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Mechanical Property Measurements on Ion-Irradiated Metals

S. J. Zinkle
W. C. Oliver

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MECHANICAL PROPERTY MEASUREMENTS ON ION-IRRADIATED METALS

S. J. Zinkle and W. C. Oliver

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MECHANICAL PROPERTY MEASUREMENTS ON ION-IRRADIATED METALS*

S. J. Zinkle and W. C. Oliver

ABSTRACT

A recently developed mechanical properties microprobe (MPM) has been used to investigate strength and elastic modulus changes in ion-irradiated metals. The indenter load and its displacement are simultaneously monitored while the indentation is being made and also during unloading. Microindentation hardness measurements have been performed on ion-irradiated copper and Cu-0.15% Zr (AMZIRC). The depth dependence of the ion damage has been investigated in selected specimens which were prepared using a cross-section technique. This procedure allows a direct comparison to be made of hardness data from different irradiation depths while the indent size is held constant. The displacement damage associated with ion irradiation caused either hardening or softening, depending on the irradiation conditions and the material.

INTRODUCTION

Heavy ion irradiation can produce high damage levels in metals in very short periods of time. This has made ion irradiation a useful tool for studying microstructural changes associated with irradiation. Unfortunately, the limited size of the ion irradiation zone (typically ≤1 μm) limits the potential for extracting direct mechanical property measurements from the damage region. There have been several investigations of the relative hardness changes associated with charged particle irradiation in metals (e.g., refs. 1-6). Due to limitations of the microhardness tester, direct measurement of the hardness in the irradiated zone was possible only for cases where the ion range was ≥10 μm. A new technique for measuring the mechanical properties of ion-irradiated materials is introduced in this paper. The present study investigates hardness and elastic modulus changes in ion-irradiated copper and Cu-0.15% Zr where the damage range is on the order of 1 μm.

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EXPERIMENTAL PROCEDURE

Annealed high-purity (99.999%) copper and 90% cold-worked-and-aged AMZIRC (Cu-0.15% Zr) specimens were irradiated with 14-MeV Cu ions to a calculated peak damage level of 40 dpa, as described elsewhere.[7,8] In addition, high-purity copper foils that were 50% cold worked were irradiated with dual beams of 0.2- to 0.4-MeV He and 4-MeV Fe ions. The displacement damage region for 14-MeV Cu ions extends to a depth of 3.5 μm (ref. 7). The maximum depth of damage for 4-MeV Fe ions in copper is about 1.5 μm. The helium injection produced a uniform concentration of 50 appm He with insignificant displacement damage (<0.01 dpa) over the depth range of 0.5 to 1.1 μm, according to an E-DEP-1 calculation.[9] The foils were mechanically polished and then electropolished in a solution of 33% HNO_3-67% CH_3OH at 5 V and −50°C prior to the irradiation. Irradiations were conducted at temperatures of 100 to 440°C. Following the irradiation, the 14-MeV Cu ion-irradiated foils were prepared for cross-section analysis using standard techniques.[10] Displacement damage rates were $\geq 2 \times 10^{-3}$ dpa/s for all of the foils.

The mechanical properties microprobe (MPM) measurements were made on a Nanoindenter system produced by Nano Instruments, Inc. This system consists of a recently developed[11,12] fully automated, ultralow load microindentation hardness tester. The indenter load and displacement were continuously monitored as the load was applied and removed, with a resolution of 0.3 μN (30 μg) and 0.16 nm, respectively. The rate of indentation during loading was controlled so that the linear strain rate of the indenter ($\dot{h}/h$, where h is the indenter displacement) was held constant at a value of either 0.05 or 0.01/s. The hardness was calculated directly from the load (L) and depth measurements without correction for elastic relaxation effects (i.e., the hardness was calculated using $H = AL/d^2$, where d is the indent depth under loading conditions and A is an experimentally measured geometric factor that relates the indenter displacement and projected area). As described elsewhere,[11] the diamond indenter is a triangular-base Berkovitch pyramid that is similar to a Vickers pyramid in terms of its relationship between indent area and penetration depth. The possibility of a chisel tip is minimized with this type of a diamond shape since the three sides of the indenter are

geometrically required to meet at a common point. Elastic modulus measurements were obtained from an analysis of the unloading curve, as detailed elsewhere.[13,14] A total of 5 to 10 indents were made in irradiated and nonirradiated regions of each specimen.

RESULTS

Figure 1 shows a typical indenter load-displacement curve that is obtained from a metal specimen using the Nanoindenter system. The portion of the curve that is marked as "A" represents loading, whereas curve "B" is produced during the process of indenter unloading. The profile of the loading curve shown in Fig. 1 is similar to the ideal parabolic shape that is expected when the hardness is independent of depth.

