Mechanical Property Changes in Ion Irradiated Metals – Part I: Ni-Cu Alloys

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MECHANICAL PROPERTY CHANGES IN ION IRRADIATED METALS - PART I: Ni-CU ALLOYS

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ABSTRACT

The effect of radiation-induced dislocation loops on hardness in ion-irradiated Ni-Cu alloys has been studied using a recently developed Mechanical Properties Microprobe (MPM). Well annealed Ni-10 at% Cu and Ni-50 at% Cu were irradiated with 14 MeV Ni ions to doses of 20 to 100 dpa peak damage (5 to 25 dpa at 1 μm) at 0.45 T_m (485°C and 425°C respectively). Ultra-low load microindentation hardness measurements and TEM were done using cross-section techniques. This method allows for direct hardness measurements of only the small irradiation zone (< 3 μm deep) which have been compared to the unirradiated material. Irradiation induced a high density of dislocation loops with the size and density of the loops dependent on composition and independent of irradiation conditions. This high dislocation loop density caused a large increase in hardness. A reasonable correlation was found between measured hardness changes and calculated changes based on dislocation loop sizes and densities.

INTRODUCTION

The Ni-Cu system has displayed a remarkable resistance to void formation under irradiation[1-6]. A recent study of two composition variations of the Ni-Cu system has shown that dislocation loops are the dominant defect resulting from heavy ion irradiation[6]. The formation of only this type of defect in an essentially homogeneous alloy provides an excellent opportunity to study the effect of loops on radiation hardening. However, until recently the narrow damage region produced by heavy ion irradiation (on the order of 1 μm deep) has made mechanical property tests on these samples impractical. A new technique has recently been introduced with which direct measurements of the mechanical properties of such regions can be made[7]. An investigation has been made using this technique to study the effect of dislocation loops on the hardness of two Ni-Cu compositions following ion irradiation, and to compare these results to the model of Ghoniem et al.[8]

EXPERIMENTAL PROCEDURE

High purity Ni-10% Cu and Ni-50% Cu specimens were irradiated with 14 MeV Ni ions to doses of 5, 10, and 25 dpa at 1 μm (20, 40 and 100 dpa peak) at 0.45 T_m (485°C and 425°C respectively)[6]. Irradiated samples were prepared for cross-sectional analysis using standard techniques[9]. TEM was used to obtain data on dislocation loop sizes and densities[6].

Ultra-low load microindentation hardness measurements were performed on a recently developed, fully automated mechanical properties microprobe (MPM)[10,11]. A schematic of the MPM is shown in Fig. 1. A load is applied and removed, and is continuously monitored along with displacement with a resolution of 2.5 μN (250 μg) and 0.4 nm respectively. Figure 2 represents a typical load-displacement curve obtained from the MPM. Hardness under load (uncorrected for elastic effects) can be calculated from the loading curves as a function of depth using $H=AL/d^2$, where d is a depth on the loading curve, L is the load at that depth and A is a geometric factor relating depth to the projected area of the indentation. A value for plastic hardness can be calculated from a load-displacement curve using the unloading part of the curve and is given by $H_p=AL_{max}/d_p^2$, where L_{max} is the maximum load applied and d_p is the maximum depth corrected for elastic effects(12).

Cross-section specimens were mechanically polished to a 0.05 μm finish and the surface electropolished using 67% CH_3OH and 33% HNO_3 at 15 V and -30 to -50°C prior to indentation. A line of indentations 5 μm apart was made at an angle of ~ 5.7° relative to the interface between the Ni-Cu foil and the Ni plating (see Fig. 3), to a depth of 150 nm, at a constant displacement rate of less than 5 nm/s. This resulted in indentations about 1 μm across spaced at intervals of 0.25 μm from the interface. Ratios of hardness to the average hardness away from the irradiated zone were calculated as a function of distance from the interface.

RESULTS

Figures 4a and 4b are examples of the microstructure of the two irradiated Ni-Cu compositions shown in cross-section. Both compositions display a high dislocation loop density in the irradiated region and are virtually defect free beyond that region. Figures 5a and 5b are enlargements of the irradiated region in Ni-10% Cu and Ni-50% Cu respectively. Table 1 shows the dislocation loop density and average diameter, and Fig. 6 shows the distribution of loop sizes. It can be seen that Ni-50% Cu has a very high density of small dislocation loops, while Ni-10% Cu has a lower density with a large range of loop sizes. Very few voids were observed in Ni-10% Cu, but the volume fraction of voids was so small that they have been ignored for the purposes of this study.

Although the two compositions start out with approximately the same hardness, they have very different radiation hardening characteristics (Figure 7). All the Ni-50% Cu samples display about a 55% increase in hardness in the irradiated region. The 5 and 10 dpa Ni-10% Cu samples have about a 25 to 30% increase in hardness while the 25 dpa sample has only about a 20% increase. All the hardness data has about a 10% standard deviation except near the end of range where the scatter is usually larger.

DISCUSSION

Void suppression in Ni-Cu alloys has been attributed to clustering of like atoms, and this suppression leads to the nucleation of dislocation loops[6]. It is thought that clustering is on a finer scale in the Ni-50% Cu relative to Ni-10% Cu, thus resulting in a higher density of smaller loops in Ni-50% Cu[6]. The loop characteristics change very little in Ni-50% Cu with increasing dose and, correspondingly, there is little change in hardness. At the highest dose, more large loops ($d > 75$ nm) are observed than for the lower doses in the Ni-10% Cu and some of these larger loops are seen to extend beyond the end of ion range. Slip of the larger loops to the surface and into the bulk may account for the lower hardness increase in the 25 dpa sample relative to the 5 and 10 dpa samples.

