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Improvement of fatigue-crack growth resistance by grain-boundary reaction precipitates at high temperature

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Effects of grain-boundary reaction precipitates on fatigue-crack growth rate were investigated using austenitic 21 wt% Cr–4 wt% Ni–9 wt% Mn heat-resisting steel at 973 K in air. Grain boundaries were serrated by the grain-boundary reaction precipitates. The crack growth rate was considerably decreased by these precipitates, especially at low crack growth rates. Fatigue cracks extended to the serrated grain boundaries or to the interface between the grain-boundary reaction nodule and the grain. Therefore, the cracks grew along zigzag paths, and brittle intergranular fracture was inhibited. The decrease in the fatigue-crack growth rate was explained by these changes in fracture mode.

1. Introduction

Grain boundaries are one of the primary sources for high-temperature fracture in solution-strengthened or precipitation-strengthened heat-resisting alloys. Therefore, the inhibition of intergranular fracture by structural control is effective in improving the high-temperature strength [1]. The grain-boundary strengthening by grain-boundary precipitates is useful to inhibit the intergranular fracture in polycrystalline heat-resisting alloys [2–6]. The grain-boundary reaction (GBR), in which grain boundaries can be considerably serrated by the GBR precipitates [6], is expected to be an effective strengthening method for industrial applications.

The strengthening effect of the serrated grain boundary with the GBR precipitates has been studied using austenitic heat-resisting steels [7–9] and cobalt-base superalloys [10] under high-temperature creep conditions. Moreover, we have found [11] in high-temperature low-cycle fatigue life that the life was largely increased by the GBR precipitates.

It is considered in the strengthening mechanisms with the GBR precipitates that the retardation of grain-boundary sliding [9] and the occurrence of the ductile fracture at the interface of the GBR nodule [8] are the important factors in improvement of the high-temperature strength. Micromechanical analysis has also been performed [12, 13] on the initiation of a wedge-type crack in alloys with serrated grain boundaries, and it was found that the effect of atomic diffusion decreases the stress concentration extent at grain-boundary triple junctions. However, the effect of serrated grain boundaries on the crack growth rate has not been studied sufficiently under high-temperature low-cycle fatigue conditions.

In this paper, low-cycle fatigue-crack growth properties were examined using an austenitic 21 wt % Cr–4 wt % Ni–9 wt % Mn heat-resisting steel with serrated grain boundaries at high temperature. The mechanism of improvement of the fatigue-crack growth resistance is discussed, based on the results of microstructural observations.

2. Material and experimental procedure

The material used was an austenitic 21Cr–4Ni–9Mn heat-resisting steel for exhaust valves in internal-combustion engines. The chemical composition is listed in Table I. Two kinds of heat-treated specimens with different amounts of GBR were used in this study. Heat treatment, the amount of GBR and the matrix hardness of those specimens are listed in Table II. The amount of GBR was estimated from the area fraction of the GBR nodule. These specimens had almost equal matrix hardness. The grain diameter was about 78 μm in all specimens. Grain boundaries were almost straight in the specimens with 2% GBR. However, they were considerably serrated by $M_{23}C_6$ carbides in the specimens with amounts of GBR of about 8% (8% GBR). Fig. 1 shows the serrated grain boundaries of the specimen with 8% GBR and the straight grain boundaries of the specimens with 2% GBR.

Sandglass-shaped test-pieces with 12 mm gauge length and 6 mm diameter were machined from these two types of heat-treated specimens. A notch was introduced at the centre of the gauge length using a thin grinder wheel (0.3 mm thick). The depth of the notch was 0.25 mm, and the surface length of it was about 2.4 mm. Fatigue-crack growth rate (FCGR) was measured after the crack extended to about

TABLE I Chemical composition of the steel used (wt %)

Steel	C	N	Cr	Mn	Ni	Si	P	S	Fe
21Cr–4Ni–9Mn	0.55	0.41	20.44	8.95	3.72	0.04	0.027	0.008	bal.