Figure 2 shows the hardness ratio of irradiated versus nonirradiated (control) copper as a function of indenter depth following a 15 dpa irradiation with 4-MeV Fe ions (50 appm He coinjection) at 220°C. The control indents were made in regions of the specimen which were masked from the ion beam by a tantalum foil. A large (70%) increase in hardness is observable in the irradiated foil for indent depths of 50 to 300 nm. The radiation-hardened surface region continued to contribute to the measured hardness for indent depths in excess of 1.5 μm. Partial recrystallization occurred in both irradiated and nonirradiated regions of the 50%-cold-worked foil during the 1-h irradiation. Examination of copper foils that had been irradiated to only 0.1 dpa (50 appm He coinjection) at 220°C resulted in a nearly identical depth-dependent hardness profile, as shown in Fig. 3.

Ion irradiation at temperatures above 300°C produced only slight radiation-hardening effects. Figure 4 shows the depth-dependent hardening associated with a 50 appm He plus 4-MeV Fe ion irradiation to 18 dpa at 440°C. The hardness increase in the near-surface depths of the irradiated region of the foil is ≤10% higher than the control hardness.

In order to obtain a more direct measurement of the depth-dependent hardening associated with ion irradiation, microindentation measurements were made on 14-MeV Cu ion-irradiated specimens that had been prepared using a cross-section technique. A constant maximum indenter depth of 150 nm was chosen, which produced indents ~1 μm in lateral dimension. A

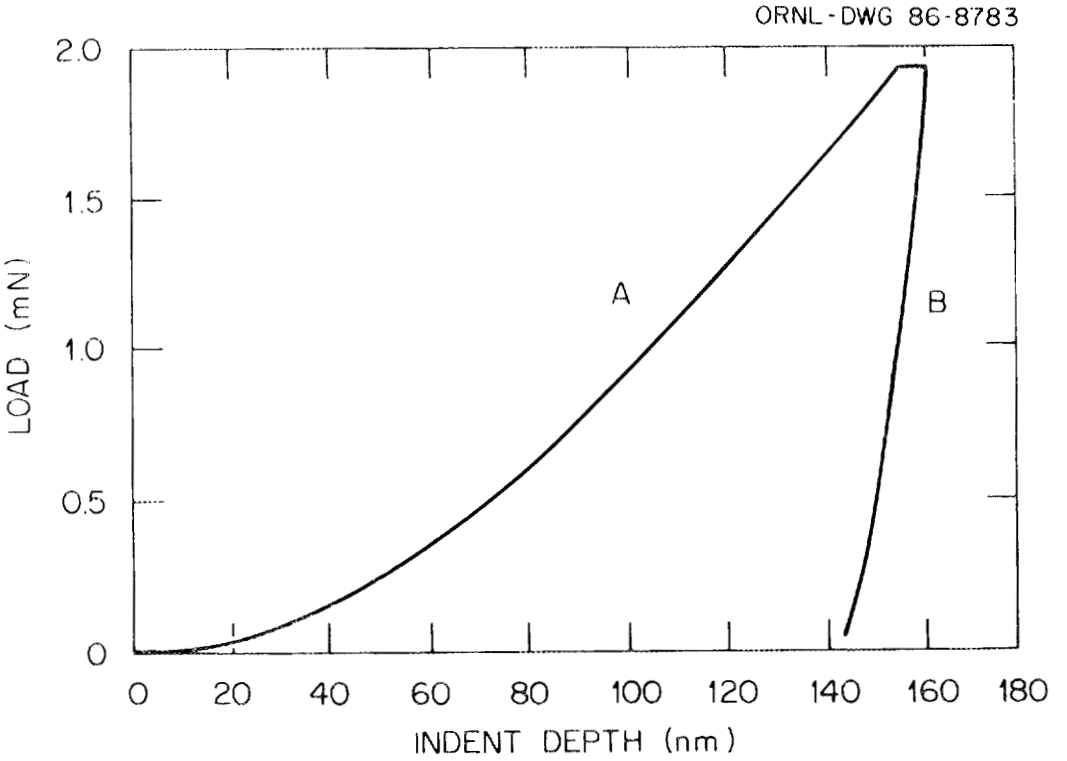

Fig. 1. Typical load versus indent depth curve for irradiated copper. A = loading portion, B = unloading.