In all of the samples tested the hardness ratio is approximately the same across the irradiated zone for indentation depths $\gtrsim 75$ nm despite the fact that dpa varies with depth. Indentation depths below 75 nm were not compared because of the large scatter in data and possible surface effects. The constant hardness ratio at indentation depths $\gtrsim$ 75 nm would seem to indicate that bulk effects were being observed. This trend was observed in another study using this technique[7] and in Part II of this study. The constant hardness ratio across the irradiated zone can, in part, be accounted for by the fact that the actual size of the indentations (~ 7 x the depth across) are smaller than the area which contributes to the hardness[13]. Thus hardness is sampled over a wide range of dpa's and any hardness change associated with the changing dpa level will be dampened. It also appears from Fig. 4a and 4b that the dislocation loop sizes and densities are the same across the irradiated zone. This, coupled with the hardness data, indicates that saturation of the radiation hardening has probably occurred. Neutron irradiations below the void swelling temperatures of Cu and Ni have shown saturation of radiation hardening at < 0.1 dpa[14-17].

Theoretical hardening due to dislocation loops can be represented by[8]:

$$\Delta\sigma_y = \sqrt{3}\Delta\tau \approx 0.5Gb(Nd)^{1/2} \text{ small loops}$$

or

$$\approx 1.2Gbd(N)^{2/3} \text{ large loops}$$

where σ_y is the yield strength, τ is the shear stress, G the shear modulus, b the Burgers vector, N the loop density and d the loop diameter. Small loops interact through short range forces while large loops interact through long range forces[8]. The cutoff for small and large loops is relatively arbitrary but is often taken to be less than 10 nm[8]. Hardness can be related to yield strength by $H \approx C\sigma_y$, where H is the hardness and C is a constant (usually taken to be 3 for diamond pyramid hardness tests)[18-20]. Thus, the change in yield strength can be determined from:

$$\Delta\sigma_y/\sigma_y \approx \Delta H/H$$

or

$$\approx H_i/H_u - 1$$

where H_i and H_u are the irradiated and unirradiated hardnesses respectively. This ratio is constant for indentation depths > 75 nm, thus absolute hardness values are not needed and the actual value for C does not need to be known. Table 2 shows the comparison of the change in yield strength calculated from theory and from hardness changes. The calculations were made using an average loop diameter and an average Burgers vector assuming half perfect and half faulted loops. The yield strength for the unirradiated samples was taken to be ≈ 150 MPa[21]. For Ni-10% Cu the results are remarkably close using either the small or large loop calculation; however, the large loop calculation is probably a more valid model for this composition. Fair agreement is achieved in Ni-50% Cu using the two models; however, even better agreement can be achieved if a combination of the two models is used ($\Delta\tau = \Delta\tau$ (small) + $\Delta\tau$ (large))[8] and it is assumed that about 85% of the loops are small (~ 5 nm) and the rest are large (~ 15 nm). With such a high density of small loops, it is possible that many loops under 5 nm were missed. Also, loop densities are probably known only to within a factor of 2 or 3 due to TEM sample thickness uncertainties.

CONCLUSIONS

The Ni-Cu system's resistance to void formation results in the nucleation of a high density of dislocation loops under irradiation. The higher density of smaller loops in Ni-50% Cu causes a hardness change twice that of Ni-10% Cu. Radiation hardening appears to saturate at or below 5 dpa for these compositions.

The MPM appears capable of making direct hardness measurements in the narrow irradiated region. Measurement of the change in hardness after irradiation compares favorably with theoretical calculations made using TEM measurements of loop sizes and densities.

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Table 1. **Initial hardness and dislocation loop characteristics for irradiated Ni-Cu**

Composition	Unirradiated MPM Hardness (300 nm) (GPa)	Temp (°C)	Dpa (1μm)	Dpa (Peak)	Dislocation Loop Density (m^{-3})	Average Loop Diameter (nm)
Ni-10% Cu	2.1	485	5	20	$1x10^{21}$	29
			10	40	$1x10^{21}$	19
			25	100	$1x10^{21}$	25
Ni-50% Cu	2.0	425	5	20	$7x10^{21}$	6
			10	40	$5x10^{21}$	7
			25	100	$5x10^{21}$	10

Table 2. Comparison of yield strength changes in irradiated Ni-Cu from MPM measurements and theoretical calculations in GPa

Composition	MPM Measurement	Small Loop Model	Large Loop Model	Large & Small Loop Model
Ni-10% Cu	38±4	32	38	
Ni-50% Cu	83±8	52	51	73