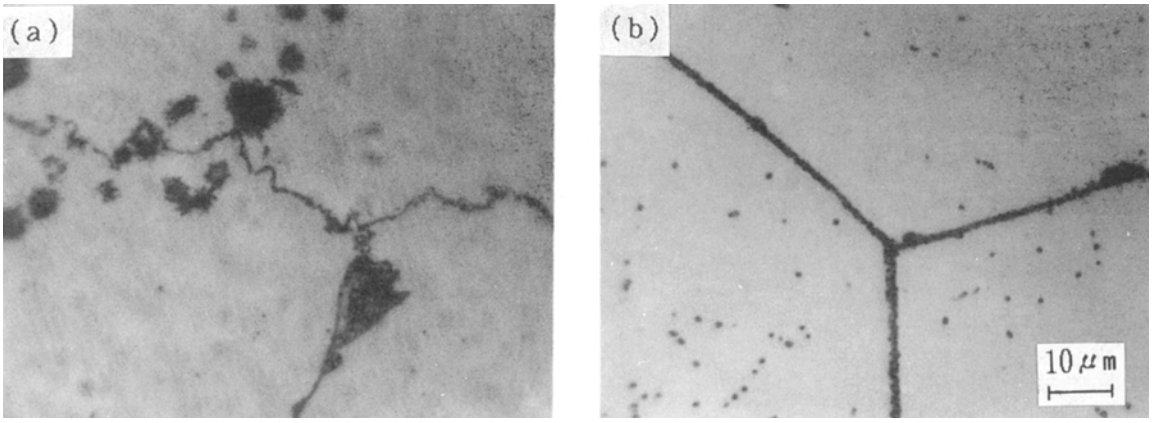

Figure 1 Optical micrographs of 21Cr–4Ni–9Mn steel. (a) 8% GBR, (b) 2% GBR.

0.15 mm from the notch root on the specimen surface, and the rate was then measured up to 6 to 7 mm surface crack length.

The FCGR was examined using strain-controlled symmetrical triangular wave shapes with the total strain range ($\Delta\varepsilon_t$) of 1.0% at 973 K in air. The tests were carried out under cyclic strain rates of $3.3 \times 10^{-3}\,\text{sec}^{-1}$ (fast–fast cycling) and $5 \times 10^{-5}\,\text{sec}^{-1}$ (slow–slow cycling). The fatigue life was about 2000 cycles for 2% GBR, about 15 000 cycles for 8% GBR in the tests with fast–fast cycling, and was about 400 cycles for 2% GBR, 4000 cycles for 8% GBR in the tests with slow–slow cycling [11]. The crack length was measured on replicas of the specimen surfaces using an optical micrograph. The crack growth rate was calculated from the gradient of the relation between the surface crack length and the number of fatigue cycles.

3. Experimental results and discussion

3.1. Crack growth rate

Fig. 2 shows the effect of serrated grain boundaries on the FCGR in the tests with fast–fast cycling. The FCGR was considerably lower in the specimens with 8% GBR. In particular, the strengthening effect was remarkable under low FCGR conditions. Fig. 3 shows the effect of serrated grain boundaries in the tests with slow–slow cycling. The effect was large under low FCGR conditions. These properties were similar to those in the tests with fast–fast cycling.

The relation between the fatigue crack growth rate and stress intensity factor range at the crack tip (ΔK) was then examined. The stress intensity factor was calculated using the results of the finite element method [14]. The configuration of the surface cracks was observed and the values of aspect ratio, λ ($\lambda = a/c$, a = crack depth, $2c$ = crack length on the specimen surface) were measured. λ was much the same in the specimens with 8% and 2% GBR, and did not change with the crack length from about 2.8 to 7.0 mm. The average value of λ was about 0.95 in the tests with fast–fast cycling and was about 1.05 in the tests with slow–slow cycling in the both kinds of specimen.

Fig. 4 shows the relation between the FCGR and the stress intensity factor range (ΔK) in the tests with fast–fast cycling. The FCGR was considerably lower in the specimens with 8% GBR, especially under conditions of low ΔK. Fig. 5 shows the relation between FCGR and ΔK in the tests with slow–slow cycling. The effect of serrated grain boundaries was considerably larger under low FCGR conditions, similar to those in the tests with fast–fast cycling. Because the period of low FCGR is relatively long in many fatigue conditions, the improvement of the crack growth resistance under low FCGR conditions is important to increase the fatigue life.

