CONF - 810831 - - 13

MASTER

TEM OBSERVATIONS OF CRACK TIP - CAVITY INTERACTIONS

J. A. Horton,[+] S. M. Ohr,[+] and W. A. Jesser[++]

[+]Solid State Div., Oak Ridge National Laboratory,* Oak Ridge, TN 37830
[++]University of Virginia, Charlottesville, VA 22901

Crack tip - cavity interactions have been studied by performing room temperature deformation experiments in a transmission electron microscope on ion-irradiated type 316 stainless steel with small helium containing cavities. Slip dislocations emitted from a crack tip cut, sheared, and thereby elongated cavities without a volume enlargement. As the crack tip approached, a cavity volume enlargement occurred. Instead of the cavities continuing to enlarge until they touch, the walls between the cavities fractured. Fracture surface dimples do not correlate in size or density with these enlarged cavities.

INTRODUCTION

The understanding of the effects of helium on physical and mechanical properties is important in the development and characterization of alloys for the first wall of a fusion reactor. The helium produced by neutron irradiation is known to affect the microstructure, and through this to affect swelling, deformation and fracture mode. However, very little is known of the interaction of dislocations resulting from propagating matrix cracks with the helium containing cavities. We have designed experiments to observe the deformation and transgranular crack propagation in type 316 stainless steel with small helium containing cavities.

Cavity enlargement is a common occurrence in several important fracture processes. High temperature fracture in unirradiated materials depends largely on the growth of grain boundary cavities [1]. In irradiated materials preexisting cavities undergo a stress assisted growth which can lead to embrittlement [2]. Microvoid coalscence or dimple rupture, which is a major fracture mode in structural materials, occurs by a process of void nucleation and void growth caused by plastic deformation processes.

Cavities can only be enlarged by acquiring vacancies either directly by diffusion or resulting from dislocation interactions. Vacancies can result from irradiation and from plastic deformation processes [3]. Stress assisted or stress biased processes can cause these vacancies to diffuse or be carried by dislocations to the cavities. It has been suggested that a dislocation could climb while intersecting a cavity and thereby add vacancies [4] in a manner similar to dislocation climb around precipitates [5]. Loop punching by a cavity can also result in its enlargement. This was first suggested for small overpressurized gas bubbles by Greenwood, et al [6]. On the other hand, slip dislocations can cut and shear a cavity without an apparent enlargement as observed for voids by Fisn, et al [7]. Ruedl and Schiller [8] have shown that slip dislocations can cut elongated cavities into pieces. The first experimental observation of cavity enlargement near a crack tip was reported by Horton, et al [9], who showed that cavity volume increased with localized strain at the crack tip.

The stress-assisted and plastic deformation processes leading to cavity growth are difficult to differentiate as they tend to occur simultaneously. In addition, these processes are possibly modified when a crack tip is near. It is the purpose of this paper to describe investigations of the mechanisms of cavity growth near a crack tip. Several possibilities are immediately apparent. The crack tip is a possible vacancy source very close to the enlarging cavity. The production and transport by dislocations of vacancies to cavities can be aided by the localized high deformation rates and strains. The existence of preexisting cavities and surfaces could change the surface energy criteria for crack propagation. The preexisting cavities could blunt the crack tip [9]. Finally, it is not known whether the cavities enlarge until they touch and thereby result in material separation or the walls between the cavities fracture by crack propagation at some critical thickness.

EXPERIMENTAL PROCEDURE

Small tension specimens (3 mm by 6.5 mm) were prepared from 0.15 mm thick type 316 stainless steel sheet. These specimens were annealed for 2.5 hours at 775 K prior to irradiation. The specimens in experiment 1 were irradiated in the ORNL Metals and Ceramics Division irradiation facility as bulk specimens and then

---

*Operated by Union Carbide Corporation under contract W-7405-eng-26 for the U. S. Department of Energy.

MGU

thinned until perforation (see Table 1). Specimens in experiment 2 and 3 were irradiated in the thinned condition at the ORNL Solid State Division accelerator facility and the University of Virginia facility, respectively.

Irradiated specimens were strained at room temperature in a Philips EM400T transmission electron microscope (TEM). Transgranular cracks were produced and progagated through cavity-containing regions of the specimen matrix. Sequential micrographs were made of crack opening displacements in increments of 10 nm.

