JOURNAL OF MATERIALS SCIENCE, 32 (1997) 1201–1205

Effect of recrystallization on environmental embrittlement of a Ni_3Al-based alloy

JIAN-QING SU, SHU-JUN GAO, BING-XI YU, JIANG-TING GUO
Institute of Metal Research, Academia Sinica, Liaoning Key Laboratory for Materials and Hydrogen

ZHUANG-QI-HU
National Key Laboratory for RSA, Institute of Metal Research, Academia Sinica, Shenyang 110015, People's Republic of China

The effects of recrystallization on room temperature tensile properties and susceptibility to test environments (air versus vacuum) of a Ni_3Al-based alloy have been investigated. The results indicate that recrystallization treatment not only increases ductility but also reduces susceptibility to test environment of the alloy. The fracture mode has also been observed in the scanning electron microscope (SEM). It was found that air promoted transgranular cleavage fracture and reduced the amount of ductile fracture for unrecrystallized and partially recrystallized microstructures. In completely recrystallized specimens, the ductile fracture mode remained unchanged when test environment changed from vacuum to air.

1. Introduction

Intermetallic compounds have long been the subject of considerable interest for high temperature applications. In particular, Ni_3Al is one of the more promising intermetallic compounds due to its anomalous dependence of strength on temperature, good oxidation resistance and lower density compared to superalloys [1–4]. However, it has a propensity to brittle intergranular fracture in polycrystalline forms under normal conditions. Recent studies by Liu *et al.* [5–7] have shown that low ductility and brittle fracture in Ni_3Al are caused not only by intrinsic factors but also by extrinsic factors. Environmental degradation, an extrinsic factor, is found to be major cause of brittle fracture in many ordered intermetallics including Ni_3Al.

Much effort has been devoted to finding out the ways of improving the ductility of intermetallics. Aoki and Izumi [8] found that the addition of a small amount of boron to polycrystalline Ni_3Al significantly improves the intrinsic ductility and changes the fracture mode from intergranular to completely transgranular fracture. It was reported that a moderate amount of boron can also eliminate the environmental embrittlement of Ni_3Al. Masahashi *et al.* [4] showed that the room temperature tensile ductility of Ni_3Al doped with 0.05 wt % B is insensitive to test environment (air versus vacuum), although Ni_3Al doped with a low level of boron (0.012 wt % B) is susceptible to environmental embrittlement (10.1% in air versus 25.5% in vacuum), as shown by Wan *et al.* (9).

Heat treatment might be another way to improve the intrinsic ductility and reduce the environmental embrittlement of intermetallics. McKamey and Pierce [10] studied the effect of recrystallization on room temperature tensile properties of an Fe_3Al-based alloy and found that the best tensile strength and ductility were attained in specimens which had been heat-treated to relieve stresses produced by the fabrication process but that had a minimum number of recrystallized grains. For Ni_3Al the influences of recrystallization upon its tensile properties have not been reported yet.

The purposes of the present study are to investigate effects of the degree of recrystallization on room temperature tensile properties of the Ni_3Al-based alloy in both vacuum and air, and to determine whether the heat treatment is an effective method for improving the intrinsic ductility and reducing the environmental embrittlement in the ordered intermetallics.

2. Experimental procedures

The alloy used for this investigation was Ni–16.5Al–8Cr–0.8Hf–0.1B–0.03Y (at %). It was melted in a vacuum induction furnace and subsequently cast into ingots with the dimension $12 \times 25 \times 250$ mm. The cast ingots were cold-rolled five times to 2 mm thick with an intermediate anneal for 2 h at 1000 °C between every two cold-rolls; the final cold-rolled reduction was 37%.

To obtain the different recrystallized microstructure, the cold-rolled sheets were annealed for one hour at different temperatures, followed by oil quenching. Four anneal temperatures were selected, i.e. 850, 950, 1050 and 1150 °C for four sets of specimens (designated as A, B, C and D, respectively). Specimens A had a stress-relieved but unrecrystallized microstructure.

