strength

Besides a low DBTT, an adequate yield strength must also be developed in an alloy which is to be considered for cryogenic applications. The yield and tensile strengths, elongation, and reduction in area both at room, dry ice, and liquid nitrogen temperatures are listed in Table IV. In addition plots of these properties as a function of manganese content are shown in Figs. 15 and 16. From these figures it can be seen that the 4%Mn, 8%Mn and 12%Mn alloys with bcc matrices exhibited fairly high yield strength but rather poor elongation. As the manganese content of the alloys was increased, the amount of the hexagonal ε phase also increased. This in turn was associated with a considerable decrease in yield strength. The room temperature yield strength of the 16%Mn alloy, which had almost 40% ε, was only 30 ksi. Increase in the manganese content to 20% resulted in stabilization of the γ phase, and no α phase was present in the as-quenched alloy. The alloy contained 66% ε and 34% γ. At room and LN temperatures yield strengths of the 20%Mn alloy were 60 and 78 ksi respectively, and elongations were 43 and 62%
respectively. Thus it appeared that with increasing amounts of $\varepsilon$ in a primarily $\alpha$ microstructure the yield strength decreased; however, when the microstructure was predominantly $\varepsilon$, and $\gamma$ replaced $\alpha$, the yield strength increased.

The results of X-ray analysis (Table III) clearly indicated that during tensile testing the $\varepsilon$ phase in the 16%Mn alloy transformed to $\alpha$. It was probable that a stress induced martensitic transformation of $\varepsilon$ to $\alpha$ contributed to the low yield strength of the 16%Mn alloy. Stress induced transformations have been reported in several metastable austenitic steels of low austenite stability. 16,20 The increase in manganese content to 20% apparently resulted in two changes. First, it led to the elimination of the $\alpha$ phase transformation and the retention of the $\gamma$ phase. As a result the alloy consisted of a mixture of $\varepsilon$ and $\gamma$ phases of apparently comparable strengths, thus preventing localized flow in either phase. Second, the $\varepsilon$ phase in the 20%Mn alloy was more stable than that in the 16%Mn alloy, thus minimizing the possible occurrence of a stress induced transformation. The higher stability of the 20%Mn alloy compared to that of the 16%Mn alloy was evident from the observation that during tensile testing a greater volume fraction of $\varepsilon$ transformed to $\alpha$ in the latter alloy (see Table III).
The inter-relationships between the DBTT and the tensile properties are shown in Fig. 17. The figure shows that the yield strength and the DBTT varied in a complex manner with increase in the manganese content. An important feature of the plot is in the region beyond 12%Mn where the hexagonal ε phase begins to form and the DBTT of the alloys begins to decrease rapidly. Unfortunately, it is in this range of compositions that the alloys begin to lose their strength. The cause of this decrease was discussed earlier.

E. Effects of Chromium

The relationship between microstructure, strength and toughness was also examined in several Fe-Mn alloys containing 8%Cr. Figure 17 relates strength and DBTT for these alloys. The relative proportions of the α, ε and γ phases in the chromium-containing alloys before and after tensile testing are indicated in Table III. Figure 18 shows the Charpy impact toughness as a function of temperature for the various as austenitized Fe-Mn alloys with and without chromium.

The fracture surfaces of the Charpy bars broken at both room (22°C) and liquid nitrogen (-196°C) temperatures are shown in Fig. 19 and correspond to the microstructures illustrated in Figs. 7(b,d,f). These fracture surfaces exhibited the same behavior at both room and liquid nitrogen temperatures as the corresponding alloys without chromium.

Figure 20 and 21 show the fracture surfaces of the fracture toughness \(K_{IC}\) specimens tested at liquid nitrogen temperature. It is interesting to note that no large shear lips were present on any of the specimens and they were in fact very difficult to observe even in the
20%Mn and 20%Mn-8%Cr alloys.

The results suggested that chromium additions to Fe-Mn alloys favored the transformation of ε and γ to the α phase during deformation.13

In the as quenched condition, the volume fraction of the α phase in the 12%Mn-8%Cr alloy was reduced by approximately 7% from the 12%Mn alloy. This is reflected in the slightly lower yield strength, shift of the DBTT to a lower temperature (see Fig. 18) and increase in fracture toughness ($K_{IC}$) (see Table V). For the 16%Mn-8%Cr alloy the volume fraction of the α phase was almost twice that in the 16%Mn alloy. The yield strength of the chromium-containing alloy was correspondingly higher. In spite of the greater volume fraction of α (and the smaller volume fraction of ε) and the higher yield strength, the chromium containing alloy had approximately the same DBTT as the alloy without chromium (see Fig. 18). However the CVN shelf energy was lower and the fracture toughness (see Table V) was reduced to slightly less than half.

