

Tensile Properties and Fracture Behavior of Thin Foils of Two Phase Ni-Base Superalloys

Hanna Borodians'ka, Masahiko Demura and Toshiyuki Hirano

National Research Institute for Metals, 1-2-1, Sengen, Tsukuba, Ibaraki 305-0047, Japan
Fax: 81-298-59-2501, e-mail: hanna@nrim.go.jp

Abstract

Tensile properties of Ni-base superalloy thin foils consisting of γ' precipitates and γ -phase solid solution matrix have been measured as a function of Al-content at room temperature. The yield strength and ultimate tensile depended on the volume fraction of γ' precipitates in the alloy, i.e. they increased gradually with increasing the volume fraction of the γ' precipitates. Similarly, the hardness depended on the volume fraction of the γ' phase. The fracture surfaces were studied by SEM. A dimple pattern was observed indicating ductile fracture. The fraction of the dimple pattern increased with decreasing the volume fraction of the γ' precipitates. These results show that the γ' precipitates play an important role in the mechanical properties of two-phase Ni-base superalloys.

Keywords: nickel alloys, microstructure, hardness, fracture surface

1. INTRODUCTION

Ni-base superalloys have been used as high temperature structural materials in the last 20 years¹⁻⁷. Ni-base superalloys, which consist of disordered phase (γ) matrix and Ni₃Al-base ordered phase (γ'), have a good creep resistance^{3,4}. Thus, they have been used in a bulk form such as turbine blade so far^{4,5}. In contrast to the bulk, thin foils of the Ni-base superalloy could be used as lightweight high-temperature structural materials, e.g. honeycomb structures⁶.

In the previous work, we presented successful foil fabrication by cold rolling of Ni-base $\gamma+\gamma'$ two-phase alloys with composition ranging from Ni-16 at% Al to Ni-22 at% Al, which can be regarded as model materials for Ni-base superalloys⁷. These alloys could be cold-rolled to 200 μm (80% reduction) in thickness from ductile ingots which were directionally solidified by the floating zone (FZ) technique⁸. This technique was originally applied to improving the ductility of single-phase γ' (Ni-25 at%Al) without any alloying elements which suffered from intergranular fracture⁹. The high ductility of the directionally solidified polycrystals was ascribed to the large fraction of low angle and low Σ -value coincidence site lattice boundaries in the columnar-grained structure¹⁰. This high ductility made it possible to cold-roll to thin foils less than 100 μm in thickness⁶. The same technique was still effective in growing ductile ingots of Ni-base $\gamma+\gamma'$ two-phase alloys as described in the previous paper⁷.

The purpose of this paper is to present the room-temperature mechanical properties of the cold-rolled thin foils of the Ni-base two-phase alloys in terms of the volume fraction and the size of γ' precipitates. These factors on the γ' phase are thought to affect the

mechanical properties such as strength and ductility in the case of Ni-base superalloys. The role of precipitates in the fracture process is of significant scientific interest because it is essential to the explanation of fracture mechanisms. On the other hand, knowledge of the effect of γ' precipitates is of practical interest, because it leads to the development of better material properties.

2. EXPERIMENTAL PROCEDURE

Ni-base $\gamma+\gamma'$ two phase alloys with composition from 16 to 22 at%Al were unidirectionally grown by the floating zone technique (FZ-UDS)⁸. From SEM microstructure the average volume fraction of γ' -phase was measured by line intercept method⁷. Cold rolling sheets (1.7 \times 8-10 mm in cross section and 80-100 mm in length) were cut from the ingots with the longest dimension parallel to the growth direction by an electrical discharge machine (EDM). The initial rolling direction and rolling plane of the sheets were determined by the Laue X-ray back reflection method. The cold rolling was carried out by about 80 % reduction in thickness along the longest dimension at room temperature by using four-high mills with a back-up roll diameter of 360 mm and a work roll diameter of 120 mm. The rolling speed employed was 3.5 m/min. The amount of reduction in thickness per pass was about 0.1 mm. The cold-rolling operations were performed without intermediate annealing. Table I. summarizes the Al content, foil thickness, cold-rolling reduction and volume fraction of γ' -phase of the alloys used.

Tensile specimens with gauge section of 10 mm-length, 5 mm-width and about 200 μm -thickness were prepared from thin the as-rolled foils by EDM. The surfaces of all the specimens were mechanically polished

Table I. Tensile properties of the as-rolled foils.