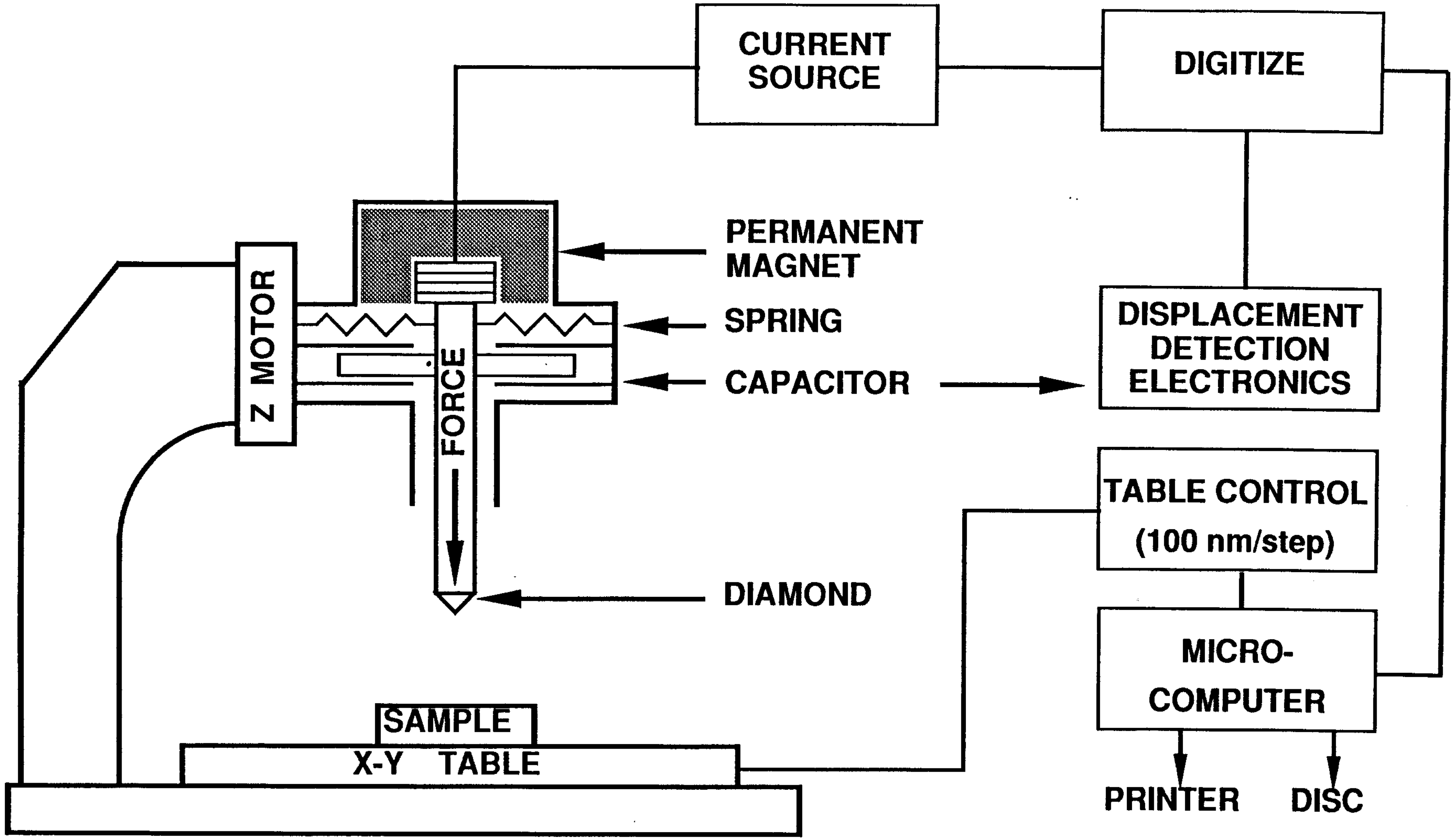

Fig. 1—Mechanical properties microprobe (MPM) schematic

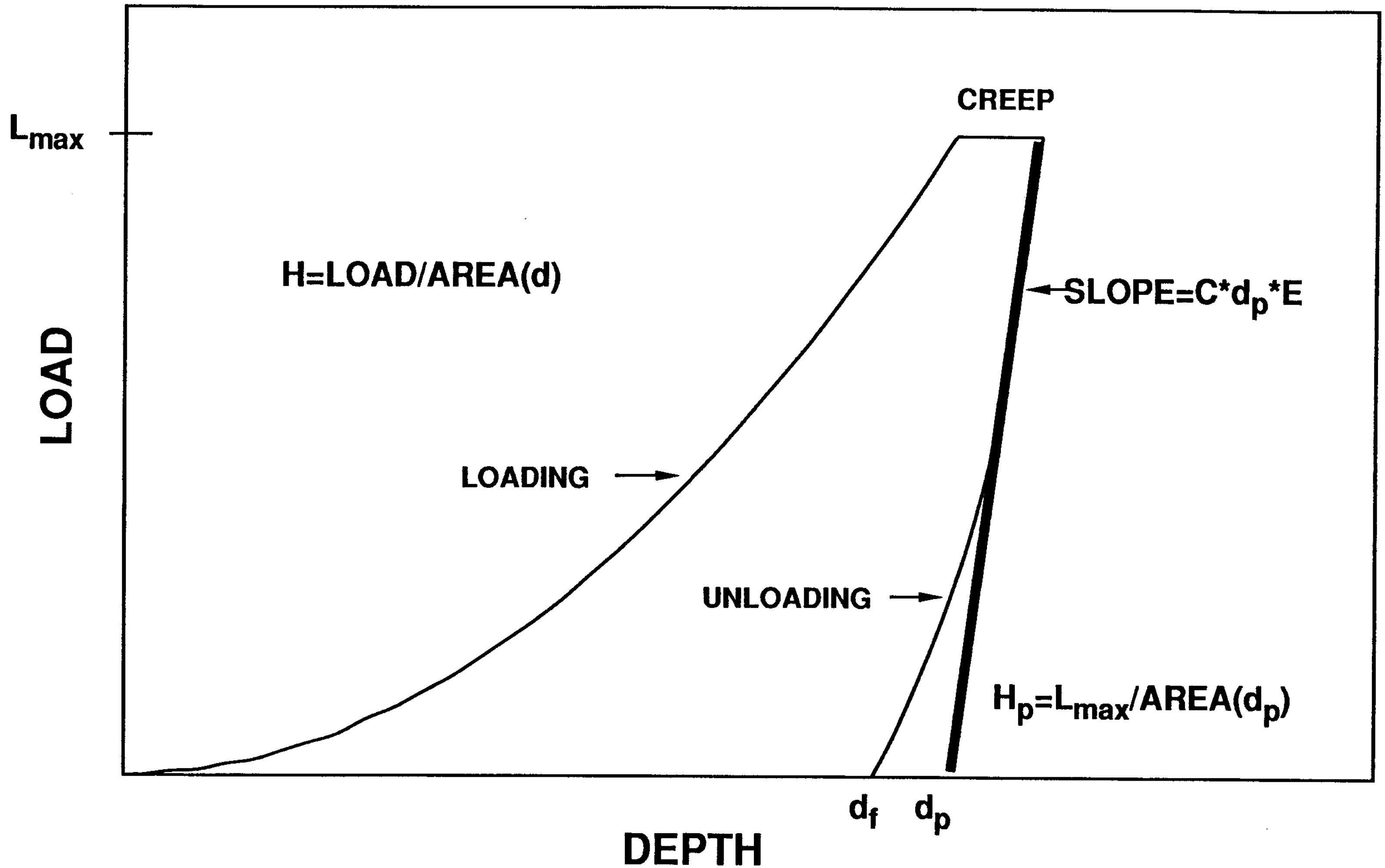

Fig. 2—Representative load-displacement curve from the mechanical properties microprobe