In the 20%Mn-8%Cr alloy there was a slight increase in gamma over the 20%Mn alloy. An additional decrease in the DBTT was observed (see Fig. 18) with little loss in strength and the fracture toughness remained unchanged (See Table V).

So from these three alloys with 8%Cr added, the chromium appears to favor the gamma phase in the 12 and 20%Mn alloys. In the 16%Mn8%Cr alloy even though the alpha was preferentially favored (which agrees with the previous dilatometry data for cooling) over either epsilon or gamma, the gamma was still favored over epsilon since the former was only reduced by 24% as opposed to 41% for the latter. The reasons for this behavior are not well understood. Nevertheless, it was evident that the 8%
chromium addition to the 16%Mn alloy was beneficial for
aattaining a superior combination of strength and toughness. These
properties coupled with the possible enhanced corrosion resistance of the
16%Mn8%Cr and 20%Mn8%Cr alloys would be very desirable in cryogenic
applications.

CVN specimens of the 20%Mn alloys with and without chromium were
also broken at liquid helium temperature. Both alloys absorbed
approximately 74 ft-lbs.

F. Preliminary Results of the Effects of
Carbon on a Mixed Structure

In the present investigation the interstitial atoms were kept to
a minimum because of their well known embrittling effect on the bcc
lattice. This is particularly important for the low manganese alloys
which are primarily or entirely bcc. However in the higher manganese
alloys since the primary phases are now HCP and FCC in structure the
role of carbon on properties is an interesting area to investigate.

In addition to the above alloys, one alloy of 16%Mn-0.05%C was
prepared and tensile (Table IV) and CVN energy (Fig. 22) tests were performed.
The main reason for the addition of carbon was to evaluate its effect on the
amounts of the phases present, on the strength, and on the toughness. From the
X-ray data in Table III it can be seen that the carbon favored the for-
mation of the epsilon and gamma phase. The gamma phase was increased by
49% and the epsilon by 26% over that in the 16%Mn alloy. The yield
strength at room temperature was increased by 75% while at L.N. temperature the yield strength was increased by 36% (Table IV) and the CVN energy was relatively unchanged except at LN temperatures where the 16%Mn alloy absorbed only 25 ft-lbs, compared with 42 ft-lbs for the 16%Mn-0.05%C alloy (see Fig. 22). The scanning electron micrographs of the CVN fracture surfaces show dimpled rupture at room temperature (22°C) and dimpled rupture and quasicleavage at LN(-196°C) temperature (see Fig. 23).

From the above results it can be seen that carbon had the beneficial effects of raising the yield strength while leaving the CVN energy relatively unchanged.
IV. CONCLUSIONS

1. A wide range of microstructures was produced in Fe-Mn alloys by varying the manganese and chromium contents.

2. In iron alloys that contained predominantly the bcc α phase, manganese additions raised the ductile-brittle transition temperature (DBTT). In Fe-Mn alloys without chromium, the formation of both hexagonal ε phase and fcc γ phase at manganese concentrations exceeding 12% resulted in a decrease in the DBTT.

3. The yield strength decreased with increasing amounts of ε in a mixed α+ε microstructure. The decrease was possibly due to localized flow in the weaker ε phase and a stress induced transformation of ε to α. The yield strength was raised when the manganese content was increased to 20%, resulting in a mixed ε+γ microstructure.

