Effect of Hydrogen on Deformation Structure of Inconel 718

Jianhong He^{*†}, Seiji Fukuyama^{*}, Kiyoshi Yokogawa^{*}
and Akihiko Kimura^{**}

^{*}Chugoku National Industrial Research Institute, AIST, MITI, 2-2-2 Hiro-suehiro,
Kure, Hiroshima 737-01, Japan

^{**}Institute for Materials Research, Tohoku University, 2-1-1 Katahira, Aoba-ku,
Sendai 980-77, Japan

Effects of hydrogen on the deformation behavior and the structure of deformation bands under monotonic loading of Inconel 718 were investigated. Hydrogen significantly reduced tensile elongation of the material and susceptibility to hydrogen embrittlement was not influenced by the aging condition. In the hydrogen-free specimen the spacing between deformation bands decreased with increasing strain, reached the minimum value at a strain of about 8% and remained constant until its fracture. Although hydrogen reduced the band spacing at the early stage of deformation, the minimum spacing was not influenced by hydrogen. Hydrogen-induced cracking occurred along the deformation bands when the band spacing reached the minimum spacing, while the deformation of the hydrogen-free specimen still continued until the fracture with the constant band spacing. It is proposed that hydrogen suppressed the cross slip and caused stress concentration within the deformation bands which were consisted of dislocations trapping hydrogen, resulting in the brittle fracture along the deformation bands.

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I. Introduction

Inconel 718 has been widely used as the main constructive material with excellent mechanical properties for hydrogen service at temperatures ranging from cryogenic to elevated temperatures⁽¹⁾⁽²⁾. However, it has been often shown that the material is rather susceptible to hydrogen embrittlement, resulting in continuous efforts to understand hydrogen effects on the material⁽²⁾⁻⁽¹⁹⁾.

Walter *et al.*⁽⁵⁾⁽⁶⁾⁽⁸⁾ and Harris *et al.*⁽²⁾⁽⁷⁾ studied hydrogen environmental embrittlement of the material to assess the contribution of hydrogen-fueled rocket engines financially supported by NASA. They found that mechanical properties of the material degraded in hydrogen atmosphere. Hicks *et al.* investigated effects of hydrogen on tensile properties⁽¹⁶⁾ and also showed that hydrogen-enhanced cracking⁽¹⁸⁾ was observed in the material after the cathodic charging with hydrogen in a molten salt bath. They observed that the internal hydrogen also reduced tensile elongation of the material⁽¹⁶⁾ and decreased threshold stress intensity for the onset of subcritical crack growth of the material⁽¹⁸⁾. Fukuyama *et al.* investigated the fatigue behavior⁽¹⁵⁾ and tensile properties⁽¹⁷⁾⁽¹⁹⁾ of the material in high-pressure hydrogen gas and found that the fatigue crack growth rate was increased by hydrogen⁽¹⁵⁾ and the crack initiation occurred at the carbides in the material⁽¹⁷⁾. Although the macroscopic behavior of the mechanical properties of the material with and without hydrogen has been shown so far, effects of hydrogen on deformation microstructure are not fully under-

stood yet⁽²⁾⁻⁽¹⁹⁾.

In this paper, correlation between tensile behavior and deformation structure in Inconel 718 was investigated after thermal hydrogen charging in high-pressure hydrogen gas in order to make clear the effect of hydrogen on the mechanical properties of the material.

II. Experimental

1. Material conditions

Inconel 718 was used as a starting material which was cold rolled to 0.37 mm thick sheet. The chemical compositions are shown in Table 1. Specimens for tensile tests with a gauge length of 25 mm were punched out from the sheets. The specimens were solution annealed at 1333 K for 3.6 ks followed by water quenching, and then aged at 1033 K for periods ranging from 3.6 to 180 ks.

Precipitation of γ' and γ'' occurred during the aging heat-treatment. Effect of aging period at 1033 K on the 0.2% proof stress of γ' and γ'' precipitation is shown in Fig. 1. The aged materials could be classified into three groups; the materials aged for less than 28.8 ks are named as underaged material, those aged for from 28.8 to 57.6 ks are named as aged material, and those for above 57.6 ks are named as overaged material.

Figure 2 shows γ'' precipitates after each aging condition. The precipitates of γ' and γ'' were identified using the reflection $\{1\ 0\ 0\}$ of γ' and γ'' and $\{1\ 1/2\ 0\}$ of γ'' on the selected area diffraction (SAD) pattern of $\langle 001 \rangle$ direction⁽²⁰⁾⁻⁽²²⁾. The shape of γ'' in underaged, aged and overaged specimen was sphere, disc, and large disc, respectively.

