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Fusion Technology Institute
University of Wisconsin
1500 Engineering Drive
Madison, WI 53706

http://fti.neep.wisc.edu

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MICROSTRUCTURE OF COPPER FOLLOWING HIGH DOSE 14-MeV Cu ION IRRADIATION

S.J. Zinkle[1], G.L. Kulcinski and R.W. Knoll[2]

Fusion Technology Institute
1500 Johnson Drive
University of Wisconsin
Madison, WI 53706-1687

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1 Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee USA 37831

2 Battelle Northwest Laboratory, Richland, Washington USA 99352

Abstract

High-purity copper has been irradiated with 14-MeV Cu ions to peak doses of 40 dpa over the temperature range of 100-500°C. Examination of the foils in a transmission electron microscope revealed that no significant amount of void formation had occurred, in conflict with previous irradiation studies. Instead, a high density of stacking fault tetrahedra (SFT) were observed. The defect cluster density is constant for irradiation temperatures $\leq$ 200°C, and the density decreases rapidly with irradiation temperature $\geq$ 300°C. It is postulated that the absence of voids is due to the low oxygen content of the copper foils (< 5 wt. ppm). The depth dependence of the ion damage was studied by using the cross-section technique. Evidence of displacement damage in the form of "black spot" defect clusters (SFT and small dislocation loops) was observed at depths well beyond that expected from ion damage range calculations. The discrepancy between the observed and calculated damage profile is most likely due to the use of too large of an electronic stopping power value in the calculation, but diffusional spreading effects may also be playing a role.

Introduction

There is currently a strong interest in copper and copper alloys for fusion reactor applications [1]. An extensive data base has been established on the properties and microstructure of copper following irradiation to doses of one displacement per atom (dpa) or less [2-5]. However, the high-dose (> 1 dpa) neutron irradiation behavior of copper is not very well characterized. In particular, the void swelling behavior of copper has never been satisfactorily determined. There are about fifteen known studies of copper or copper alloys at neutron irradiation conditions relevant for void formation [5]. Only three of these studies were performed at doses greater than 1 dpa, and they were each confined to a single irradiation temperature [6,7,8]. Low-fluence neutron results indicate that void formation occurs in copper for irradiation temperatures between 220 and 550°C (0.35 - 0.61 T_M).

High-dose (> 1 dpa) electron and ion irradiation studies have generally found [5] that void formation occurs easily in copper for irradiation temperatures of 300 to 550°C. However, there have been several investigations of copper alloys where no voids were observed over this temperature range even after ion irradiation to 40 dpa [9-13]. Previous ion and electron irradiation studies by Glowinski and coworkers suggested that gas is also necessary for void formation in pure copper [14-17]. However, only a limited temperature range was studied, which opened the possibility that the absence of gas merely shifts the void swelling regime to different (lower) temperatures. The present report is intended to address the issue of whether void formation is possible in high purity (gas free) copper during charged particle irradiation. Specifically, the microstructure of pure copper has been examined following self-ion irradiation at dose and temperature conditions relevant for void swelling: 10 to 40 dpa and 100 to 500°C. All of the irradiated foils were examined in cross-section in order to observe the depth-dependent nature of the radiation damage.

Experimental Procedure

Foils of MARZ grade (99.998% purity including interstitial impurities) copper obtained from Materials Research Corporation were used for the irradiations. The nominal impurity content of the manufacturer's stock material is given in Table 1. The as-received foils were annealed at 800°C for 1.5 hours and cooled to room temperature in a hydrogen atmosphere prior to their preparation for irradiation [13]. The pre-irradiation treatment consisted of mechanical polishing followed by electropolishing for 5 seconds at an applied potential of 5 V in a solution of 33% HNO_3/67% CH_3OH cooled to -50°C. The low polishing temperature was chosen to prevent gas from being introduced into the foils during electropolishing. The foils were irradiated with 14-MeV Cu^{3+} ions using the University of Wisconsin tandem Van de Graaf accelerator. Figure 1 shows the depth-dependent damage profile for 14-MeV Cu ions incident on a copper target as calculated by the BRICE code [18]. The average flux for all samples was about 5 x 10^{16} ions/m^2-s, with fluences ranging from 7 x 10^{19} to 3 x 10^{20} ions/m^2. This corresponds to a calculated peak damage rate of 7 x 10^{-3} dpa/s and peak damage levels of 9 to 40 dpa. Table 2 lists the irradiation conditions for this study. The vacuum during irradiation was about 1 x 10^{-7} torr. The foils were irradiated in a thermal shielded carousel specimen holder [13] which limited the amount of post-irradiation annealing (maximum of 100 to 200°C for 2 hours).

Following the irradiation, all of the samples were electroplated with copper and prepared for cross-section analysis using methods that are described in detail elsewhere [13,19]. The cross-section specimens were jet-electropolished in a solution of 33% HNO_3/67% CH_3OH cooled to -30°C at an applied potential of 20 V (90 mA current) and were examined in a JEOL TEMSCAN-200CX electron microscope.

Results

Ion irradiation of pure copper over the temperature range of 100-500°C (0.28-0.57 T_M) resulted in "black spot" damage and the formation of dislocation tangles and loops, but no significant void formation. A sparse distribution of voids was observed in pure copper irradiated to a peak damage level of 40 dpa at 400°C. The average void diameter for this condition was $\lesssim$ 100 nm, with an estimated density of only $10^{17}/m^3$ ($\Delta V/V \lesssim 5 \times 10^{-5}$). No voids were observed in pure copper for any of the other irradiation conditions over the temperature range of 100 to 500°C. A room temperature preimplantation of 45 appm He into copper followed by a 40 dpa peak damage ion irradiation at 300°C resulted in the formation of small cavities in the He implanted region (0.5-1.5 μm depth) [12]. The damage level in the He implanted region was roughly constant at 10 dpa (see Fig. 1). The visible cavities were of small size, d = 2.6 nm, and high density, $n \gtrsim 6 \times 10^{21}/m^3$ ($\Delta V/V \simeq 5 \times 10^{-5}$). Conversely, preimplantation of 45 appm H in pure copper did not produce any observable cavity formation following a 300°C, 40 dpa peak damage irradiation [12].

