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Materials

TEM Study of Microcrack Nucleation and Propagation for 310 Stainless Steel*

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Abstract: TEM in situ tensile tests of 310 stainless steel show that a dislocation free zone (DFZ) forms if the displacement keeps constant after dislocations are emitted from a crack tip. The elastic DFZ is gradually thinned and the stress in the DFZ will reach the cohesive strength, resulting in nucleation of nanocracks in it and their bluntness into voids. If continuously tensioning, the inhomogeneously thinning ahead of the crack tip, initiating and connecting of microcracks or microvoids will be observed rather than a DFZ, nanocracks' initiation and bluntness into voids. The inverse pile-up ahead of a loaded crack tip can move back to the crack tip when unloading.
Key words: 310 stainless steel, in situ TEM tension, dislocation free zone, initiation of nanocrack

In situ TEM observation is a powerful method to study microcrack nucleation and propagation. Our In situ tensile tests by TEM[1~3] showed that in either ductile or brittle materials, many dislocations could be emitted from a loaded crack tip and a dislocation free zone (DFZ) formed after keeping constant displacement. The DFZ is an elastic zone. The stress within it is very large. Even though keeping constant displacement, the thermal activity could also facilitate dislocations emitting, multiplicating and moving, resulting in the region near the crack tip inhomogeneously thinning gradually. The stress within a certain area will be up to or larger than the cohesive strength, causing nanocrack nucleating in the DFZ. For ductile materials[1,4], the nanocracks that nucleate in DFZs will blunt into voids. For brittle materials[2,3], however, the nanocracks will cleave continuously.

The previous work of in situ tensile tests[5~9] indicated that, for fcc ductile metals, the zone was thinned continuously ahead of the loaded crack tip by crossing slip, leading to shear voids which would propagate in two directions and then connect with the main crack, resulting in a zig-zag crack propagation. In these tests, dislocations emitted from crack tip could not reach equilibrium because of tensioning continuously. So DFZ, as well as initiation and bluntness of nanocracks within a DFZ would not be observed. In a word, the early process of nanocracks nucleation in DFZs could not be observed under quick tension. The present work is to certify the opinion mentioned above, i. e. for ductile materials, nanocracks nucleate and then blunt into voids under constant displacement after the crack tip emits dislocations. If continuously tensioning, however, only were microvoids initiation and connection through duplex slip observed.

1 Experimental Procedures

Stable austenitic stainless steel of type 310 was used. Its chemical compositions are (mass fraction,%): Cr-24.48, Ni-21.33, Nb-0.23, C-0.06. The thin strip with thickness of 0.2 mm was austenitized at 1 050℃ in argon atmosphere for 20 min followed by quenching into water at room temperature.

Firstly, the sheet was thinned to about 50 μm by chemical polishing in a solution consisting of HCl (30 ml), H_2SO_4 (25 ml) and H_2O (25 ml). Then the specimens were electropolished until a hole was created in a methanol solution containing 10% H_2SO_4. Finally, the hydrogen introduced during electropolishing was removed by ion bombardment, as well as the specimens being cleaned. In situ tensile tests were carried out in a H-800 microscope which minimum elongation rat is 0.05 μm/s. In order to investigate the formation of DFZs as well as the nucleation of nanocracks or microvoids within, most observations were made

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under constant displacement.

2 Results and Discussions

During electropolishing, some microcracks were formed around the hole of a specimen. Once the applied stress intensity K_I exceeds a critical value K_{Ie}, the crack that was vertical to the tensile axis will emit dislocations during tensioning (Fig. 1a). In Fig. 1(a), all the dislocations with bow shape are protruding towards the direction $\overrightarrow{AN}$, which proves that dislocations are moving away from the crack tip. If unloading a little, the crack tip would stop emitting dislocations, and all the dislocations are bowed oppositely, which indicates that dislocations are moving towards the crack tip. The oppositely bowing is due to the attraction of dislocation image force. In addition, in Fig. 1(a), the extended dislocation E is in the middle of symbol NN. In Fig. 1(b), however, E has been at the end of sign NN. This shows that the extended dislocation E has moved about 100 nm towards the crack tip during unloading.