3.2. Microstructural observation

Fig. 6 shows the microstructures near the crack tip of specimens in the tests with fast–fast cycling. Loading direction was horizontal in the photographs. Cracks mainly propagated intergranularly, though they partially grew transgranularly in both kinds of specimen with 8% and 2% GBR. The cracks propagated along the serrated grain boundaries or along the interfaces between the GBR nodule and the grain in the specimens with 8% GBR. The cracks were then largely deflected by the GBR precipitates. The height and width of the deflected portions of the cracks were from about a few to 10 μm on the serrated grain boundaries, and from about 10 to 30 μm at the GBR nodule. On the other hand, the cracks propagated along straight grain boundaries in the specimens with 2% GBR.

Fig. 7 shows the microstructures near the crack tip of specimens in the tests with slow–slow cycling. Cracks similarly propagated on the serrated grain boundaries or on the interfaces between the GBR nodule and the grain in the specimens with 8% GBR. The cracks were largely deflected by the GBR

TABLE II Heat treatment, amount of grain-boundary reaction (GBR) and matrix hardness of specimens

Heat treatment	Amount of GBR(%)	Matrix hardness (5 N)
1473 K × 1 h → AC + 1023 K × 3 h → AC	2	285 Hv
1473 K × 1 h → AC + 1073 K × 30 min → AC	8	283 Hv

AC = air cooled.

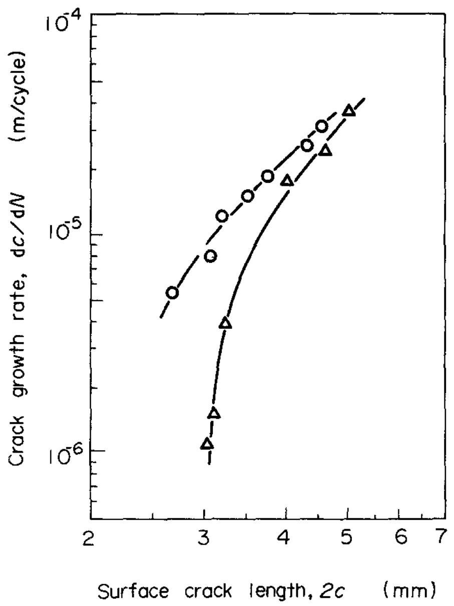

Figure 2 Surface-crack growth rate in tests with fast–fast cycling (△) 8% GBR, (○) 2% GBR.

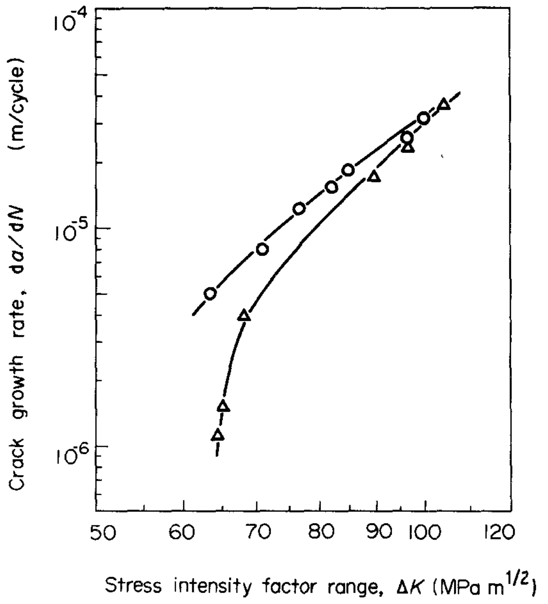

Figure 4 Relation between surface-crack growth rate and stress intensity factor range, ΔK, in the tests with fast–fast cycling. (△) 8% GBR, (○) 2% GBR.

precipitates. The height and the width of the deflected portions of the cracks were much the same in the tests with fast–fast cycling. Some intergranular wedge-type cracks were observed ahead of the main crack as shown in Fig. 7b.