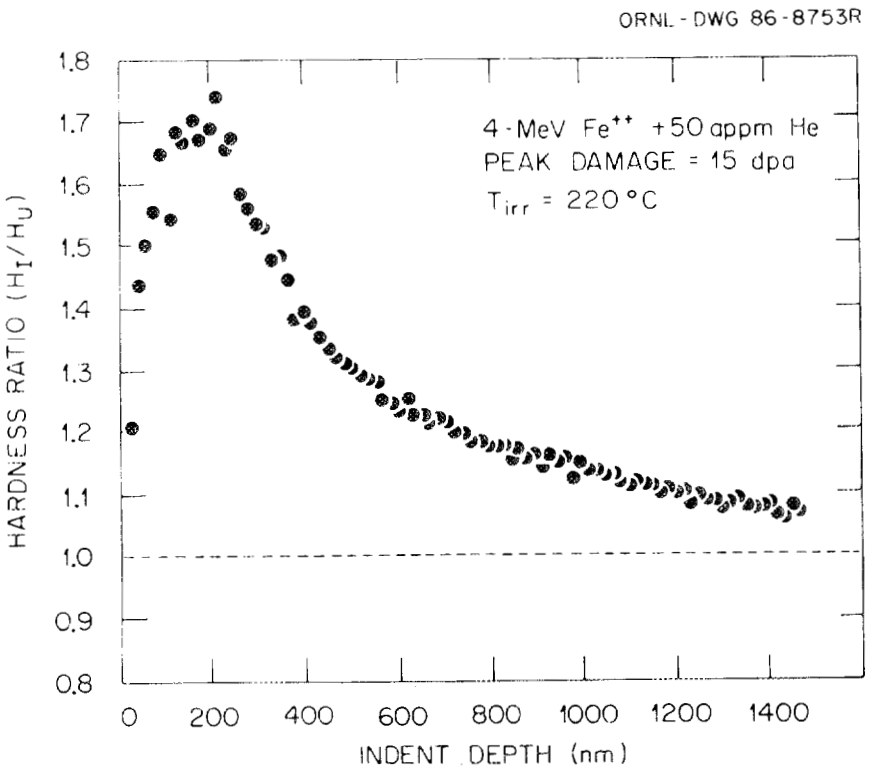

Fig. 2. Irradiated-to-nonirradiated hardness ratio of copper as a function of indent depth. Indents are made normal to the irradiated surface.

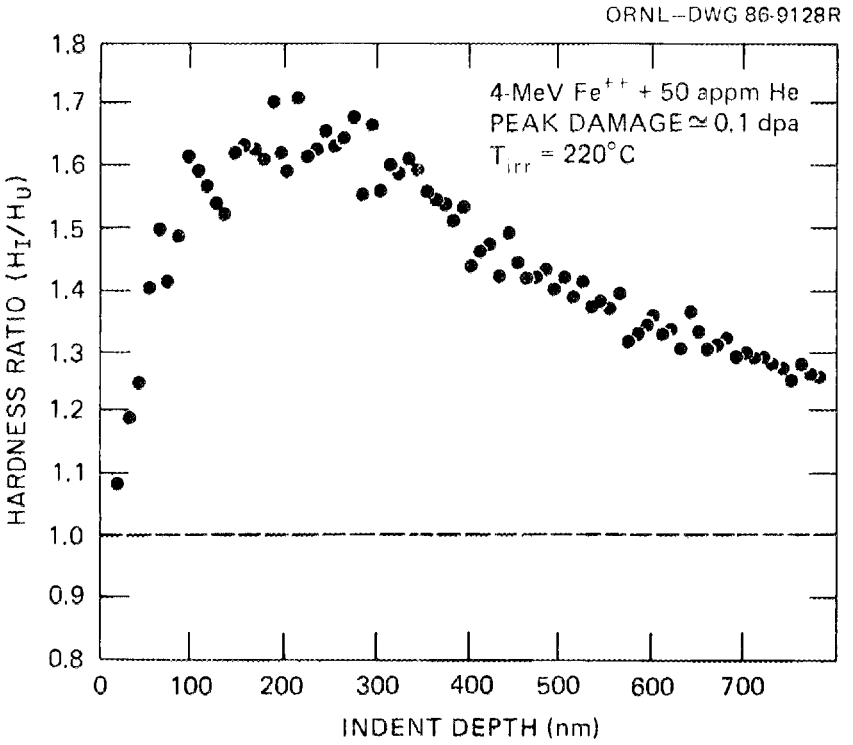

Fig. 3. Irradiated-to-nonirradiated hardness ratio of copper as a function of indent depth. Indents are made normal to the irradiated surface.