0022–2461

TABLE I Effect of recrystallization on room temperature tensile properties of the Ni_3Al-based alloy

Heat treatment (h/°C)	f_r^a (%)	Test environment	Yield (MPa)	Ult. (MPa)	Elong. (%)	I^b (%)
1/850 + 2/500	0	Vacuum	916	1552	13.9	15.3
		Air	908	1550	11.7	
1/950 + 2/500	20	Vacuum	766	1489	17.4	8.0
		Air	760	1462	16.0	
1/1050 + 2/500	50	Vacuum	695	1430	19.4	5.2
		Air	690	1415	18.4	
1/1150 + 2/500	100	Vacuum	630	1518	32.2	0.3
		Air	628	1526	32.1	

[a] f_r recrystallized fraction.
[b] I embrittlement index.

Partial recrystallization was produced in specimens B and C, while specimens D exhibited a completely recrystallized microstructure.

It is known that boron has a strong tendency to segregate to the grain boundaries, and that boron significantly influences the environmental embrittlement of Ni_3Al. To isolate the effect of the degree of recrystallization on environmental embrittlement, therefore, keeping the segregation amount of boron constant in all the test specimens is necessary. According to the data in literature [11, 12], the effect of heat history on the segregation of boron in Ni_3Al was reversible and the maximum equilibrium segregation of boron in Ni_3Al was reached by annealing for 100 min at 500 °C. Thus, in this study, a final heat treatment of annealing for 2 h at 500 °C followed by oil quenching was performed for all the test specimens. The tensile specimens with a guage section of $15 \times 3 \times 2$ mm were prepared after the final annealing. The longitudinal direction of specimens was parallel to the rolling direction. The room temperature tensile tests with strain rate of $5 \times 10^{-4}\ s^{-1}$ were performed on a Gleeble 1500 machine equipped with a vacuum chamber. Environment effects were investigated under two conditions: vacuum of about 6.6×10^{-3} Pa and air with the relative humidity of 57% at 15 °C. Fracture surfaces were examined using a scanning electron microscope operated at 20 kV. The microstructures were studied using optical metallography techniques. The recrystallized fraction was estimated by visual inspection.

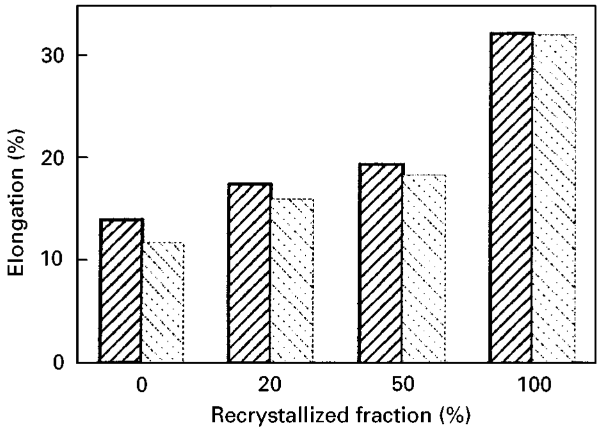

Figure 1 Variation of elongation with recrystallized fraction. (▨) vacuum; (▤) air.

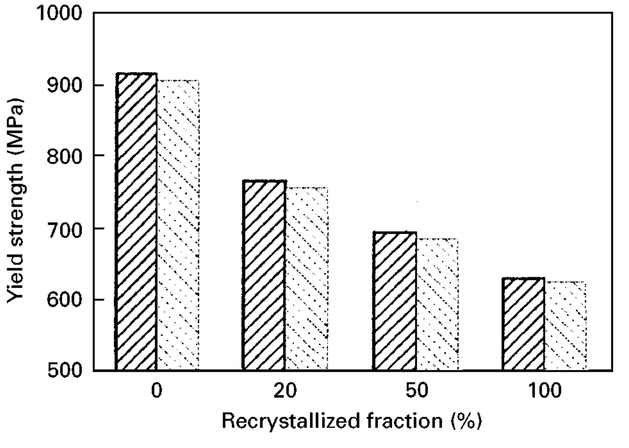

Figure 2 Variation of yield strength with recrystallized fraction. (▨) vacuum; (▤) air.

3. Results and discussion

3.1. Tensile properties

Mechanical properties of Ni–16.5Al–8Cr–0.8 Hf–0.1B–0.03Y alloy were determined by tensile tests at room temperature in vacuum and air. Table I summarizes the tensile results and indicates the recrystallized fraction of the corresponding microstructures. Each data point was an average of three tests. As shown in Table I the ductility of the alloy tested both in vacuum and in air increases with recrystallized degree. The unrecrystallized microstructures possess the lowest elongation, while the completely recrystallized microstructures produce the highest ductility. The variation of elongation with the recrystallized degree is illustrated in Fig. 1.

