Subsequent recrystallizations technique of local plastic strain in 304 stainless steel

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The recrystallization technique has been successfully used to observe and measure local plastic zone and plastic strain in engineering materials: mild steel [1], high-tensile strength steel [2], austenitic stainless steel [3, 4], heat-resistant alloys [5], etc. Plastic strain within the plastic zone is to be determined from the recrystallized grain size by use of the relationship between strain and the grain size. However, the accuracy of the measured strain is not good because of the large scatter of the grain size for a given strain and the small variation of the grain size on strain, especially in the high-strain region.

To avoid these ambiguities and to reveal plastic zones with different amounts of strain more clearly, a multiple recrystallizations technique was developed in the present study; the recrystallization temperature is subsequently increased stepwise in order to observe at first the plastic zone with high plastic strain, next the plastic zone with lower plastic strain, and so on.

The present experiment was carried out on 304 stainless steel, a commercially available 304 stainless steel plate (5.8 mm thick). To find the suitable recrystallization conditions, the indentation technique [5] was adopted, i.e. the polished surface was indented with a conical indentor with angle of 60° under a compression load of 1470 N. The indented surface was polished carefully using waterproof emery papers to #1500. The specimens were annealed in a vacuum furnace for 86.4 ks, first at 1023 K, secondly at 1173 K and finally at 1223 K. Conventional recrystallization annealing (only at one temperature) was also carried out to check whether the multiple heat treatments affect the recrystallization response.

Fig 1. shows the recrystallization response. In the annealing at 1023 K and 1123 K, thermal etching was poor. The specimens were therefore polished and etched in a etchant (C_2H_5OH:HCl:HNO_3 = 100:15:30). In the annealing at 1173 and 1223 K, thermal etching during annealing was good. The micrographs of the thermal etched surface are shown in Fig. 1. It is seen that the recrystallized zone increases with increasing temperature. This means that the plastic zone with and above a critical plastic strain, ε_{CR}, can be observed (ε_{CR} being the critical strain for the occurrence of recrystallization).

In the annealing at 1023K (Fig. 1b), the recrystallized zone is only near the indentation edge, about 30–40 μm. The darkly etched zone around the recrystallized zone would be due to the carbide precipitation in the strained grains. In the annealing