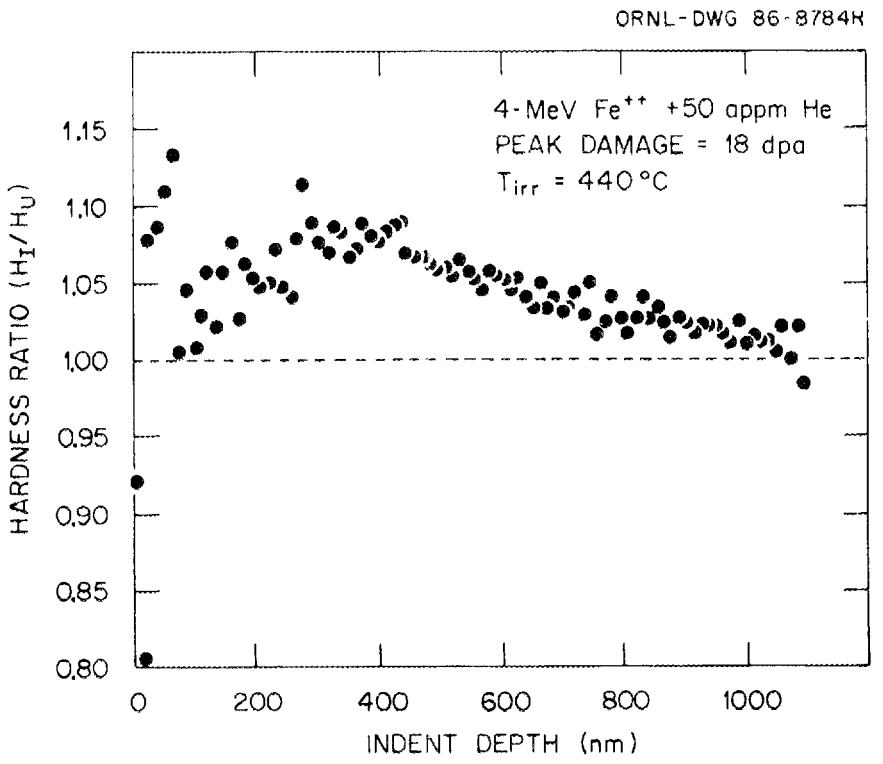

Fig. 4. Irradiated-to-nonirradiated hardness ratio of copper as a function of indent depth. Indents are made normal to the irradiated surface.

row of indents that were spaced ~5 μm apart were made such that the axis of the row was at an angle of 5.7° with respect to the interface between the irradiated foil and the copper plating (Fig. 5). This produced a net displacement of 0.5 μm between successive indents with respect to the interface.

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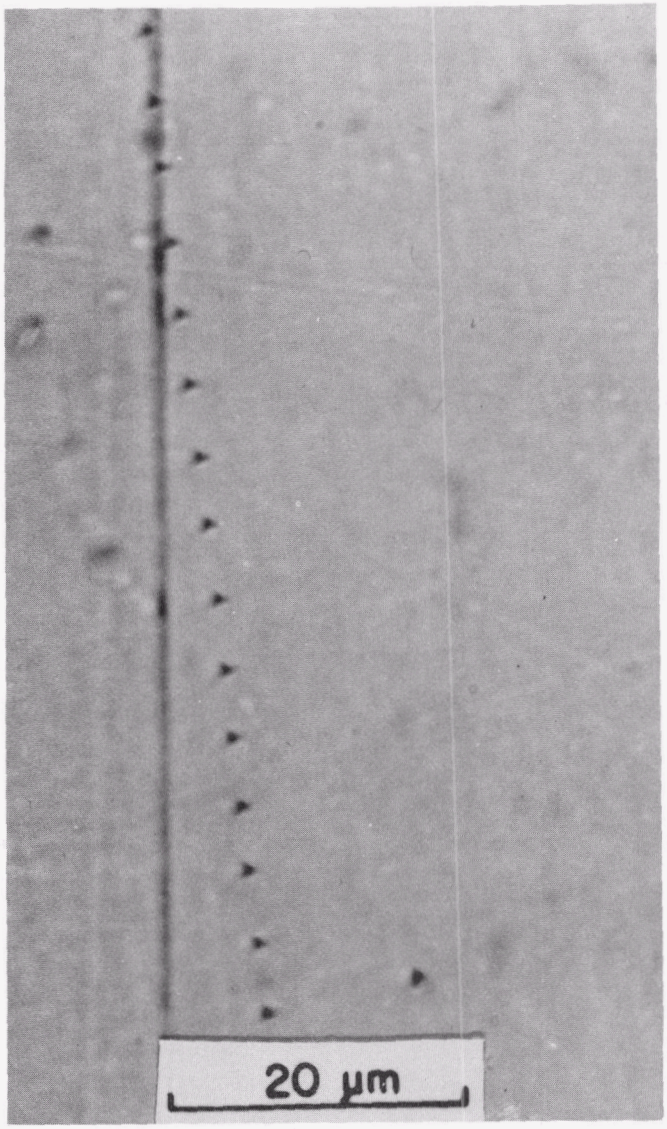

Fig. 5. Optical micrograph of indentations made in a cross-sectioned copper specimen.

