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Small Specimen Procedures for Determination of Deformation Maps

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REFERENCE: Farrell, K., Byun, T. S., Jones, J. W., Gibson, L. T., Sitterson, R. G., Hashimoto, N., Bailey, J. L., and Gardner, J., **"Small Specimen Procedures for Determination of Deformation Maps,"** *Small Specimen Test Techniques: Fourth Volume, ASTM STP 1418,* M. A. Sokolov, J. D. Landes, and G. E. Lucas, Eds., ASTM International, West Conshohocken, PA, 2002.

Abstract: Irradiation-hardened metals undergo changes in plastic deformation modes that degrade mechanical properties. Variation of deformation mode as a function of fluence and level of strain can be tracked with deformation mode maps. Mapping requires many tensile tests and TEM specimens. To streamline the process, a special tensile test cradle and refined TEM specimen preparation techniques have been developed. The 0.25-mm-thick sheet tensile specimen with dogbone shape is sufficient to ensure bulk mechanical behavior and eliminates grinding operations for the TEM pieces. Extraneous strains that might arise in such a small specimen from pin loading or friction gripping are avoided by loading the specimen under its shoulders in a small, sliding cradle which also prevents accidental damage during remote handling in and out of the tensile machine. After straining, three TEM pieces each 1.5 mm square are cut from the gauge section in one pass in a special jig using a slow speed rotary saw fitted with ganged blades. To accommodate these square specimens during electrochemical thinning, the specimen holder is custom altered. Finally, for TEM examination, the thinned specimen is supported by an auxiliary platform constructed from molybdenum foil rings.

Keywords: radiation damage, tensile test jig, sub-size TEM pieces, plastic deformation maps, strain localization

Introduction

It has been known since the very earliest investigations of the effects of irradiation on the mechanical properties of metals [*1*] that irradiation can strengthen a metal and at the same time reduce its ductility and alter the mode of deformation. A great deal of

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effort has gone into showing that the irradiation strengthening is caused by clusters of radiation-induced point defects that impede the movement of slip dislocations during post-irradiation straining. Less attention has been paid to explaining the changes in ductility, which must be related to the mechanism(s) of plastic deformation. Metals are ductile because they undergo plastic flow, or deformation, by the generation and movement of dislocations on slip planes within the atomic lattice of the metal. Usually, many intersecting slip planes are operative. The dislocations can move from one slip plane to another and they become entangled into a three-dimensional network of dislocation cells. This ability to develop a network of dislocation cells ensures that the material work hardens and deforms in a homogenous manner. That ability is lost when the materials are hardened by irradiation. In an irradiated and strained specimen the deformation cell structure gives way to narrow, two-dimensional slip bands, or channels, of order 0.1 μm wide, in which the radiation defect structure has been erased by the passage of numerous dislocations in the channels. Large blocks of material between the channels are relatively undisturbed [2,3]. Plastic shear strains of several hundred percent have been measured in the channels in specimens that displayed only about 5% bulk strain [3]. Therefore, dislocation channeling is intensive strain localization on a microscopic scale. The occurrence and nature of the channels are dependent on radiation dose and temperature, strain level, test temperature, and other parameters. Although there is clearly a connection between this localization of strain and the reduced ductility of irradiated metals, quantitative correlations have not been made. One way to establish a firm foundation for a comprehensive correlation is to construct deformation mode maps like those developed by Ashby [*4,5*] to define creep regimes. Such maps for irradiated metals could form the basis to connect changes in mechanical properties to deformation modes through common parameters, and to bring some predictive capability into the picture.

Presently, deformation mode maps for irradiated materials exist only for neutron-irradiated nickel and gold [*6*]. The maps were prepared by straining irradiated tensile specimens to prescribed elongations then discontinuing the test and examining TEM specimens cut from the gauge sections. Okada et al's map for gold is reproduced in Figure 1. In it, the regimes of deformation by dislocation cell formation and by dislocation channel deformation (DCD) are displayed in elongation-fluence space. It can be seen that at low doses below 7×10^{20} n.m^{-2} the specimens deform solely by cell formation, the cell size decreasing with increasing elongation. The cell size at 2.5% elongation is ~0.5μm. In an unirradiated control specimen the corresponding cell size was 1μm. For an exposure of 3×10^{20} n.m^{-2}, deformation was by DCD, changing to cell structure at an elongation around 10%. At higher doses, DCD prevails to higher strains. These changes can be correlated with reductions in work hardening rates and uniform strain parameters.