Figure 2 shows the cross-section microstructures of ion-irradiated copper for irradiation temperatures of 100-400°C. Evidence of radiation damage exists to depths in excess of 3.5 μm, which is significantly deeper than the predicted damage range obtained from a Brice calculation for 14-MeV Cu ions incident on a copper target (Fig. 1). The defect cluster density was roughly constant for irradiation temperatures of 100 and 200°C. Irradiation at temperatures above 300°C resulted in a very low defect cluster density.

The low temperature (100-300°C) damage microstructure consisted of localized regions of large defect clusters which are superimposed on a background of small defect clusters. It is possible that these enlarged clusters are due to subcascades or the agglomeration of small planar loops. Dislocation loops of ~ 30 nm diameter and dislocation tangles were observed following ion irradiation at 400°C and 450°C. The damage at 450°C was limited to the depth range of ~ 1.5 to 2.8 μm; the depth range of 0 to 1.5 μm was denuded of any dislocation loops or tangles. There was no observable sign of radiation damage in the copper foil irradiated at 500°C.

A more detailed analysis of the black spot damage revealed several significant features. Figure 3 shows enlarged black spot defects in irradiated copper that are located in the near vicinity of dislocation lines. Since dislocation lines are known to be a preferential sink for interstitials, it appears likely that the larger defect clusters in Fig. 3 may be interstitial loops which have grown as a result of a favorable incident point defect flux. The observation of enhanced cluster sizes on dislocations in ion irradiated copper agrees very well with experimental results by Yoshida et al. [20] on 14-MeV neutron irradiated copper. They found that about 80% of the large clusters located near dislocations were interstitial-type loops.

Figure 4 gives the high-magnification bright field microstructure of pure copper irradiated with Cu ions to 10 dpa at 200°C. Triangle-shaped stacking fault tetrahedra (SFT) are visible along with other black spot defects that have no definite shape.

There does not appear to be any substantial zone that is denuded of SFT in the vicinity of grain boundaries. Figure 5 illustrates the weak beam dark field microstructure of irradiated copper at a grain boundary. Formation of SFT occurs right up to the boundary, although there is possibly a lower SFT density within the first 10 nm of the grain boundary. Previous studies [21] have found that there is no denuded zone at grain boundaries for small defect clusters (< 5 nm) observed in copper following neutron irradiation. A denuded zone of ~ 100 nm width was observed for defects with sizes greater than 5 nm (predominantly interstitial-type clusters) [21].

Irradiation of copper at 100°C resulted in a coarsened damage microstructure in the peak damage region compared to the midrange damage microstructure (see Fig. 2). This difference in the peak and midrange damage microstructures was not evident at the other (higher) irradiation temperatures that were investigated, i.e. 200-400°C. A similar coarse peak damage microstructure was observed in a Cu-Cr-Zr alloy irradiated at 100°C [12]. Figure 6 compares the peak and midrange damage microstructures of copper irradiated at 100°C. The larger defect clusters in the peak damage region appear to be dislocation loops, with an average diameter of 12 nm and a density of $\gtrsim 1 \times 10^{21}/m^3$.

The measured defect cluster density in ion-irradiated copper at a depth of 1 μm (10 dpa damage level) is plotted as a function of irradiation temperature in Fig. 7. Representative error bars are indicated on the data points. The main source of error was due to uncertainties in the foil thickness, which was determined from thickness fringes with an estimated ±20% accuracy. Both the SFT and total cluster density were constant for irradiation temperatures $\leq$ 200°C with values of $9 \times 10^{22}/m^3$ and $1.4 \times 10^{23}/m^3$, respectively. Irradiation at $\geq$ 300°C resulted in a sharp decrease in the cluster densities. The general trend of the cluster density versus irradiation temperature curve is in good agreement with previous investigations of ion [22] and neutron irradiated [23] copper, i.e. the defect cluster density is constant for irradiation temperatures below 250°C and the density decreases rapidly for irradiation temperatures above 300°C. The fraction of defect clusters that are resolvable as SFT is $\gtrsim$ 60% for irradiation temperatures of 100-200°C (Fig. 7). This fraction decreases at higher irradiation temperatures, becoming about 30% for irradiation at 400°C.

Figure 8 is a histogram showing the size and frequency of defect clusters that were observed at a depth of 1 μm (10 dpa damage level). The measurements were made from bright field and weak beam dark field micrographs with a total print magnification of about 1 x 10^6. A total of 500 to 1500 defect clusters were counted using a Zeiss particle analyzer for each of the irradiation temperatures from 100-300°C. Only 150 clusters were counted for the 400°C irradiation condition due to the low cluster density. The SFT and total cluster size distributions were very similar for irradiation temperatures of 100-300°C. The size distributions appear to be close to log-normal, with a peak at about 2.0 nm. The mean SFT and total cluster diameters were determined to be 2.6 and 2.8 nm, respectively, over this temperature range. The mean defect size was $\gtrsim$ 4 nm following irradiation at 400°C. Other researchers have reported mean SFT sizes of 2.2 to 2.6 nm following neutron [20,23] and Cu^+ ion [24] irradiation of copper and copper alloys, in agreement with the present results.

