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RECEIVED BY TIC APR 4 1977

Presented at the USSR-US Seminar on
Applied Problems of Low-Temperature
Materials and the Manufacture of
Welded Cryogenic Structures, Kiev,
USSR, October 12 - 14, 1976,
E. O. Paton Inst. of Electro-Welding

LBL-5767

Conf-761085--2

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December 1976

Prepared for the U. S. Energy Research and
Development Administration under Contract W-7405-ENG-48

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THE USE OF MICROSTRUCTURE CONTROL TO TOUGHEN FERRITIC STEELS FOR CRYOGENIC USE. I. Fe-Ni STEELS

by

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ABSTRACT

Alternation of austenitization and austenite + ferrite two-phase decomposition treatment in a cyclic thermal treatment allows the achievement of ultra-fine grain size in steels containing 8-12% Ni. The grain refinement leads to a substantial improvement in cryogenic mechanical properties. The ductile-brittle transition temperature of a ferritic Fe-12Ni-0.25Ti alloy was suppressed to below liquid helium temperature by this grain refinement procedure; the transition temperature of commercial "9Ni" cryogenic steel was similarly reduced by combining the grain refinement with a final temper which introduces a small admixture of retained austenite.

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I. INTRODUCTION

The quest for a strong ferritic steel which remains tough to liquid helium temperature is made difficult by the ductile-brittle transition behavior common to materials of BCC crystal structure. The most promising of the available ferritic cryogenic alloys, Fe-Ni base alloys of intermediate nickel content, exhibit a ductile-brittle transition near liquid nitrogen temperature (77°K). The transition temperature is, however, known to decrease as the grain size of the steel is made smaller. The available evidence also suggests that the transition temperature may be lowered by incorporating a small amount of retained austenite into the ferritic steel. In the research reviewed here these two techniques were exploited to suppress the transition temperature of Fe-12Ni and Fe-9Ni alloys to below 4-6°K.

II. GRAIN REFINEMENT THROUGH THERMAL CYCLING

Research by Porter and Dabkowski(1) and by Saul, Robertson and Adair(2) demonstrated that steels of intermediate nickel content can be grain refined through cyclic use of the martensite reversion reaction. The grain refinement accomplished by the $\alpha' \rightarrow \gamma \rightarrow \alpha'$ cycle is presumably due to two factors: enhanced martensite nucleation at defects introduced into the austenite by shear reversion and refinement of the martensite packet size to relieve internal stress in the reverted γ. A single $\alpha' \rightarrow \gamma \rightarrow \alpha'$ cycle accomplishes an appreciable grain refinement in an Fe-12Ni alloy.(3) Repetition of the cycle refines the grain size further. Succeeding cycles are, however, less efficient. The grain size of an Fe-12Ni alloy appears to stabilize at ~10 μm mean grain diameter

(ASTM #11) after 3 to 4 cycles. The saturation of the grain refinement achieved through repetition of the $\alpha' \rightarrow \gamma \rightarrow \alpha'$ cycle is qualitatively similar to the saturation of transformation-induced strengthening in cyclically reverted austenite(4) and would appear to arise from the same cause: a saturation of the transformation-induced defect structure in reverted austenite. It is hence unlikely that ultrafine grain sizes can be obtained in this way.

Ultrafine grain size can, however, be achieved in Fe-Ni alloys through a technique employed by Miller(5) in which the alloy is severely cold-worked, then decomposed by annealing in the two-phase $(\alpha + \gamma)$ range. The grain refinement mechanism in this instance is the fine-scale nucleation of γ in the boundaries of the martensite lathes, which have been severely deformed during the working operation. Mean grain diameters less than 1 μm (ASTM #17-19) were obtained by this technique.

The research reported here sought a thermal cycling treatment to establish a grain size comparable to that achieved through mechanical processing. The approach taken was to alternate the familiar $\alpha' \rightarrow \gamma \rightarrow \alpha'$ reversion cycle with a two-phase decomposition at a temperature just below the A_s temperature at which the shear reversion to austenite begins. The treatment employed is shown schematically in Fig. 1, and the evolution of microstructure during the processing of an Fe-12Ni-0.25Ti research alloy is illustrated by the series of micrographs in Fig. 2. An ultrafine structure of non-aligned grains of mean diameter ∿1 μm (ASTM #17) results.