Two programs are underway at Oak Ridge National Laboratory to obtain deformation maps. One program covers eight pure metals, Al, Cu, Fe, Mo, Nb, Ni, V, and Zr. The other is focused on three reactor construction alloys A533B ferritic steel, 316 austenitic stainless steel, and Zircaloy-4. Preparation of even a single deformation mode map requires many irradiations and tensile tests, and a great deal of TEM work. This paper describes the experimental techniques developed to expedite these tasks.

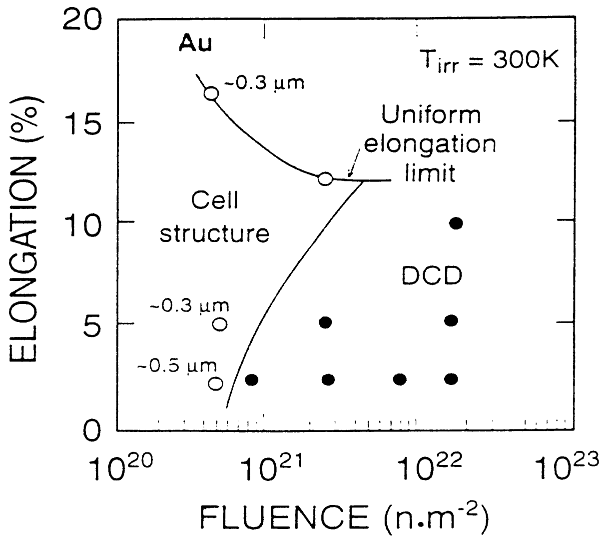

Figure 1 - *Deformation modes for gold as functions of fluence and elongation. After Okada et al [6]*

Techniques

Our irradiations were conducted in the hydraulic tube facility at the High Flux Isotope Reactor where the irradiation capsule is limited to a sealed aluminum tube called a rabbit, with internal dimensions of 53 mm length and 9.6 mm diameter. For this size rabbit to accommodate a reasonable payload of specimens the specimen had to be smaller than usual. It was also desirable to use small specimens in order to keep the radioactivity levels to a minimum for reduction of radioactive waste and to comply with ALARA (as low as reasonably achievable) personal protection requirements. The low activities of most of our specimens allowed them to be tested and cut in a C zone, which is quicker and less costly than a hot cell and permits more flexibility of operations. Small specimens are also advantageous for minimization and dissipation of gamma heat for control of irradiation temperature.

Gamma heating rates are high in the hydraulic facility. To maintain the temperature of the tensile specimens as close as possible to the temperature of the rapidly flowing water, the rabbit was redesigned with many perforations drilled through the walls to allow the cooling water to be in direct contact with the specimens. However, for some materials, contact with water has its drawbacks. Ferritic materials will rust. Other materials such as zirconium are sensitive to hydrogen absorbed from the water. To protect specimens of these susceptible materials from the depredations of the cooling water, they were enclosed in aluminum foil envelopes. This wrapping technique developed for specimen protection may be useful elsewhere, and is described below.

Protective Envelopes

Aluminum has high thermal conductivity and good aqueous corrosion resistance. Aluminum can be made soft enough to be pressed tightly against a tensile specimen by low forces that will not damage the specimen. The envelopes for the water-sensitive materials were made by an electron beam welding technique from 0.125 mm thick foils of six-nines purity aluminum. Trials with other grades of aluminum and with high purity aluminum foils purchased from commercial sources showed them to be inferior with regard to welding and corrosion pitting resistance. The foil was annealed at 500°C for one hour to reveal the presence of gas bubbles, which tend to be a feature of high purity aluminum. Regions with bubbles were discarded. Rectangular pieces of the foil were folded flat along their length axes. They were trimmed to a size of 8 mm wide and 23 mm length. One 8 mm edge and the 23 mm edge were sealed by electron beam welding. The flat, welded envelopes were annealed for 30 min at 350°C to soften the aluminum. Their welds were examined carefully under a microscope for flaws. The envelopes were then shaped into rectangular section sleeves by easing a mandrel of stainless steel strip, 5 mm wide x 0.05 mm thick with a wedge-shaped nose, into the unwelded 8 mm end until it reached the other end. It was worked back and forth until it was a loose fit. The blunt end of the mandrel was used to square off the sealed end of the sleeve. The sleeves were annealed for 30 min at 350°C.

