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THE SUPPRESSION OF LOW TEMPERATURE INTERGRANULAR BRITTLENESS IN FERRITIC Fe-Mn ALLOYS

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ABSTRACT

Methods of enhancing the cryogenic toughness of an Fe-12Mn-0.2Ti alloy steel were investigated. The initial intergranular brittleness was found to have a two-fold origin. Either rapid quenching from above M_f temperature or an extended holding below this temperature caused the brittle intergranular fracture at -196°C. It was found possible to minimize the embrittling effect by a controlled cooling with a resulting increase in the cryogenic impact resistance of the steel.

I. INTRODUCTION

In recent years a number of investigations [1-6] have studied the cryogenic mechanical properties of Fe-Mn alloys. This research shows that over the range of manganese content over which the alloys are essentially martensitic in the as-cooled conditions (6-12 wt %) the Fe-Mn binary alloys show excellent cryogenic strength. ~~This~~ cryogenic toughness is, however, poor; the alloys exhibit the characteristic ductile-brittle transition at temperatures near room temperature.

The composition range 8-12% Mn is of particular interest since alloys within this range exhibit the highest strengths, in the as-cooled condition, of the Fe-Mn binary system. As the Mn content is increased from 8 to 12% [2] the yield strength increases while the ductile-brittle transition temperature decreases slightly. The fracture mode below the ductile-brittle transition also changes, from predominantly quasi-cleavage to a strikingly intergranular fracture mode.[5,6] This composition range is also characterized by the first appearance of the hexagonal ε-martensite in the BCC (α') martensite matrix. In the as-quenched condition ε-martensite is first noticed at ∿10% Mn. The ε-martensite fraction increases to ∿15% by volume at 12% Mn and rapidly becomes the dominant phase at higher Mn contents. There is an interesting coincidence between the intrusion of the ε-phase and the shift to an intergranular fracture mode, though it has not yet been conclusively shown that the two phenomena are causally related.

Previous research in this laboratory has concentrated on the use of alloy processing to improve the cryogenic toughness of ferritic 8-12%Mn alloys.[5-7] In the case of the 12% Mn alloy it was found that the intergranular fracture at cryogenic temperatures could be partially

suppressed by tempering to introduce a distribution of retained austenite, and fully suppressed by a cold work-plus-temper [5,6] or controlled rolling treatment, [7] with a concomitant improvement in cryogenic toughness. Grain refinement, accomplished through thermal cycling, was shown to decrease the ductile-brittle transition temperature of 8% Mn alloys.[6,7]

The present paper reports a method for obtaining good cryogenic toughness in an Fe-12Mn-0.2Ti alloy in the as-cooled condition. The property improvement is obtained through a controlled cooling technique identified during the course of fundamental research on the origin of intergranular embrittlement in Fe-Mn alloys.

II. TECHNICAL APPROACH

The modes of fracture of Fe-8Mn, Fe-10Mn, and Fe-12Mn-0.2Ti in the as-cooled condition at -196°C are shown in Fig. 1. The 8Mn alloy fractures in a predominantly transgranular mode, the 10Mn alloy exhibits a mixed-mode fracture, and the 12Mn alloy fails in a strikingly brittle intergranular mode.

Given the appearance of the fracture surface in 12Mn alloys it is tempting to ascribe the embrittlement to a chemical segregation along prior austenite grain boundaries. However, the available evidence [7] strongly suggests that this is not the case. The Auger electron energy spectrum obtained from the intergranular fracture surface of a quenched Fe-12Mn-0.2Ti alloy is shown in Fig. 5(a). The spectrum is essentially identical to that obtained from transgranular surfaces (compare Fig. 5(b)) indicating the absence of significant grain boundary segregation. It is possible, though unlikely, that the embrittlement is due to a segregant too subtle to be detected by Auger spectroscopy. In this case the segregation would necessarily occur either during austenitization,

in which case it could be diminished by raising the austenitization temperature, or during cooling, in which case it could be suppressed by increasing the cooling rate. In fact, neither an increase in austenitization temperature nor an increase in cooling rate (to iced brine quenching of three specimens) affects the intergranular failure of Fe-12Mn-0.2Ti in any significant way.[7] We have therefore concluded that the intergranular embrittlement of Fe-12Mn is not due to chemical segregation.