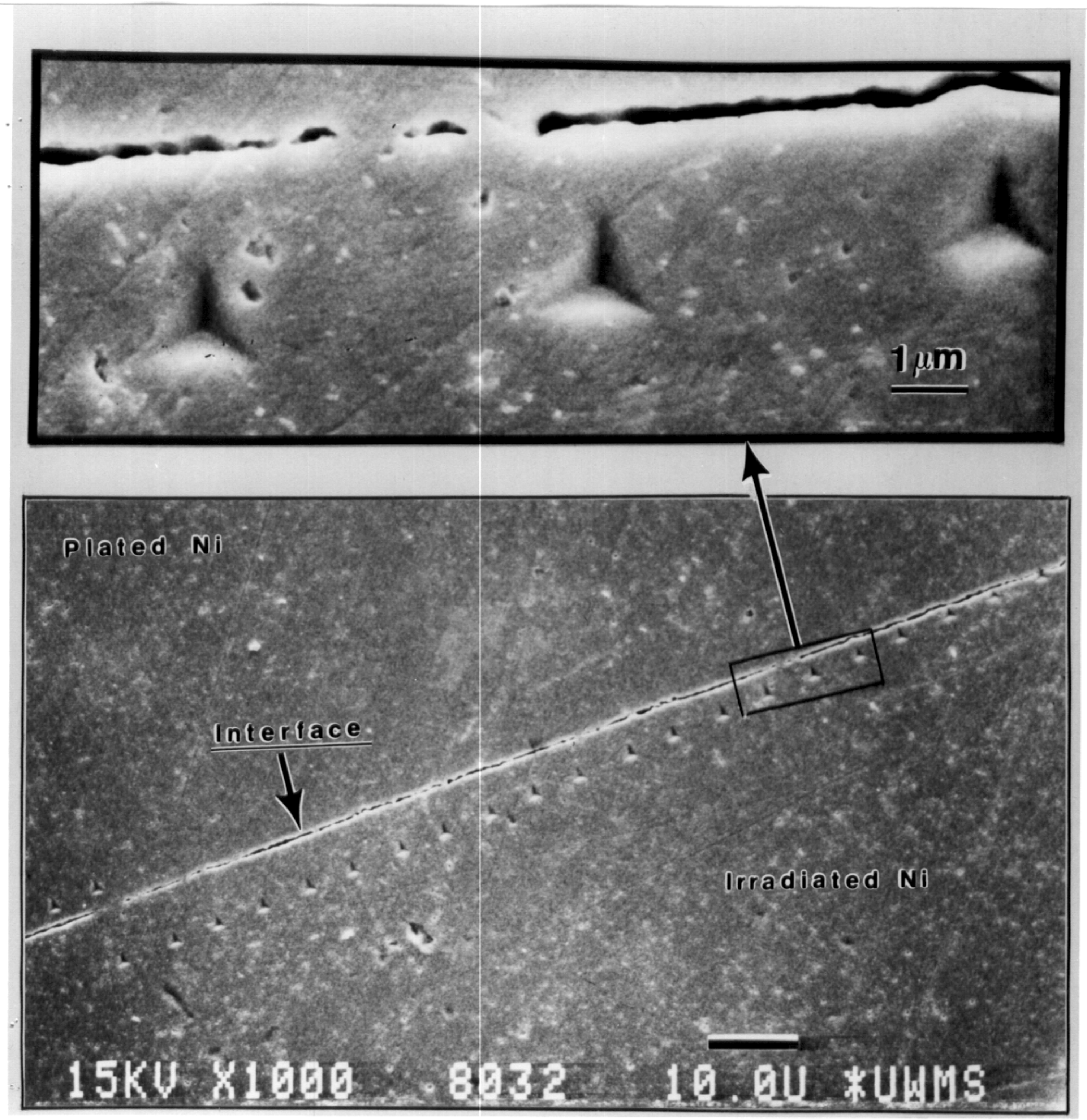

Fig. 3—SEM micrographs of indentations made in a cross-sectioned sample.

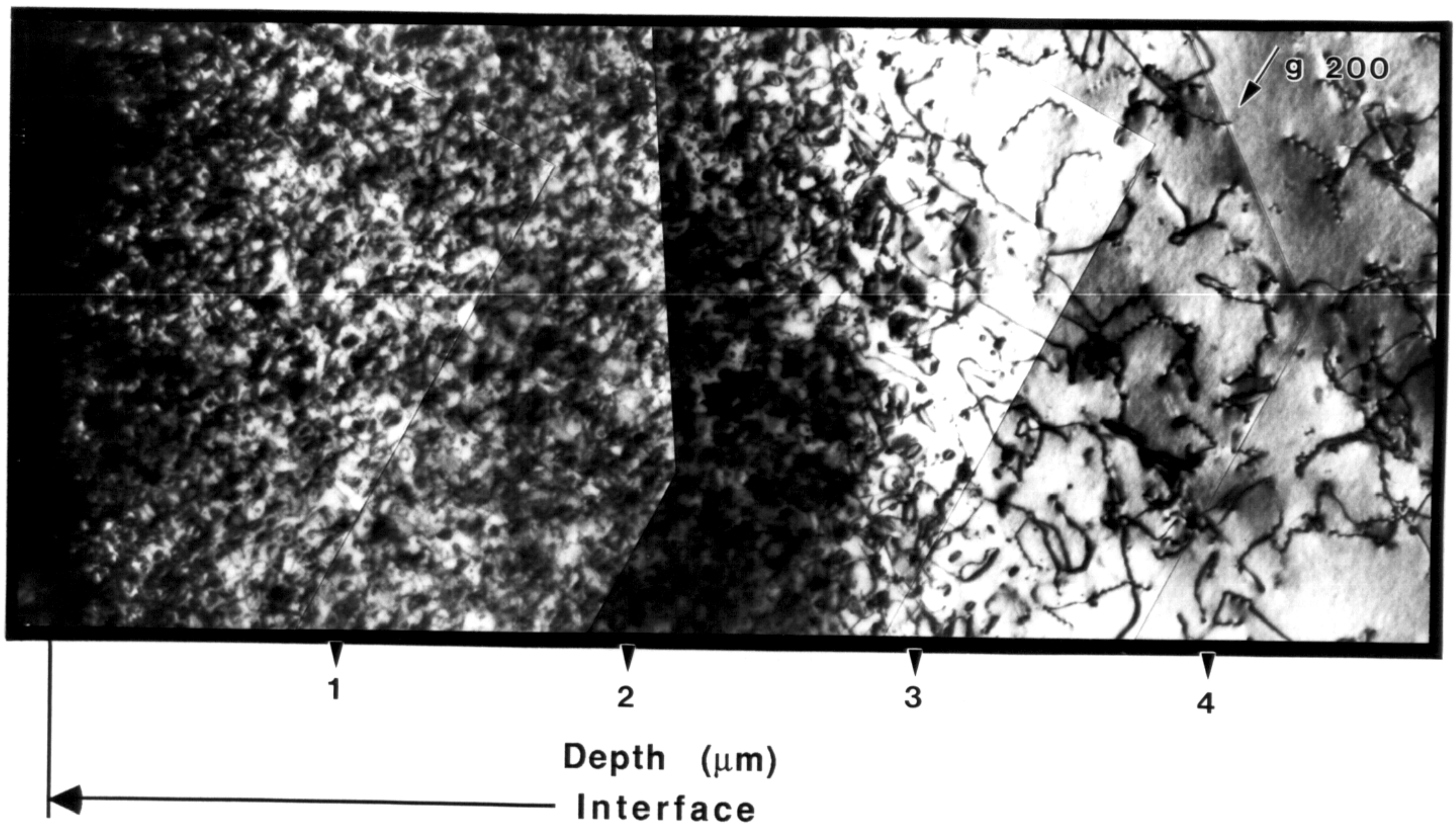

Fig. 4a—TEM micrograph of Ni-10% Cu irradiated to 10 dpa at 1 μm at 0.45 Tm in cross-section showing the entire irradiated region.