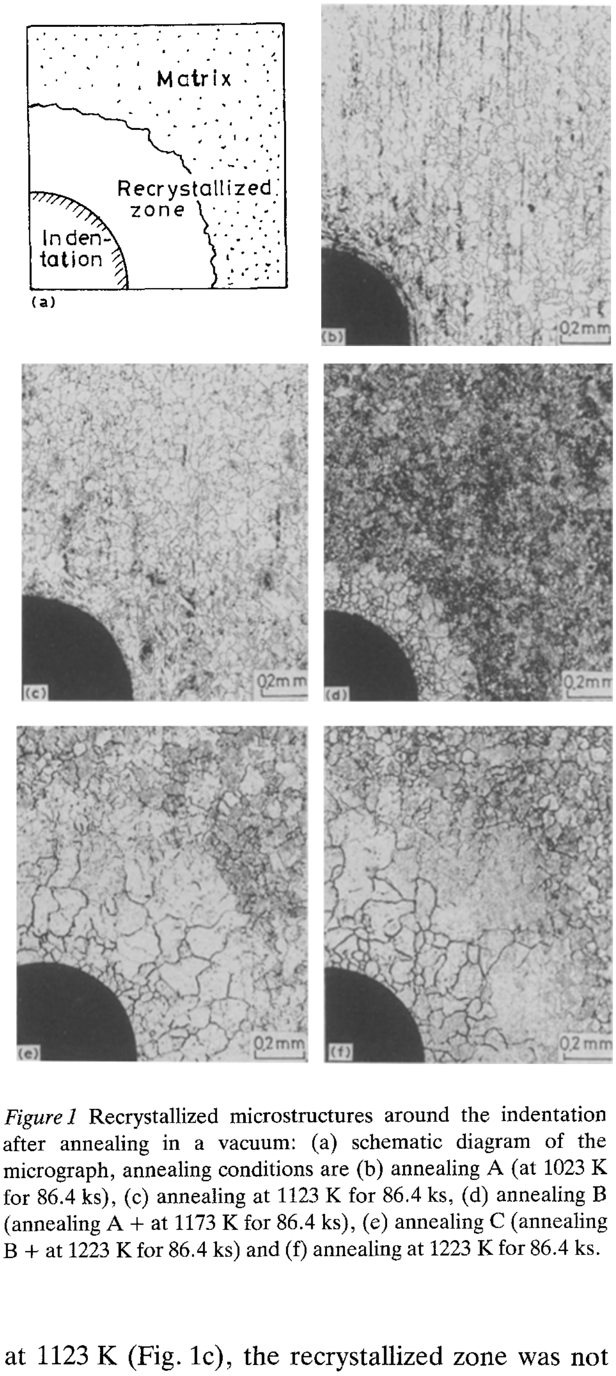

Figure 1 Recrystallized microstructures around the indentation after annealing in a vacuum: (a) schematic diagram of the micrograph, annealing conditions are (b) annealing A (at 1023 K for 86.4 ks), (c) annealing at 1123 K for 86.4 ks, (d) annealing B (annealing A + at 1173 K for 86.4 ks), (e) annealing C (annealing B + at 1223 K for 86.4 ks) and (f) annealing at 1223 K for 86.4 ks.

at 1123 K (Fig. 1c), the recrystallized zone was not clear, so the heat treatment was not done thereafter in the present experiment. It should be noted that the recrystallization response is quite similar between the subsequent annealing (Fig. 1e), and the single annealing (Fig. 1f).

To determine ε_{CR}, smooth tensile specimens were tensile-loaded to various amounts of true strain ε at 295 K. After cutting, polishing and annealing (in the case of annealing at 1023 K, further etching), the

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recrystallization response was examined. We refer hereafter the annealing conditions by upper-case letters; A = at 1023 K for 86.4 ks, B = A + at 1173 K for 86.4 ks and C = B + at 1223 K for 86.4 ks. The result is shown in Fig. 2. The very highly strained zone with $\varepsilon \geqslant 0.5$ can be observed by annealing A, a highly deformed zone with $\varepsilon \geqslant 0.12$

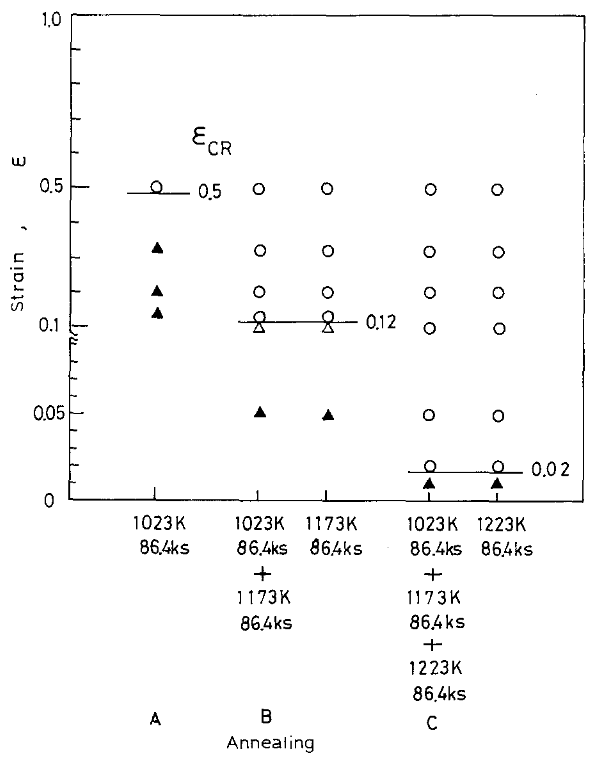

Figure 2 Critical strain for recrystallization, ε_{CR}, versus recrystallization conditions: (▲) no recrystallization, (△) 50% recrystallization and (○) full recrystallization.