Figure 6 shows the cross-sectional hardness profile of copper at an indent depth of 150 nm following a 14-MeV Cu ion irradiation to a peak damage level of 40 dpa at 100°C. Cross-section traverses were made for two specimen conditions. In the first case, the cross-sectioned surface was mechanically polished using 0.05 μm alumina powder. The second treatment consisted of the same mechanical polish followed by a light electropolish with 33% HNO_3–67% CH_3OH at 5 V and −30°C for 2 s. As shown in Fig. 6, the choice of specimen preparation technique did not have a significant effect on the measured hardness in the irradiated portion of the cross-section foil. However, the mechanical polish treatment produced significantly higher hardness values than those for the electropolish condition in the nonirradiated regions of the cross-section foil. The hardness in the irradiated region was constant even though the damage level varied from 5 to 40 dpa at different depths from the surface. This is an indication that "black spot" hardening effects saturate at moderate damage levels.

An examination of the irradiated to nonirradiated hardness ratio as a function of indenter depth for the cross-section hardness data given in Fig. 6 revealed differing behavior depending on the polish treatment. Measurements made on electropolished specimens showed a constant hardness ratio for indenter depths ≥50 nm (Fig. 7). On the other hand, measurements from a mechanically polished specimen resulted in a hardness ratio that increased in direct proportion with indent depth up to the maximum depth that was studied, 150 nm (Fig. 8).

Cross-section hardness measurements were also made on an electropolished AMZIRC specimen that had been irradiated at 400°C with 14-MeV Cu ions to a peak damage level of 40 dpa. As shown in Fig. 9, the irradiated region of the AMZIRC specimen is softer than nonirradiated regions. The irradiated-to-nonirradiated hardness ratio obtained from several series of cross-section measurements was 0.82 for an indenter depth of 150 nm. Carefully positioned deep indents made on the same specimen showed that the hardness ratio was constant with a value of ~0.82 for indent depths of 100 to 500 nm. (Indents deeper than 500 nm were not attempted in the damage region since they would geometrically begin to overlap the electroplated and nonirradiated regions of the cross-section specimen.) A

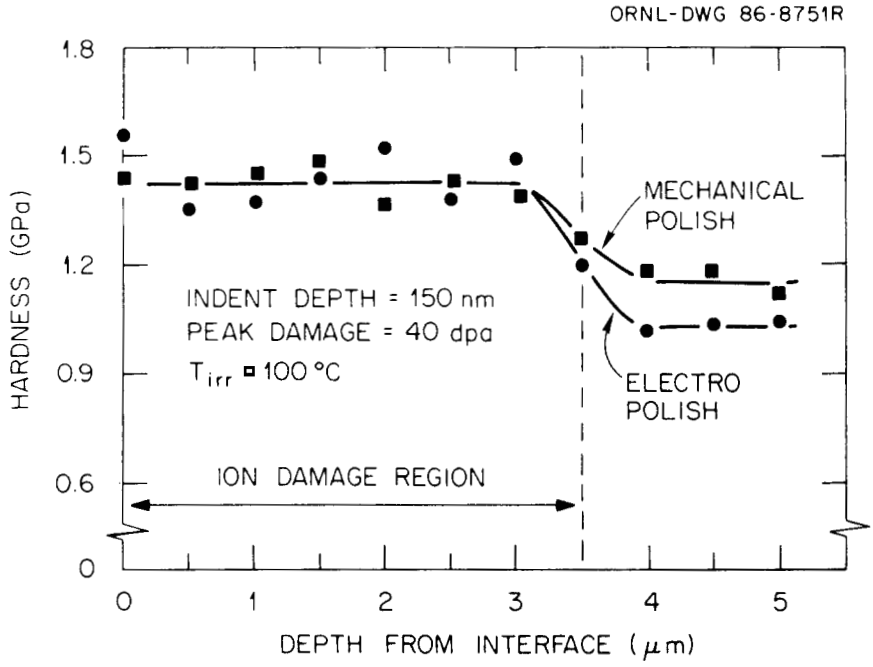

Fig. 6. Cross-section hardness of annealed copper following 14-MeV Cu ion irradiation for two different specimen surface treatments.

