NICKEL-FREE Fe-12Mn-0.2Ti ALLOY STEEL FOR CRYOGENIC APPLICATIONS

by

S. Hwang, S. Jin, and J. W. Morris, Jr.

Department of Materials Science and Engineering and Materials and Molecular Research Division, Lawrence Berkeley Laboratory; University of California, Berkeley, California 94720 USA

ABSTRACT

A nickel-free Fe-12Mn-0.2Ti alloy steel was investigated for cryogenic applications. The systematic control of the grain size and the microstructural distribution of the retained γ phase was made through $\alpha+\gamma$ heat treatments in combination with mechanical working. Substantial improvements of important low temperature mechanical properties were obtained by these processings. Our results indicated a good possibility of developing a new, inexpensive cryogenic steel.

> This report was prepared as an extension of packparatesed by the Users Covernment, Making and Santa Covernment, Making and Santa Covernment, Making and Santa Covernment, Making and Santa Covernment, Santa Santa Santa Covernment, Santa Covernment, Santa Covernment, Santa Santa Covernment, Santa Cov

DISTRIBUTION OF THIS DOCUMENT IS UNLIMIT

I. INTRODUCTION

Recent advances in cryogenic devices for use in modern energy and transportation systems have created an increasing need for new alloys which retain good engineering properties at very low temperatures. Virtually all commercially available cryogenic steels contain a significant amount of nickel and/or chromium but these create economic difficulties for large quantity applications. The present paper deals with the design of a nickel-free, Fe-12Mn alloy steel for cryogenic purposes. The main approach to this goal is based on systematic characterization and control of the microstructure and phase distributions. By focussing particular attention on the retained austenite, a promising set of mechanical properties (yield stress 1179 MPa with K_{IC} 316 MNm^{-3/2}) were obtained at -196°C.

II. EXPERIMENTAL

Ingots of nominal composition Fe-12 wt.% Mn-0.2 wt.% Ti were melted under an argon gas atmosphere, homogenized at 1200°C for 24 hours under vacuum, and then upset cross-forged at 1100°C. Solution treatment was conducted at 900°C for 2 hours under argon gas flow followed by brine quenching. (Hereafter these specimens will be designated as AS.)

III. RESULTS AND DISCUSSION

The prior austenite (γ) grain size ranged from 50 to 70 µm. X-ray diffraction identified 15 vol. % of the ε (hcp) phase. By TEM (Transmission Electron Microscopy) study it was confirmed that the

-1-

substructure consisted of essentially dislocated α^{-} (martensite) plates and the blocky α^{-} morphology co-existing with the lenticular α^{-} form (1), the former phase being bound by ε platelets. (The latter point was confirmed by dark field analysis.) The yield stress of the AS specimens at -196°C was comparable to other commercial, ferritic Fe-Ni cryogenic steels (2). However, a sharp ductile-to-brittle transition was observed at -50°C in the standard Charpy test. Below the transition temperature (T_r) the fracture mode was 100% intergranular.

A preliminary study to suppress T_c through grain refinement was carried out by a thermal cycling technique (3,4). While this technique was extremely effective in the ferritic Fe-Ni systems, it was unsuccessful in Fe-12Nn primar-ly due to the $e^{x}\gamma$ reverse transformation. The next approach was to directly utilize the strong γ stabilization ability of manganese.

2. Simple Two-Phase $(\alpha+\gamma)$ Treatment: Retained γ Phase

While the beneficial effect of the retained γ has been demonstrated in many Fe-Ni systems (4-7), no serious attempt appears in the literature to exploit this effect in the Fe-Mn alloy. The retained α can be introduced into the Fe-12Mn system in an analogous way to the ferritic Fe-Ni systems; α is held in the $\alpha+\gamma$ temperature range so that the diffusion-controlled $\alpha^{+}+\alpha^{+}+\alpha^{+}\gamma$ transformation occurs. The relative amounts of resultant phases at -196°C after 4 hour heat treatments are shown in Fig. 1 as a function of temperature. Inserted are the corresponding CVN (Charpy impact energy) at -196°C. The coincidence of the CVN peak and the retained γ peak is quite obvious. The stability of