Fig. 8 shows the effect of the GBR nodule on the detour of crack path in the tests with slow–slow cycling. The same detours were observed in the tests with fast–fast cycling. Loading direction was longitudinal and the fatigue cracks propagated from right to left in the photographs. Fig. 8a shows that intergranular fracture is difficult at the interface between the GBR nodule and the neighbouring grain. The crack extended around the GBR nodule. Fig. 8b shows the zigzag crack path on the interface between the GBR nodule and the forward grain. Fig. 8c shows the fatigue cracks which look like transgranular cracks. These were, however, also related to the fracture on the interface between the GBR nodule and the forward grain.

These microstructural observations show that the brittle intergranular fracture, which occurred on the straight grain boundaries, is inhibited by the GBR precipitates. This inhibition may be explained by the retardation of grain-boundary sliding [9] and the decrease of the stress concentration at grain-boundary triple junctions by the serrated grain boundaries [13]. The occurrence of ductile fracture at the interface of the GBR nodule [8] is also an important factor to improve the crack growth resistance.

The strengthening by the GBR precipitates was effective in low FCGR conditions of less than about 30 μm/cycle in both the fast–fast and slow–slow cycling tests, as shown in Figs 2 to 5. Namely, the effects of serrated grain boundaries were large under low crack growth rate conditions. The value of 30 μm agrees with the size of the deflected portion of the crack at the GBR nodule. Under these conditions, where ΔK was relatively low, grain-boundary sliding may be inhibited by the serrated grain boundaries.

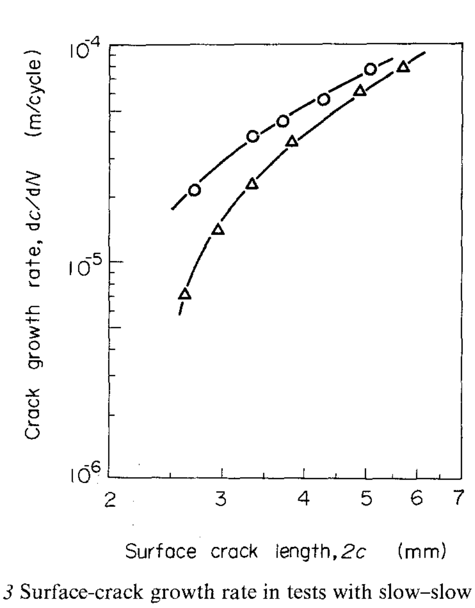

Figure 3 Surface-crack growth rate in tests with slow–slow cycling. (△) 8% GBR, (○) GBR.

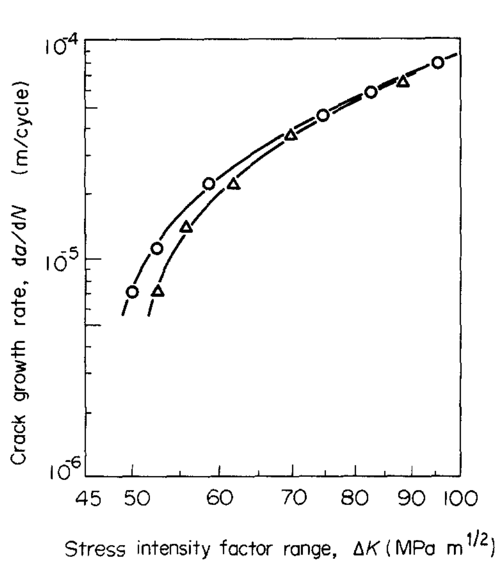

Figure 5 Relation between surface-crack growth rate and stress intensity factor range, ΔK, in the tests with slow–slow cycling. (△) 8% GBR, (○) 2% GBR.