On the other hand, the yield strength of alloy decreases with the increase in recrystallized degree. The maximum yield strength is attained for the unrecrystallized material, while the completely recrystallized material possesses the lowest yield strength, as shown in Fig. 2.

Unrecrystallized microstructures possess the highest yield strength and the lowest ductility can attribute to intrinsic effects of strain-hardening arising from an increased dislocation density in the matrix due to the extrinsic influence of cold deformation. The course of

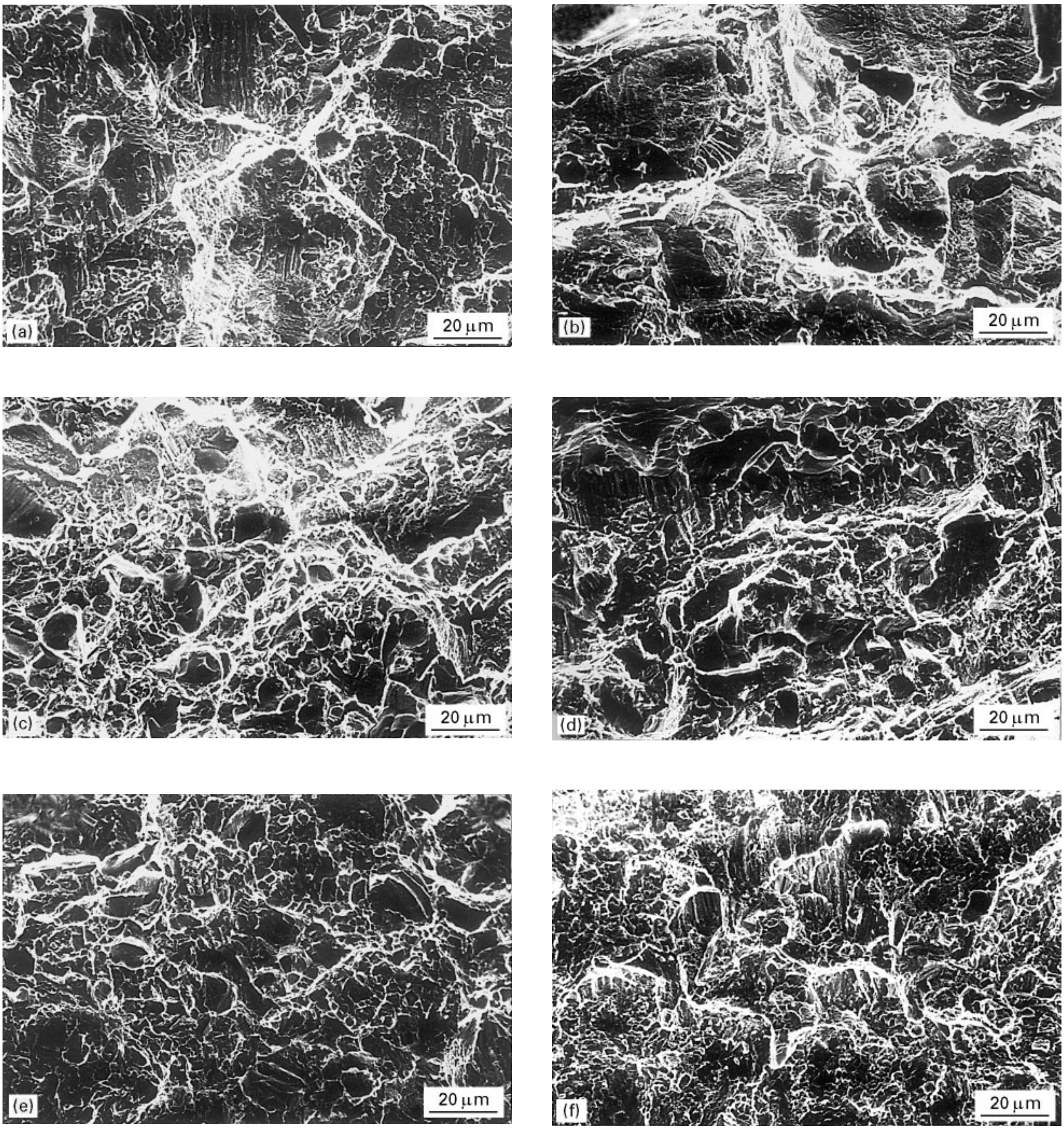

Figure 3 Fractographs of specimens A(a,b,1 h/ 850 °C + 2 h/500 °C), B(c,d,1 h/950 °C + 2h/500 °C) and D(e,f,1 h/1150 °C + 2 h/500 °C) tested at room temperature in (a,c,e) vacuum and (b,d,f) air.

recrystallization relief of the intrinsic effects of strain hardening. A reduction or elimination of strain-hardening effects results in degradation in yield strength with a resultant improvement in ductility.

3.2. Fracture behaviour

Scanning electron microscope (SEM) examination of fracture surfaces revealed that the fracture mode is dependent on both recrystallization degree and test environment. As shown in Fig. 3, the specimens with non-recrystallization microstructure, when tested in vacuum, exhibited a mixed fracture mode of transgranular cleavage fracture and transgranular ductile fracture (Fig. 3a). With increasing the degree of recrystallization, transgranular cleavage fraction decrease and transgranular ductile fracture increase (Fig. 3c), the completely recrystallized specimens show mainly transgranular ductile fracture mode (Fig. 3e). As to the effect of test environment, it was found that air promotes transgranular cleavage fracture and reduces the amount of