-2-

the reverted γ is indirectly inferred from the rapid increase of the slope of the ε curve above 550°C, which indicates decomposition of the reverted γ to ε during cooling. Isothermal study at 500°C (optimum for γ stability) again showed good correlation between CVN and the retained γ . The 500°C/10 hr. treatment appeared to be optimal for the CVN. The cryogenic mechanical properties were measured for this treatment. As listed in Table I, every property measured was improved. T_c decreased by 100°C, K_{IC} at -196°C increased by 28 MNm^{-3/2} and uniform elongation at -196°C increased by 7% with a simultaneous slight increase in the yield stress. The fraction of the intergranular fraction mode at -196°C was cut in half.

Although the simple $\alpha + \gamma$ treatment results in the improvement of various cryogenic mechanical properties, the extent of the improvement may not be sufficient for practical applications. Therefore our efforts were next directed towards controlling the size and distribution of the phases.

3. Finer Grain Size, Uniform y Distribution: Cold Working

The effectiveness of previous cold working on the final $\alpha+\gamma$ processed structure has been demonstrated by Miller (8). Cold rolling should be significantly more effective in the Fe-12Mn system because of the ε phase intrusion along the α' boundaries. The cold working not only increases the driving force for nucleation but also removes the undesirable $\varepsilon \rightarrow \gamma$ continuous transformation (9), which is the source of the severe directionality of the resultant γ (see Fig. 2). A typical microstructure resulting from the cold rolling (at room temperature)

-3-

plus α + γ treatment is shown in Fig. 3. The extreme fineness ($\sim 0.5 \text{ µm}$) and uniformity of the phases are evident. Furthermore, the phase analysis (Fig. 4) characterized a noticeable increase in stability of the reverted γ against the γ + ε reaction (compare with Fig. 1). Again the correlation between the CVN at -196°C and the retained γ content is excellent. The cryogenic mechanical properties evaluated for a particular processing (50% reduction plus 600°C/4 hrs.) showed substantial improvements over the AS state. As Table I shows, an extremely high yield stress (1179 MPa) with an attractive K_{IC} value (316 MNm^{-3/2}) and CVN (55 ft-1b) were obtained at -196°C. As illustrated in Fig. 5, T_c was suppressed very close to -196°C (half upper-shelf energy criterion).

A preliminary study on the grain boundary strengthening by molybdenum modification was conducted with an Fe-12Mn-0.2Ti-1Mo composition. While a set of ingots showed an extensive suppression of T_c (below -150°C for AS) as well as excellent K_{IC} (450 MNm^{-3/2} at -196°C for processed specimens), another set showed little improvement. The search for the origin of this scatter is underway.

To summarize, a quite promising combination of cryogenic strength and toughness can be obtained in the Fe-12Mn-0.2Ti steel alloy through control of the grain size and the distribution of the retained austenite. Molybdenum addition might further improve these properties so that a nickel-free, cryogenic Fe-12Mn steel can actually be used for practical applications in the near future.

In Provide State

-4-

ACKNOWLEDGMENT

The support of the National Aeronautics and Space Administration, Lewis Research Center under Contract No. NASA-NGR-05-003-562 and of the Energy and Research Development Administration through the Molecular and Materials Research Division of the Lawrence Berkeley Laboratory are gratefully acknowledged.

REFERENCES

- Bolton, J. D., Petty, E. R. and Allen, G. B., Met. Trans., vol. 2, 2915 (1971).
- Tobler, R. L., Mikesell, R. P., Durcholz, R. L., and Reed, R. P., ASTM STP 579, 261 (1975).
- Jin, S., Morris, J. W., Jr., and Zackay, V. F., Met. Trans. A, vol. 6A, 141 (1975).
- Jin, S., Hwang, S. K., and Morris, J. W., Jr., Met. Trans. A, vol. 6A, 1721 (1975).
- Hwang, S. K., Jin, S., and Morris, J. W., Jr., Met. Trans. A, vol. 6A, 2015 (1975).
- Marschall, C. W., Heheman, R. F., and Troiano, A. R., Trans. ASM, vol. 55, 135 (1962).
- Antolovich, S. D., Saxena, A., and Chanani, G. R., Met. Trans., vol. 5, 623 (1974).
- 8. Miller, R. L., Met. Trans., vol. 3, 905 (1972).
- 9. Parr, J. Gordon, J. Iron and Steel Inst. B, vol. 283, 137 (1952).