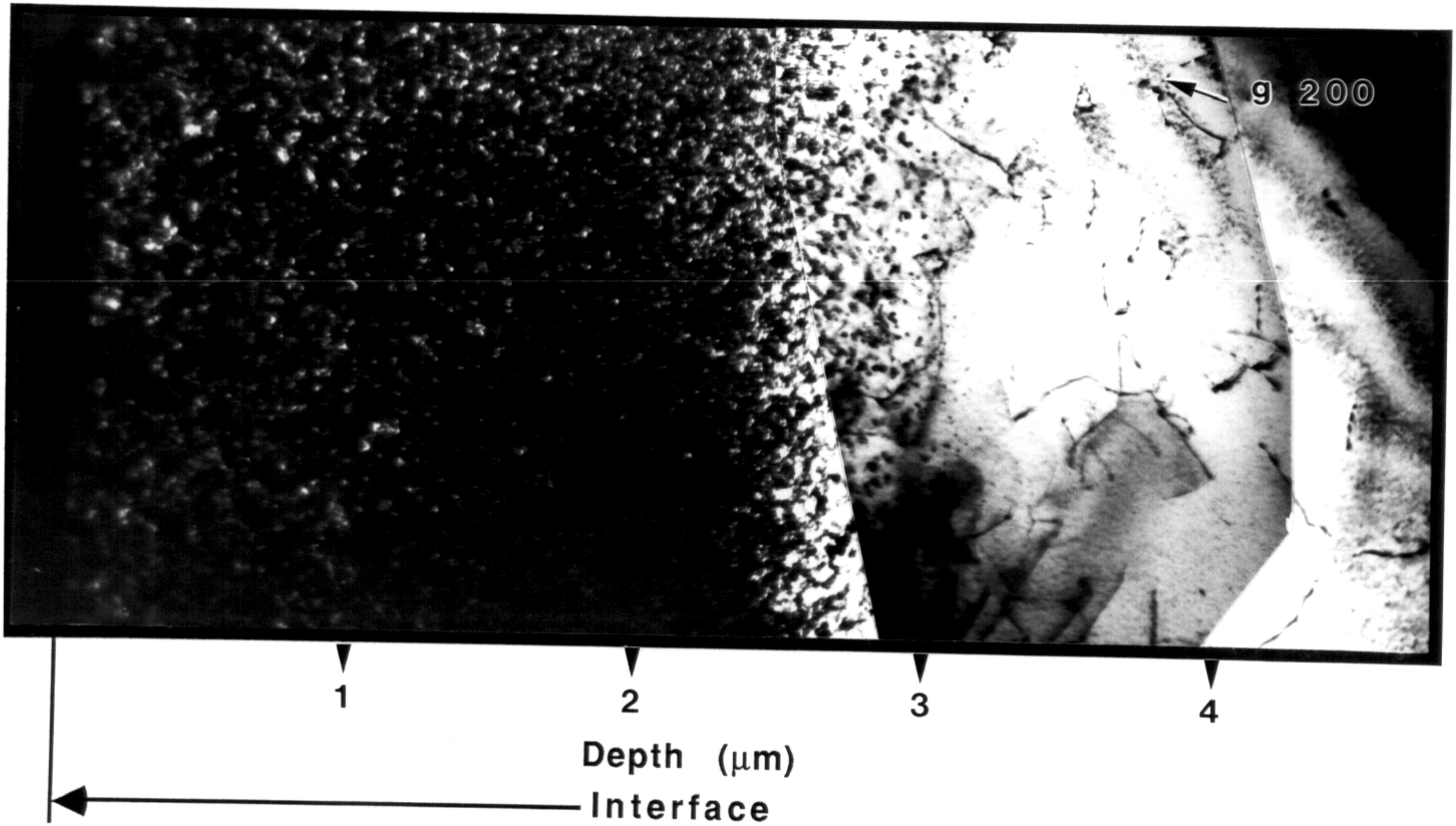

Fig. 4b—TEM micrograph of Ni-50% Cu irradiated to 10 dpa at 1 μm at 0.45 Tm in cross-section showing the entire irradiated region.