An alternative possibility is that the embrittlement of Fe-12Mn is a consequence of the martensite transformation itself, through mechanisms analogous to those responsible for quench cracking in structural steels, but in this case complicated by the intrusion of the ε-martensite phase. If this hypothesis is correct it should be possible to suppress the intergranular embrittlement through controlled cooling to prevent the build-up of internal stresses along grain boundaries.

On the other hand Bolton[4] reported that some BCC Fe-Mn alloys were susceptible to tempered martensite embrittlement. Our data[7] confirm this. The increase in the ductile-brittle transition temperature of Fe-12Mn by heat treatment at 350°C is shown in Fig. 2. This embrittlement does appear to have a chemical origin whose nature is under investigation.

The assembled evidence therefore suggests that there are two independent intergranular embrittlement mechanisms operative in Fe-12Mn alloys. The first is a transformation-induced embrittlement which should be controlled by reducing the rate of cooling through the martensite transformation. The second is a tempered martensite embrittlement should be controllable by reducing the residence time of martensite at intermediate temperatures. This analysis in turn suggests that a step

cooling technique may be used to render both mechanisms inoperative, resulting in a tough Fe-12Mn alloy in the as-cooled condition. To test this possibility the following experiment was conducted.

III. EXPERIMENTAL PROCEDURE

Fe-12% Mn (by weight) and Fe-12Mn-0.2Ti alloys were induction melted in an argon gas atmosphere. Typical impurity levels were 0.001% C, 0.028% O, 0.007% S, 0.007% P, 0.010% Mo, 0.050% Ni, 0.002% Cr, 0.010% V, and 0.005% Nb. Each ingot underwent vacuum homogenization at 1200°C for 24 hours. The ingots were then upset cross-forged at 1100°C and finish-rolled into plates. Blanks of Fe-12Mn-0.2Ti alloy received a solution treatment at 1100°C for 2 hours. They were then furnace cooled for specific periods (therefore, to specific temperatures) followed by air cooling to room temperature. V-notched ASTM standard Charpy specimens were machined from the heat treated blanks, longitudinal to the rolling direction, and broken at -196°C. The results are shown in Fig. 3 with the corresponding cooling curves.

The specimens which were interrupted from the furnace cooling either too early or too late showed low impact energy. The fracture modes of these specimens were intergranular as shown in Fig. 4. Significantly, however, the specimens that were air cooled after 11∿12 hours of furnace cooling showed a ductile fracture mode with relatively high impact energy at -196°C. The temperatures of the furnace at these moments were in the range of 120∿100°C, which is close to the martensite finish temperature (∿120°C, by dilatometry) for this alloy. Another comparatively small increase of impact energy was observed in a specimen air cooled after 9 hours of furnace cooling. This datum may represent experimental scatter.

The AES spectra obtained from the Fe-12Mn-0.2Ti alloys subjected to three different modes of cooling are shown in Fig. 5. Each spectrum closely represents the chemistry of the fracture surfaces shown in Fig. 4. In the quenched specimen no significant segregation was observed on the denuded grain boundary facets. In the completely furnace cooled specimen some substructures were observed in the spectrum, including a nitrogen peak, although the intensities were not high.

IV. DISCUSSION

The experimental results show that it is possible to use a step cooling technique, consisting of a slow furnace cool through the martensite transformation followed by a more rapid air cool to room temperature, to suppress intergranular fracture in Fe-12Mn-0.2Ti and substantially improve cryogenic toughness.

The experimental data support the conclusion of a two-fold cause for grain boundary embrittlement in Fe-12Mn. The first type of the embrittlement is encountered when the alloy is rapidly cooled through the martensite transformation. It appears to have a mechanical origin associated with the phase transformation possibly similar to that cited for quench-cracking. The mechanism is, however, probably more complicated since the 12Mn alloy contains approximately 15 vol.% of HCP ε phase in the as-quenched structure. If $\gamma \rightarrow \varepsilon \rightarrow \alpha'$ [10,11] transformation route is operative the alloy would undergo a violent density change because ε is the densest phase. This may explain why the 12Mn alloy is susceptible to the intergranular fracture while the 8Mn alloy, which contains no ε phase, fractures transgranularly as shown in Fig. 1.

