JOURNAL OF MATERIALS SCIENCE 27 (1992) 2636–2640

Creep–rupture properties and grain-boundary sliding in wrought cobalt-base HS-21 alloys at high temperatures

M. TANAKA, H. IIZUKA, F. ASHIHARA
Department of Mechanical Engineering, Mining College, Akita University, 1-1, Tegatagakuen-cho, Akita 010, Japan

The effects of serrated grain boundaries on the creep-rupture properties of wrought cobalt-base HS-21 alloys were investigated at 1311 and 1422 K. The amount of grain-boundary sliding and the initiation and growth of grain-boundary cracks were also examined during creep at 1311 K. Specimens with serrated grain boundaries exhibited longer rupture life and larger rupture ductility than those with straight grain boundaries, but these specimens had almost the same rupture life and rupture ductility under lower stresses at 1422 K, because serrated grain boundaries were also formed in specimens with originally straight grain boundaries. The average amount of grain-boundary sliding during creep at 1311 K increased with time (or with creep strain), but was almost the same in both specimens with serrated grain boundaries and those with straight grain boundaries at the same creep strain. Grain-boundary cracks or voids initiated in the early stage of creep in those specimens at 1311 K. Therefore, the strengthening by serrated grain boundaries at high temperatures above about 1311 K was attributed to the retardation of growth and linkage of grain-boundary cracks and voids.

1. Introduction

Serrated grain boundaries are effective in improving creep–rupture properties of heat-resistant alloys. The strengthening mechanisms of serrated grain boundaries are principally (1) the inhibition of grain-boundary sliding [1] which leads to the initiation and growth of grain-boundary cracks, (2) the retardation of grain-boundary crack initiation caused by the decrease of stress concentration at grain-boundary triple junctions owing to the decrease of sliding grain-boundary length [2–4] and dynamic recovery at serrated grain boundaries [5], and (3) the decrease of crack growth rate owing to the decrease of the stress intensity when a crack propagates on serrated grain boundaries [6–10]. The other important mechanisms are (4) the occurrence of ductile grain-boundary fracture [4, 11], (5) the lengthening of crack path [9], and (6) the crack arrest at serrated grain boundaries [12]. However, it is not clear whether the strengthening by serrated grain boundaries is still effective and what strengthening mechanisms work at very high temperatures above the usual service temperature of heat-resistant alloys (typically below 1373 K (1100 °C).

In this study, the effects of serrated grain boundaries on the creep–rupture properties were investigated using wrought cobalt-base HS-21 alloys at 1311 K (1038 °C) and 1422 K (1149 °C). The HS-21 alloys were chosen as test material because of their high oxidation resistance. The amount of grain-boundary sliding and the initiation and growth of grain-boundary cracks were also examined on specimens with serrated grain boundaries and those with straight grain boundaries crept at 1311 K. The strengthening mechanisms of serrated grain boundaries at these temperatures were then discussed on the basis of the experimental results.

2. Experimental procedure

The commercial wrought cobalt-base HS-21 alloys used in the previous study were also chosen as test materials in this study (Table I) [13]. Specimens with serrated grain boundaries were obtained by furnace cooling to 1323 K (1050 °C), followed by water quenching after solution heating for 3.6 ks at 1523 K (1250 °C) [13]. Specimens with normal straight grain boundaries were solution-heated for 3.6 ks at 1523 K and then water quenched. Fig. 1 shows the microstructure of heat-treated specimens of HS-21 alloys.

TABLE I Chemical composition of cobalt-base alloys used (wt%)

Alloy	C	Cr	Ni	Mo	Mn	Fe	Si	P	S	B	Co
HS-21	0.27	26.71	2.37	5.32	0.64	0.09	0.59	< 0.005	0.007	0.003	Balance

0022–2461

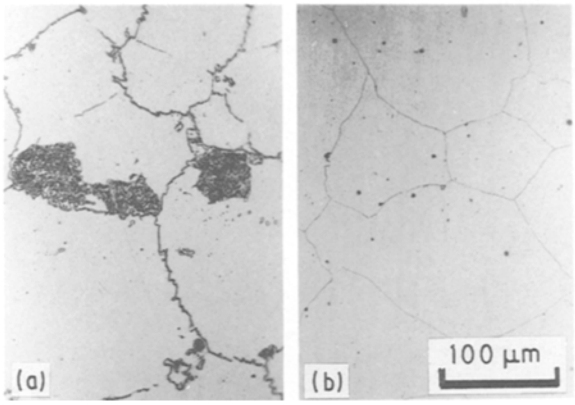

Figure 1 Optical micrographs of heat-treated specimens of HS-21 alloys (electrolytically etched with 10% aqueous chromic acid (a) specimen with serrated grain boundaries (b) specimen with straight grain boundaries.