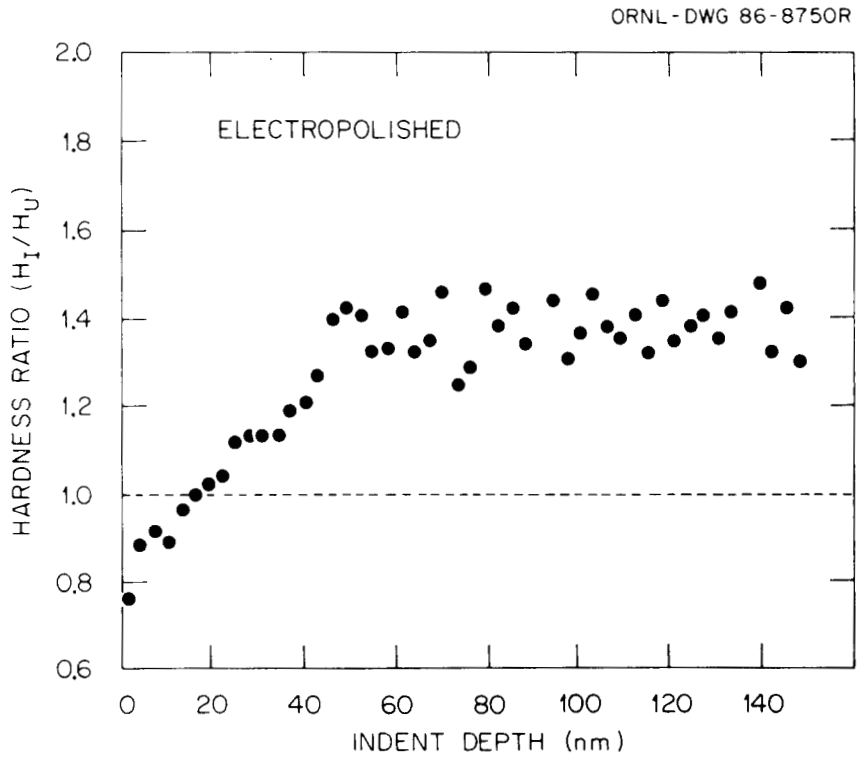

Fig. 7. Hardness ratio of 14-MeV ion-irradiated copper cross-section specimen in the electropolished condition as a function of indent depth.

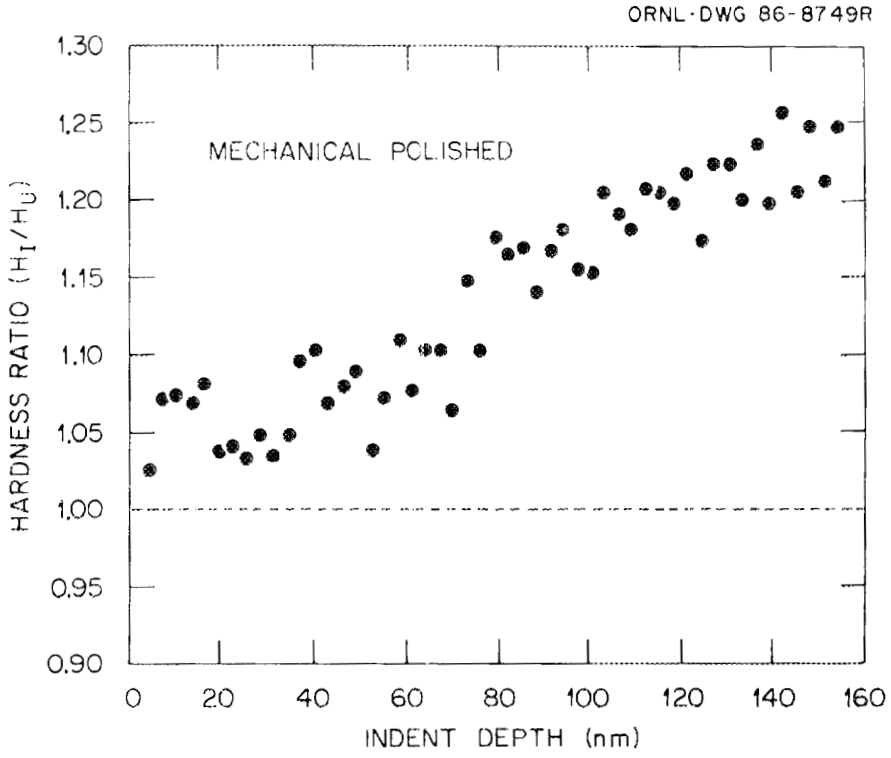

Fig. 8. Hardness ratio of 14-MeV ion-irradiated copper cross-section specimen in the mechanical polished condition as a function of indent depth.