4. Chromium additions of 8% did not cause appreciable changes in the DBTT. In the 12%Mn alloy the chromium addition reduced the yield strength at LN temperature by 8%, increased the fracture toughness by 14%, and shifted the DBTT to a slightly lower value. In the case of the 16%Mn alloy, an 8%Cr addition nearly doubled the yield strength at the same DBTT while lowering the CVN shelf energy and reducing the $K_{IC}$ fracture toughness to less than one half. Chromium additions did not significantly change the yield strength, DBTT or fracture toughness of the 20%Mn alloy. The enhanced corrosion resistance that probably would be obtained with chromium additions is considered desirable for potential cryogenic applications. The results suggested that chromium additions to Fe-Mn alloys favored the transformation of ε and γ to the α phase during deformation.
5. The addition of 0.05% carbon to the 16%Mn alloy raised the yield strength substantially without changing the Charpy V-notch impact toughness appreciably (see Fig. 22).
V. RECOMMENDATIONS FOR FUTURE RESEARCH

1. Investigation of the effect of varying chromium in high manganese alloys on the strength, toughness and corrosion resistance.

2. Use of an element which will combine with manganese to form small finely dispersed noncoherent precipitates for strengthening.

3. Investigation of the effects of carbon on strength and toughness in high manganese alloys.

4. Developing an alloy with optimum mixtures of phases through an austenitizing and normalizing process with the objective of eliminating the ice brine and L.N. quenching presently used.

5. Investigate further alloys with 20\%Mn and with chromium varying between 8-12\% and carbon varying between 0.05 to 0.1\%.
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This research was performed partially under the auspices of the U. S. Atomic Energy Commission through the Inorganic Materials Research Division of the Lawrence Berkeley Laboratory and partially under the auspices of the National Aeronautics and Space Administration.
For alloys ranging in composition from 4 to 12% Mn the dilatometric curves showed one phase transformation upon heating and cooling. This was the alpha (bcc) to gamma (fcc) transformation which resulted in a contraction in length during heating. On cooling the reverse transformation occurred at a lower temperature indicated by an expansion in length. The 16% Mn alloys showed two transformations upon heating. The first at approximately 200°C showed an expansion in length upon transformation. The second at approximately 600°C showed a contraction in length upon transformation this as in the above material was the alpha (bcc) to gamma (fcc) phase transformation. The temperatures for the expansion matched the reported temperatures in the literature for the epsilon (hcp) to gamma (fcc) phase transformation. However an expansion was not expected since there is no mention in the literature that the epsilon (hcp) is denser than the gamma (fcc) phase.

Hot stage X-ray analysis was performed to confirm this transformation. A run was first made at room temperature (20°C) showing the presence of the three phases epsilon, alpha, and gamma. The specimen was then heated up to 320°C and held for twenty minutes. At this temperature no epsilon peaks appeared and the gamma peak doubled in intensity while the alpha peak remained relatively unchanged. The specimen was then cooled and the reverse results were noted. The densities of the three phases were then calculated using Troiano and McGuire's data of gamma a₀ = 3.584Å, alpha a₀ = 2.862, and epsilon c/a = 1.605 and the location of the epsilon (100) peak from the X-ray data. The calculated densities were:
These calculations showed that epsilon is indeed the densest phase.

Upon cooling this alloy down to room temperature the reverse transformation of gamma to alpha did not occur. However, in the alloy containing 8% chromium the reverse reaction did occur above room temperature.

For the 20% Mn alloys only one transformation occurred upon heating and that was the epsilon to gamma transformation. The reverse transformation occurred in both alloys with and without chromium, however it was suppressed to a lower temperature in the chromium containing alloy.
APPENDIX B

X-Ray Analysis

In order to compare the integrated diffraction intensities of the peaks from the different phases Back Laue X-ray diffraction pictures were first obtained to determine if preferred orientation existed which would render the quantitative analysis inaccurate. Examination of the pictures showed the diffraction rings to have uniform intensity all around. It was then safe to conclude that no excessive preferred orientation existed.

To obtain large peak areas to increase the accuracy of the quantitative analysis the X-ray scans were run at 1/2 degree per minute and a full scale intensity range of 200 counts per second. The areas were then determined using a Dietzgen Compensating Optical Reading Polar Planimeter.

The mathematical technique used is explained by Cullity. The basic intensity equation is used and the parameters in it are separated as follows:

\[ I = KRV \]

where \( I \) is the integrated intensity, \( K \) is a constant, \( V \) is the volume of the phase being analyzed, and \( R \) is a grouping of variables as follows:

\[ R = \frac{1}{\nu} f^2 P(L)e^{-2M} \]

where \( \nu \) is the volume of a unit cell, \( f \) is the structure factor, \( P \) is the multiplicity, \( L \) is the Lorentz-polarization factor, and \( e^{-2M} \) is the temperature factor.