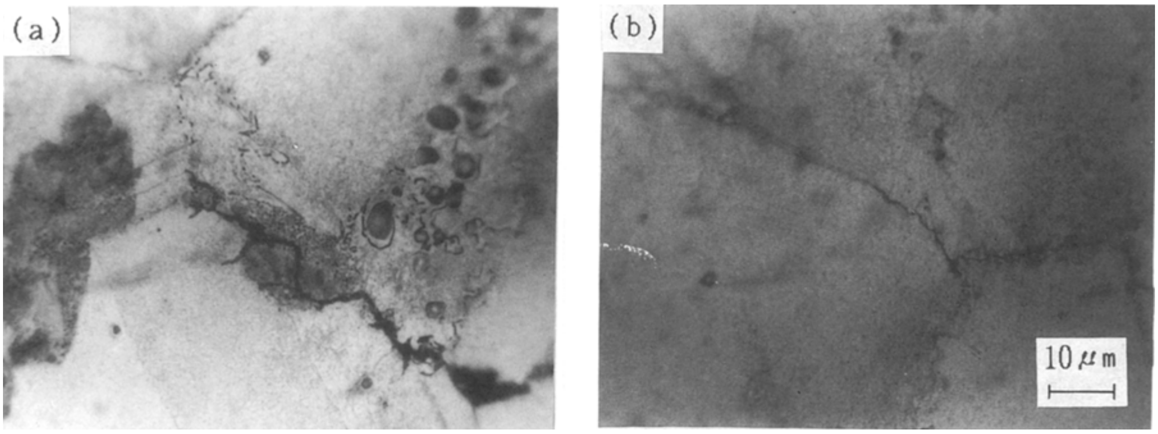

Figure 6 Microstructures near the crack tip in the specimens fatigued by fast–fast cycling (crack depth, $a \simeq 1.5$ mm). (a) 8% GBR, (b) 2% GBR.

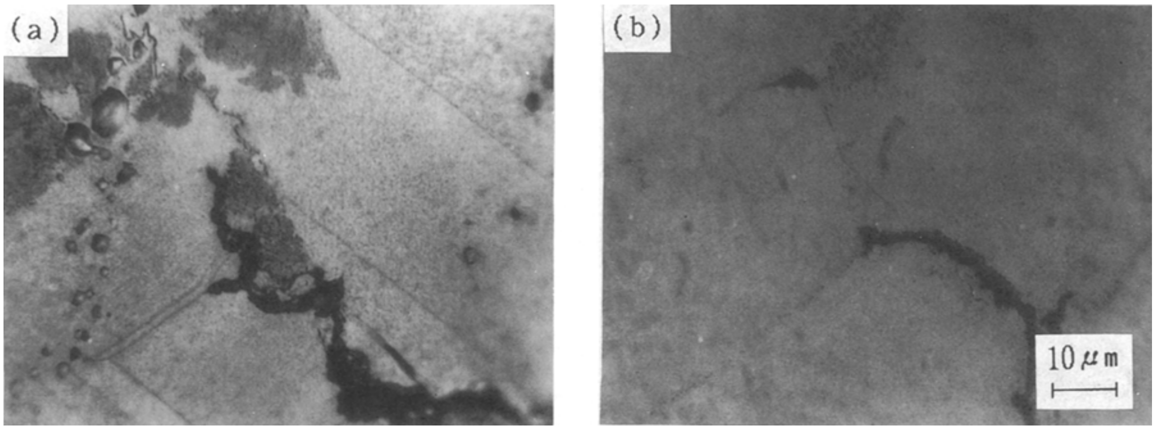

Figure 7 Microstructures near the crack tip in the specimens fatigued by slow–slow cycling (crack depth, $a \simeq 1.5$ mm). (a) 8% GBR, (b) 2% GBR.