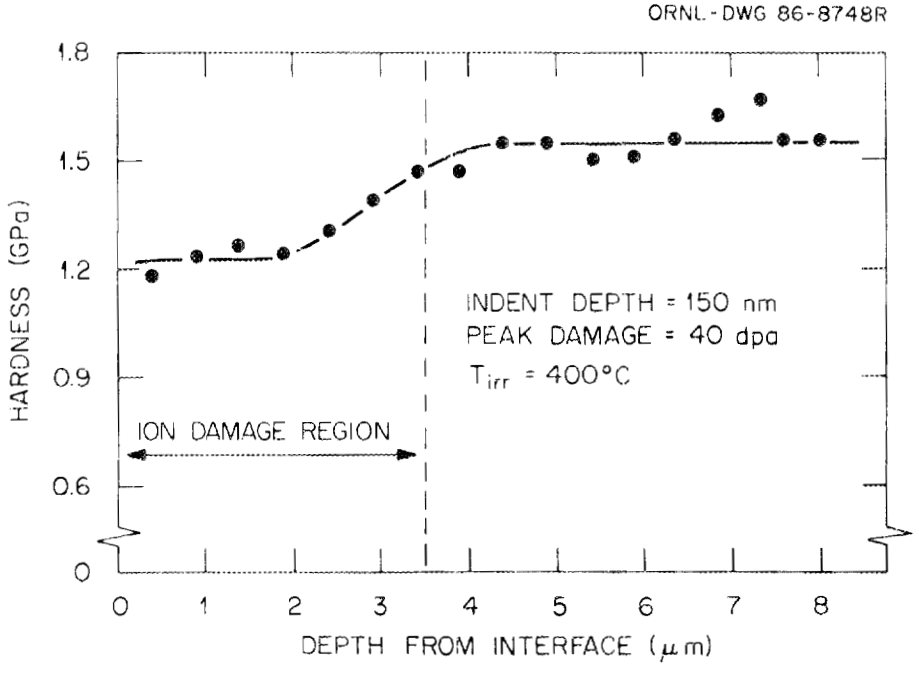

Fig. 9. Cross-section hardness of AMZIRC following 14-MeV Cu ion irradiation at 400°C.

series of cross-section microindentations were also made on the same specimen in a near-surface region which had not been exposed to the ion beam. The hardness was found to be constant for indents in the region 0.5 to 10 μm from the foil plating interface, which indicates that the observed softening is due to an irradiation effect and not a surface effect.

The effect of strain rate on the measured hardness value was examined using the electropolished copper cross-section specimen that had been irradiated with 14-MeV Cu ions to 40 dpa at 100°C. Decreasing the constant strain rate from 0.05 to 0.01/s caused a decrease in the microhardness numbers in both the irradiated and nonirradiated regions by about 10% compared to the base case (0.05/s). This corresponds to a stress exponent of $n \simeq 16$, which is about the expected value for copper at room temperature. Similar rate effects in copper and other materials have been reported in the literature.[15]

The elastic modulus of annealed copper following Cu-ion irradiation at 100°C to a peak-damage level of 40 dpa was determined from the slope of the unloading curve. For a constant indent size, the composite elastic modulus (specimen plus indent system) is directly proportional to this slope.[13,14] Measurements were made at room temperature in both irradiated and nonirradiated regions of a cross-section specimen. The measured nonirradiated value of the elastic modulus was 106 GPa, which is in fair agreement with the handbook value for copper (117 GPa). The small absolute error is probably due to uncertainties in the stiffness of the indentation system (including specimen mounting effects). The relative modulus changes investigated in the present study should not be significantly affected by this uncertainty since both the irradiated and nonirradiated measurements were made on the same (cross section) specimen under constant geometry conditions. Comparison of the irradiated and nonirradiated modulus values indicated that the 100°C irradiation produced an ~10% increase in the elastic modulus of copper. A similar analysis of the AMZIRC cross-section specimen produced a nonirradiated modulus value of 133 GPa which compares well with the manufacturer's value of 129 GPa. Ion irradiation to a peak damage level of 40 dpa at 400°C produced an almost 20% decrease in the elastic modulus.

DISCUSSION

It is well known that low-load hardness measurements performed using standard techniques are sensitive to the indenter load.[16,17] A recent study[13] has found that most of the load dependence is due to strain-rate effects, and that this anomalous behavior can be minimized by controlling the linear strain rate of the indentation (as was done in the present case). Under the usual experimental conditions, the average linear strain rate of the indenter ($\dot{h}/h$) can be much greater at low loads (< 1 g) than it is at high loads (> 1 kg) due to the difference in the final indent depth (h). Examination of the depth-dependent hardness for indents made in irradiated cross-section specimens that have been electropolished (where the results should be independent of indent depth in the ideal case) indicates that constant irradiated-to-nonirradiated hardness ratios are obtained with the present experimental conditions for indenter depths (h) that are greater than 50 to 100 nm (see Fig. 7). This implies that the results obtained using $h \geq 100$ nm should be quantitatively comparable to bulk radiation hardening measurements. The depth-dependent hardness at lower indent depths may be due to other experimental effects that were not controlled in this study.

It is well established that mechanical polishing produces a work-hardened surface region that can cause misleading hardness results,[16] particularly at low indenter loads. Figures 7 and 8 clearly show that a mechanical polish treatment using a 50-nm alumina abrasive has an effect on the measured microhardness for indent depths in excess of 150 nm. Figures 6 through 8 reaffirm the conclusion[16] that electropolish preparation techniques should be used for microindentation hardness measurements on metals.