Discussion

A. Microstructure of Copper Following Ion Irradiation

The observed temperature dependence of the "black spot" density of irradiated pure copper (Fig. 7) is in good agreement with previous experimental and theoretical studies [22,23]. The fall-off in the defect cluster density with increasing irradiation temperature has been attributed to thermal emission of vacancies. Muncie et al. [23] reported that vacancy loops were the dominant vacancy cluster morphology following an 80°C neutron irradiation of copper. Irradiation at higher temperatures resulted in an increasing fraction of SFT, with 99% of the vacancy clusters identified as SFT at 300°C. This type of temperature dependence with regard to cluster morphology was not observed in the present investigation. The difference in the observed neutron and ion irradiation behavior may be due to damage rate effects. The increased atomic mobility associated with ion irradiation, compared to neutron irradiation, may allow vacancy loops to dissociate into SFT more readily at the low irradiation temperatures.

The observed defect cluster size distribution was peaked at about 2.0 nm (Fig. 8). It is believed that this is a true measure of the most probable defect cluster size. However, this peak is close to the microscope resolution limit of ~ 1 nm and might possibly be a microscopy artifact.

It is now well established that void formation and growth can be suppressed during ion irradiation in the region where the injected ions are deposited, which roughly coincides with the peak damage region [25]. Temperature and dose rate dependent surface effects are also known to result in void denuded regions to depths on the order of 0.5 μm [26]. The use of high-energy ions for irradiation (14-MeV in the present case) produces a region at intermediate depths (~ 1 μm) that is free of both injected ion and surface effects. Cross-sectional analysis of these irradiated foils provides conclusive evidence that significant levels of void formation do not occur during charged particle irradiation of high purity copper.

The absence of appreciable void formation in the ion-irradiated copper foils may initially be regarded as surprising in light of the numerous [5] experimental charged particle irradiation studies which have shown that void formation occurs easily in copper. However, theoretical calculations indicate that the most stable morphology of a vacancy cluster in pure copper is in a planar configuration (such as a SFT or a dislocation loop) and not as a void [27-29]. We have recently reexamined [12,30] the relative energies of various vacancy clusters in copper using the formalism of Sigler and Kuhlmann-Wilsdorf [27]. The results of the calculation are reproduced in Fig. 9. The continuum mechanics calculation predicts that stacking fault tetrahedra are the most stable configuration of vacancy clusters in pure copper, in agreement with the present experimental results. There have been several recent observations of SFT in copper following electron [31,32] and neutron [20,23,33] irradiation. Stathopoulos et al. [24] did not observe SFT in pure copper following ion irradiation, but instead found Frank loops that were dissociated towards stacking fault tetrahedra.

It is proposed that the lack of significant void formation in the present case is due to the low oxygen content (< 5 ppm) of the copper foils that were irradiated. Glowinski and co-workers observed that copper containing 50 ppm oxygen formed voids easily following electron [14] and ion [15-17] irradiation. However, void swelling was shifted to lower temperatures or else completely eliminated in Cu foils that had been outgassed prior to irradiation [15-17,34]. A thermodynamic model has been recently developed which predicts that a matrix oxygen content of at least 1 ppm O is needed in order to make void formation energetically stable in pure copper [12,30]. The stacking fault tetrahedron is predicted to be the most stable vacancy cluster configuration for copper when there is an insufficient oxygen concentration.

It appears that the bulk of the more than twenty previous charged particle irradiation studies of pure copper and copper alloys that have reported void formation may need to be re-evaluated with regard to their initial gas content. High purity (electrolytic grade) copper that has not been deoxidized typically contains $\gtrsim$ 200 ppm oxygen and copper is classified as "oxygen free" even if it contains up to 10 ppm (~ 40 appm) oxygen [35]. Differences in initial gas concentration may have a strong influence on the types of radiation damage defect clusters that occur during irradiation and could conceivably mask the effects of other irradiation parameters being studied.

B. Comparison of Theoretical and Experimental Ion Damage Range

Irradiation of copper with 14-MeV Cu ions produced damage which generally extended to depths greater than expected from BRICE code calculations (Fig. 1). The BRICE code predicts that the damage range for 14-MeV Cu ions incident on copper should be $\lesssim$ 2.8 μm (Fig. 1). Black spot damage profiles gave a clear measurement of the distribution and range of the cumulative displacement process for the copper foils irradiated at 100 to 300°C. The maximum range of 14-MeV Cu ions incident on copper was determined to be 2.7 to 4.4 μm from black spot damage measurements, with most range measurements centered at about 3.5 μm. More than 25 measurements were made on cross-section specimens that were irradiated at low temperatures (100 to 300°C). There was no apparent temperature dependence of the damage range within the scatter of the data.

The rather large variation in the experimentally measured damage range may be due to several different sources. The lower values of the damage range were obtained on specimens where the foil-plating interface was not intact due to an oversized TEM hole. Although procedures were established to ensure that the edge of the foil coincided with the original interface, it is possible that as much as 0.2 to 0.3 μm of the foil surface may have been missing. The cross-section foils were always placed in the electron microscope with the foil-plating interface parallel to the tilt axis. Translation along the intact interface in thicker regions of the foil provided a check on whether a substantial portion of the original foil surface had been removed during the electrothinning process (estimated accuracy limit ≈ 0.2 μm). All cross-section measurements were corrected for the tilt angle in the microscope. A previous investigation of ion-irradiated copper reported an even larger variation in the measured damage range than is given here. In that study [36], displacement damage extended from 0 to 16 times the calculated range depending on the irradiation temperature.

Cross-section studies of ion-irradiated metals by other researchers have often found that the measured damage range exceeds the calculated value by a significant amount [37-43]. Knoll [13] observed evidence of displacement damage (loss of coherence of precipitates) to depths of 3.4 to 4.3 μm in copper alloys irradiated with 14-MeV Cu ions. This extended damage observation is in good agreement with the present results, although Knoll postulated that the observed effects may simply be due to long-range migration of interstitials. The occurrence of ion displacement damage beyond the calculated end of range has generally been attributed to stopping power errors [37-41] and diffusional spreading effects [42,43].