4. Conclusions

The effects of serrated grain boundaries caused by grain-boundary reaction precipitates on the improvement of high-temperature low-cycle fatigue strength were investigated using an austenitic 21Cr–4Ni–9Mn steel at 973 K in air. The results obtained were as follows:

1. The fatigue life was longer in specimens with serrated grain boundaries than in those with straight grain boundaries in tests with fast–fast and slow–slow

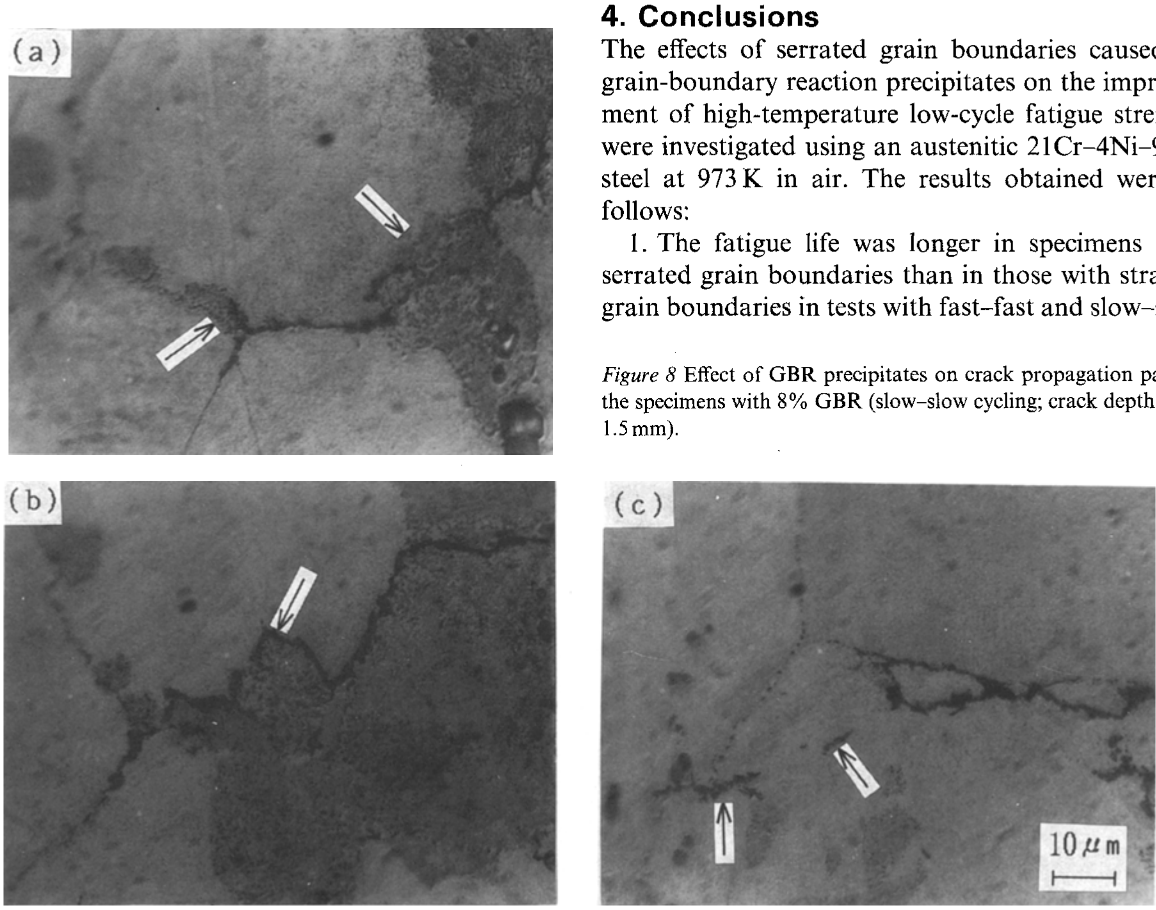

Figure 8 Effect of GBR precipitates on crack propagation path in the specimens with 8% GBR (slow–slow cycling; crack depth, $a \simeq 1.5$ mm).

cycling. The effects of serrated grain boundaries were large, especially under low fatigue-crack growth-rate conditions.

2. In specimens with serrated grain boundaries, cracks mainly propagated along the serrated grain boundaries or along the interfaces between the grain-boundary reaction nodule and the grain. Therefore, the cracks grew along zigzag paths, and brittle intergranular fracture was inhibited.

3. The effects of serrated grain boundaries on the fatigue-crack growth resistance were especially large under low crack growth rate conditions in which the amount of crack extension in one fatigue cycle was less than one deflected portion of the crack at the grain-boundary reaction nodule.

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