Effect of magnetic field on strain aging of Co-Ni-based superalloy

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Compressive tests have been conducted at room temperature to study variation in strength of a paramagnetic fcc type Co-Ni-based superalloy subjected to strain aging treatments in the temperature range from 943 to 1033K with an applied magnetic field of 10T, or without magnetic field. Alloy aged under the magnetic field exhibits higher strain age hardening than that aged without magnetic field. Dislocation pinning caused by the segregation of the solute atoms into stacking faults bounded by the Shockley partials can be promoted in an applied magnetic field, resulting in enhanced strain age hardening. A mechanism of enhancement in strength of strain aged alloy in magnetic field is discussed.

1. INTRODUCTION

The Co-Ni-based superalloy with an fcc crystal structure exhibits high work hardening rate both at room temperature and at elevated at temperatures[1]. In addition, the dynamic strain aging (DSA) characteristics mainly due to the strong chemical interaction between solute atoms and stacking faults bounded by the Shockley partial dislocations is observed in the temperature range from approximately 773K to 1000K[1]. It has also been established that the static strain age hardening occurs in the prestrained alloy after being annealed at temperatures where the DSA appears[2]. Thus the purpose of the present investigation is to examine influence of an application of high magnetic field on the static strain aging behavior in a paramagnetic Co-Ni-based superalloy with the fcc type crystal structure.

2. EXPERIMENTAL PROCEDURE

The sample of the alloy SPRON 510 (hereafter designated by Co-Ni-based alloy) was supplied from Seiko Electric Company, Japan. The Co-Nibased alloy having chemical composition listed in Table 1 was melted by a vacuum induction melting furnace. The ingot was forged into rod with a diameter of 20 mm at a temperature of approximately 1323K. The rod shaped alloy was annealed at 1323K for 24h to attain chemical homogeneity and was subsequently swaged from 20 mm to 13 mm in diameter at room temperature to introduce the prestrain by approximately 35%reduction in diameter. The rectangular parallel piped specimens with typically $2.0 \times 2.0 \times 5.0$ mm³ in sizes were cut by spark wire cutting machine. The aging treatments for the prestrained samples were performed at temperatures of 943, 983, 1013 and 1033K under an applied magnetic field of 10T or without magnetic field. The

Table I Chemical composition of specimen.

Co	Ni	Cr	Mo	Mn	Nb	Fe	Si	Ti	С
bal.	30.4	21.0	10.0	0.5	1.5	2.1	-	0.8	-

samples for aging with the magnetic field were set in the center of an electric furnace where a uniform magnetic field is applied. Compression tests of the aged specimens and as-prestrained specimen were carried out at room temperature with an imposed strain rate of $1.7 \times 10^{-3} \text{s}^{-1}$.



Figure 1: The optical micrograph for the Co-Nibased alloy.

An optical microstructure of recrystallized specimen obtained from annealing the prestrained specimen at 1323K for 24h was observed to examine the existence of the second phase after abrading the surface of the specimens using emery paper up to # 06 and etched in a solution of 4HCI-1HNO₂-1acetone-1H₂O.

To examine the deformation microstructures, transmission electron microscope (TEM) observations were conducted. Thin discs with a thickness of approximately 1mm for TEM were cut by a wire-cut spark machine from the deformed samples and mechanically polished with emery papers to a thickness of approximately 0.15mm. The thin foils for TEM observations were prepared by jet-electropolishing the disc at a voltage of 12V at 243K in a solution of 90 parts methanol plus five parts sulphric acid. TEM observations were performed using Hitachi H-800 at an accelerated voltage of 200kV.

3. RESULTS

3.1 Microstructure observations

An optical micrograph for the present Co-Nibased alloy is shown in Figure 1. As seen in the figure, equi-axed grains with an average grain size of approximately 100μ m, containing no appreciable second phase, can be seen and annealing twin boundaries are frequently found, indicating that the present alloy consist of a single phase and has a low stacking fault energy (SFE). Figure 2 shows a TEM micrograph of the aged

sample in 10T at 1013K after being prestrained at room temperature by 35% reduction in diameter. Beam direction, **BD**=110 and foil normal, FN=110. A bright field image in Fig. 2 (a) shows that the microstructure consists of dislocations with high density and a number of thin deformation twins observed as thin plate-like microstructures. An electron diffraction pattern taken from the same area of the thin foil is shown in Fig. 2 (b) where diffraction spots due to both matrix and twins are indexed. The corresponding dark field image is shown in Fig. 2 (c), which was taken using diffraction spot indicated by an arrow in Fig. 2 (b). As seen in the figure, a number of thin deformation twins are clearly observed. The microstructures of the aged samples at temperature lower than 1013K in an applied magnetic field of 10T and 0T are almost the same as those of the as-prestrained sample. We cannot find the discernible difference between TEM microstructures aged in magnetic field and without magnetic field in the temperature range upto 1013K.

Figure 3 shows a TEM micrograph of the dislocation arrangement in a tensile-deformed sample by 10% at room temperature. The incident electron beam direction, **BD**, is $[10\bar{1}]$ and the foil normal, **FN**, is approximately [101]. According to the trace analysis, the slip plane of the observed dislocations is found to be a primary (111) plane and screw and edge orientations of the dislocations can be indicated in the figure by solid lines. As seen in the figure, planar dislocation array without dissociation are seen and several sets of parallel slip bands where mobile edge dislocations are aligned are observed, forming edge dipoles and multipoles.