The calculated ion damage range depends on the choice of the available electronic stopping powers [38]. Figure 10 shows the calculated depth-dependent displacement damage profiles for 14-MeV Cu incident on a copper target using LSS [44] and Firsov [45] electronic stopping power (esp) values. The use of Firsov's esp values leads to a broadened damage distribution and a deeper ion damage range as compared to LSS, in agreement with the present experimental results. However, the predicted damage range is still significantly less than the observed maximum range. Narayan et al. [38] and others [46,47] have found that modifications of the electronic stopping power result in calculated ion ranges that are in good agreement with experimental findings.

Farrell et al. [42] have proposed that the discrepancy between observed and calculated damage profiles in ion irradiated metals is primarily due to the neglect of diffusional spreading effects in the calculations. However, Whitley [39] concluded that diffusional spreading alone could not cause the observed increased damage range. In general, the high dose levels of the irradiated cross-section specimens in the present study do not allow a distinct determination of damage level versus depth to be made -- there are no distinguishable depth-dependent changes in defect cluster density. However, the irradiated microstructure at 100°C did exhibit a different end of range behavior (Figs. 2,6). It seems reasonable to associate this end of range (EOR) microstructure with the peak damage and/or injected ion region. The "coarse" EOR microstructure extended from a depth of 2.8 μm to 3.6 μm. If diffusion spreading was by itself responsible for the observed extended damage depth in ion-irradiated copper, then the coarse EOR microstructure should begin at $\lesssim$ 2 μm (corresponding to the calculated EOR damage) and extend out to the maximum observed damage depth (~ 3.6 μm). The fact that the coarse EOR microstructure is not observed at the calculated peak damage region indicates that the damage range calculations (electronic stopping power) are underestimating the actual mean ion range. It is possible that diffusional spreading may be further enhancing this discrepancy, but by itself it cannot account for the experimental observations.

In summary, it appears that the difference between the observed and calculated ion displacement damage profiles may be attributed to the use of too large an electronic stopping power in the calculations, along with possible diffusion spreading effects. Failure to take into account this difference between the calculated and actual damage profile can result in wrong dose values. In particular the extended ion damage range means that at a given depth the actual displacement damage is less than the calculated value on the near-surface side of the damage peak. This may be one contributing source to the well-known difference [5,48] between ion and neutron void swelling rates.

Conclusions

Ion irradiation of high-purity copper over the temperature range of 100-500°C results in the formation of stacking fault tetrahedra and dislocation loops and tangles. The defect cluster density decreases rapidly with irradiation temperature ≥ 300°C. The absence of any significant void formation is attributed to the low oxygen content of the copper foils (< 5 ppm O). Future irradiation studies on copper and copper alloys should be performed on foils with a well-known and preferably low gas concentration. The observed displacement damage extends to depths that are about 30% greater than expected from a BRICE damage calculation. The discrepancy between the observed and calculated damage profile is most likely due to the use of too large of an electronic stopping power value in the calculation.

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Table 1. Impurity Analysis of Copper Foils as Determined by the Manufacturer in Weight Parts per Million (ppm)

Element	ppm	Element	ppm
C	6	Cr	0.3
H	< 1.0	Fe	0.8
O	< 5.0	Mn	< 0.1
N	< 1.0	Ni	< 0.1
Ag	0.5	Pb	0.7
Al	1.0	S	0.5
Ca	0.4	Si	0.3
Cd	< 0.1	Sn	< 0.1
Co	< 0.1	Zn	1.5

Table 2. Irradiation Parameters for the Ion-Irradiated Copper Foils

Irradiation Temperature	Calculated Damage (dpa) 1 μm	Peak (2 μm)
100°C	10	40
200°C	10	40
300°C	10	40
400°C	10	40
400°C	2	9
450°C	7	30
500°C	2	9