A detailed discussion of this grain refinement procedure is given elsewhere.(6) An examination of the microstructural evolution during the cycling procedure has been undertaken to determine the controlling mechanisms of the grain refinement.(7) Its essential features are, however, evident in Figs. 1 and 2. The initial, annealed structure has a grain size of ∿40 μm. This is reduced to ∿10 μm by an $\alpha' \rightarrow \gamma \rightarrow \alpha'$ reversion cycle, giving the structure 1A. The 1A structure is decomposed by a nucleation and growth process at 650°C in which austenite grains form in the prior austenite grain boundaries and along the martensite lath boundaries to yield a fine structure of lath-like aligned grains (1B) while the martensitic matrix undergoes an extensive recovery by cell wall formation which decomposes the martensite lath with an aggregate of subgrains. The preferential alignment of these grains is largely broken up by a second austenite reversion cycle with a short anneal at 730°C (2A). The grain refinement is completed through a final two-phase decomposition at 650°C (2B). After cooling to room temperature the alloy consists of fine grains of dislocated martensite. No residual austenite is detected by X-ray diffraction or transmission electron microscopy.

The grain refinement has a marked beneficial effect on the cryogenic mechanical properties of the Fe-12Ni-0.25Ti alloy.(8) In the 2B condition the alloy exhibits an outstanding combination of strength and toughness in liquid helium, as shown by the data presented in Table I, where the cryogenic tensile and toughness properties of grain-refined Fe-12Ni-0.25Ti (2B) are compared to those of the alloy in the unrefined condition (1A) and to those of commercially processed "9Ni" and 304 stainless steels. The fracture mode of the grain-refined alloy in liquid helium is essentially pure ductile rupture, as illustrated by the comparative fractographs in Fig. 3.

III. TOUGHENING CRYOGENIC ALLOYS OF LOWER NICKEL CONTENT

Alloys of lower nickel content may be processed to ultrafine grain size through a thermal cycling procedure essentially the same as that used for the 12Ni alloy, adjusting the treatment temperatures to reflect the shift in the martensite reversion temperatures (A_s and A_f) with composition. This procedure has been successfully used to form ultrafine grained microstructures in commercial "9Ni" steel(9) and in an 8Ni research alloy.(10) In both cases grain refinement leads to a substantial decrease in the ductile-brittle transition temperature (e.g. Fig. 5), but this temperature remains above 4°K. The transition temperature may, however, be lowered further by adding a final tempering treatment in the lower portion of the $(\alpha + \gamma)$ field, which has the consequence of introducing a fine distribution of retained austenite in the grain boundaries and martensite lath boundaries. Such a temper is commonly used in the commercial processing of ferritic cryogenic steels (e.g., ASTM A353-72).

Commercial "9Ni" steel (Nippon 9Ni-0.6Mn-0.06C-0.25Si) was grain-refined using the same treatment diagrammed in Fig. 1 but with one hour holding time at each step, and given a final temper for one hour at 575°C.(9) The temper introduces a significant fraction (~10-15%) of retained austenite (Fig. 4) which is thermally and mechanically quite stable at temperature down to 4°K. The reprocessed alloy has an excellent combination of strength and toughness in liquid helium (Table II), and does not exhibit the transition behavior observed in the conventionally processed alloy.

A similar treatment applied to an Fe-8Ni-2Mn-0.25Ti research alloy(10) gave ambiguous results. The introduction of ∿5% retained austenite (treatment 2Br in Fig. 5) into the grain-refined alloy (treatment 2B in Fig. 5) suppressed the ductile-brittle transition temperature, as measured by Charpy impact test, from ∿-118°C to below liquid helium temperature, -267°C. However, the transition temperature measured by the fracture toughness test is not significantly reduced by the presence of austenite and remains well above liquid helium temperature as shown in Fig. 5. This anomalous effect of austenite is now under investigation.