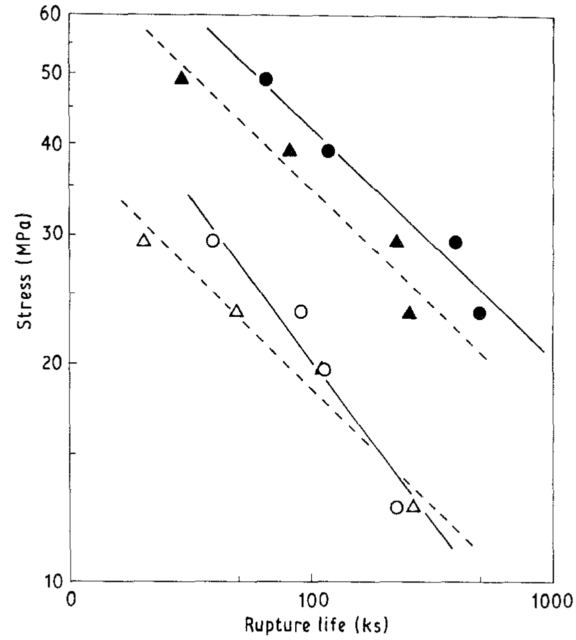

Figure 2 Rupture lives of HS-21 alloys at (●, ▲) 1311 and (○, △) 1422 K. (○, ●) serrated grain boundaries, (▲, △) straight grain boundaries.

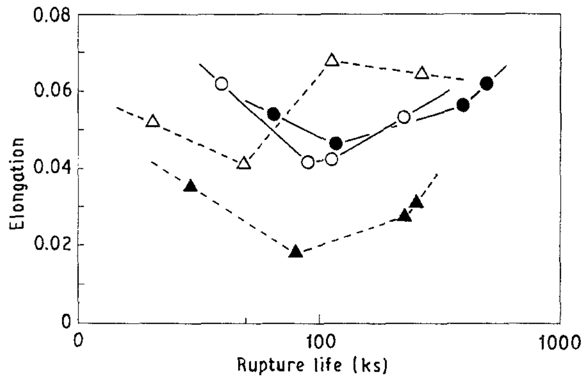

Figure 3 Creep ductility of HS-21 alloys at (●, ▲) 1311 and (○, △) 1422 K. (○, ●) serrated grain boundaries, (▲, △) straight grain boundaries.

Grain boundaries are considerably serrated by precipitation of $M_{23}C_6$ carbide particles in the specimen with serrated grain boundaries (Fig. 1a) [13], but there are virtually no precipitates in the interior of grains except a very small amount of residual carbides in both specimens (Fig. 1a and b). Both specimens have the same grain diameter of about 130 µm.

Creep and creep–rupture tests were performed using the usual single-lever type of creep–rupture equipment with a capacity of 19.6 kN at 1311 K (1038 °C) and 1422 K (1149 °C) in air. Smooth round-bar specimens with 30 mm gauge length and 5 mm diameter were used for both experiments. All these specimens were initially loaded after 10.8 ks holding at test temperature. Marker lines were scratched on the surface of some specimens with a knife-edge at a spacing of about 0.2 mm for the measurement of grain-boundary sliding. These lines were repeatedly placed on the specimen surface during a series of the measurements. According to Taira *et al.* [14], the average amount of grain-boundary sliding was obtained on both specimens with serrated grain boundaries and those with straight grain boundaries during creep at 1311 K [1]. The initiation of grain-boundary cracks on the specimen surface during creep were also examined at 1311 K. The length of the largest grain-boundary crack which was observed along the gauge portion of longitudinally sectioned specimens ruptured at 1311 K was measured using an optical microscope. The relation between largest crack length and local creep strain where the crack was found was then examined.