In another specimen, after the crack tip emitted dislocations, it was observed that a group of dislocations piled up ahead of crack tip A and reached equilibrium under constant displacement, as shown in Fig. 2. During largely tilting, no dislocation could be observed between A and B. Hence AB is a dislocation free zone (DFZ). Our experimental observation showed that, even in the same specimen, the widths of DFZs were different ahead of different crack tips.

Since the DFZ is an elastic zone, the stress ahead of crack tip is[6]

$$\sigma_y = \frac{K_{Ia}}{\sqrt{2\pi r}} - \int_c^d \frac{\mu b}{2\pi(t-r)} \cdot \left(\frac{t}{r}\right)^{1/2} \cdot f(t)\mathrm{d}t \qquad (1)$$

where the first term is stress concentration caused by the applied load, and the second is contribution of the piled up dislocations. t is the distance from crack tip to the leading dislocation of the inverse pile-up. $f(t)$ is the density of piled up dislocations which piling length is $d-c$. It is known from equation (1) that the inverse pile-up reduces the notch stress $K_{Ia} / \sqrt{2\pi r}$ without changing the singular character of crack tip (i. e. when $r \to 0$, $\sigma \to \infty$). In common cases, after emitting dislocations, the crack tip will become a sharp notch which root radius is ρ. If the coordinate at notch is $r = \rho / 2$ and $\theta = 0$, the stress concentration ahead of notch would be $\frac{K_{Ia}}{\sqrt{2\pi r}} \cdot \left(1 + \frac{\rho}{2r}\right)$[10]. When there

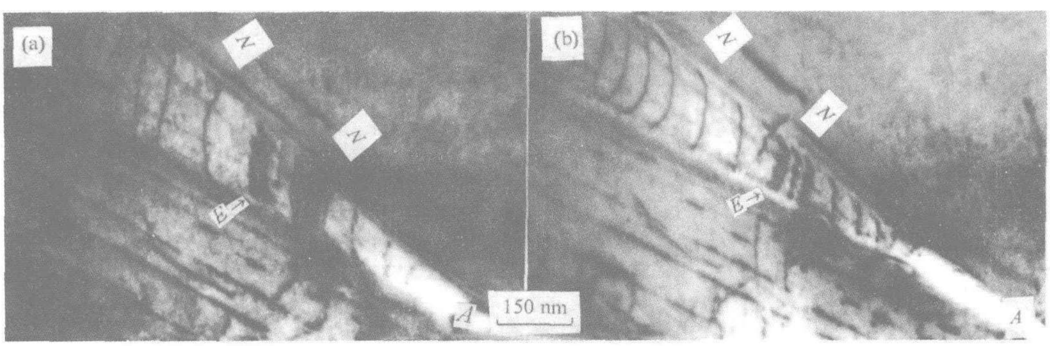

Fig. 1 Inversely piled up dislocations ahead of a loaded crack tip *A* (a) and their movement towards the crack tip *A* after unloading a little (b)

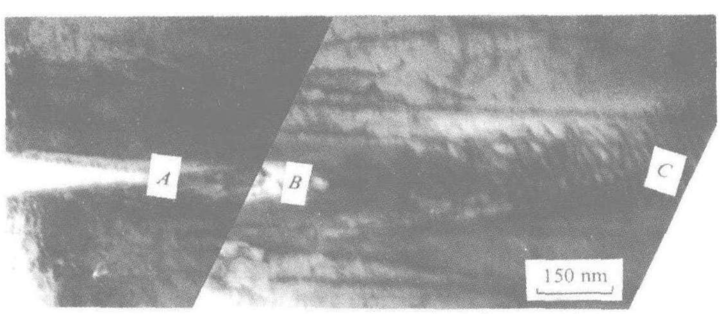

Fig.2 Inversely piled up dislocations *BC* and the dislocation free zone *AB* ahead of a loaded crack tip *A*

exists a DFZ ahead of notch, the equation(1) would become

$$\sigma_y=\frac{K_{\mathrm{Ia}}}{\sqrt{2\pi r}}\cdot\left(1+\frac{\rho}{2r}\right)-\int_c^d\frac{\mu b}{2\pi(t-r)}\cdot\left(\frac{t}{r}\right)^{1/2}f(t)\,\mathrm{d}t \quad (2)$$