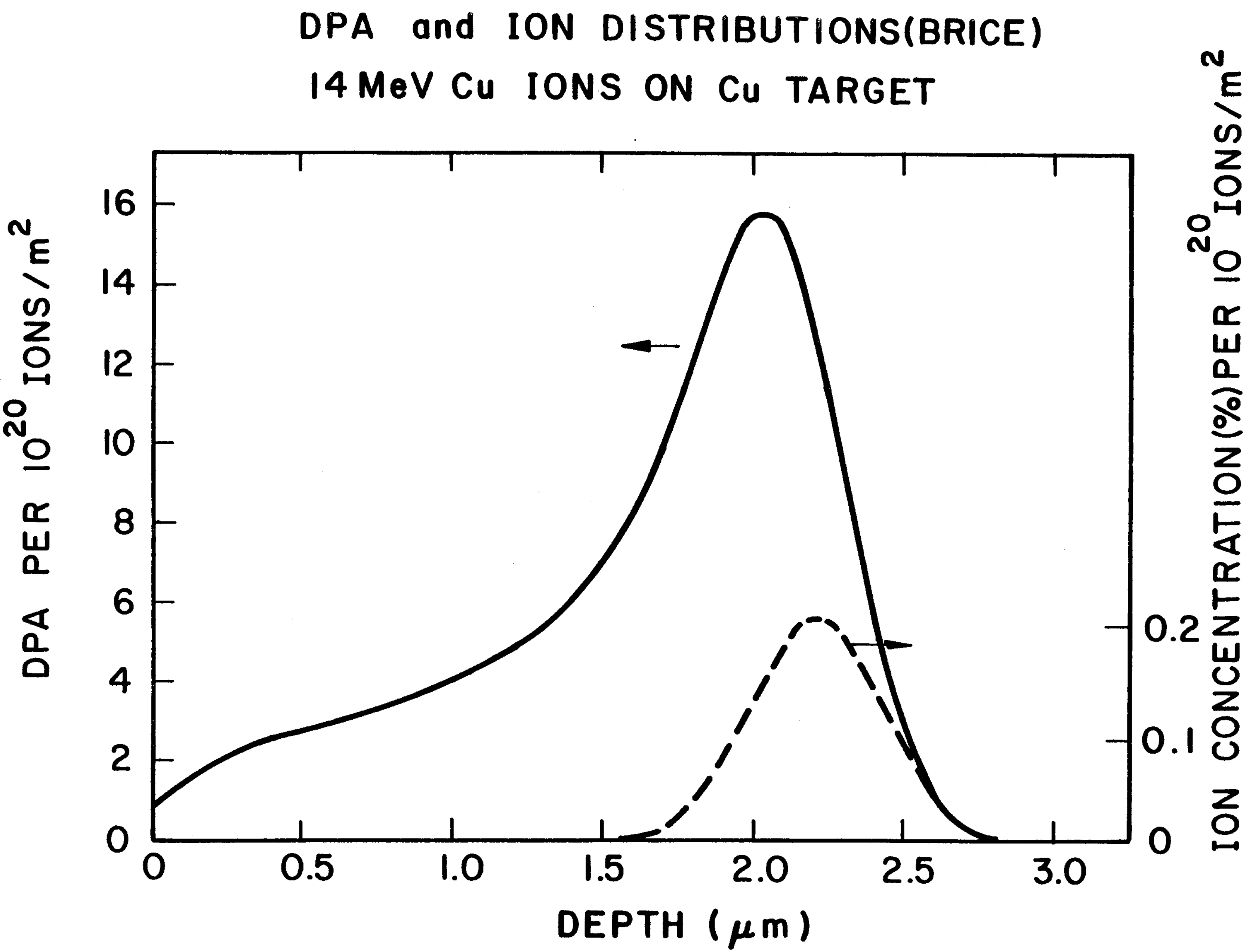

Figure 1. Calculated 14-MeV Cu ion displacement damage profile and injected ion distribution for a copper target as determined from the BRICE code [18]. The calculation assumes a displacement energy of E_d = 29 eV and a displacement efficiency of K = 0.8.

CROSS-SECTION MICROSTRUCTURE OF ION-IRRADIATED COPPER

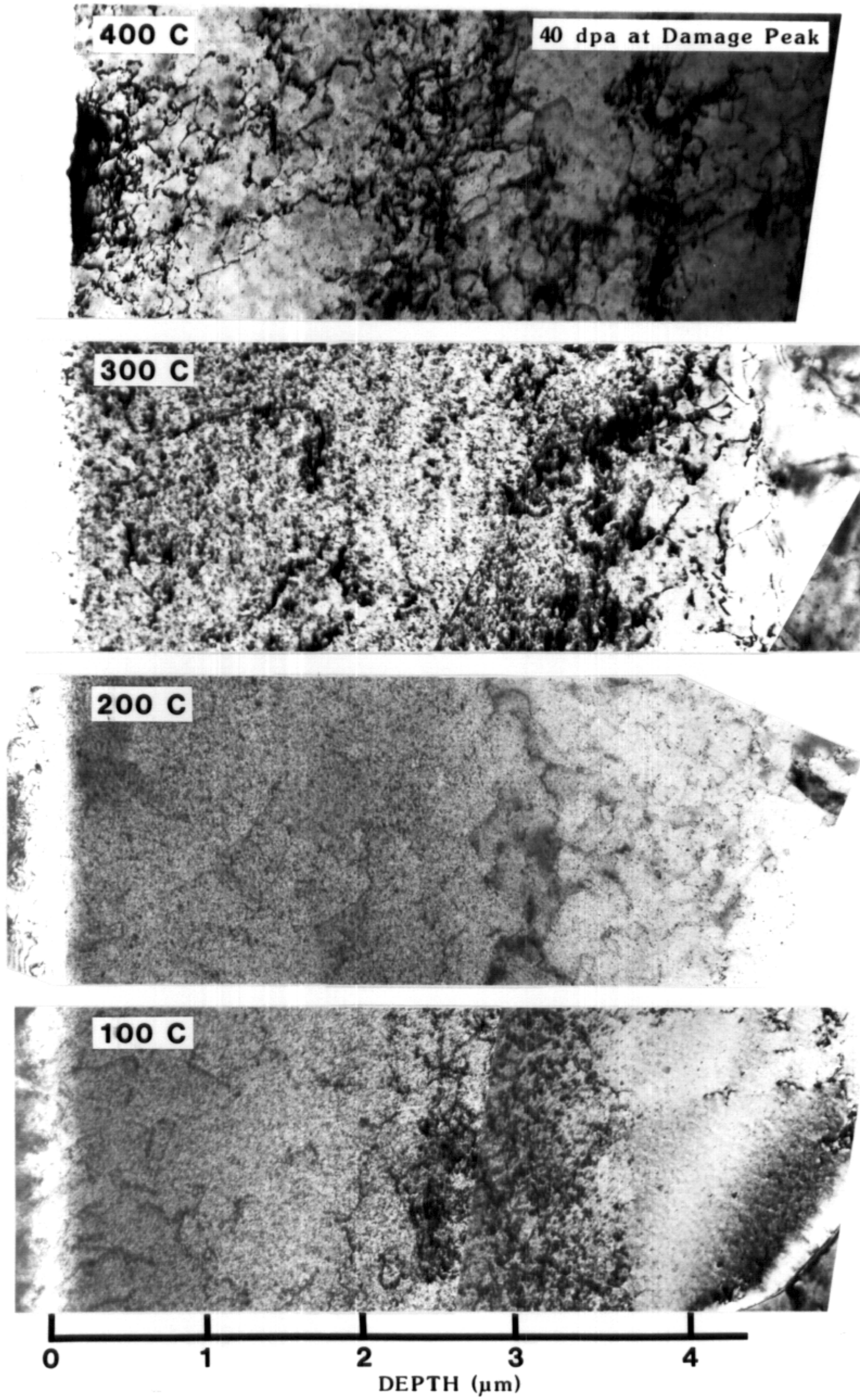

Figure 2. Cross-section microstructure of copper following ion irradiation to a peak damage level of 40 dpa at temperatures of 100-400°C.