The following equations can then be used representing each phase:

\[ I_\alpha = KR\nu_\alpha \]
\[ I_Y = K_1 R_Y V_Y \]
\[ I_\varepsilon = K_1 R_\varepsilon \varepsilon \]

\[ V_\alpha + V_\gamma + V_\varepsilon = 1 \]

So:
\[ V_\alpha = \frac{I_\alpha}{K_1 R_\alpha} \quad V_\gamma = \frac{I_Y}{K_1 R_\gamma} \quad V_\varepsilon = \frac{I_\varepsilon}{K_1 R_\varepsilon} \]

\[ \frac{I_Y}{I_\varepsilon} = \frac{R_\gamma V_\gamma}{R_\varepsilon V_\varepsilon} \quad \frac{I_\alpha}{I_\varepsilon} = \frac{R_\alpha V_\alpha}{R_\varepsilon V_\varepsilon} \]

Therefore:
\[ V_\gamma = \frac{I_Y}{I_\varepsilon} \frac{R_\varepsilon}{R_\gamma} V_\varepsilon \quad V_\varepsilon = \frac{I_\alpha}{I_\varepsilon} \frac{R_\varepsilon}{R_\alpha} V_\varepsilon \]

Since
\[ V_\varepsilon = 1 - (V_\alpha + V_\gamma) \]

Therefore:
\[ V_\varepsilon = 1 - \left( \frac{I_\alpha}{I_\varepsilon} \frac{R_\varepsilon}{R_\alpha} \right) V_\varepsilon - \left( \frac{I_Y}{I_\varepsilon} \frac{R_\varepsilon}{R_\gamma} \right) V_\varepsilon \]

\[ V_\varepsilon \left[ 1 + \left( \frac{I_\alpha}{I_\varepsilon} \frac{R_\varepsilon}{R_\alpha} \right) + \left( \frac{I_Y}{I_\varepsilon} \frac{R_\varepsilon}{R_\gamma} \right) \right] = 1 \]

Finally:
\[ V_\varepsilon = \frac{I_\varepsilon}{I_\varepsilon + \left( \frac{R_\varepsilon}{R_\alpha} \right) I_\alpha + \left( \frac{R_\varepsilon}{R_\gamma} \right) I_Y} \]
Where only two phases exist the same manipulation results in:

\[ V_\gamma = \frac{I_\gamma}{I_\gamma + \left(\frac{R_\gamma}{R_\alpha}\right) I_\alpha + \left(\frac{R_\gamma}{R_\varepsilon}\right) I_\varepsilon} \]

\[ V_\varepsilon = \frac{I_\varepsilon}{I_\varepsilon + \left(\frac{R_\varepsilon}{R_\gamma}\right) I_\gamma} \]

for the presence of only the epsilon and gamma phases, and

\[ V_\varepsilon = \frac{I_\varepsilon}{I_\varepsilon + \left(\frac{R_\varepsilon}{R_\alpha}\right) I_\alpha} \]

for the presence of only the epsilon and alpha phases. The results of this analysis are shown in Table II.
APPENDIX C

Fracture Toughness Testing

Of the compositions tested only two met the plane strain fracture toughness criteria as specified by ASTM for the determination of $K_{IC}$.

To analyze the other compositions Witt's Equivalent Energy theory was used. Using this theory the lower bound fracture toughness is given by:

$$K_{IC} = \frac{P_Q \sqrt{A_1/A_2} f(a/w)}{B \sqrt{w}}$$

Where $P_Q$ is the load at any point on the curve in the linear portion and $A_2$ is the area under this part of the curve. $A_1$ is the area under the curve to maximum load and the quantities $a$, $w$, and $B$ are respectively the crack length, the width, and the thickness of the specimen. The geometric shape factor $f(a/w)$ is evaluated in the ASTM specifications. The results of the tests are listed in Table V. Pictures of the resulting fracture surfaces can be seen in Fig. 20, 21. From the pictures it can also be seen that the 20%Mn alloys with and without chromium experienced a bowing out of the fatigue precrack which could not be prevented.
APPENDIX D

Ductile to Brittle Transition Temperature (DBTT)

The ductile to brittle transition temperature was taken to be that temperature at which the Charpy V-notch energy was half that of the highest measured Charpy V-notch energy.
REFERENCES


Table I. Chemical compositions of alloys.