Figure 4 shows a dislocation structure of the aged sample at 943K for 2h after tensiledeformation by approximately 10 % at room temperature. It should be noted that unlike the dislocations without aging treatment as shown in Fig.3, all the dislocations accompany a wide ribbon of stacking fault showing itself as a set of alternating light and dark fringes, indicating that the dislocations dissociate into the Shockley



Figure 2. TEM micrographs of the aged sample in 10T at 1013K after being prestrained at room temperature by 35% reduction in diameter. (a) the bright field image, (b) the diffraction pattern, and (c) the dark field image. The dark field image (c) was taken using a diffraction spot, [111]T due to twins, indicated by an arrow in the diffraction pattern (b).

partials. It is likely that these dislocation dissociations result from the aging treatment. During aging, solute atoms migrating to the dislocations segregate into the stacking fault bounded by the two Shockley partials, resulting in



Figure 3 TEM micrograph of the dislocation arrangement in a sample tensile-deformed by 10% at room temperature (**BD** = $\overline{101}$ and **g**=11 $\overline{11}$).



Figure 4. The dislocation structure observed in an aged alloy at 943K for 2h. The specimen was prestrained by 10% in tension at room temperature before the aging treatment. The widely dissociated dislocations having stacking fault fringes are seen in the figure.

the reduction of the SFE and widening the width of the stacking fault [4].

3.2 Compression tests

Figures 5 (a), (b) and (c) show the room temperature compressive true stress-true strain curves of the sample aged at 943K, 1013K and 1033K for 2h, respectively, in an applied magnetic field of 10T and 0T. The stress-strain curve of as-prestrained sample is also indicated for comparison. As can be seen in these figures, the strengths of the aged samples are higher than those of the as-prestrained sample. The increment in strength of the prestrained sample by aging treatment occurs by strain aging [2]; the dislocations induced by plastic deformation at room temperature are pinned due to high-temperature annealing, resulted from the segregation of the solute atoms to stacking faults bounded by the Shockley partials. In Fig. 4, we find that the dissociation width of the dislocations induced at room temperature is expanded by aging treatment at 943K. This observation on variation in dissociation width of the dislocations convinces us that the age hardening observed in the present alloy arises from the strain aging. At every aging temperature we can find that the yield stresses and the flow stresses of the samples aged in an applied magnetic field of 10T are higher than those of the samples aged without magnetic field, indicating that the strain age hardening in the present Co-Ni-based alloy is enhanced by an application of magnetic field. A further point to note is that remarkable yield point phenomenon is found in the aged samples, arising from the abrupt increase in mobile dislocation density resulted from unlocking of dislocations pinned due to strain aging. When the applied stress reach high enough to unlock the pinning, the immobilized dislocations begin to move and the number of the mobile dislocations are increased abruptly. As a result, the yield drop phenomenon appears as observed in bcc alloys.

4. DISCUSSIONS

In the present study, it is found that aging in a magnetic field enhances the strain age hardening. In this section, we discuss a mechanism of the magnetic effect on the strain aging in the present Co-Ni based alloy. Pure Co undergoes phase transformation from paramagnetic fcc to ferromagnetic hcp crystal structure at temperatures below 693K. However, the transformation temperature of pure Co can be lowered to temperatures below room temperature by alloying Ni [4] so that the fcc crystal structure remains stable in Co-Ni alloy even below room temperature. Although the magnetic property of the binary Co-Ni alloy is ferromagnetic, Cr addition more than 20% to the Co-Ni alloy render the binary alloy paramagnetic.

Fig.6 shows the stacking order of ABCABCA... of the close packed (111) plane in fcc crystal containing a dissociated dislocation consisting of the two Shockley partials bounding stacking fault. As illustrated in the figure, the stacking fault can be considered to be mono-layered hcp phase formed in the paramagnetic fcc matrix. Thus in the present Co-Ni-based alloy containing Cr, since a hcp Co phase is ferromagnetic, a stacking fault bounded by the Shockley partials is assumed to be ferromagnetic so that a magnetic field enhances



Figure 5 The room temperature compressive true stress-true strain curves of the sample aged at (a) 943K, (b) 1013K and (c) 1033K in an applied magnetic field of 10T and 0T. The stress-strain curve of as-prestrained sample is also indicated for comparison.



Figure 6 The stacking sequence of close packed (111) planes and stacking fault bounded by the two Shockley partials of a fcc crystal structure.

the stability of a stacking fault, resulting in widening dissociation width of the dislocations in an applied magnetic field as compared to in nonmagnetic field. Thus the dislocation pinning caused by the segregation of the solute atoms into the stacking faults can be promoted in an applied magnetic field, resulting in enhancing the strain age hardening.

5. CONCLUSIONS

- 1. The yield stresses and the flow stresses of the Co-Ni-based alloy aged in an applied magnetic field of 10T are higher than those of the samples aged without magnetic field, indicating that the strain age hardening in the present Co-Ni-based alloy is enhanced by the application of the magnetic field.
- 2. It is assumed that a stacking fault bounded by the Shockley partials has ferromagnetic nature in a paramagnetic matrix of the Co-Ni-based alloy containing Cr. Thus the stacking fault becomes stable in magnetic field, resulting in wider dissociation of dislocations.
- 3. In the Co-Ni-based alloy, dislocation pinning effect due to solute segregation into stacking faults bounded by the Shockley partials is enhanced by an application of magnetic field, resulting in enhancement of the strain age hardening in an applied magnetic field.

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REFERENCES

- [1] A. Chiba, X. G. Li and M. S. Kim, *Phil.* Mag. A, **79**, 1533-1554 (1999).
- [2] T. Ono, A. Chiba, S. Takahashi, M. Sato, and Y. Koyama, Abstracts of the Japan Institute of Metals, Fall meeting 115, 229 (1994).
- [3] C. Hitzenberger and H. P. Karnthaler, *Phil. Mag.* A, 64, 151-163 (1991).
- [4] H. Suzuki, Sci. Rep. Res. Inst., Thohoku Univ., A, 4, 453-463 (1952).

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