ENHANCED CLUSTER SIZE ON DISLOCATIONS IN ION-IRRADIATED COPPER

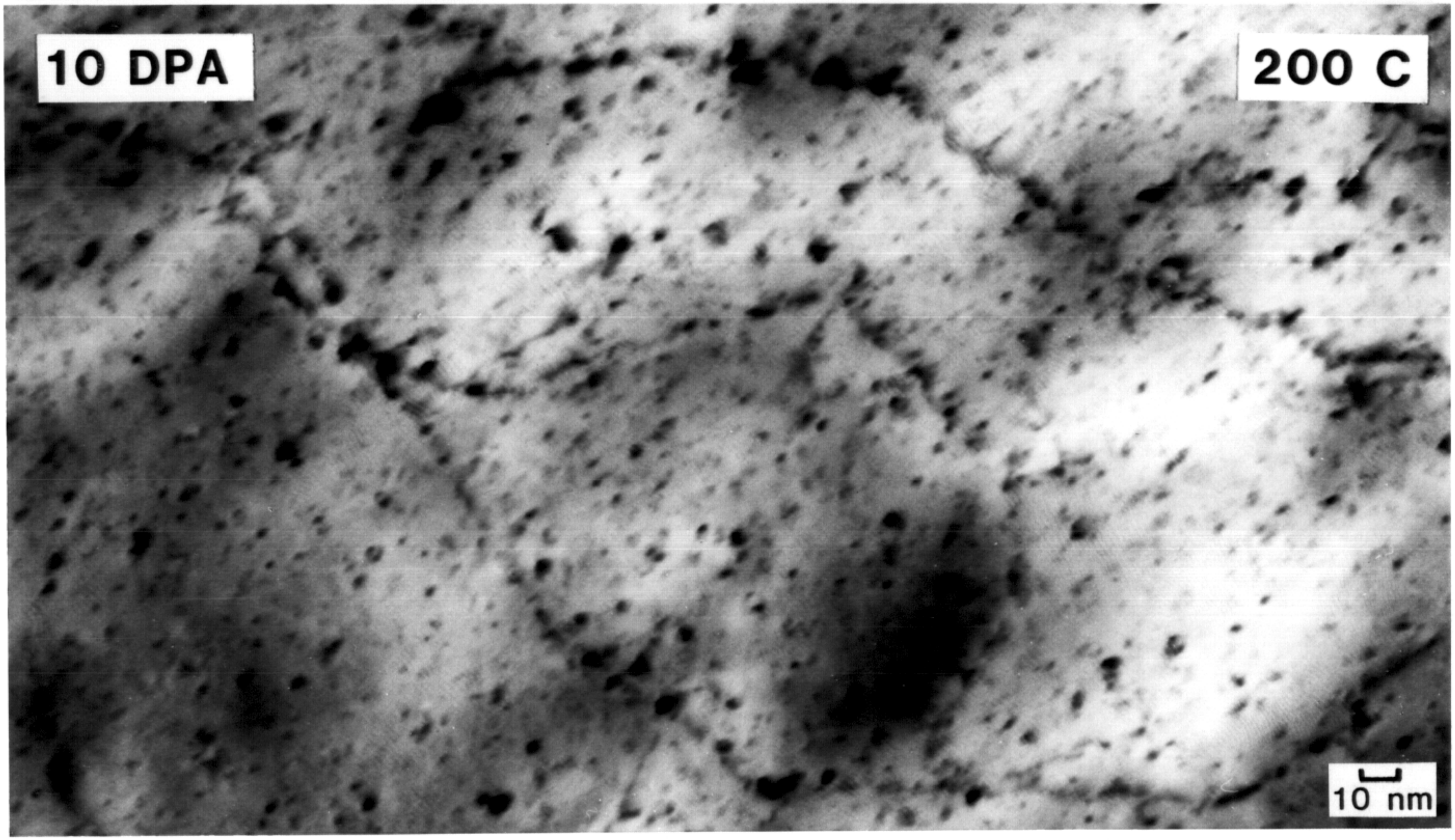

Figure 3. Enlarged defect clusters on dislocations in copper following ion irradiation to 10 dpa at 200°C.