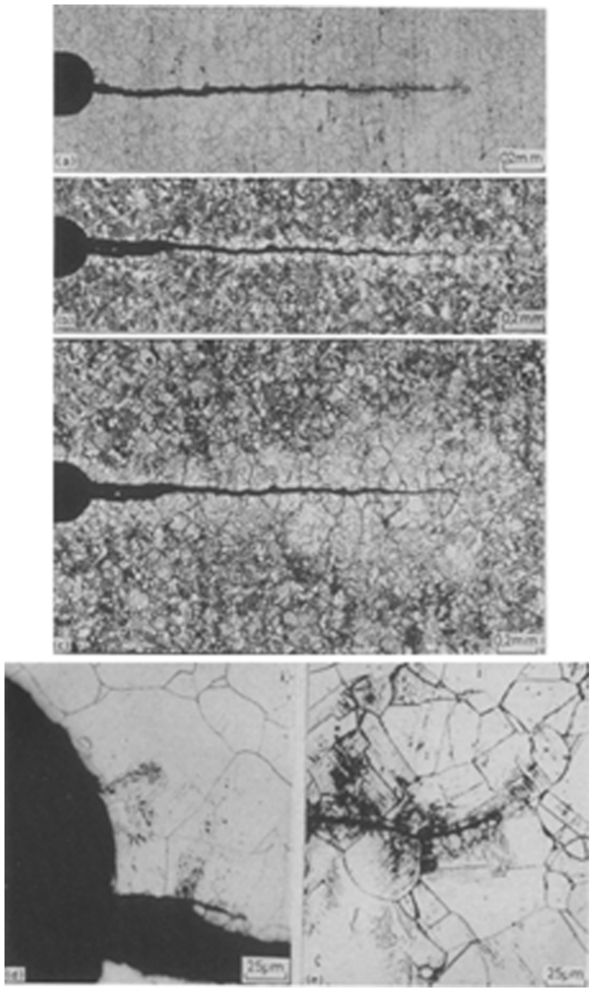

Figure 3 Microstructures around a fatigue crack after annealing. Anneal conditions are (a) A, (b) B, (c) C, and (d) and (e) A. (d) Very tiny fatigue crack in a grain at notch root (enlarged view of notch root in a) and (e) crack tip (very small recrystallized grains are formed).

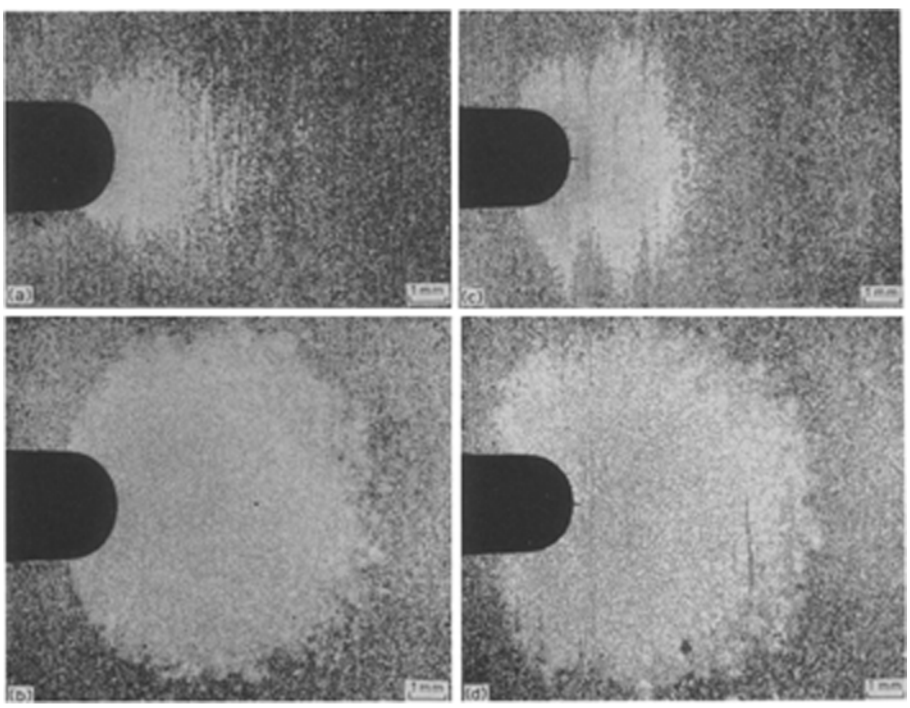

Figure 4 Macroscopic recrystallized zones in monotonic tension (a, b) and low-cycle fatigue (c, d). Annealing conditions are (a) and (c) B, (b) and (d) C. The ratio of notch length a to ligament w of the CT specimen was 0.6. Maximum load in tension was 6.25 kN. Maximum tensile and compressive load was 6.25 kN and −6.25 kN. Number of cycles was 45 (= visible crack initiation). Unloaded from the tensile part of the cycle.

by annealing B or by a single annealing at 1173 K for 86.4 ks, and a plastic zone with $\varepsilon \geqslant 0.02$ by annealing C or by a single annealing at 1223 K for 86.4 ks. Even if the specimen is only one, the above plastic zones can be measured by this subsequent recrystallization technique; first annealing A, secondly annealing B and finally annealing C. The recrystallized zone in the fatigued specimen is, as shown in [3], the accumulated plastic zone and the corresponding strain is the accumulated plastic strain ε_{ac}.