Now, the tip of notch (which coordinate is $r_0=\rho/2$) is no longer the stress singular character site. When ρ is very little, however, the stress at the tip and in the vicinity of notch is still very large. In equation (2), the stress intensity K_{Id} corresponding to the second term is negative, so efficient stress intensity K_{If} becomes to be $K_{\mathrm{Ia}}+K_{\mathrm{Id}}$. Lin calculated K_{If} in another way and obtained more succinct form[11]: $K_{\mathrm{If}}=K_{\mathrm{Ia}}\cdot(c/d)^{1/2}$. Then equation (2) can be expressed as

$$\sigma_y=K_{\mathrm{Ia}}(c/d)^{1/2}(1+\rho/2r)/\sqrt{2\pi r} \quad (3)$$

The previous tests[1~4] showed that a crack tip would turn to be a sharp notch, $\rho\le 10$ nm, after dislocation emission and DFZ formation. The dimension ratio of a DFZ and the piled up dislocation was $c/d\ge 0.1$, and the initiation position within the DFZ was $r\le 25$ nm. Let the applied stress intensity K_{Ia} equalize to 50 MPa · $\mathrm{m}^{1/2}$, substituting these data into equation (3), σ_y could be equal to the cohesive strength $\sigma_{\mathrm{th}}=(0.1-0.043)E$[12] (where E is modulus of elasticity), resulting in nanocracks nucleating within the DFZ. For the unilateral notches specimen

$$K_{\mathrm{Ia}}\approx 1.12(P/BW)\sqrt{\pi a} \quad (4)$$

where P is the applied load, which keeps constant under the constant displacement, B is the DFZ thickness, W is the specimen width, and a is the crack primitive length.

After DFZ formation, even though keeping constant displacement, the thermal activity could also facilitate dislocations to emit, multiply and move in plastic regions, resulting in the DFZ inhomogeneously thinning continuously, as shown in Fig. 3(a). According to equation (4), K_{Ia} increases steady and reaches up to the largest value at the thinnest site with DFZ thinning inhomogeneously. In Fig. 3(b), the stress calculated by means of equation (3) is equal to or larger than the cohesive strength σ_{th} at the thinnest zone, resulting in microcrack 'a' nucleating within the DFZ ahead of crack tip A. For some ductile materials such as stainless steel, even if keeping constant displacement, microcracks initiated ahead of the crack tip would blunt into voids (Fig. 4a), and then connect each other, resulting in ductile propagation (Fig. 4b).

If continuously tensioning, the whole configuration could not reach equilibrium state. Then a DFZ and the

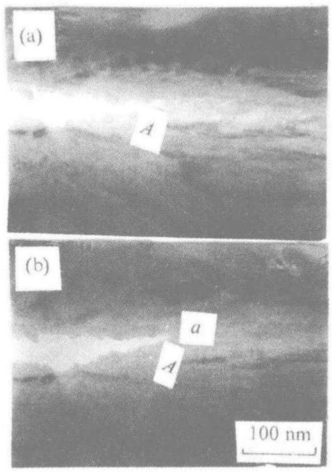

Fig. 3 Inhomogeneously thinning zone ahead of the crack tip A (a) and initiating a nanocrack 'a in the inhomogeneously thinning zone (b)

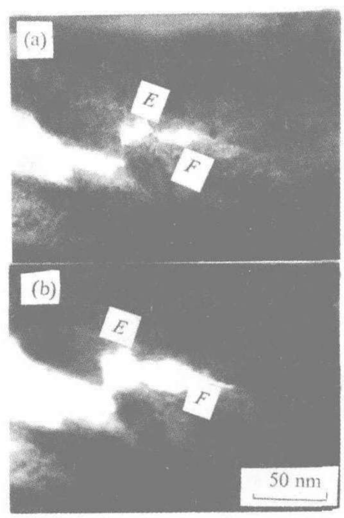

Fig. 4 Nanocracks' blunting and connecting after their nucleating, (a) blunted voids E and F; (b) voids connected with main crack

original procedure of microcrack initiation would not be observed. In addition, it was observed that the zone ahead of the loaded crack tip was gradually thinned by dislocation moving, and microcraks initiated and grew up into microvoids in the thin area, resulting in the zig-zag propagation, as shown in Fig. 5(a). On one side of zig-zag crack, there exists some slip lines and $(1\bar{1}1)[110]$ edge dislocations (Fig. 5(b)). On the other side, $(1\bar{1}1)[121]$ twins caused possibly by quick tension are parallel to the crack (Fig. 5(c)). Worth of being mentioned, in Fig. 6, after the crack propagated, a series of screw dislocations that are vertical to crack could be observed on the side of crack.