3. Results and discussion

3.1. Effect of serrated grain boundaries on creep–rupture properties

Fig. 2 shows the creep–rupture strength of specimens with serrated grain boundaries and those with straight grain boundaries of HS-21 alloys at 1311 and 1422 K. The strengthening by serrated grain boundaries is effective under higher stresses at 1422 K, which is only about 100 K below the solution temperature of specimens, while the strengthening effects of serrated grain boundaries are larger at 1311 K. Fig. 3 shows the rupture ductility of the HS-21 alloys at 1311 and 1422 K. The elongation of specimens with serrated grain boundaries is larger than that of specimens with straight grain boundaries at 1311 K, but the elongation is almost the same in both specimens under lower stresses at 1422 K.

Fig. 4 shows the optical micrographs of specimens of HS-21 alloys ruptured under a stress of 19.6 MPa at 1422 K. The tensile direction is vertical in the micrographs. Grain-boundary cracks and voids are visible near fracture surfaces of both the specimen with serrated grain boundaries (Fig. 4a) and that with straight grain boundaries (Fig. 4b). Serrated grain boundaries are retained even after high-temperature exposure of the specimen (Fig. 4a). Grain boundaries in the specimen with originally straight grain boundaries (Fig. 1a) are also serrated with large grain-boundary precipitates (Fig. 4b). This suggests that specimens with originally straight grain boundaries were strengthened by serration of grain boundaries due to the carbide precipitation on grain boundaries during high-temperature long-term exposure. Carbide precipitates are

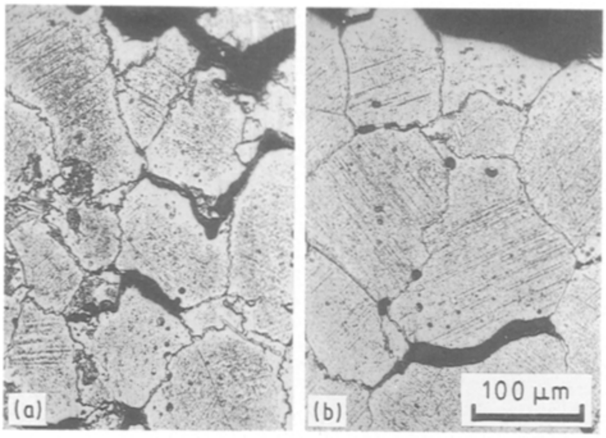

Figure 4 Optical micrographs of specimens of HS-21 alloys ruptured under a stress of 19.6 MPa at 1422 K (electrolytically etched with 10% aqueous chromic acid. (a) Specimen with serrated grain boundaries. (b) Specimen with straight grain boundaries.

also visible in the grains of both specimens (Fig. 4a and b).

3.2. Grain-boundary sliding in specimens during creep at 1311 K

Fig. 5 shows the average amount of grain-boundary sliding in specimens of HS-21 alloys during creep at 1311 K. The average amount of grain-boundary sliding increases with time in both specimens, but it is not always smaller in specimens with serrated grain boundaries under both stresses of 39.2 and 23.5 MPa. These experimental results are very different from the earlier observations on the austenitic 21Cr–4Ni–9Mn steels [1] in which the amount of grain-boundary sliding is considerably reduced by serrated grain boundaries during creep at 973 K.

Fig. 6 shows the relation between average amount of grain-boundary sliding and creep strain in those specimens of HS-21 alloys crept at 1311 K. The amount of grain-boundary sliding at a given creep

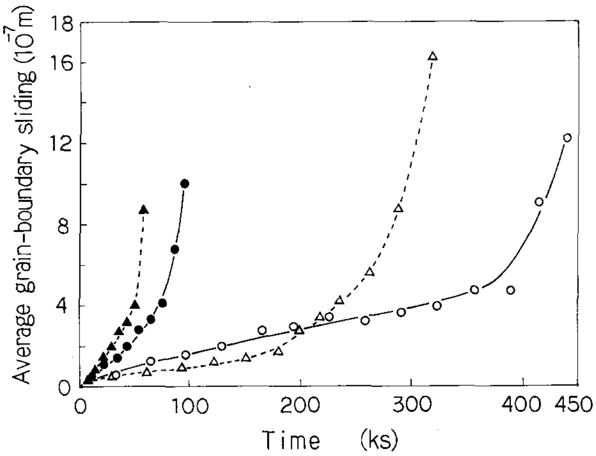

Figure 5 The average amount of grain-boundary sliding in specimens of HS-21 alloys during creep at 1311 K, and (▲, ●) 39.2 MPa and (△, ○) 23.5 MPa. (▲, △) Straight grain boundaries, (●, ○) serrated grain boundaries.