Some examples of the local plastic zones are illustrated in the following. The specimen is 0.8 CT (0.8 in compact tension specimen 5.8 mm thick). Fig. 3 shows the accumulated plastic zone near mid-thickness around a high-cycle fatigue crack. Very localized highly deformed zone (the small grains at crack) is seen by annealing A (Fig. 3d and e), which is not visible at low magnification (Fig. 3a) and would never be revealed by annealing at higher temperatures. A plastic zone with accumulated plastic strain $\varepsilon_{ac} \geqslant 0.12$ (Fig. 3b) and 0.02 (Fig. 3c) is clearly seen.

Figs 4–6 are comparisons of the monotonic plastic zones in tension and the accumulated plastic zones in

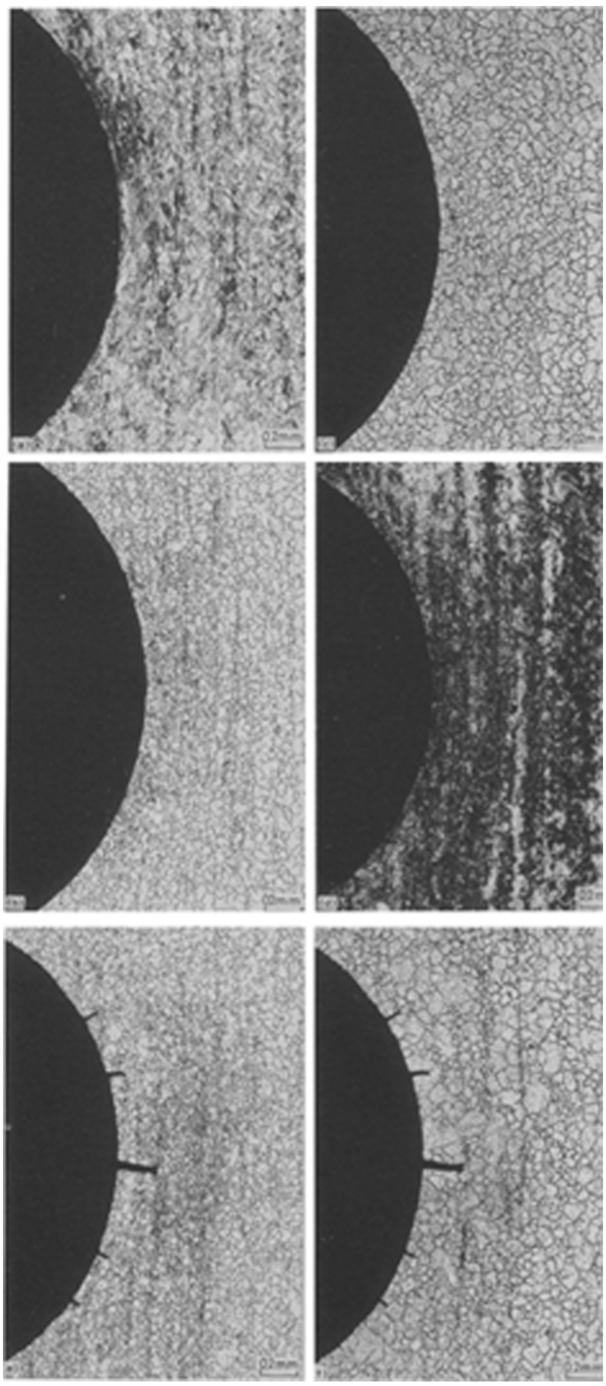

Figure 5 Microstructures around notch tip after annealing in monotonic tension (a–c) and in low-cycle fatigue (d–f). Annealing conditions are (a) and (d) A, (b) and (e) B, and (c) and (f) C. (b) and (c) are enlarged views of the notch tip in Fig. 4a and b. (e) and (f) are enlarged views of the notch tip in Fig. 4c and d. The occurrence of the secondary recrystallization by annealing C is obvious by comparing (b) and (c), and (e) and (f).