ACKNOWLEDGMENTS

This research was supported by the Office of Naval Research under Contract N00014-75-C-0154 and by the Energy Research and Development Administration through the Materials and Molecular Research Division of the Lawrence Berkeley Laboratory.

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Table I. Tensile and Toughness Properties at 6°K

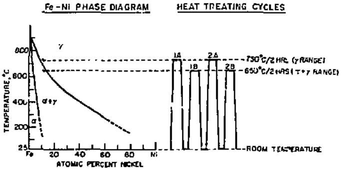
Alloy	Treatment	YS		TS		C_v		K_{IC}	
		(ksi)	(MPa)	(ksi)	(MPa)	(ft-lb)	(Kgfm/cm^2)	(ksi$\sqrt{in}$)	(MPa$\sqrt{m}$)
Fe-12Ni-0.25Ti	2B	195	1346	219	1511	99	17.1	232*	255*
Fe-12Ni-0.25Ti	1A	182	1256	207	1428	55	9.5	75	83
9Ni	ASTM A353-72	189	1304	204	1408	75	13.0	72	79
304	As Quenched	105	726	270	1863	128	22.1	168*	185*

*Estimated from Equivalent Energy(11) as described in Ref. 8.

Table II. Tensile and Toughness Properties of 9Ni Steel at 6°K

Treatment	YS		TS		C_v		K_{IC}	
	(ksi)	(MPa)	(ksi)	(MPa)	(ft-lb)	(Kgfm/cm^2)	(ksi$\sqrt{in}$)	(MPa$\sqrt{m}$)
ASTM A353-72	189	1304	204	1408	75	13.0	72	79.2
2B + 575°C, 1 hr.	191	1318	223	1539	117	20.2	166*	183*

*Estimated from equivalent energy, as described in Ref. 9.



XBL 739-1684

Fig. 1. The thermal cycling procedure.

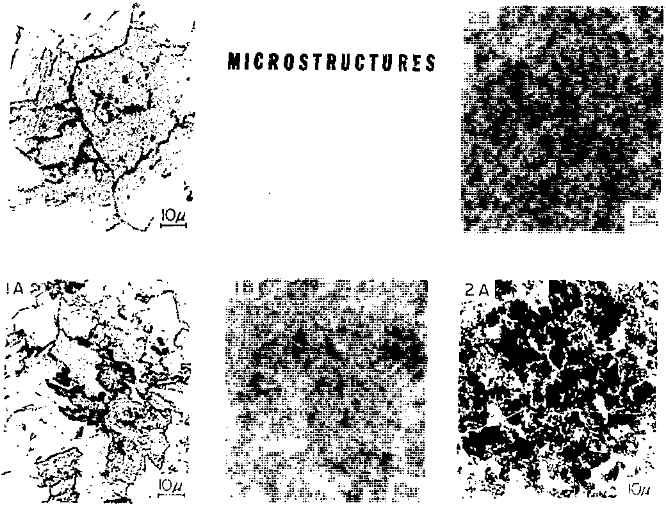


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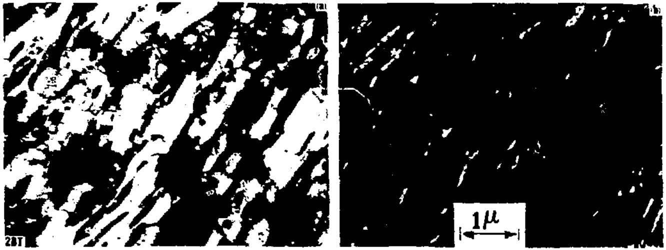
Fig. 2. The evolution of microstructure during thermal cycling.

(XBB 739-5684)



XBB 762-1123

Fig. 4. Retained Austenite in 9Ni Steel (2BT)

(a) Bright Field TEM.

(b) Dark field TEM from $(200)_\gamma$ peak.

(XBB 762-1123 (top))