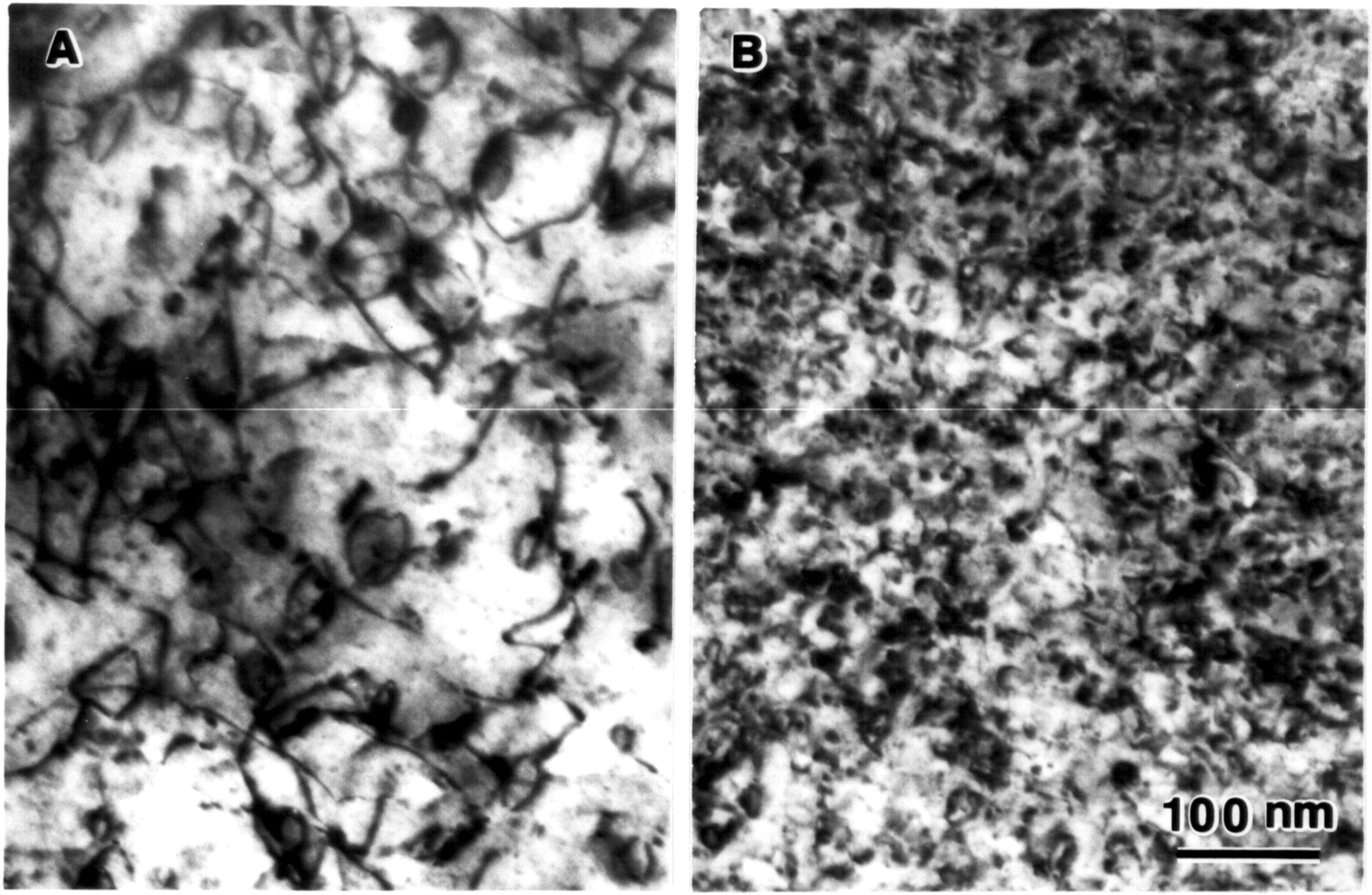

Fig. 5—Enlargements of the irradiated regions from Fig. 4 for Ni-10% Cu (a) and Ni-50% Cu (b).