[†] Present address: Tsinghua University, Beijing, P. R. China.

Table 1 Chemical compositions of Inconel 718 (mass %).

C	Mn	Si	P	S	Cr	Co	Mo	Cb+Ta	Ti	Al	B	Cu	Ni	Fe
0.05	0.21	0.12	<0.006	<0.002	18.1	0.4	3.01	5.08	1.02	0.55	0.004	0.05	52.7	Bal.

Fig. 1 Effect of aging period at 1033 K on 0.2% proof stress of Inconel 718.

Fig. 3 Effect of aging period at 1033 K on the density and diameter of γ'' precipitation in Inconel 718. Fig. 2 Typical γ'' of underaged, aged and overaged Inconel 718. (a: aged for 3.6 ks (underaged), b: aged for 28.8 ks (aged), c: aged for 180 ks (overaged) at 1033 K, ($g=[1\ 1/2\ 0]$, $B=[0\ 0\ 1]$)

Figure 3 shows the change in the density and diameter of the γ'' precipitates due to the aging at 1033 K. In underaged materials, the diameter of γ'' monotonically in-

creased, while the density increased at first, then reduced after 3.6 ks of aging, which resulted in a rapid increase in 0.2% proof stress at the beginning of aging and a moderate increase thereafter. In both aged and overaged specimens, the density and the diameter increased and decreased, respectively, although the rates were much higher in aged materials.

2. Tensile testing and TEM observation

Hydrogen was charged into specimens in high-pressure hydrogen gas of 11 MPa at 573 K for 259 ks in a pressure vessel. The specimens were tensile tested at an initial strain rate of $3.3 \times 10^{-5} \text{ s}^{-1}$ in air at ambient temperature. The fracture surfaces were observed by a scanning electron microscope.

All foils for the transmission electron microscope (TEM) examination using JEOL 2000FX were sanded to a thickness of 0.15 mm, followed by the final polishing and thinning by twin-jet method in the electrolyte of 8% solution of HClO_4 in ethanol at low temperatures.

III. Results

1. Effect of hydrogen on stress-strain curve

The effect of hydrogen on the stress-strain curve of aged specimen is shown in Fig. 4. Hydrogen reduced the fracture stress significantly without showing marked effects on the flow stress, which resulted in a significant reduction of the elongation.

The effect of aging on elongation of specimens with and without hydrogen is shown in Fig. 5. Elongation of the hydrogen-charged specimen (ϵ_H) and the hydrogen-free specimen (ϵ_{NOH}) decreased with increasing aging

Fig. 4 Effect of hydrogen on the stress-strain curve of aged Inconel 718.

Fig. 5 Effect of aging period on elongation and the ratio of elongations ($\epsilon_H/\epsilon_{NOH}$) of Inconel 718 aged at 1033 K.

time, i.e. ϵ_{NOH} decreased from 27.5% to 21.5% and ϵ_H also decreased from 10.8% to 8.2%. The resultant ratio of elongations ($\epsilon_H/\epsilon_{NOH}$) remained almost constant at 0.38, which suggests that the susceptibility to hydrogen embrittlement of the material does not depend on the aging time.

Typical fracture modes of the hydrogen-charged specimen (a) and the hydrogen-free specimen (b) of aged Inconel 718 are shown in Fig. 6. Planar cleavage facets were found on the fracture surface of the hydrogen-charged specimen. In their study on fatigue crack growth of hydrogen-charged Inconel 718, Clavel *et al.* identified the orientation of the planar facets as $\{1\ 1\ 1\}$ s using the stereographic observation techniques⁽²³⁾. Hicks *et al.* also derived the same conclusion using the cross sectioning and etching technique⁽¹⁶⁾⁽¹⁸⁾. The fracture mode of the hydrogen-free specimen was completely ductile showing dimples on the fracture surface. Fracture mode did not depend on the aging time in both the hydrogen-charged and hydrogen-free specimens.

2. Effect of hydrogen on deformation bands

A typical morphology of deformation bands of the

Fig. 6 Effect of hydrogen on the fracture mode of aged Inconel 718. (a: hydrogen-charged specimen, b: hydrogen-free specimen)

hydrogen-free specimen of aged Inconel 718 is shown in Fig. 7 with the SAD pattern. The deformation bands along two different $\langle 1\ 1\ 0 \rangle$ directions were observed as reported in the previous papers^{(23)–(40)}. The spacing between the deformation bands was measured using the image observed in the direction of $[0\ 1\ 1]$.