The second type of embrittling reaction is active below the $M_{f\alpha'}$ temperature, 120°C. This embrittlement phenomenologically resembles the

tempered martensite embrittlement described above (Fig. 2), which is presumed to have a chemical origin. Some evidence of grain boundary segregation is, in fact, apparent in the Auger spectrum shown in Fig. 5(c). On the other hand, the nature of this embrittlement is puzzling because of the very low temperatures involved (<120°C) and the modest segregation indicated by the Auger spectra. This phenomenon requires further study.

V. CONCLUSIONS

From the present investigation the following conclusions may be made:

1. The sources of brittle intergranular fracture in an Fe-12Mn-(0.2Ti) alloy is two-fold: non-chemical and chemical. The former is active during quenching from austenitizing treatment while the latter is operating in martensitic structure.
2. The two embrittling zones can be located in a continuous furnace cooling curve with respect to a critical temperature around 100∿120°C. This temperature coincides with $M_{f\alpha}$, temperature.
3. The embrittlements can be minimized by a stepwise cooling. This consists of a furnace cooling down to 100∿120°C followed by fast cooling to ambient temperature. By this method a high impact toughness can be obtained in as-cooled condition without intergranular brittleness.

ACKNOWLEDGMENT

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FIGURE CAPTIONS

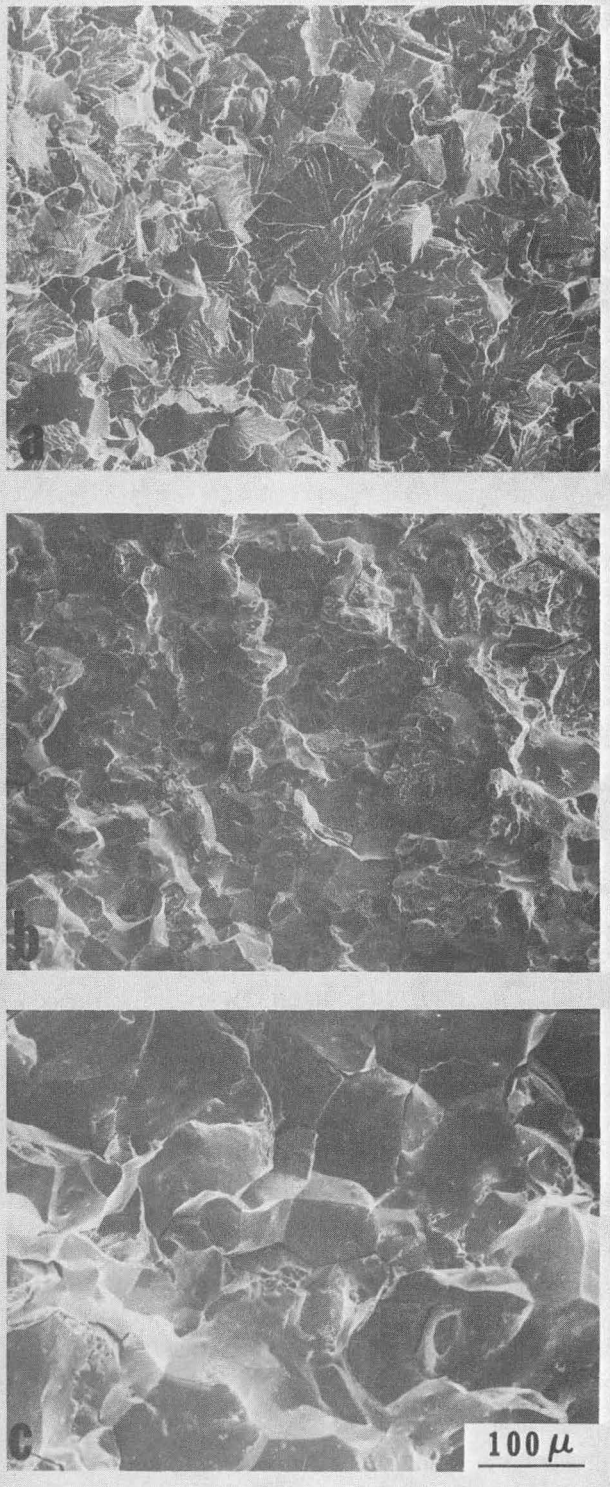
Fig. 1. Fracture modes of Fe-Mn alloys at -196°C. SEM
(a) Fe-8Mn. (b) Fe-10Mn. (c) Fe-12Mn-0.2Ti.
All solution annealed condition (900°C/2h/IBQ).