Two tensile specimens, one seated exactly atop the other, were eased into the mouth of the sleeve and were guided to the end of the passage using the blunt end of the mandrel. The loaded envelopes were aligned with their open ends facing upwards in a jig in the jaws of a vice in the vacuum chamber of the welding machine. After air was evacuated from the chamber and the envelopes, a closure bar on the jig was remotely operated to bring the edges of the open end of an envelope together and hold them whilst they were sealed with an electron beam weld. When the welding chamber was opened, atmospheric pressure pressed the soft envelope against the specimens. Further contact of envelope and specimens was assured by gently kneading the package between two fingers. In the reactor, the water pressure of about 30 atmospheres keeps the foil tight against the specimens. The flattened end of the package provides a tab for engraving identity marks with a round-tip scriber.

The Tensile Specimen

The dimensions of the tensile specimen are shown in Figure 2. Tolerances for the R and W dimensions are +/- 0.0013 mm, and +/- 0.05 mm elsewhere. The specimen thickness of 0.25 mm was chosen because it is adequate to give bulk tensile properties yet is thin enough for excision of TEM pieces that require no further reduction in thickness by grinding, thereby eliminating the grinding operation and its associated radioactive dust. The major features of note are the broad heads and the absence of pinholes in the heads. These heads are designed for under-the-shoulder load application in order to avoid using pin loading and friction gripping techniques, which have the potential for introducing extraneous plastic strains that might confound strain control and measurement in thin specimens with short gauge lengths. The 5 mm width of the head allows a generous radius at the interactions of the head and the gage section so as to minimize stress concentrations there, and provides a loading area 3.33 times the gage section area. A bonus is that a 3 mm diameter disk can be punched from the head for use in damage microstructure study or disk-bend tests. The gage section dimensions of the tensile specimen, 1.5 mm wide x 8 mm long, were chosen to allow three square (1.5 mm x 1.5 mm) TEM samples to be taken from the strained gage section.

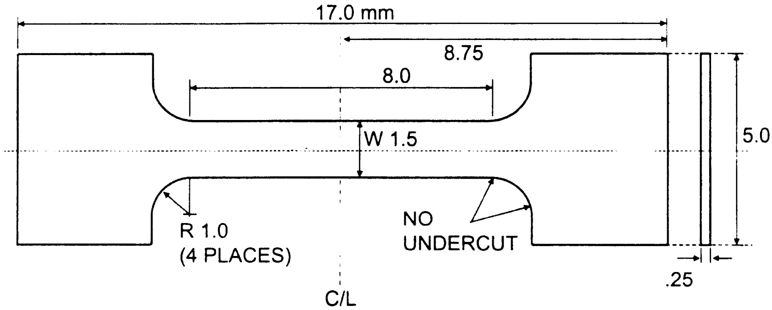

Figure 2 - *Dimensions of the miniature tension specimen. (Unit in mm)*

The Tensile Test Cradle

Testing of radioactive tensile specimens requires the use of tongs or manipulator arms to maneuver the specimen into and out of the tensile machine. With miniature specimens the risk of accidental damage to the specimen from these insertion operations is high. A tensile test cradle can abolish or minimize such risk. The cradle pictured in Figure 3 is constructed to protect the specimen from accidental bending or torsion moments that might occur during manual positioning of the specimen in the tensile machine. It aligns the specimen, applies the load under its shoulders during the test, and requires only easy movements to release the tested specimen. The tensile specimen is placed in the cradle simply by dropping the specimen into an open recess in the cradle while the cradle is lying on a workbench.

The cradle with a specimen inside is pictured in Figure 3. The cradle is constructed from cold worked stainless steel. Its two headpieces move on a track made from two rod rails. The rails pass through clearance holes drilled in the heads. C-clips in grooves at the ends and center of the rails limit the movement of the heads and keep them on the rails under normal operation. In the event of an unplanned excessive separation of the machine cross head, the C-rings will be jerked out of their grooves and destruction of the cradle will be avoided. The head pieces have extension arms with end knobs that have convex shoulders radiused to mate with concave seats of the pull rods of the tensile machine.