CAVITY-DISLOCATION INTERACTIONS

Deformation produced cracks initiated at the edge of the electropolishing hole and emitted dislocations on planes coplanar with the crack. These dislocations were observed to travel distances of 10 μm or more into the thicker regions of the specimen. This general mechanism is very similar to that which occurs in unirradiated stainless steel. These cracks have been reported to emit screw dislocations in a manner which shears the crystal along a relatively thir set of slip planes [10]. In the cavity containing specimens from experiment 1, these dislocations were unimpeded by the cavities along a distinct set of slip planes. The cavities were observed to elongate during this process (see fig. 1). In unirradiated stainless steel, many of the dislocations travel along a distinct set of slip planes as partials. In the cavity containing material very little splitting was observed except for the "front" few dislocations. These showed only a small amount of separation. When intersecting a cavity surface the partial dislocations recombined at the cavity surface.

The ($\bar{1}1$ slip planes in figure 1 are inclined at 53° to the plane of the specimen. The direction of cavity elongation is [110] which corresponds to the probable Burgers vector of the slip dislocations involved in the process. These sheared cavities are about 6 um in front of the crack tip. The original foil thickness was 120 nm. The shear displacement in the [110] direction is approximately 50 nm. The cavities were also sheared in the same direction by this amount. Stereo observations showed that cavities directly above or below the slip planes were unsheared. Furthermore, stereo viewing showed the elongated cavities to be planar. They also exhibited little mass thickness contrast differences. These observations suggest that cross sections of the cavities were offset by the dislocation interaction as depicted in the sketch in figure 1. This intersection does not result in volume enlargement. If enough slip planes are involved, this process should continue until the cavity has been sheared to a sharp-pointed cone (fig. 2). The number of atomic planes contributing to the shear is apparently equivalent to at least one half the cavity diameter or about 15 to 30 nm.

A region of more generalized deformation is shown in figure 3 from a specimen irradiated in experiment 2. The bands of elongated cavities are in front of and in line with a propagating crack, lie in a $[\bar{2}1\bar{1}]$ direction, and exhibit a twin relationship to the regions without elongated cavities. Since the twins contained only elongated cavities, they were formed by deformation after the irradiation. The average shear strain of 300%, (shear angle 72°) requires the passage of the equivalent of two complete dislocations and one twinning dislocation per atomic plane. Again there was no apparent cavity enlargement.

CRACK TIP - CAVITY INTERACTIONS

In the region near a crack tip, the more intense plastic deformation caused a cavity elongation nearly perpendicular to the crack propagation direction. These elongated cavities have undergone a volume enlargement as evidenced by mass thickness contrast differences. The cavities do not continue to grow until they touch each

TABLE 1. Irradiation Conditions and Resulting Microstructures

Experiment #	1	2	3
Condition	Bulk	Prethinned	Prethinned
Ion Species and Energies	5 MeV Fe^{++}, 200-400 keV He^{+}	80 keV He^{+}	80 keV He^{+}
Damage Level and Helium Conc. or Flux $m^{-2}s^{-1}$ and Fluence m^{-2}	50 dpa, 500 appm He	$1x10^{18}$, $5x10^{21}$	$3x10^{18}$, $7x10^{21}$
Irradiation Temp K	950	no additional specimen heating	875
Cavity Diameters nm	5 to 60	2 to 12	20 to 40
Cavity Density m^{-3}	$9x10^{20}$	2×10^{22}	$1.5x10^{22}$
Cavity Volume Fraction %	1.	.2	12.
Average intercavity spacing nm	100	37	40

Figure 1: a. TEM micrograph of elongated cavities 6 μm from the crack tip (experiment 1). b. Same area with dislocations in contrast. Electron beam direction (B) near [011]. c. Sketch illustrating possible formation mechanism.

Figure 2. Elongated cavities in front of the crack tip in an irradiated stainless steel type 316 specimen (experiment 1). B near [013].

Figure 3: Sheared cavities in a deformation twin in front of a crack tip (experiment 2). B near [001].

other but rather at some stage of growth an intercavity fracture occurs in a brittle fashion. Figure 4a shows an interbubble fracture occurring in a wall of 70 nm thickness between two approximately 70 nm diameter cavities. The volume enlargement of the cavity in front of the crack tip is approximately 2.2 assuming that the cross-sectional area is unchanged. In figure 4b the wall thickness is about 180 nm and the cavity in front of the crack tip has enlarged by a factor of 6. These cavities have now become fracture surface dimples. No ligament formation occurred as the walls fractured.

The specimens with the small (2 to 12 nm) cavities (experiment 2) gave more meaningful results since the cavity diameters were small relative to the specimen thickness at the crack tip. Indeed larger volume increases were evident for the cavities in these specimens. For example, one dimple, shown in figure 5, is approximately 6 nm in diameter and 12 nm long but originated as a 3 nm diameter cavity as measured on a preceding micrograph. The volume has increased by a factor of 20, assuming a circular cross section. The amount of cavity enlargement also suggests local strains of at least 400%.