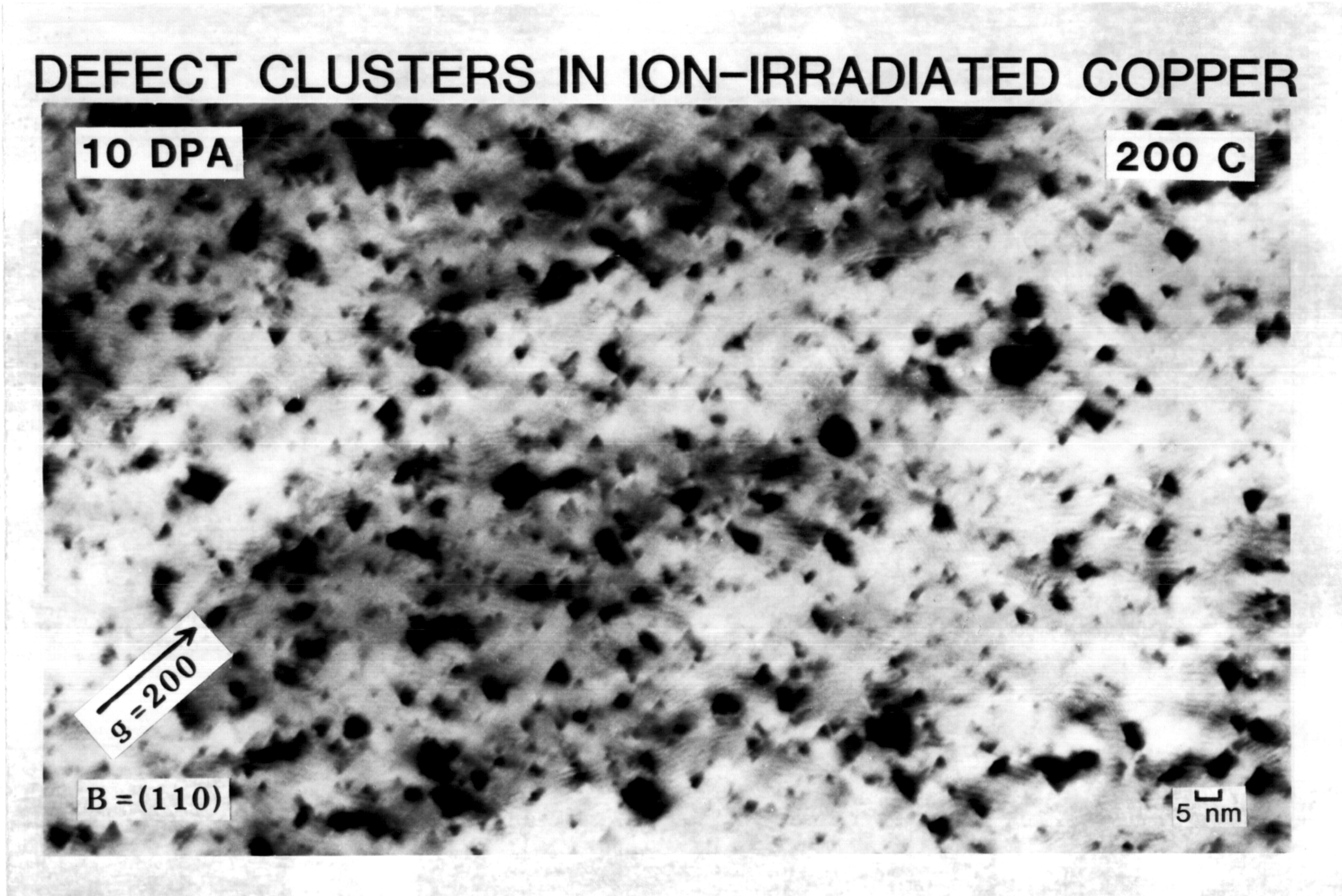

Figure 4. Bright field microstructure of copper irradiated to 10 dpa at 200°C showing triangle-shaped stacking fault tetrahedra.