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Table II. Hardness (Rockwell C scale).

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Table III. Determination of volume percent of phase present by X-ray Analysis.

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Table IV. Tensile data.

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Table V. Fracture Toughness ($K_{IC}$)

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*Calculated using Witt's equivalent energy theory (see Appendix C).
Fig. 1. Standard ASTM E23-72 Charpy V-notch specimen.
Fig. 2. Standard ASTM E8-69 tensile specimen.
Fig. 3. Standard ASTM E399-72 fracture toughness ($K_{IC}$) specimen.
Fig. 4. Double cantilever beam gage and method of mounting on large single edge notched fracture toughness ($K_{IC}$) specimens for displacement measurement.
Fig. 5. Phase transformation temperatures for Fe-Mn alloys of the present investigation. Also shown are some results of other investigations. 12,18
Fig. 6. Optical micrographs of as austenitized 4% Mn (a) and 8% Mn (b) alloys.
Fig. 7. Optical micrographs of as austenitized 12% Mn (a) 12% Mn-8% Cr (b) 16% Mn (c), 16% Mn-8% Cr (d), 20% Mn (e) and 20% Mn-8% Cr (f) alloys.
Fig. 8. Optical micrographs of 8% Mn after heat treatments of (a) 900°C (hold for 2 hrs) ice brine quench—(as austenitized), (b) 550°C (hold for 1/2 hr) to 730°C (hold for 1/2 hr) ice brine quench—repeated 4 times (c) 550°C (hold for 1/2 hr) to 700°C (hold for 1/2 hr) ice brine quench—repeated 4 times.
Fig. 9. Charpy V-notch impact toughness vs testing temperature for the as austenitized and grain refined 8% Mn alloy.
Fig. 10. Ductile to brittle transition temperature vs \((\text{grain size})^{-1/2}\) for the as austenitized and grain refined 8\% Mn alloy.
Fig. 11. Charpy V-notch impact toughness vs testing temperature for the as austenitized 4 through 20% Mn Alloys without chromium.
Fig. 12. Scanning electron fractographs of as austenitized 4% Mn (a) tested at 22°C, (b) tested at -196°C, and 8% Mn (c) tested at 22°C, (d) tested at -196°C Charpy fracture surfaces.
Fig. 13. Scanning electron fractographs of as austenitized 12% Mn (a) tested at 22°C, (b) tested at -196°C, 16% Mn (c) tested at 22°C, (d) tested at -196°C, and 20% Mn (e) tested at 22°C, (f) tested at -196°C Charpy fracture surfaces.
Fig. 14. Ductile to brittle transition temperature vs percent manganese in Fe-Mn alloys.
Fig. 15. Yield and ultimate tensile strength at both 22°C and -196°C vs percent manganese in Fe-Mn alloys.
Fig. 16. Percent elongation and reduction in area of both 22°C and -196°C vs percent manganese in Fe-Mn alloys.
Fig. 17. Ductile to brittle transition temperature vs yield and ultimate tensile strength for Fe-Mn and Fe-Mn-Cr alloys. Also indicated are the phases present in the alloy.
Fig. 18. Charpy V-notch impact toughness vs testing temperature for the as austenitized 12 to 20% Mn alloys with and without chromium.
Fig. 19. Scanning electron fractographs of as austenitized 12% Mn-8% Cr (a) tested at 22°C, (b) tested at -196°C, 16% Mn-8% Cr (c) tested at 22°C, (d) tested at -196°C, and 20% Mn-8% Cr (e) tested at 22°C, (f) tested at -196°C Charpy fracture surfaces.
Fig. 20. Fracture surfaces of as austenitized 4% Mn (a) 12% Mn (b), and 12% Mn-8% Cr (c) fracture toughness ($K_{IC}$) specimens.
Fig. 21. Fracture surfaces of as austenitized 16% Mn (a), 16% Mn-8% Cr (b), 20% Mn (c), and 20% Mn-8% Cr (d) fracture toughness ($K_{IC}$) specimens.
Fig. 22. Charpy V-notch impact toughness vs testing temperature for the as austenitized 16% Mn and 16% Mn-0.05% C alloys.
Fig. 23. Scanning electron fractographs of as austenitized 16% Mn-0.05% C (a) tested at 22°C, (b) tested at -196°C Charpy fracture surfaces.
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