Alloys	Al content (at%)	Foil thickness (μm)	Cold rolling reduction (%)	Volume fraction of γ' phase (%)	Yield stress (MPa)	Ultimate tensile stress (MPa)	Total elongation (%)
Ni-16Al	15.9	202	80	36	1246	1259	0.93
Ni-18Al	18.1	244	76.6	42	1458	1491	0.72
Ni-20Al	19.7	214	80	68	---	1528	0.63
Ni-22Al	21.6	283	73	87	---	1639	---
Ni ₃ Al *	25	302	83	100	---	1929	---

*- stoichiometric γ' ¹¹

with emery papers and 0.05 μm alumina powder suspension, then electropolished in order to remove surface damages by EDM. Vickers hardness was measured on the longitudinal section of the foils. Tensile tests were carried out up to fracture at room temperature in air at an strain rate of $8.33 \times 10^{-4} \text{s}^{-1}$ using Instron-type testing machine. The load data were collected at a frequency of 5Hz with a computer. Duplicate tensile tests were performed for each alloy. The fracture surfaces of tensile specimens were examined by scanning electron microscopy (SEM).

3. RESULTS AND DISCUSSION

3.1. Mechanical properties of the as-rolled foils

Vickers hardness tests were done on the longitudinal section on the foils. Vickers are shown in Fig. 1 as a function of Al content. The hardness increases markedly with increasing the Al content, in other words the volume fraction of γ' -phase.

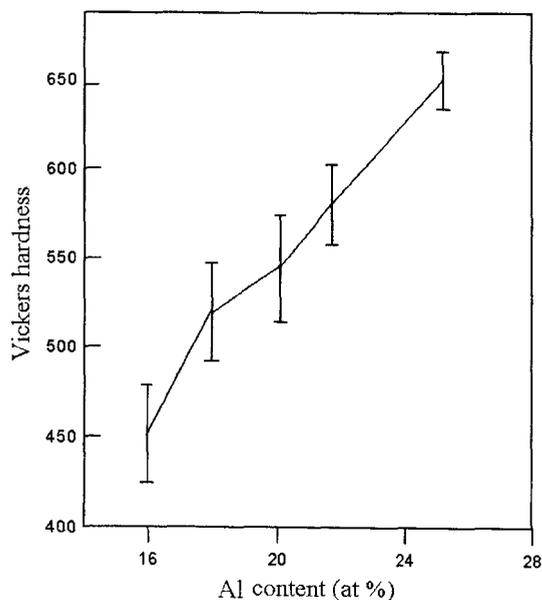


Fig. 1. Vickers hardness as a function of Al content

The tensile properties of the as-rolled foil specimens at room temperature are listed in Table I. The stress-strain curves are shown in Fig. 2 compared with that of a single phase γ' (stoichiometric Ni₃Al).¹¹

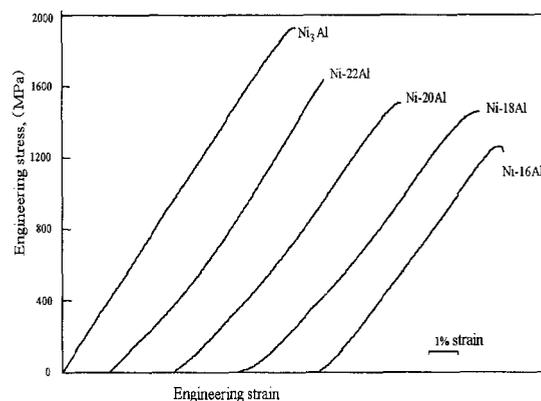


Fig. 2. Stress-strain curves of the as-rolled foils

Two foil specimens, Ni-16Al and Ni-18Al, showed nearly 1% of elongation. The foil specimens with high Al content Ni-20Al and Ni-22Al, fractured without plastic elongation in the same way as stoichiometric Ni₃Al foil. Since the foils were heavily cold rolled, the ultimate tensile stress was less than that of stoichiometric Ni₃Al foil.¹¹ Ultimate tensile stress depends on the volume fraction of γ' precipitates in the alloy, which increases with increasing the Al content.

There is some difference in the evolution of the slip bands between the foil specimens. First all the foils formed fine slip bands over the entire gauge section of the specimens at the beginning of the tensile tests. The slip bands developed from the surfaces as well as from within the crystal. Most of the slip bands did not spread the whole surface of the specimens. The following deformation step was the propagation of slip bands along the tensile directions. The propagation of the slip bands resulted in the formation of neck in the case of the Ni-16Al and Ni-18Al specimens (Fig. 3).

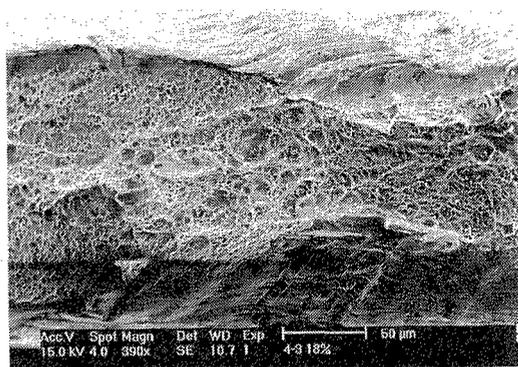


Fig. 3. Neck formation in Ni-18Al.