The cavity enlargement apparently only occurs for cavities intersected by the crack itself. Cavities observed as close as 12 nm (experiment 2) from the final fracture surface appeared unaffected by the fracture process. This estimate was made possible by TEM micrographs which are projected images of the crack flank. In these micrographs elongated cavities overlap undeformed cavities. Measurements were made of the distance from an undeformed cavity to the crack flank surface (see fig. 6).

FRACTURE SURFACE DIMPLES AND CAVITY ENLARGEMENT

One can compare fracture surface dimple diameters with the original cavity diameters. The present TEM observations suggest the dimples have a diameter up to about twice the original cavity diameter and have the same planar density. Observations by others of fracture surface dimple and original cavity sizes and densities have been limited to TEM specimens prepared close to the fracture surface but not actually of the fracture surface [11]. These observations showed a greater dimple density than original cavity density [12]. A large fracture surface (experiment 3) in the electron transparent region which formed during a 875 K

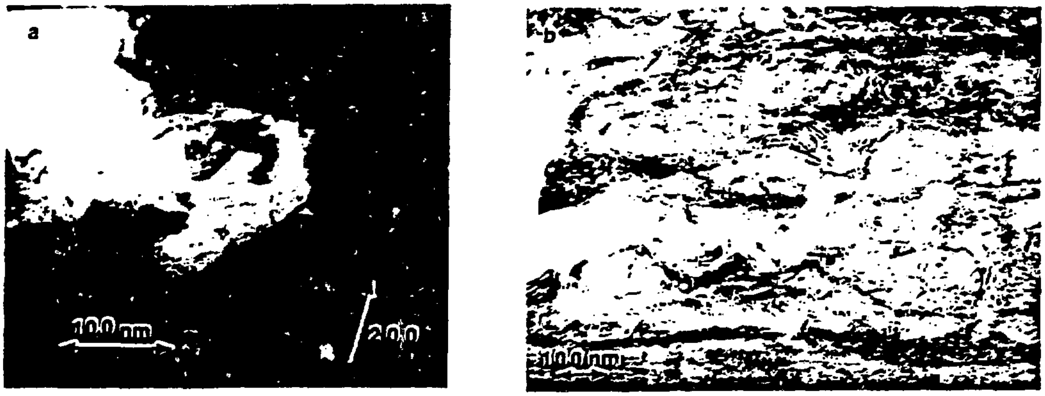

Figure 4 a b: Elongated cavities at the crack tip (experiment 1). Note the wall of material between the cavities is fracturing. B near [031].

Figure 5: TEM micrograph of crack tip - cavity interactions in experiment 2. The arrow points to a fracture surface dimple which originated as a 3 nm diameter cavity as observed in a preceding micrograph. B near $[\bar{1}14]$.

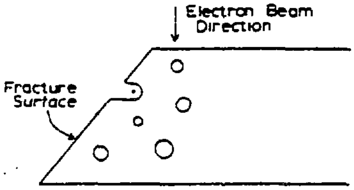

Figure 6: Sketch of the profile of the crack flank depicting how an estimate could be made of the extent of plastic deformation.

helium irradiation at the Univ. of Virginia facility was examined concurrently by imaging with scanning transmission electron microscopy (STEM) and secondary electrons (SED). The surface examined resulted from a blistering process and is on the ion beam exit side of the specimen and hence the impinging ion beam has not modified the observed surface. The larger fracture surface dimples (up to 400 nm in diameter - Fig. 7) bear little relation in size or density to the original cavities. These dimples are the ones commonly observed by scanning electron microscopy. The smallest fracture surface dimples observed were about 20 nm in diameter which is close to the original cavity size. Surprisingly, there appears to be a much lower density of small dimples than small bubbles.

The mechanisms of cavity enlargement is fundamental to many fracture processes. The results presented here give some indication of the conditions at which the cavities enlarge near a crack tip. Calculations of the number of vacancies per lattice site required for this observed cavity enlargement, divided by the amount of localized strain, yield about .1 to .2 vacancies per atom per unit strain. Similar results have been reported previously [13]. This compares to 1 x 10^{-3} vacancy concentration per unit strain quoted for resistivity measurements at low strains. A conservative estimate was made of the possible vacancy production by assuming that all of the work done during the localized plastic deformation goes to creating vacancies with a 1 eV vacancy formation energy. This conservative estimate would not create a sufficient number of vacancies to account for the observed enlargement [13]. Clearly the presence of the crack tip, nearby surfaces, and pre-existing helium containing cavities alters the mechanisms of vacancy production in bulk material.