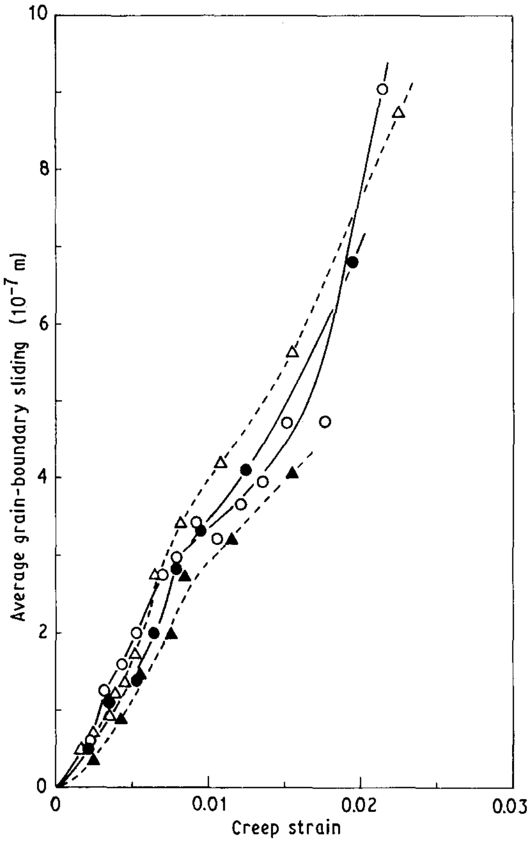

Figure 6 The relation between average amount of grain-boundary sliding and creep strain in specimens of HS-21 alloys during creep at 1311 K, and (▲, ●) 39.2 MPa, and (△, ○) 23.5 MPa. (▲, △) Straight grain boundaries, (●, ○) serrated grain boundaries.

strain is almost the same in both specimens with serrated grain boundaries and those with straight grain boundaries. The amount of grain-boundary sliding in both specimens is somewhat larger under the lower stress (23.5 MPa) at small creep strains.

Fig. 7 shows examples of grain-boundary sliding observed on specimens of HS-21 alloys crept under a stress of 39.2 MPa at 1311 K. Displacement of scratch markers at grain boundaries is visible in both the specimen with serrated grain boundaries (Fig. 7c) and the one with straight grain boundaries (Fig. 7d), although it is not clear in the micrographs of low magnification (Fig. 7a and b). The recrystallization which was probably induced by work hardening in scratching marker lines on the specimen surface also occurred along some marker lines. The measurement of grain-boundary sliding was not made on these lines, because the recrystallization affects the grain-boundary sliding during creep [15].

The amount of grain-boundary sliding generally increases with an increase of temperature [16, 17]. Langdon and Vastava [16] discussed the correlation of grain-boundary sliding with diffusion of atoms. The serrated grain boundaries may be ineffective in blocking the grain-boundary sliding owing to the enhanced diffusional recovery at very high temperatures. This is why the average amount of grain-boundary sliding during creep at 1311 K is almost the same in both specimens with serrated grain boundaries and those with straight grain boundaries.