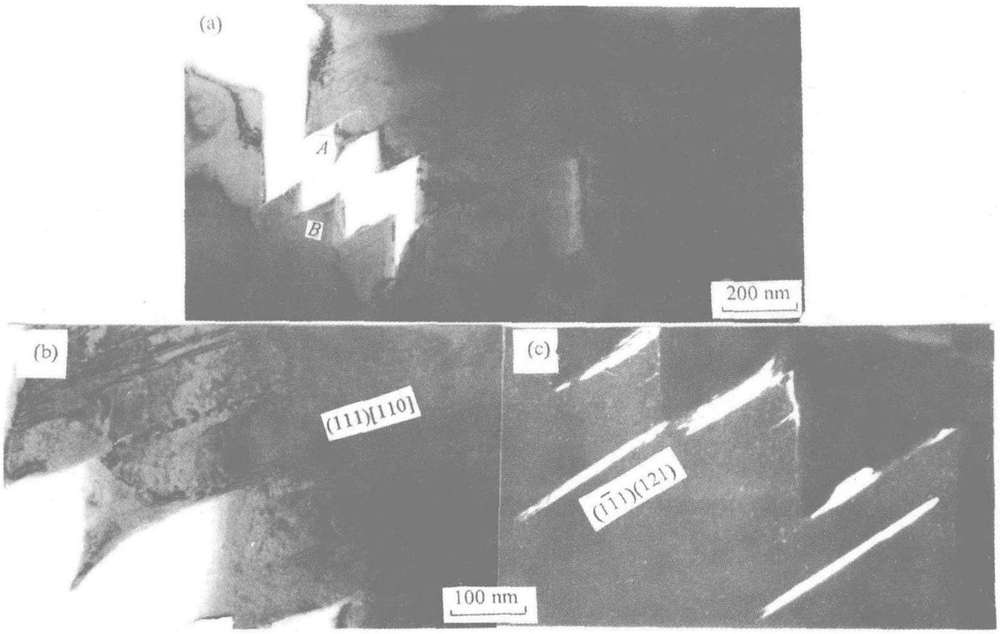

Fig. 5 Dislocations and twins beside the zig-zag crack, (a) zigzag crack; (b) slip lines and edge dislocations near the crack *A*; (c) twins near the crack *B* (dark field image)

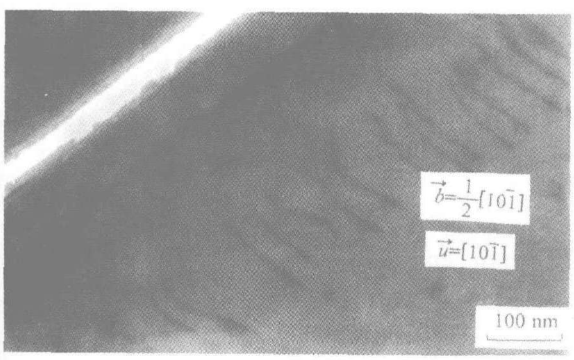

Fig. 6 Screw dislocations beside a propagating crack

From the mentioned above, only under constant displacement, could a DFZ be observed, as well as nucleating and blunting of nanocracks. If continuously or quickly tensioning, it is only observed that dislocations emanate and move, microcracks or microvoids initiate and connect through shearing.

3 Conclusions

(1) After a loaded crack tip emits dislocations, while keeping constant displacement, a DFZ forms ahead of the crack tip and nanocracks initiate and blunt into voids within the DFZ. If continuously tensioning, it is only observed that the zone ahead of the crack tip is thinned gradually and microcracks or microvoids nucleate and connect through shearing.

(2) After dislocations reach an equilibrium state, unloading would result that the dislocations ahead of crack tip move back towards the crack tip, even disappear at the crack tip.

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