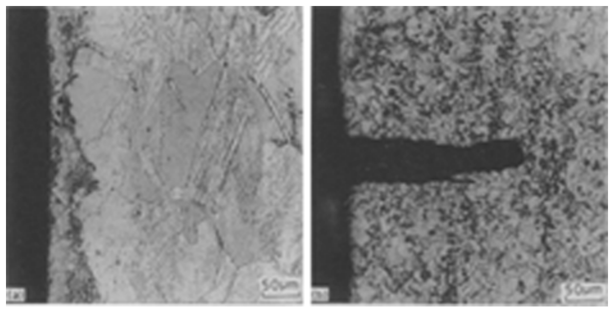

Figure 6 Enlarged view of (a) Fig. 5a and (b) Fig. 5d. Very small recrystallized grains are seen at the very edge of the notch tip in tension (a) and around the low-cycle fatigued notch tip (b).

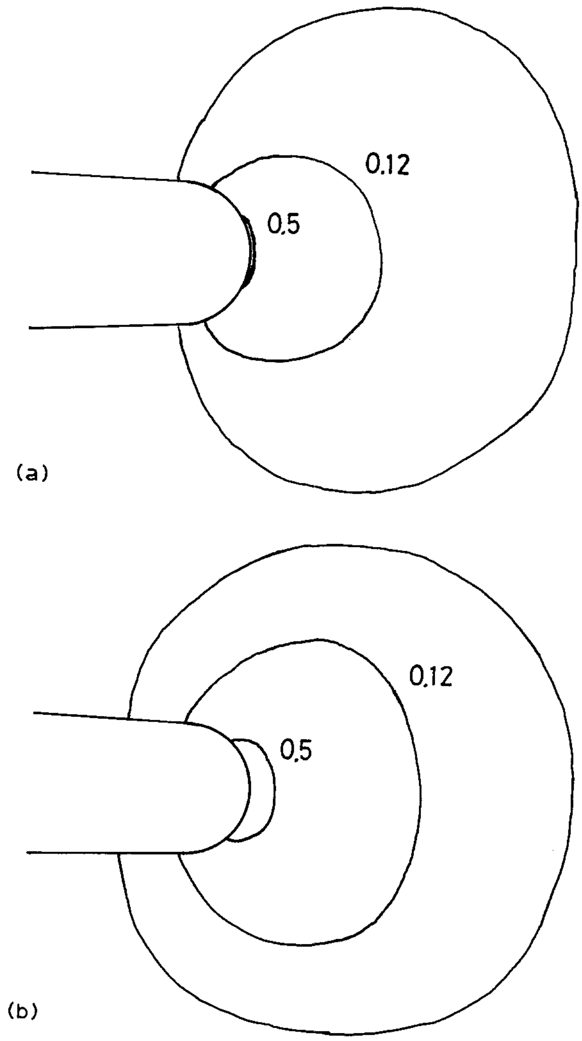

Figure 7 Distribution of plastic strain around the notch in tension ($\varepsilon = 0.02$) (a) and of accumulated plastic strain around the notch in low-cycle fatigue ($\varepsilon_{ad} = 0.02$) (b).

low-cycle fatigue (fully reversed load control) near mid-thickness around the notch (radius $\rho = 1$ mm), which was electric-discharge-machined followed by polishing with emery papers. The plastic zone with $\varepsilon \geqslant 0.02$ and $\varepsilon_{ac} \geqslant 0.02$ is almost the same for tension and fatigue (Fig. 4b and c). However, fatigue damage accumulation near the notch tip is significant. The size of the accumulated plastic zone with $\varepsilon_{ac} \geqslant 0.12$ (Fig. 4c) is larger that that ($\varepsilon \geqslant 0.12$) in monotonic tension (Fig. 4a). The size of the highly deformed zone with $\varepsilon_{ac} \geqslant 0.5$ is significantly large in fatigue (compare Fig. 5a and d; i.e. the distance from the notch tip to the zone ($\varepsilon \geqslant 0.5$) front is 10–20 μm in tension (Fig. 6a) and that to the zone ($\varepsilon_{ac} \geqslant 0.5$) front is 400–450 μm in low-cycle fatigue (Fig. 5d).

From the above observations of plastic zones with various values of ε_{CR}, the stain distribution around the notch of the present specimens can be drawn as in Fig. 7.

This technique is now being used to study the process of low-cycle fatigue damage accumulation around notches.

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