Figure 8 shows the dark field image taken by using the $(2\ 2\ \bar{2})$ reflection of the matrix of underaged specimen which was deformed to a strain of 6% following hydrogen charging. A crack was observed along the deformation band which was perpendicular to the $\langle 1\ 1\ 1 \rangle$ direction.

The effect of the amount of strain on spacing between the deformation bands in the hydrogen-free specimens of underaged, aged and overaged Inconel 718 is shown in Fig. 9. Spacings of all the specimens decreased with increasing the strain and remained to be 180 nm above the strain of 8%. The minimum spacing of deformation bands was smaller than that of the two-step aged material and larger than that of the homogenized material strained by monotonic loading⁽³⁴⁾. The spacing decreases

Fig. 7 Typical deformation bands with SAD pattern of hydrogen-free specimen of aged Inconel 718. (a: $g=[0\ 2\ 0/0\ 4\ 0]$, $B=[0\ 0\ 1]$, with a total strain of 3%) (b: SAD pattern of (a))

Fig. 9 Effect of strain on the spacing between the deformation bands in hydrogen-free specimens of underaged, aged and overaged Inconel 718.

with increasing the aging time at a given strain below the strain of 8%, which is consistent with the previous reports that the band spacing was reduced by overaging heat-treatment⁽²⁷⁾⁽²⁸⁾⁽³⁰⁾⁽³²⁾. The fracture strain appears to decrease with increasing the aging time.

Effect of hydrogen on the band spacing in aged Inconel 718 is shown in Fig. 10. Hydrogen reduced the spacing at the early stage of the deformation below the strain of 8%. However, the minimum spacing was not influenced by hydrogen. The strain where the spacing reached the minimum value was also reduced by hydrogen from 8 to 6%. The hydrogen-charged specimen fractured when the band spacing reached the minimum value, namely 180 nm, while the hydrogen-free specimens still deformed keeping the band spacing constant.

Effect of hydrogen on dislocation structures of overaged Inconel 718 is shown in Fig. 11. Dislocation pile-ups were frequently observed in the hydrogen-charged specimen, while dislocation cross slipping were

Fig. 8 Crack along deformation band of hydrogen-charged specimen of underaged Inconel 718 after the deformation up to a total strain of 6%.

Fig. 10 Effect of hydrogen on the spacing between the deformation bands of aged Inconel 718.

Fig. 11 Effect of hydrogen on dislocation structures of overaged Inconel 718 with a total strain of 3%. (a: hydrogen-charged specimen, b: hydrogen-free specimen; $g = [1 \bar{1} 1/2 \bar{2} 2]$, $B = [0 1 1]$)

observed around γ'' in the hydrogen-free specimen. These suggest that hydrogen suppresses the cross slip of dislocation in deformation bands.

IV. Discussion

Hydrogen effects on deformation behavior of Inconel 718 are summarized as follows. (1) Hydrogen reduces tensile elongation significantly, while no effect was observed on flow stress. (2) Hydrogen causes the change in the fracture mode from ductile to brittle fracture. (3) Hydrogen reduces the band spacing at the beginning of the deformation, but no change was observed for the minimum spacing. (4) Hydrogen-induced cracking occurs when the band spacing reaches the minimum value. (5) Hydrogen appears to suppress the cross slip of dislocation motion. (6) No effects of γ'' precipitates morphology on the extension of hydrogen embrittlement was observed.