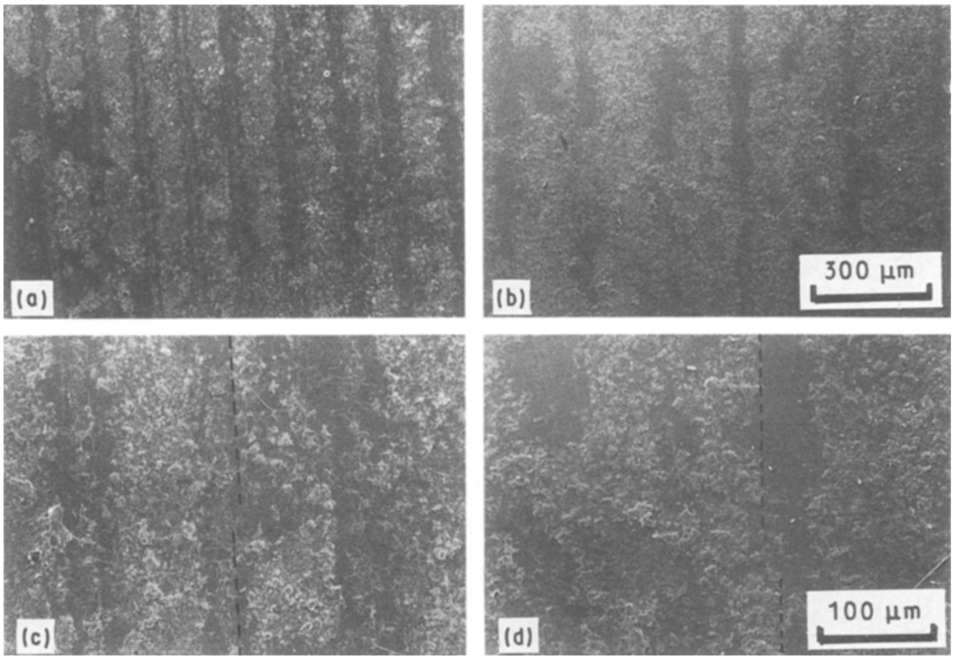

Figure 7 Examples of grain-boundary sliding in specimens of HS-21 alloys crept for 43.2 ks under a stress of 39.2 MPa at 1311 K. (a, c) Specimen with serrated grain boundaries. (b, d) Specimen with straight grain boundaries.

3.3. Initiation and growth of grain-boundary cracks

The serrated grain boundaries are still effective in retarding the growth of grain-boundary cracks at very high temperatures. Fig. 8 shows examples of creep curves of HS-21 alloys at 1311 K. Grain-boundary cracks or voids (arrowed) initiated in the early stage of creep due to serious oxidation in both specimens with serrated grain boundaries and those with straight grain boundaries. The grain-boundary cracks or voids were found at about 30%–40% of the rupture life of specimens crept under a stress of 39.2 MPa, but these were detected at only 13%–18% of the rupture life under a stress of 23.5 MPa. Thus, the strengthening by serrated grain boundaries is effective in retarding the growth and linkage of grain-boundary cracks or voids at high temperatures above 1311 K.

Fig. 9 shows grain-boundary cracks and voids observed on specimens of HS-21 alloys crept under a stress of 39.2 MPa at 1311 K. The tensile direction is horizontal in the micrographs. Grain-boundary voids can be observed along the grain boundaries in the specimen with serrated grain boundaries (Fig. 9a), while a small crack is visible in the specimen with straight grain boundaries (Fig. 9b). Grain-boundary voids similar to those found in specimens with serrated grain boundaries were also observed on specimens with straight grain boundaries ruptured at 1422 K (Fig. 4) and those ruptured under lower stresses at 1311 K. The previous study [13] showed that the ductile grain-boundary fracture occurred in specimens with straight grain boundaries, as well as in those with serrated grain boundaries, owing to enhanced diffusional recovery under lower stresses at 1311 K. Therefore, the occurrence of ductile fracture [4, 11] may

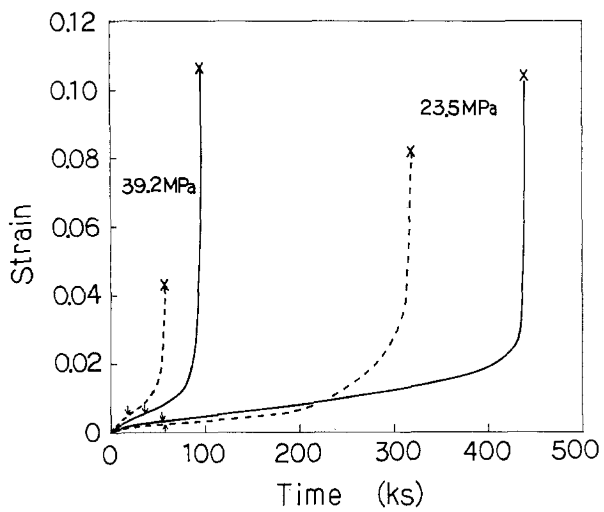

Figure 8 Examples of creep curves of HS-21 alloys at 1311 K. (- - -) Straight grain boundaries, (——) serrated grain boundaries. Arrows indicate crack initiation.