The fracture process is possibly affected by the helium pressure with the cavities. Even assuming all of the helium stopped in the specimen (fig. 7) migrated to the visible cavities, the cavities were underpressured [14]. Blistering has resulted from underpressured bubbles in a foil with thickness less than the range of the incident helium ions [13].

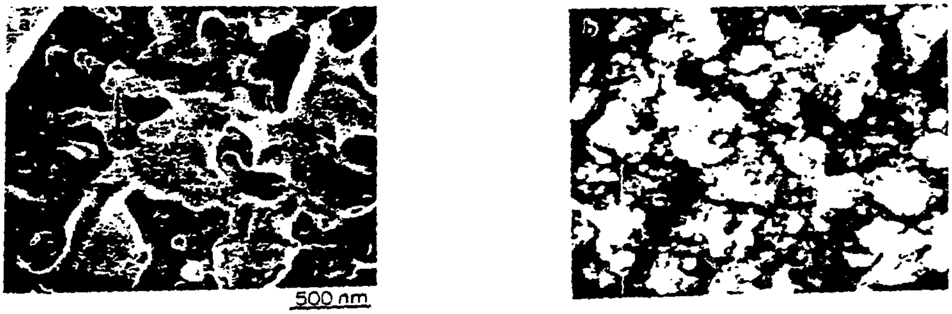

Figure 7: Concurrent SED (a) and STEM (b) images of the same area under a blister cap showing the relationship between preexisting cavities and fracture surface dimples.

CONCLUSIONS

1) Localized slip confined to a few parallel slip planes in front of crack tips cut and sheared cavities without a measurable cavity volume enlargement.
2) Cavities above or below the zone of localized slip were not disturbed.
3) In the thin areas of irradiated, cavity-containing material, dislocations were emitted from the crack tip on a distinct slip plane that is coplanar with the plane of the crack. The process by which this occurs is similar to that operating in unirradiated material.
4) Only bubbles or voids which were very near or intersected by the crack tip underwent a volume enlargement. The localized flow left cavities close to the fracture surface undeformed. Volume enlargements up to a factor of 20 have been observed.
5) Although the volume enlargement was substantial when tested at room temperature, the cavities did not enlarge until they touch but instead the walls between the cavities fractured in a brittle manner.
6) Although the cavities were observed to enlarge, no size or density correlation was found with the fracture surface dimples observed by normal SEM, as determined by concurrent STEM and SED imaging of an electron thin fracture surface.

ACKNOWLEDGEMENTS

The authors wish to thank J. M. Williams and M. B. Lewis for performing the irradiations, T. C. Estes for specimen preparation, F. W. Young and F. W. Wiffen for reviewing the manuscript.

REFERENCES

[1] Raj, R. and Ashby, M. F., Acta Metall. 23 (1975) 653-666.
[2] Barnes, R. S., J. Nucl. Mater. 11, 2 (1964) 135-148.
[3] Damask, A. C. and Dienes, G. J., Point Defects in Metals, Gordon and Breach, New York, NY (1971) 220-221.
[4] Jagannadham, K., A Mechanism of Void Growth in the Region of the Plastic Zone ahead of a Crack Tip, submitted to Int. J. of Fracture.
[5] Ashby, M. F., in Physics of Strength and Plasticity, ed. by A. S. Argon, MIT Press (1969) 133.
[6] Greenwood, G. W., Foreman, A. J. E., Rimer, D. E., J. Nucl. Mater., 1, 4 (1959) 305-324.
[7] Fish, R. L., Straalsund, J. L., Hunter, C. W., and Holmes, J. J., ASTM STP 529 (1973) 149-164.
[8] Ruedl, E. and Schiller, P. J. Nucl. Mater. 85 & 86 (1979) 769-773.
[9] Horton, J. A., Bennetch, J. I., and Jesser, W. A., J. Nucl. Mater. 85& 86 (1979) 829-833.
[10] Ohr, S. M. and Narayan, J., Phil. Mag. A 41, 1 (1980) 81-89.
[11] Trinkaus, H. and Ullmaier, H., Phil. Mag. A 395 (1979) 563-580.
[12] Sagues, A. A., Schroeder, H., Kesternich, W., and Ullmaier, H., Breeder Reactor Structural Materials, ed. by M. L. Bleiberg and J. W. Bennett (NY: TEM of AIME, 1977) 367-374.
[13] Horton, J. A., Ph.D. Thesis, Univ. of Virginia, Nov. 1979.
[14] Cost, J. R. and Chen, K. Y., J. Nucl. Mater., 67 (1977) 265.