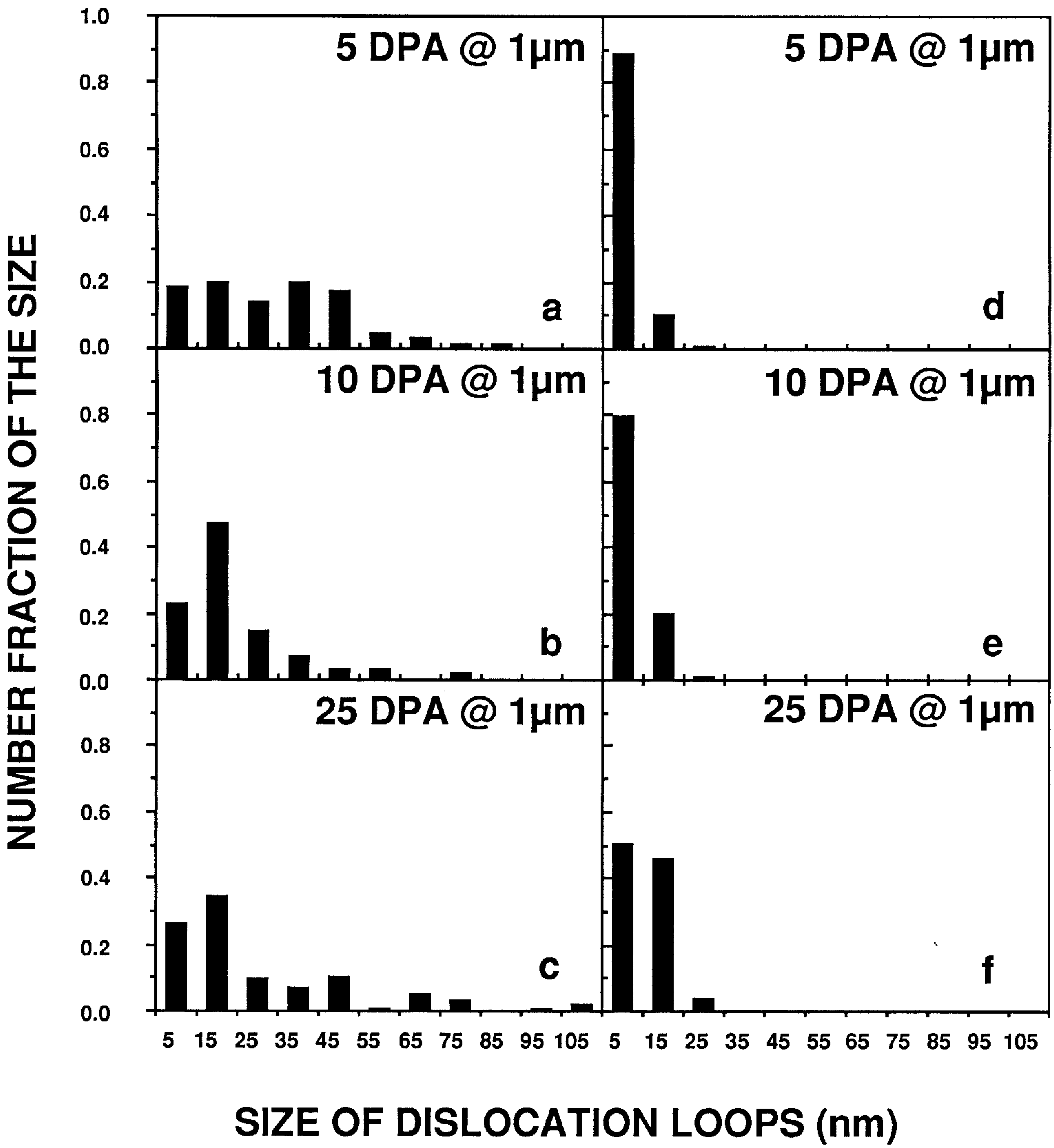

Fig. 6—Distribution of dislocation loops in irradiated Ni-10% Cu (a-c) and Ni-50% Cu (d-f) for different fluences.