1. Hydrogen effect on the deformation bands

A marked hydrogen-induced reduction of tensile elongation appears to be correlated with the change in the structure of deformation bands due to hydrogen. The significant effects of hydrogen were observed on the band spacing; the spacing was reduced in the hydrogen-charged specimens, which can be interpreted in terms of the hydrogen-induced suppression of cross slipping of dislocations. One of the present authors⁽⁴¹⁾ showed that the slip trace morphology became sharper by the addition of hydrogen in high purity iron single crystals. Duesbery *et al.*⁽⁴²⁾ showed in their computer simulation study that hydrogen caused the dissociation of dislocation core in iron so as to suppress their cross slip. Although the deformation of fcc Inconel 718 is controlled by the motion of edge dislocations, hydrogen may suppress the cross slip of edge dislocations in a similar manner to that in iron. Gilman demonstrated that, in an asymmetrical bicrystal of Zn at 77 K⁽⁴³⁾, the blockage of edge dislocations at the grain boundary which might be due to the hydrogen-induced suppression of the cross slip of the dislocation led to dislocation splitting along the glide plane. At the initial stage of the deformation, the spacing decreased with increasing strain, and TEM observation revealed that the dislocations frequently pile up in the hydrogen-charged specimens, as shown in Fig. 11. This suggests that hydrogen suppresses the cross slip of the dislocations and the resultant dislocation pile-up forces the back stress to the dislocation source in the dislocation band. For the progress of the further deformation the other dislocation source will be activated to produce another deformation band. The spacing therefore becomes small in the hydrogen charged specimens at the early stage of the deformation. The minimum value of the spacing, however, was not influenced by hydrogen, suggesting that the deformation above the strain where the spacing reached the minimum is not affected by cross slip of the dislocation.

2. Hydrogen-induced brittle fracture

As previously shown, the hydrogen-induced cracks in Inconel 718 propagate along the deformation bands. Since hydrogen is often strongly bounded by precipitates, it is easily expected that γ'' precipitates trap

hydrogen and might act as the nucleation sites of the brittle fracture. As shown in Fig. 5, however, the ratio of elongations ($\epsilon_H/\epsilon_{NOH}$) does not depend on the aging time, while the morphology of the precipitates is significantly influenced by the aging time (Fig. 2). This suggests that the precipitates do not play a role on the hydrogen-induced fracture. Solid solution hydrogen can be swept by dislocations in the deformation bands. Piling-up of dislocations with hydrogen clouds cause local build up of hydrogen in the slip plane. Hydrogen-induced dissociation of the dislocations may enhance the planar distribution of hydrogen. Under these conditions, hydrogen can cause the decohesion⁽⁴⁴⁾ at the deformation band. The resultant decrease in the fracture stress causes the brittle fracture. It is considered that in the hydrogen-free specimen the deformation still continues after the spacing reached the minimum value, while the deformation of hydrogen-charged specimen was interrupted by the brittle fracture at the strain, since the fracture stress was reduced and the back stress to dislocation motion was increased by hydrogen.

V. Conclusion

Tensile testings of Inconel 718 were conducted after hydrogen charging in high-pressure hydrogen of 11 MPa at 573 K. The results obtained are as follows;

(1) Hydrogen significantly reduced the tensile elongation accompanied by the change in the fracture mode from ductile rupture to brittle fracture along the deformation bands. Susceptibility to hydrogen embrittlement did not depend on the aging time, namely the morphology of precipitates.

(2) Hydrogen reduced the spacing between the deformation bands at the early stage of the deformation, while no effect was observed on the minimum spacing. Hydrogen induced fracture occurred when the spacing of the deformation bands reached the minimum value.

(3) It is proposed that hydrogen suppressed the cross slip of dislocation and reduced the fracture stress along deformation bands in Inconel 718.