Only the Ni-16Al and Ni-18Al specimens showed necking in fracture. Near the neck multiple slips were observed as shown in Fig. 4. Just before the fracture, a coarse shear band was observed on the specimen surface adjacent to the neck as shown in Fig. 5. The necking occurred through the specimens.

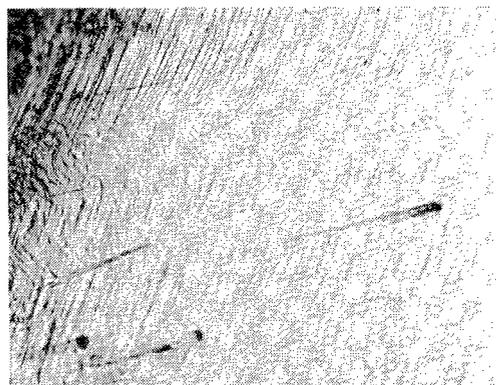


Fig. 4. The generation of multiple slips in Ni-18Al.

In contrast, the Ni-20Al and Ni-22Al specimens did not show the neck formation. The multiple slip were not observed in the Ni-20Al and the Ni-22Al specimens.

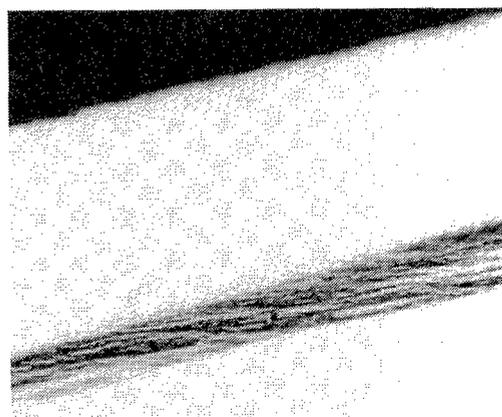


Fig. 5. An optical micrograph of the coarse shear band in Ni-18Al.

3.2 Observation of fracture surfaces

SEM observation of the fracture surfaces revealed the presence of dimple pattern over the fracture surface of the Ni-16Al specimen, indicating a trace of ductile fracture (Fig. 6(a)). The fracture surface of Ni-18Al specimen, which had higher ultimate tensile strength and less plastic elongation than Ni-16Al, also shows a dimple pattern (Fig. 6(b)). The Ni-20Al specimen shows a dimple pattern and brittle fracture pattern, which occurred at grain boundaries (Fig. 6(c)). The grain boundary surfaces have dimple structure. In the Ni-22Al specimen, cleavage fracture predominates, although a dimple pattern is partly observed (Fig. 6(d)). Thus, the fraction of the dimple pattern shows a tendency of increasing with decreasing the volume fraction of γ' -phase.

The presence of the dimple pattern over the fracture surface of the $\gamma+\gamma'$ two-phase alloys is the result of the initiation and growth of voids at the γ/γ' interfaces.¹² Clearly, that the size of the γ' precipitates and their volume fraction are important factors. Precipitates in the Ni-20Al and Ni-22Al specimens are large γ' Ni₃Al intermetallic constituents (1-25 μm), which start cracking already after little straining when fracture of the γ matrix is still remote. Cracking of these precipitates results in a large number of small voids around the tip of an existing crack.¹³ The other type of precipitates are a sub-micron size (0.1-0.5 μm) in Ni-16Al and Ni-18Al foils. Final separation of the materials took place upon decohesion of these particles because the voids show almost spontaneous growth and coalescence.^{13,14}

The γ' -phase particles tend to cluster so that the larger dimples are also clustered. Thus, it should be expected that the different size of dimples present on the region of high stretch in front of a crack tip. This was suggested by the SEM fractograph as shown in Fig 6 (b). This view was obtained from the fracture region in the tension Ni-18Al specimen, which γ' -phase size of 0.3-0.5 μm . In this figure, row of small voids connects with regions of large dimples. The small dimples had size ranging from 0.8 to 1.2 μm , and large dimples had size ranging from 2 to 10 μm .