Microhardness measurements made normal to the surface of ion-irradiated specimens do not directly reproduce the depth-dependent hardness profile due to the fact that the indentation is sensitive to the specimen hardness for distances up to 10 times the indent depth.[18] The measured hardness is generally taken to be roughly equal to the specimen hardness at a depth of ≥4 times the indent depth. This empirical observation is confirmed by the measurements presented in Figs. 2 through 4.

These figures show that radiation-hardening effects begin to decrease from the maximum plateau value for indent depths ~300 to 400 nm. This may be compared to the maximum damage range for 4-MeV Fe ions incident on a copper target, which is ~1.5 μm. A detailed comparison of the hardness profile in conventional and cross-sectioned specimens is in progress.

The hardness of copper is increased by about 70% following ion irradiation at 220°C for damage levels of both 0.1 dpa (Fig. 3) and 15 dpa (Fig. 2). This indicates that radiation-hardening effects at this temperature have already reached a saturation level for a damage dose of ~0.1 dpa, which is in good agreement with neutron irradiation studies of copper in this temperature range.[19-21] As mentioned earlier, the cross-section hardness results presented in Fig. 5 (100°C irradiation) also show that radiation hardening in copper is independent of the damage level for moderate doses. The maximum increase in the yield strength of copper following high dose neutron irradiation at low temperatures (≤100°C) is $\Delta\sigma_y \simeq 300$ MPa. The measured hardness increase for cold-worked copper irradiated at 220°C with 4-MeV Fe ions to a damage level of either 0.1 or 15 dpa (50 appm He coimplant) was $\Delta H \simeq 700$ MPa (70 kg/mm^2). Using hardness-yield strength correlations found in the literature,[17,22] this corresponds to $\Delta\sigma_y \simeq 210$ MPa which is in fair agreement with the neutron irradiation results. The implanted helium should contribute slightly to the measured strength increase, but the magnitude of its contribution is not known.[23] A similar analysis of the radiation-hardening measurements performed on the 14-MeV Cu ion-irradiated cross-section specimen (Fig. 6) produces a correlated yield strength (due to displacement effects alone) of $\Delta\sigma_y \simeq 150$ MPa.

A significant amount of softening was observed in the AMZIRC copper alloy following ion irradiation at 400°C (Fig. 9). As discussed elsewhere,[24] this loss in strength is attributed to radiation-enhanced recrystallization effects. The decrease in hardness number at an indenter depth of 150 nm was $\Delta H = -330$ MPa. This corresponds to a correlated yield stress change in AMZIRC of $\Delta\sigma_y \simeq -100$ MPa.[22,25] The measured yield stress of nonirradiated AMZIRC following a 1 h anneal at 400°C is 350 MPa.[26] This annealing treatment is similar to that seen by the nonirradiated (control) regions of the AMZIRC cross-section specimen. The

estimated yield strength in the irradiated region of the AMZIRC specimen is therefore $\sigma_y \simeq 250$ MPa. This radiation-enhanced softening observation is in agreement with measurements made on AMZIRC irradiated with neutrons at 385°C.[27]

A previous high-temperature (100 to 500°C) electron irradiation study on copper found that the elastic modulus (E) increased by about 4% following a low dose irradiation,[28] in general agreement with the present results which found a 10% increase for a high dose irradiation. The increase in E can be attributed to dislocation pinning effects.[28,29] The decrease in the elastic modulus of AMZIRC following high-dose ion irradiation at 400°C might be due to radiation-enhanced recovery and precipitate overaging effects which would decrease the contribution of dislocation pinning. The measured value of E for AMZIRC following the irradiation was only a few percent higher than the measured value for nonirradiated copper.

CONCLUSIONS

The conclusions of this study are as follows:

1. Low-load microhardness results on electropolished specimens may be quantitatively related to bulk hardness changes if strain rate effects are properly controlled.

2. Low-temperature (100 to 220°C) ion irradiation of copper produces correlated strength increases that are somewhat less than results found in neutron-irradiated copper. Radiation-hardening effects in copper saturate for doses ≥0.1 dpa at these irradiation temperatures. Irradiation of copper at elevated temperatures (440°C) produces only slight (~10%) radiation-hardening effects.

3. Radiation softening occurs in a commercial high-strength copper alloy (AMZIRC) following ion irradiation at 400°C. The softening is in agreement with predictions based on radiation-enhanced recrystallization effects observed in the microstructure.

4. Preliminary measurements indicate that the elastic modulus of annealed copper is increased by 10% following a high-dose Cu-ion irradiation at 100°C. The modulus of cold-worked and aged Cu-0.15% Zr is decreased following ion irradiation at 400°C.

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