| | | Yield Stress (MPa) | Ultimate Stress (MPa) | Uniform Elong. (%) | Total Elong. (%) | Reduction in Area (%) | ĸıc |
|---------------------------|---|--------------------------|-----------------------------|--------------------------|------------------------|-----------------------------|-----|
| AS | • | 883 | 1351 | 11 | 25 | 54 | 194 |
| 500°C/10 hr. | | 952 | 1358 | 18 | 33 | 62 | 222 |
| C.W. (50%)+600°C/4h. 1179 | | 1503 | 26 | 38 | 66 | 316 | |

Table I. Mechanical Properties of Fe-12Mn-0.2Ti Steel at -196°C

nav y constraint anticia

a fregerige

.

.

.

Contraction and the

- 【な破綻集】 低い日本 (成本) 「「「」」 「「」」 「」」 「」」 「」」 「」」 「」」 「」」

.

· .

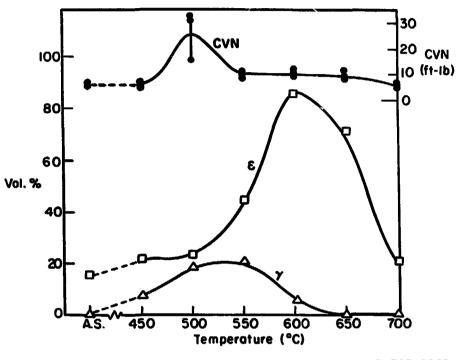
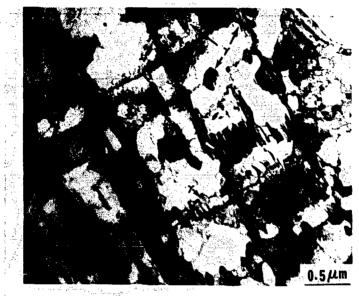




Fig. 1. Phases and CVN at -196°C after 4 hr. treatments at temperatures.

.

.



XBB 762-1740

Fig. 2. Reverted γ phase after 10 hours at 500°C (TEM). (XBB 762-1740 bottom)

Ċ

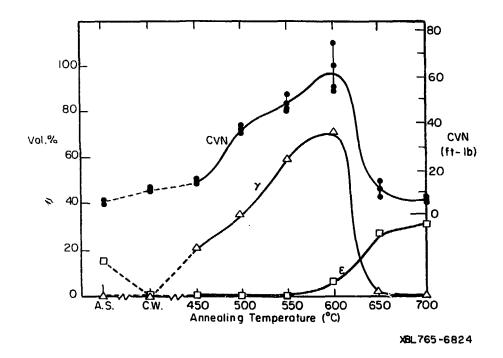


Fig. 4. Phases and CVN at -196°C after 50% reduction and annealed 4 hours at temperatures.

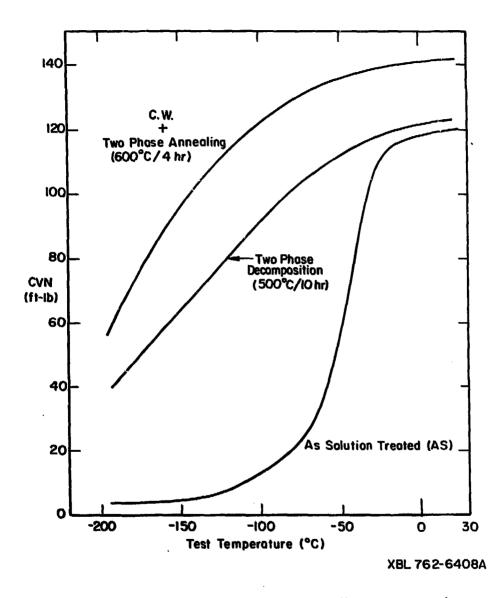


Fig. 5. CVN measured at various temperatures for different processings.