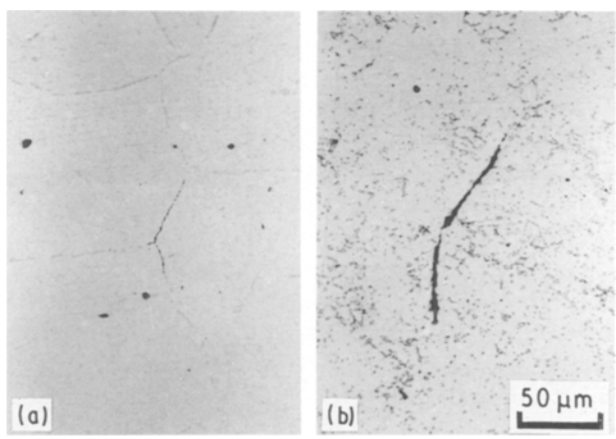

Figure 9 Grain-boundary cracks and voids observed in specimens of HS-21 alloys crept under a stress of 39.2 MPa at 1311 K (not etched). (a) Specimen with serrated grain boundaries (t = 36.0 ks). (b) Specimen with straight grain boundaries (t = 32.4 ks).

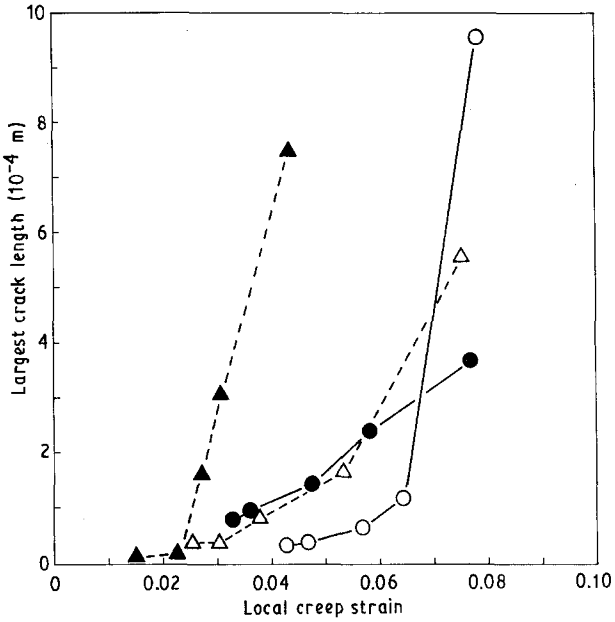

Figure 10 Relation between length of the largest grain-boundary crack and local creep strain in specimens of HS-21 alloys ruptured at 1311 K, and (▲, ●) 39.2 MPa and (△, ○) 23.5 MPa. (▲, △) Straight grain boundaries, (●, ○) serrated grain boundaries.

contribute little to the strengthening at high temperatures above about 1311 K.

Fig. 10 shows the relation between length of the largest crack and local creep strain in specimens of HS-21 alloys ruptured at 1311 K. The crack length at a given local creep strain is longer in specimens with straight grain boundaries than in those with serrated grain boundaries under both stresses of 23.5 and 39.2 MPa, except at the largest creep strain close to fracture strain. These results also indicate that the strengthening by serrated grain boundaries at very high temperatures above about 1311 K is attributed to the retardation of growth and linkage of grain-boundary cracks and voids. The strengthening mechanisms may be related to the decrease of crack growth rate owing to the decrease of the stress intensity when a crack propagates on serrated grain boundaries [6–10], the lengthening of crack path [9], and the crack arrest at serrated grain boundaries [12].