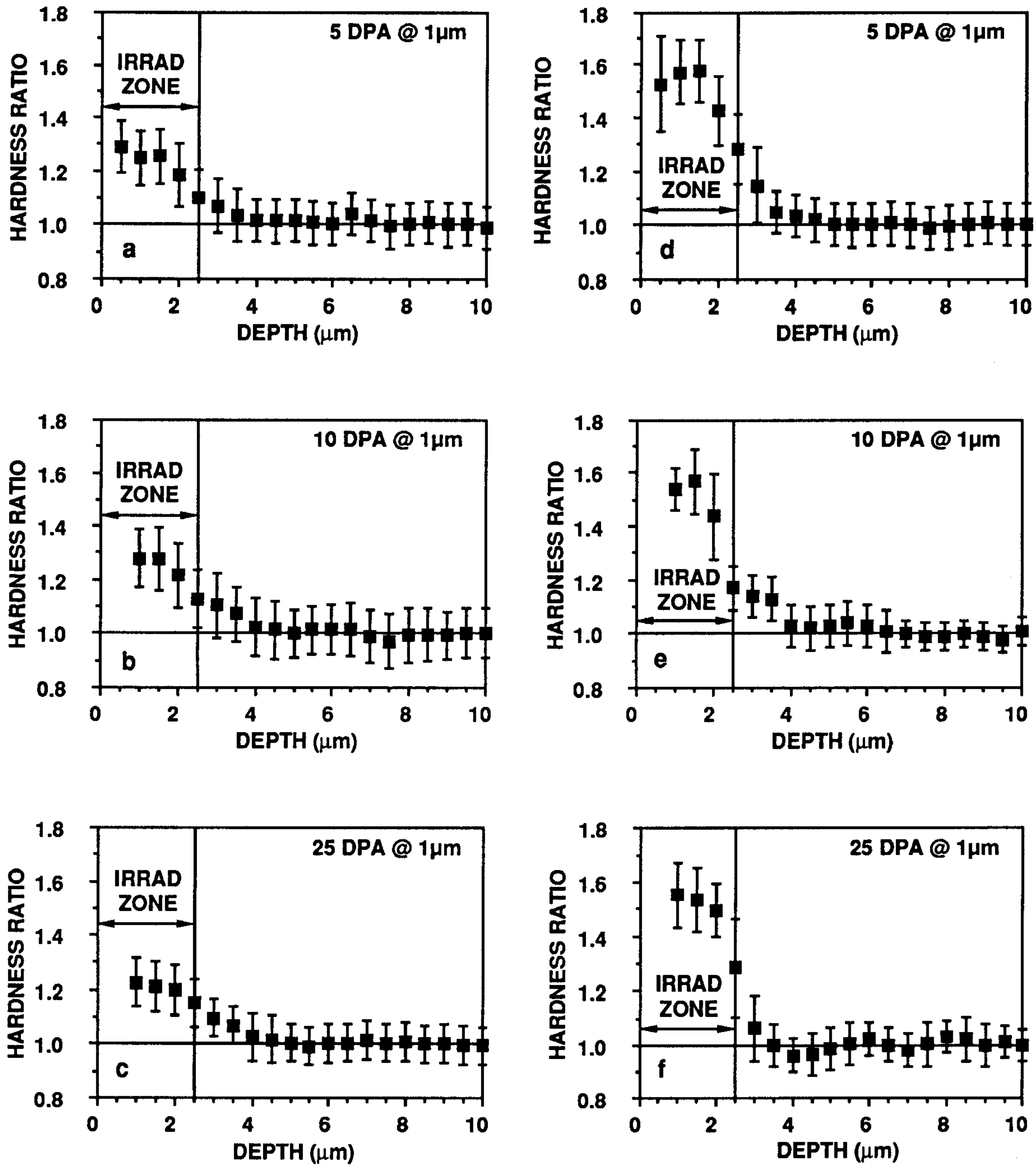

Fig. 7—Ratio of hardness to average unirradiated hardness versus depth in Ni-10% Cu (a-c) and Ni-50% Cu (d-f) irradiated at 0.45 Tm to various fluences.