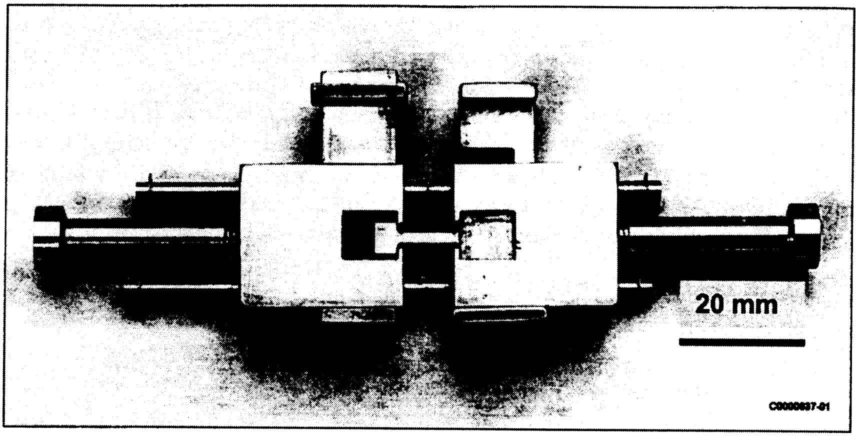

Figure 3 - *The tensile cradle.*

The recessed cavities in the headpieces are shaped to accept the heads of the tensile specimen, and have a short, 1.5 mm wide entry channel to accept the specimen gauge section. The bottom of the recess is milled to a depth of 0.125 mm below the tensile axis of the cradle. This ensures that the tensile axis of the cradle corresponds with the mid-thickness of the tensile specimen. The junction of the recess and channel has a 1 mm radius to match the radius of the shoulder in the tensile specimen. The channel is 1.5 mm wide at its bottom and is flared out near its top to make it easier to place the specimen into the channel and headpieces.

Applying the test load to a thin sheet specimen under its shoulders can promote buckling or fluting in the specimen heads. To prevent such distortion we installed sliding gates in the headpieces. The gates slide through each headpiece in slots at right angles to the tensile axis and they straddle the specimen heads. The bottom of the gate slot is 0.30 mm above the bottom of the recess, giving 0.05 mm clearance above the specimen. There is a cutout in the gate to permit installation of the specimen. The gates have sturdy push plates for operation with manipulators or tongs. The gates not only deter plastic buckling of the heads, they assure that the specimen is seated properly and they retain the pieces of a specimen broken in test. Tests to failure with trial specimens, and subsequently with many irradiated specimens, have shown no distortion of their heads.

To prepare a specimen for testing, the cradle is laid on its back on a bench with the gates open and the headpieces touching one another. The specimen is picked up with tweezers or vacuum tweezers and is oriented above the channel and released. It falls into the channel and the head cavities. Seating of the specimen is checked though a mirror and the heads are separated with tongs until resistance is felt. Using tongs, the gates are closed and the cradle is lifted by one of its extension arms and is placed in the machine pull rod thimbles.

Tensile testing was performed at room temperature in a screw-driven machine at a crosshead speed of 0.008 mm/sec, corresponding to a nominal strain rate of 10^{-3} s^{-1}. Engineering strains were calculated from the recorded crosshead separation, using a nominal gauge length of 8 mm. Engineering stresses were calculated as the load divided by initial cross-sectional area measured before irradiation.

Comparison of the tensile properties of our miniature specimens tested in the support cradle with those of larger specimens show very good reproduction of strength properties but reduced elongation values due to the short gauge length. Duplication of property values in multiple tests is excellent. Yield point drops and Lüders strains are reproduced. Examples of tensile curves are illustrated in Figure 4.