Fig. 2. Fe-12Mn. The increase of ductile-brittle transition temperature by martensite embrittling treatment (350°C/2h/WQ).
CVN: V-notched Charpy impact energy.

Fig. 3. Fe-12Mn-0.2Ti. The furnace cooling curve and Charpy impact energy measured at -196°C. Specimens were soaked at 1100°C for 2 hours followed by furnace cooling. They were then taken out from the furnace at specified time and temperature and air cooled.

Fig. 4. Left: Fe-12Mn-0.2Ti. SEM fractographs. After soaking treatment (1100°C/2h) specimens were furnace cooled for (a) 6 hours, (b) 11 hours, (c) 15 hours followed by air cooling.

Fig. 5. Right: Fe-12Mn-0.2Ti. AES spectra. After soaking treatment (1100°C/2h) specimens were (a) water quenched, (b) 12 hour furnace cooled/air cooled, (c) completely furnace cooled.



XBB 775-5057

Fig. 1

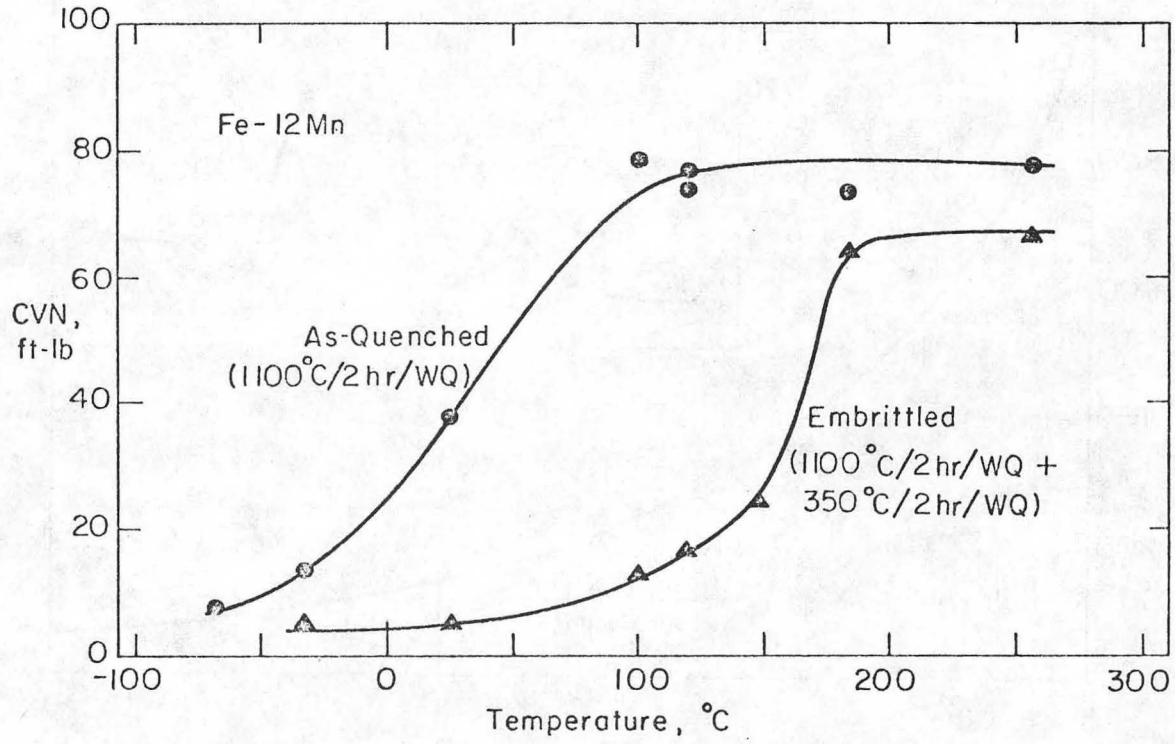

Fe-12Mn
As-Quenched
(1100°C/2hr/WQ)
Embrittled
(1100°C/2hr/WQ +
350°C/2hr/WQ)
CVN,
ft-lb
Temperature, °C
100
80
60
40
20
0
-100
0
100
200
300
XBL774-5344