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REFERENCES

- (1) H. L. Eiselstein: ASTM STP369, ASTM, (1965), p. 62.
- (2) J. A. Harris, Jr. and M. C. VanWanderham: NASA CR-124394, (1973).
- (3) P. M. Lorenz: NASA CR-100208, (1969).
- (4) V. Frick, G. R. Janser and J. A. Brown: Space Shuttle Materials, SAMPE, (1971), p. 567.
- (5) R. J. Walter and W. T. Chandler: NASA CR-124410, (1973).
- (6) R. P. Jewett, R. J. Walter, W. T. Chandler and R. P. Frohberg: NASA CR-2163, (1973).
- (7) J. A. Harris, Jr. and M. C. VanWanderham: ASTM STP543, ASTM, (1974), p. 198.
- (8) W. T. Chandler and R. J. Walter: ASTM STP543, ASTM, (1974), p. 170.
- (9) H. J. Saxton, A. J. West and A. W. Thompson: *Int. Conf. Effect of Hydrogen Behavior Materials*, TMS USA, (1977), p. 631.
- (10) R. J. Walter and W. T. Chandler: *Environmental Degradation of Engineering Materials*, ed. by M. R. Louthan, Jr. and R. P. McNitt, Virginia Polytechnique Inst., (1977), p. 513.
- (11) W. T. Chandler: *Recent Advances in Structures for Hypersonic Flight*, NASA Conf. Pub. 2065, Part I, (1978), p. 195.
- (12) W. T. Chandler, R. J. Walter, C. E. Moeller and H. W. Carpenter: *Plating and Surface Finishing*, **65** (1978), No. 5, 63.
- (13) W. T. Chandler: *Advanced Earth-to-Orbit Propulsion Technology Conf.*, Huntsville, U.S.A., (1986).
- (14) NASDA: Report of Joint Study with GIRIC, MRP88-57, (1987).
- (15) S. Fukuyama, K. Yokogawa, M. Araki, Y. Koyari, H. Aoki and Y. Yamada: *J. Soc. Mater. Sci. Jpn.*, **38** (1989), 539.
- (16) P. D. Hicks and C. J. Altstetter: *Metall. Trans. A*, **21A** (1990), 365.
- (17) S. Fukuyama, K. Yokogawa, M. Araki, Y. Koyari and Y. Yamada: *J. Soc. Mater. Sci. Jpn.*, **40** (1991), 736.
- (18) P. D. Hicks and C. J. Altstetter: *Metall. Trans. A*, **23A** (1992), 237.
- (19) S. Fukuyama, K. Yokogawa, Y. Yamada and T. Iida: *J. Iron and Steel Inst. Jpn.*, **78** (1992), 860.
- (20) R. Cozar and A. Pineau: *Metall. Trans.*, **4** (1973), 47.
- (21) J. M. Oblak, D. F. Paulonis and D. S. Suvall: *Metall. Trans.*, **5** (1974), 143.
- (22) Yafang Han, P. Deb and M. C. Chaturvedi: *Met. Sci.*, **16** (1982), 555.
- (23) M. Clavel and A. Pineau: *Metall. Trans. A*, **9A** (1978), 471.
- (24) D. F. Paulonis, J. M. Oblak and D. S. Duvall: *Trans. ASM*, **62** (1969), 611.
- (25) G. R. Leverant, B. H. Kear and J. M. Oblak: *Metall. Trans.*, **4** (1973), 355.
- (26) H. F. Merrick: *Metall. Trans.*, **5** (1974), 891.
- (27) D. Fournier and A. Pineau: *Metall. Trans. A*, **8A** (1977), 1095.
- (28) S. Floreen and R. H. Kane: *Fatigue Eng. Mater. Struct.*, **2** (1980), 401.
- (29) T. H. Sanders, Jr., R. E. Frischmuth and F. T. Embley: *Metall. Trans. A*, **12A** (1981), 1003.
- (30) J. P. Pedron and A. Pineau: *Mater. Sci. Eng.*, **56** (1982), 143.
- (31) M. Clavel and A. Pineau: *Mater. Sci. Eng.*, **55** (1982), 157.
- (32) D. D. Krueger: Report R85AEB133, General Electric Aircraft Engine Business Group, Cincinnati, OH, U.S.A., (1985).
- (33) M. Sundararaman, P. Mukhopadhyay and S. Banerjee: *Acta Metall.*, **36** (1988), 847.
- (34) D. W. Worthem, I. M. Robertson, F. A. Leckie, D. F. Socie and C. J. Altstetter: *Metall. Trans. A*, **23A** (1990), 3215.
- (35) W. W. Milligan and S. D. Antolovich: *Metall. Trans. A*, **18A** (1987), 85.
- (36) J. M. Oblak, D. F. Paulonis and D. S. Duvall: *Metall. Trans. A*, **5A** (1974), 143.
- (37) Y. F. Han, P. Deb and M. C. Chaturvedi: *Metal Sci.*, **16** (1982), 555.
- (38) I. Kirman and D. H. Warrington: *Metall. Trans.*, **1** (1970), 2667.
- (39) S. Mahajan and G. Y. Chin: *Acta Metall.*, **21** (1973), 1353.
- (40) C. Nourtier and G. Saada: *Fundamental Aspects of Dislocation Theory*, ed. by J. A. Simmons *et al.*, U.S.A., National Bureau of Standards, (1970), p. 1259.
- (41) A. Kimura, H. Matsui and H. Kimura: *Mat. Sci. Eng.*, **58** (1983), 211.
- (42) M. S. Duesbery, V. Vitek and D. K. Bowen: *Proc. Roy. Soc.*, **A332** (1973), 85.
- (43) J. J. Gilman: *Trans. TMS-AIME*, **212** (1958), 783.
- (44) C. J. McMahon, Jr.: *Proc. Int. Conf. Hydrogen Effects in Metals*, TMS-AIME, (1981), p. 219.