ductile fracture for both unrecrystallized and partially recrystallized microstructure (Fig. 3b, d) except the completely recrystallized specimens, in which the ductile fracture mode remains unchanged when test environment changes from vacuum to air (Fig. 3e, f). It can be considered as one type of hydrogen embrittlement and a decrease in susceptibility of the alloy to the hydrogen embrittlement due to recrystallization, as discussed later.

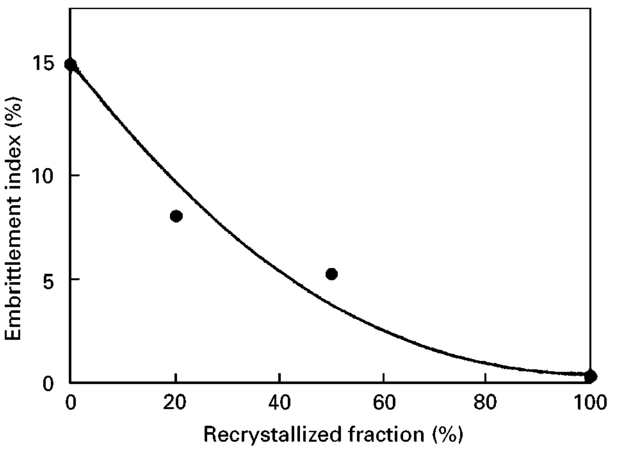

Figure 4 Effect of recrystallization on embrittlement index.

3.3. Effect of environment

The experimental results indicate that the ductility of the alloy is reduced by air (versus vacuum), in other words, the alloy used in this study also exhibits the susceptibility to environment embrittlement like many other Ni_3Al-based intermetallics. The extent of susceptibility to environmental embrittlement can be described with the embrittlement index I

$$I = (\delta_v - \delta_A)/\delta_v \quad (1)$$

Where δ_v and δ_A are the elongations in vacuum and air, respectively. The effect of recrystallization degree on the embrittlement index is shown in Fig. 4.

Recent studies have revealed that a lot of ordered intermetallics containing aluminium are very susceptible to environmental embrittlement. The phenomenon of environmental embrittlement in aluminides [13–15], as well as in aluminium- based alloy [16, 17], is explained by the following chemical reaction

$$2Al + 3H_2O \rightarrow Al_2O_3 + 6H^+ + 6e^- \quad (2)$$

Aluminium atoms in the aluminides react with moisture in air, resulting in the generation of atomic hydrogen that causes embrittlement. Because Ni_3Al-based alloy also contains the reactive element Al, we believe that the above reaction should occur in this alloy.