CONCLUSION

The mechanical properties of the cold-rolled foils of two-phase $\gamma+\gamma'$ alloys with no ternary additions were examined by tensile- and Vickers hardness- tests. The following results are obtained:

1. The cold-rolled foils had extremely high ultimate tensile strengths (UTS) and Vickers hardness numbers. The UTS and Vickers hardness number increased with increasing the volume fraction of γ' phase. This indicates that the γ' phase acts as a hard particle in the cold-rolled

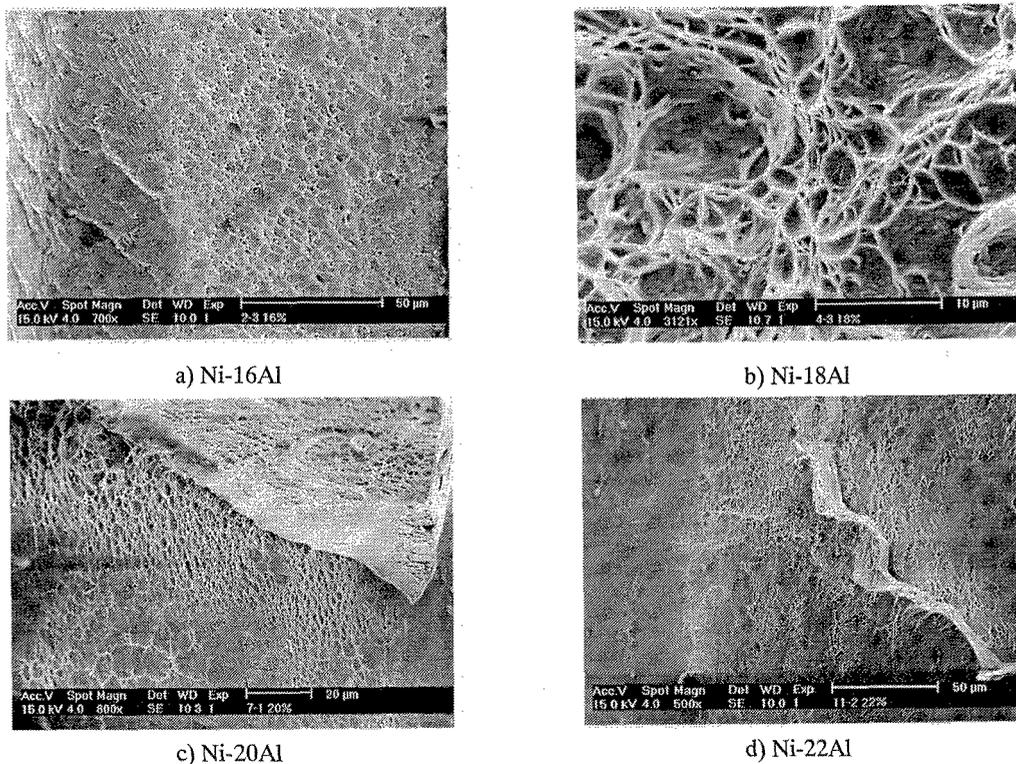


Fig. 6. SEM fractographs of fracture surfaces.

foils.

2. The cold-rolled foils of Ni-20Al and Ni-22Al fractured with almost no tensile elongation. The cold-rolled foils of Ni-16Al and Ni-18Al showed 1% tensile elongation to fracture with necking. This ductility is probably because of the low volume fraction of the γ' phase.

REFERENCES

- [1] D. P. Pope and S. S. Ezz, *Int. Mater. Rev.*, **29**, 136-167 (1984).
- [2] N. S. Stoloff, *Int. Mater. Rev.*, **34**, 153-183 (1989).
- [3] H. Harada, M. Yamazaki and Yu. Koizumi, *Tetsu-to-Hagane*, **65**, 1049-1058 (1979) in Japanese
- [4] M.G. Ardakani, M. McLean and B.A. Shollock, *Acta mater.*, **47**, 9, 2593-2602 (1999).
- [5] C.C. Law and A.F. Giamei, *Metall. Trans. A*, **7A**, 5-16 (1976)
- [6] M. Demura, Y. Suga, K. Kishida, O. Umezawa, E.P. George, T. Hirano and, *Intermetallics*, (2000) accepted.
- [7] H. Borodianska, T. Hirano, *Trans. MRS-J*, to be submitted (2000)
- [8] T. Hirano, *Acta metall.*, **38**, 2667-2671 (1990)
- [9] T. Hirano, *Scripta mater.*, **25**, 1747-1750 (1991)
- [10] T. Watanabe, T. Hirano, T. Ochiai, H. Oikawa, *Materials Science Forum*, 157-162, 1103-1108 (1994)
- [11] M. Demura, K. Kishida, O. Umezawa, E. P. George, and T. Hirano, Mechanical properties of structural films, STP 1413, ed. by Chris Muhlstein and Stuart Brown, ASTM symposium book, to be submitted, (2000)
- [12] A.S. Tetelman and A.J. McEvily, "Fracture", Vol.6, ed. by H. Liebowitz, Academic press, (1972) pp. 156-161.
- [13] D. Broek, *Eng. Fract. Mech.*, **5**, 55-63 (1973).
- [14] Honeycombe R.W.K. "The Plastic Deformation of Metals", 2nd edn. Edward Arnold, London, 1985, pp.85

(Received December 7, 2000; Accepted January 31, 2001)