4. Conclusions

The effects of serrated grain boundaries on the creep–rupture properties of wrought cobalt-base HS-21 alloys were investigated at 1311 and 1422 K. The amount of grain-boundary sliding and the initiation and growth of grain-boundary cracks or voids were also examined on specimens of the HS-21 alloys crept at 1311 K. The following results were obtained.

1. The rupture strength and ductility were improved by serrated grain boundaries even at very high temperatures above 1311 K. The strengthening by serrated grain boundaries was still effective at 1422 K, which is only about 100 K below the solution temperature of specimens (1523 K). The strengthening effects are larger at 1311 K. Specimens with originally straight grain boundaries had almost the same rupture life and rupture ductility under lower stresses at 1422 K, because these specimens were strengthened by serrated grain boundaries with carbide precipitations formed during high-temperature long-term creep.

2. The average amount of grain-boundary sliding during creep at 1311 K was almost the same in specimens with serrated grain boundaries and those with straight grain boundaries at the same creep strain. The amount of grain-boundary sliding in both specimens increased with time (or with creep strain), and it was a little larger under lower stress at small creep strains. The serrated grain boundaries were not effective in inhibiting the grain-boundary sliding of specimens at high temperatures above about 1311 K.

3. The strengthening by serrated grain boundaries was attributed to the retardation of growth and linkage of grain-boundary cracks and voids. The length of the largest crack observed in specimens ruptured at 1311 K was larger in the specimen with straight grain boundaries than in the one with serrated grain boundaries at a given local creep strain. Grain-boundary cracks and voids initiated in the early stage of creep probably due to severe oxidation of specimen surface at high temperatures above about 1311 K.

Acknowledgements

The authors are grateful to Mr A. Hosoi for his assistance in the creep experiments. They also thank Mitsubishi Metal Corporation for supplying HS-21 alloy bars used in this study.

References

1. M. TANAKA, O. MIYAGAWA, T. SAKAKI, H. IIZUKA, F. ASHIHARA and D. FUJISHIRO, *J. Mater. Sci.* **23** (1988) 621.
2. M. YAMAZAKI, *J. Jpn Inst. Metals.* **30** (1966) 1032.
3. M. KOBAYASHI, O. MIYAGAWA, T. SAGA and D. FUJISHIRO, *Tetsu to Hagané* (*J. Iron Steel Inst. Jpn*) **58** (1972) 751.
4. M. YAMAMOTO, O. MIYAGAWA, M. KOBAYASHI and D. FUJISHIRO, *ibid.* **63** (1977) 1848.
5. M. TANAKA and H. IIZUKA, *Trans. Jpn. Soc. Mech. Eng.* **53** (1987) 483.
6. M. TANAKA, H. IIZUKA and F. ASHIHARA, *J. Mater. Sci.* **23** (1988) 129.
7. H. KITAGAWA and R. YUUKI, *Trans. Jpn Soc. Mech. Eng.* **41** (1975) 1641.
8. M. ISHIDA, *ibid.* **45** (1979) 306.
9. S. SURESH, *Metal. Trans.* **14A** (1983) 2375.
10. V. VITEK, *Int. J. Fracture* **13** (1977) 481.
11. M. TANAKA, O. MIYAGAWA, T. SAKAKI and D. FUJISHIRO, *Tetsu to Hagané* (*J. Iron Steel Inst. Jpn*) **65** (1979) 939.
12. R. OHTANI, M. OKUNO and R. SHIMIZU, *J. Soc. Mater. Sci. Jpn* **31** (1982) 505.
13. M. TANAKA, H. IIZUKA and F. ASHIHARA, *J. Mater. Sci.* **24** (1989) 1623.
14. S. TAIRA, M. FUJINO and M. YOSHIDA, *J. Soc. Mater. Sci. Jpn* **27** (1978) 447.
15. M. R. DRURY, F. J. HUMPHREYS and S. H. WHITE, *J. Mater. Sci.* **24** (1989) 154.
16. T. G. LANGDON and R. B. VASTAVA, ASTM STP 765 (American Society for Testing and Materials, Philadelphia, PA, 1982) p. 435.
17. K. U. SNOWDEN, in "Cavities and Cracks in Creep and Fracture", edited by J. Gittus (Applied Science, London, 1981) p. 259.

Received 9 July 1990
and accepted 6 February 1991