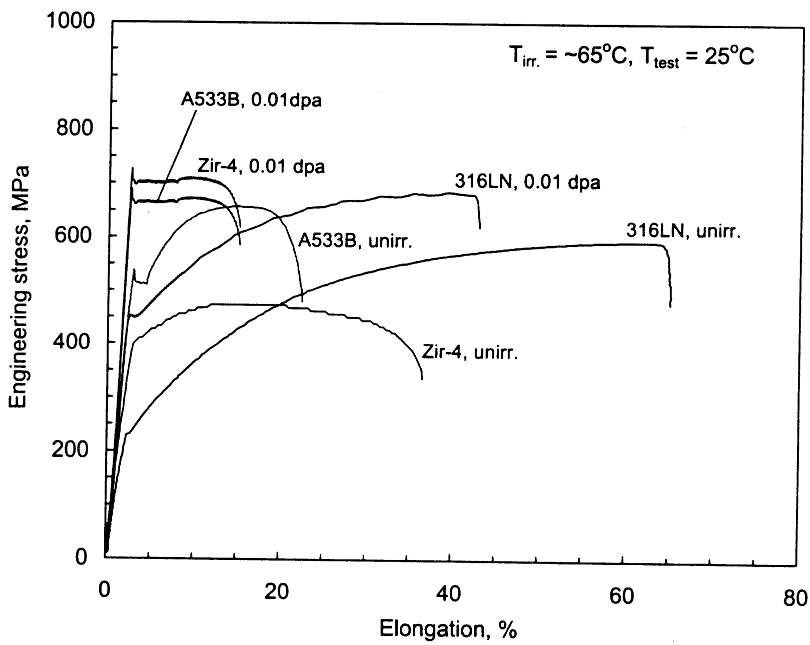

Figure 4 - *Examples of room temperature engineering stress-elongation curves.*

Cutting the TEM Pieces

The TEM pieces are taken from the gauge section of the specimen and are 1.5 mm squares, much smaller than a regular 3 mm disk. To avoid introduction of extraneous dislocations during excision of the TEM pieces, they must be cut, not punched or sheared. And the specimen must be held so that its gauge section is fully supported during cutting, and so that the cut pieces are not thrown off by the cutting tool. A slow-speed, water-cooled Buehler Isomet® rotary saw was chosen to cut a tensile piece into TEM and shoulder pieces using a custom holder to support the tensile piece. To improve the production rate, the standard, single circular saw blade was replaced with a gang of four ceramic blades separated by 1.5 mm thick spacer disks.

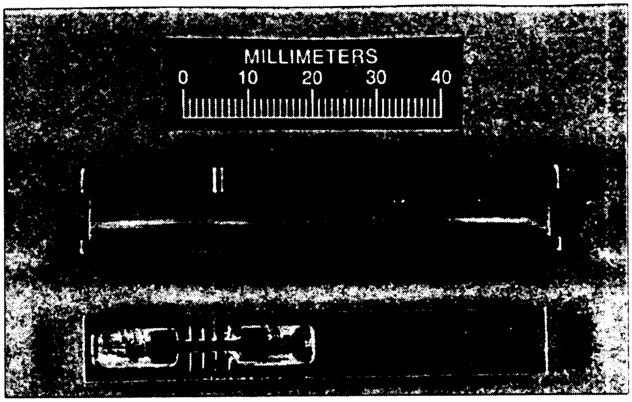

Figure 5 - *TEM cutting holder with tensile specimen in the specimen cavity.*

The specimen is held in the two-piece, aluminum holder shown in Figure 5. One of the pieces is a flat bar with a shallow, specimen-shaped cavity milled in it to accept the tensile specimen. The cavity is elongated to accommodate stretched specimens. The channel depth is 0.75 mm so that the specimen sits down in the cavity. The other piece of the holder is a U channel. It has four slots cut through one of its upright arms using the ganged saw blade assembly. The channel is laid on its side with the slots facing up, and the bar with its specimen is slide into the channel opening with the specimen facing and abutting the saw slots. The channel is raised to an upright position. The force of the saw blades is sufficient to hold the specimen during sawing. To present the specimen to the rotating saw blades, the specimen holder is gripped in the jaws of a blade dressing chuck clamped to the frame of the saw. The handle of the chuck's manual screw feed is cranked to deliver the enclosed specimen to the saw blades. After cutting, the severed TEM pieces remain in position in the gauge section groove of the holder cavity and are picked off one at a time with tweezers. A slight burr is removed by light sanding.

Thinning the TEM Piece

The 1.5 mm square TEM disk cannot be thinned to electron transparency in our Struers Tennupol® regular specimen holder which is designed for 3 mm disks. The holder requires modification. Two changes are necessary: The holes in the dished inserts of the holder, which define the polishing "window," must be reduced, and the seating depression for the TEM disk in the platinum electrode strip must be reshaped. The window holes in the standard 3 mm disk inserts are 1.5 mm diameter. For a 1.5 mm square TEM disk, a window hole of 0.85 mm diameter proved satisfactory. It also worked best when the rim of the hole was chamfered on the dished side of the insert at an angle of 45° or when the dish at the hole was deepened with a spherical burr.

The regular platinum strip electrode is sandwiched between the flat surfaces of the dished inserts. Its hole is larger than the 1.5 mm window holes in the inserts. It has a circular shelf depression to seat the 3 mm diameter disk. For 1.5 mm square samples, a 0.85 mm hole was drilled through a platinum strip. Then the platinum strip was annealed in vacuum for 30 minutes at 1050°C to soften it. Next, a short length of a drill rod was machined into a 1.55 mm square section tool and was used to make a square depression on the annealed platinum strip laid on a softwood anvil. By tapping the tool with a light hammer a square depression of the correct dimensions was forged around the hole. The depression was made carefully to be shallower than the 0.25 mm thickness of the TEM piece. This precaution is necessary to guarantee that the specimen will stay it in contact with the platinum when the holder is assembled.