The effects of recrystallization on environmental embrittlement could be explained in terms of this mechanism and the hydrogen trap concept as follows. In unrecrystallized specimens, a large number of defects (such as dislocations, vacancies and grain boundaries) produced by cold work deformation still exists even after stress-relieving treatment. Because these defects are all the traps for hydrogen, atomic hydrogen generated by chemical reaction 2 would be distributed over grain (including both grain interior and grain boundary). The hydrogen atoms inside the grain weaken the cohesive strength of cleavage plane and result in transgranular cleavage fracture. The hydrogen atoms in grain boundaries also weaken the cohesive strength of grain boundary, but boron segregated to grain boundary will compensate for the loss of the cohesive strength, because boron can strengthen the grain boundary cohesive [18]. Thus, the specimens with unrecrystallized microstructure, when tested in air, would exhibit more transgranular cleavage fracture.

In recrystallized specimens, the defects inside grain decrease with increasing the degree of recrystallization, hence, the quantity of hydrogen trapped by the defects also decreases. This will result in a decrease in the susceptibility to environmental embrittlement. Therefore, the unrecrystallized microstructure would be of the most susceptible to test environment. In completely recrystallized specimens, the amount of trapped hydrogen inside the grain is so small that the alloy is insensitive to test environment and exhibits a mainly ductile fracture.

4. Conclusions

1. The ductility of the Ni_3Al-based alloy tested at room temperature both in vacuum and in air increased with the increase in the degree of recrystallization. Unrecrystallized and partially recrystallized microstructure were susceptible to test environment. Increasing the recrystallization degree resulted in a decrease in susceptibility to environmental embrittlement. The complete recrystallization can fully eliminate this susceptibility.
2. Air promoted transgranular cleavage fracture and reduced the amount of ductile fracture for unrecrystallized and partially recrystallized microstructure. In completely recrystallized specimens, the ductile fracture mode remained unchanged when test environment changed from vacuum to air.
3. The recrystallization treatment is an effective method for increasing the intrinsic ductility and reducing the environmental embrittlement for the Ni_3Al-based intermetallics.

Acknowledgements

The authors wish to thank the National Natural Science Foundation of China and the National Advanced Materials Committee of China for financial support.

References

1. J. A. HORTON, C. T. LIU and M. SANTELLA, *Metall. Trans.* **18A** (1987) 1265.
2. C. LALL, S. CHIN and D. P. POPE. *ibid.* **10A** (1979) 1323.
3. M. TAKEYAMA and C. T. LIU, *Acta Metall.* **36** (1988) 1241.
4. N. MASAHASHI, T. TAKASUGI and D. IZUMI, *ibid.* **36** (1988) 1823.
5. C. T. LIU, *Scripta Metall. Mater.* **27** (1992) 25.
6. E. P. GEORGE, C.T. LIU and D. P. POPE, *ibid.* **7** (1992) 365.
7. *Idem., ibid.* **28** (1993) 857.
8. K. AOKI and O. IZUMI, *Nippon Kinzoku Gakkaishi* **43** (1979) 1190.
9. X. J. WAN, J. H. ZHU, and K. L. JING, *Scripta Metall.* **26** (1992) 473.
10. C. G. McKAMEY and D. H. PIERCE, *Scripta Metall. Mater.* **28** (1993) 1173.
11. A. CHOUDHURY, C. L. WHITE and C. R. BROOKS, *Acta Metall. Mater.* **40** (1992) 57.

12. D. L. LIN, D. CHEN and H. LIN, *Acta Metall.* **39** (1991) 523.
13. C. T. LIU, E. H. LEE and C. G. McKAMEY, *Scripta Metall.* **23** (1989) 875.
14. C. T. LIU, C. G. McKAMEY and E. H. LEE, *ibid.* **24** (1990) 385.
15. C. T. LIU and E. P. GEORGE, *ibid.* **24** (1990) 1285.
16. M. P. SPEIDEL, in "Hydrogen Damage", edited by C. D. Beachem (ASM publication, 1977), p. 329.
17. R. J. GEST and A. R. TROIANO, *Corrosion* **30** (1974) 274.
18. C. T. LIU and C. L. WHITE, in Proceedings of the Symposium on High-Temperature Ordered Intermetallic Alloys Vol. 39 (MRS Publications; 1985) p. 365.

Received 6 February
and accepted 21 May 1996