Fig. 2

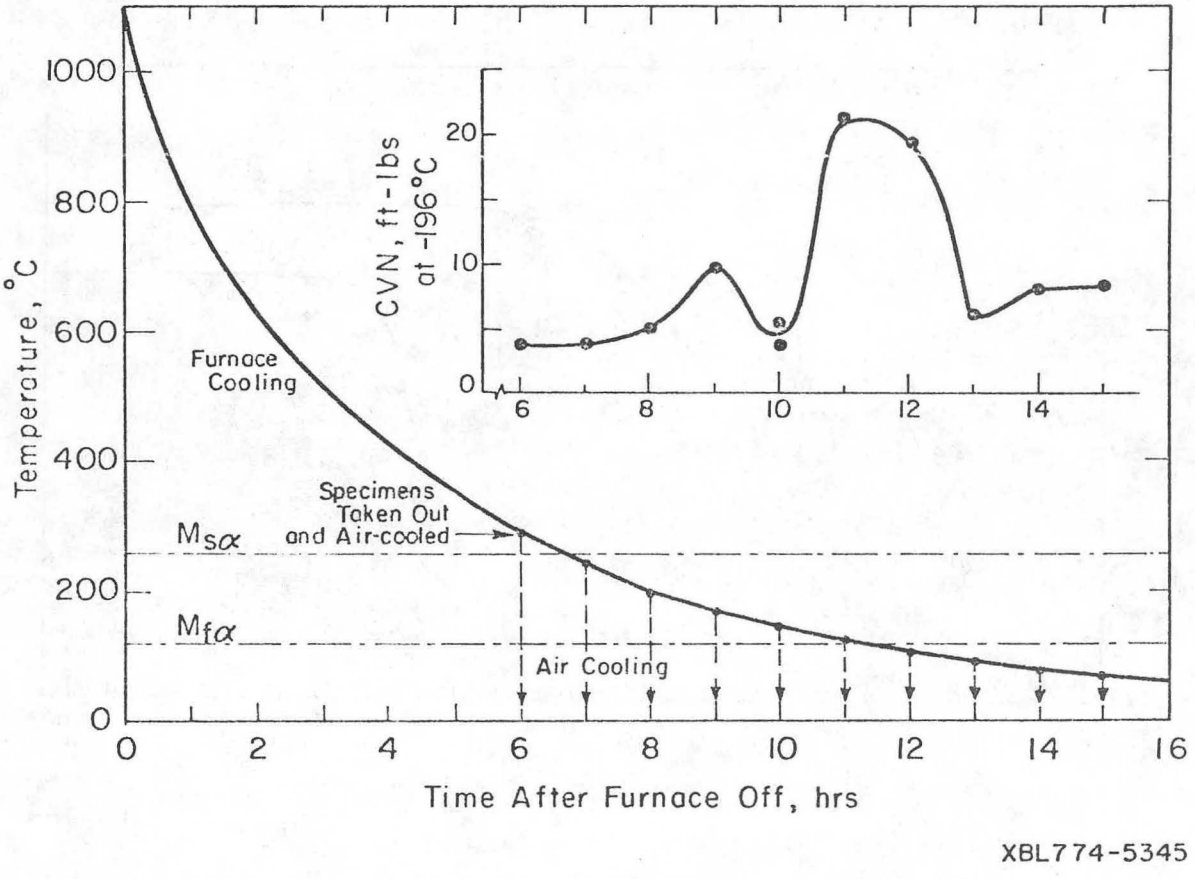
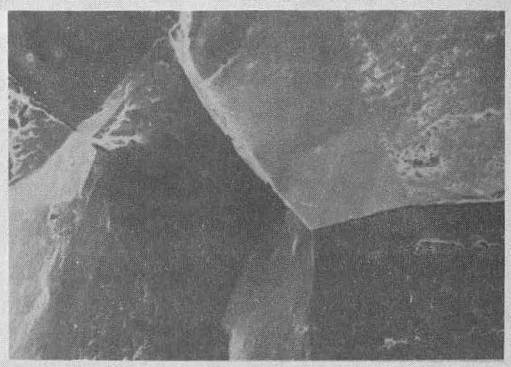
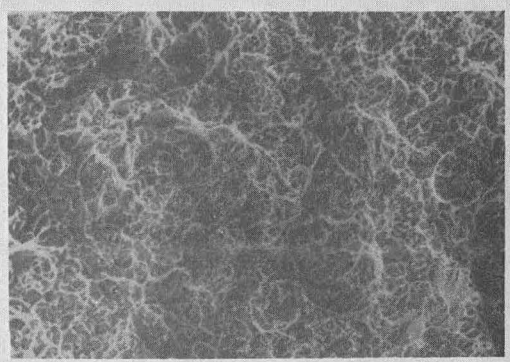