When making these modifications to the holder, the shallow raised platforms around the window holes on the flat surfaces of the inserts must not be erased or damaged. These rims are essential to provide seals around the windows that prevent the electrolyte from seeping onto the covered areas of the specimen where it will cause corrosion.

Supporting the Specimen During TEM Examination

Supporting the small, square specimen during TEM examination requires use of an auxiliary platform inside the microscope standard specimen holder. The platform must be thin enough to allow the screw-in retainer ring of the standard specimen holder to maintain sufficient room for clamping by the threaded retainer ring of the holder. A satisfactory platform was constructed from circular microscope grids. Molybdenum foil grids 3-mm size, 0.005 mm thick with a single 1 mm round hole and 0.01 mm thick with a 1.4 mm round hole were available. The 1.4 mm hole was enlarged to a 1.55 mm square hole. Using a 1 mm round hole grid as a base, a square hole grid was laid on top of it and fixed permanently in place with four laser spot welds spaced around the periphery. This composite forms the receptacle for the square TEM disk. The receptacle assembly is placed in the specimen holder and the TEM piece is positioned in the square. A 1 mm hole grid is laid on top of the specimen and the retainer ring is screwed in. This ring acts as a secondary retainer and as a barrier against friction from the retainer screw. This arrangement secures the specimen, allows an unobstructed viewing area of 1 mm diameter, and imposes little limitation on tilting capability.

Some Results

Our deformation mapping programs are still in their infancies and it will be some time before we have sufficient data for complete maps. Most of the tensile tests have been made for the three commercial materials, and the TEM studies are underway. An example of the deformation microstructure observed in 316 stainless steel irradiated to 0.01 dpa and strained 6% can be seen in Figure 6. The deformation is heterogeneous, largely confined to very narrow bands lying on [111] planes. The bands are not simple, cleared dislocation channels like those found in other fcc metals. Piled-up faulted dislocations can be resolved in the bands. Strong streaking characteristic of twinning is evident in the diffraction patterns, confirming that the bands consist of packets of very fine twins, or microtwins.

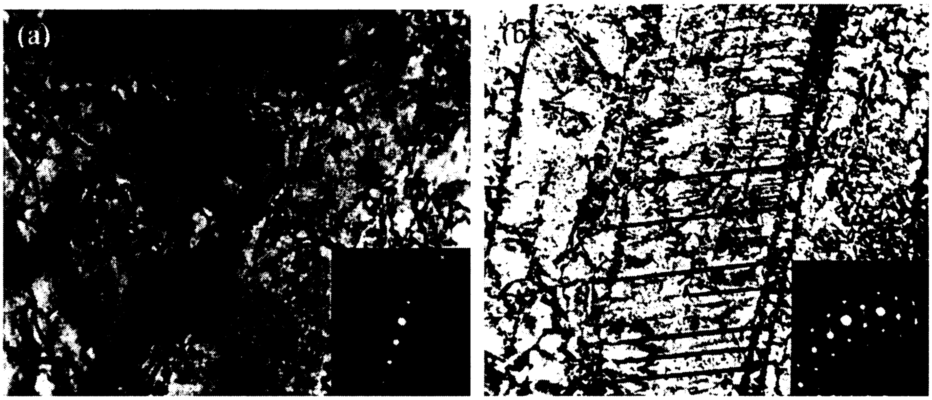

Figure 6 - *Deformation microstructures of 316 stainless steel after 6% elongation; (a) unirradiated and (b) irradiated to 0.15 dpa at about 65°C.*

This type of microtwinning in irradiated stainless steel has been seen elsewhere [*7-9*]. In unirradiated control specimens the deformation microstructure consists of a mix of planar dislocation bands and many randomly dispersed dislocations between the bands. Planar deformation is typical of stainless steel and is generally attributed to the steel's low stacking fault energy which results in Shockley partial dislocations on the [111] habit planes and restricts their ability to cross slip. It is contended, however, that short-range order might be a more likely reason [*10*]. A detailed description of deformation microtwinning in irradiated, austenitic stainless steel has been submitted for publication [*11*].

Acknowledgement

This research was sponsored by the U. S. Department of Energy in part by contract number DE-AC05-00OR22725 between the Division of Materials Sciences and UT-Battelle Corporation, and in part by the Division of Nuclear Energy through NERI project number IWO MSF 99-0072.

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