Temperature, °C
1000
800
600
400
200
0
Furnace Cooling
Specimens Taken Out and Air-cooled
$M_{s\alpha}$
$M_{f\alpha}$
Air Cooling
0
2
4
6
8
10
12
14
16
Time After Furnace Off, hrs
CVN, ft-lbs at -196°C
20
10
0
6
8
10
12
14
XBL774-5345

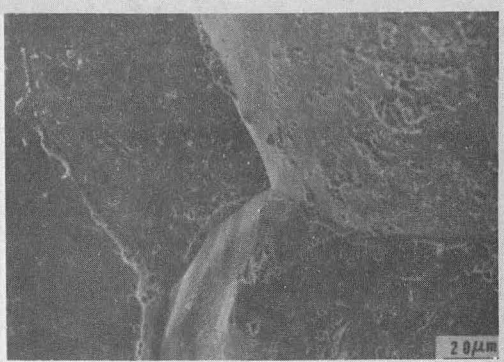
Fig. 3



a



b



c

Fig. 4

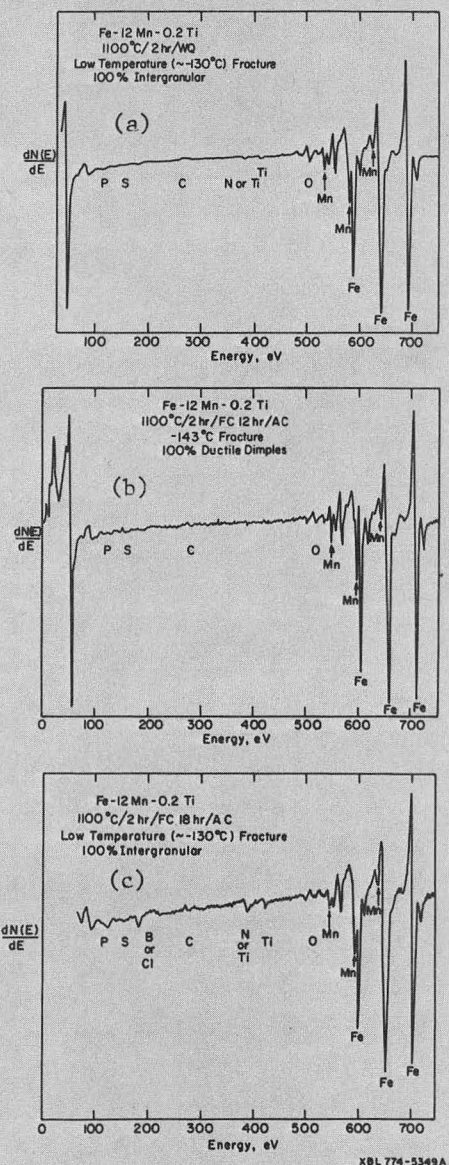


Fig. 5

XBB 777-6925

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