DAMAGE MICROSTRUCTURE IN COPPER ADJACENT TO A GRAIN BOUNDARY

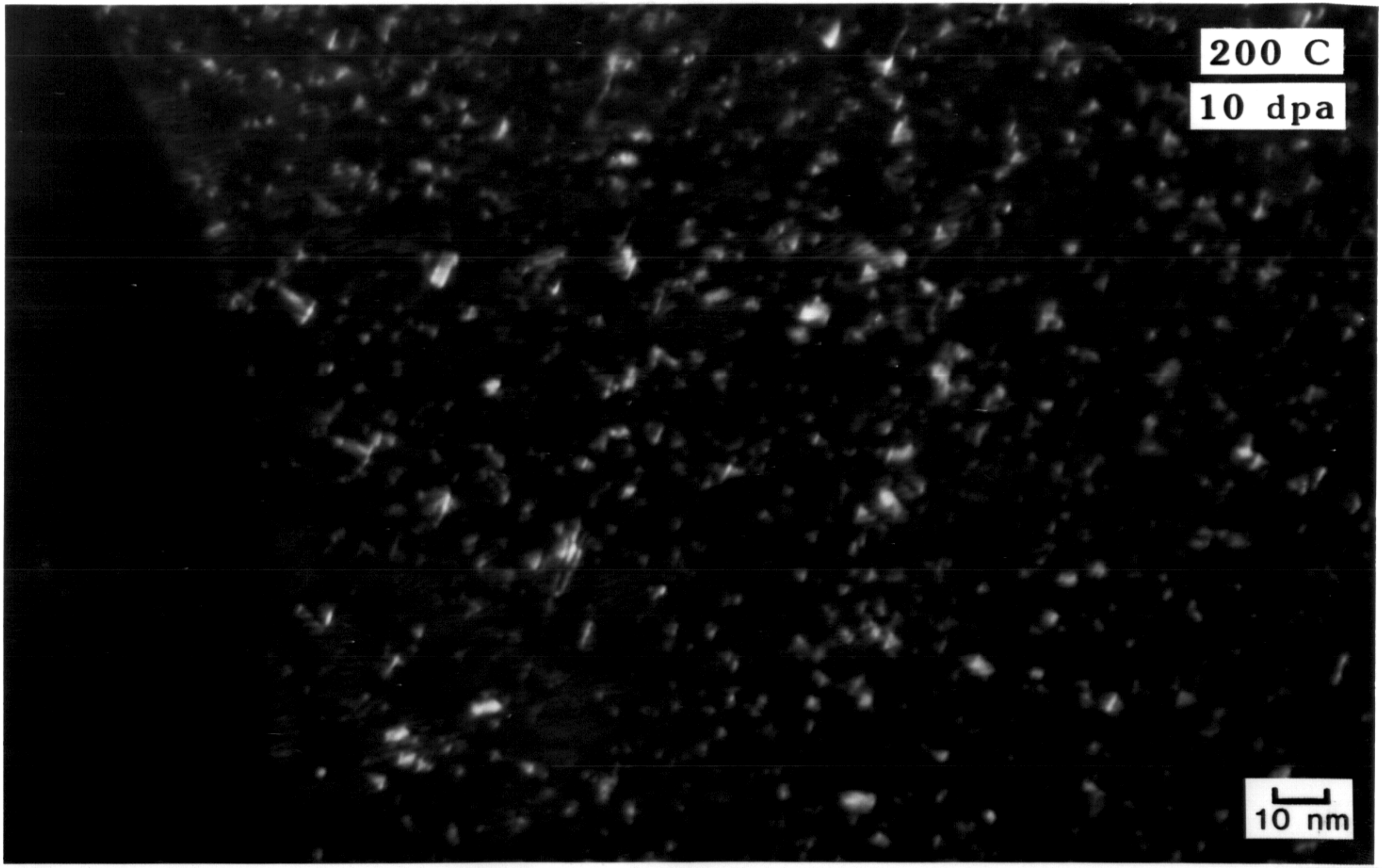

Figure 5. Weak beam (g,3g) microstructure of defect clusters adjacent to a grain boundary in copper irradiated to 10 dpa showing no significant denuded zone.

DEPTH-DEPENDENT MICROSTRUCTURE OF ION-IRRADIATED COPPER

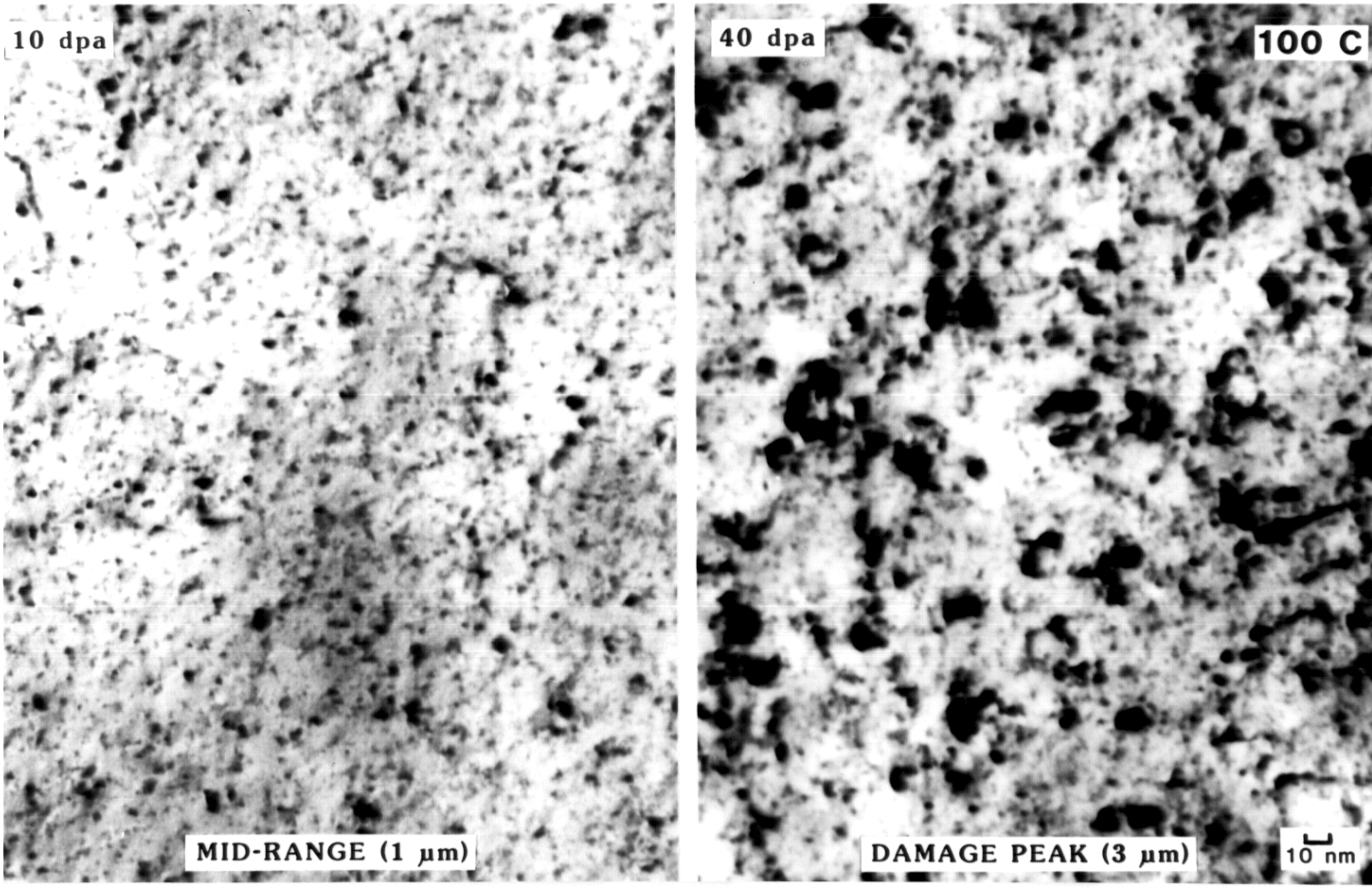

Figure 6. Mid-range and peak damage microstructure of copper irradiated with 14 MeV Cu ions to a fluence of 3×10^{20} ions/m^2 at 100°C.

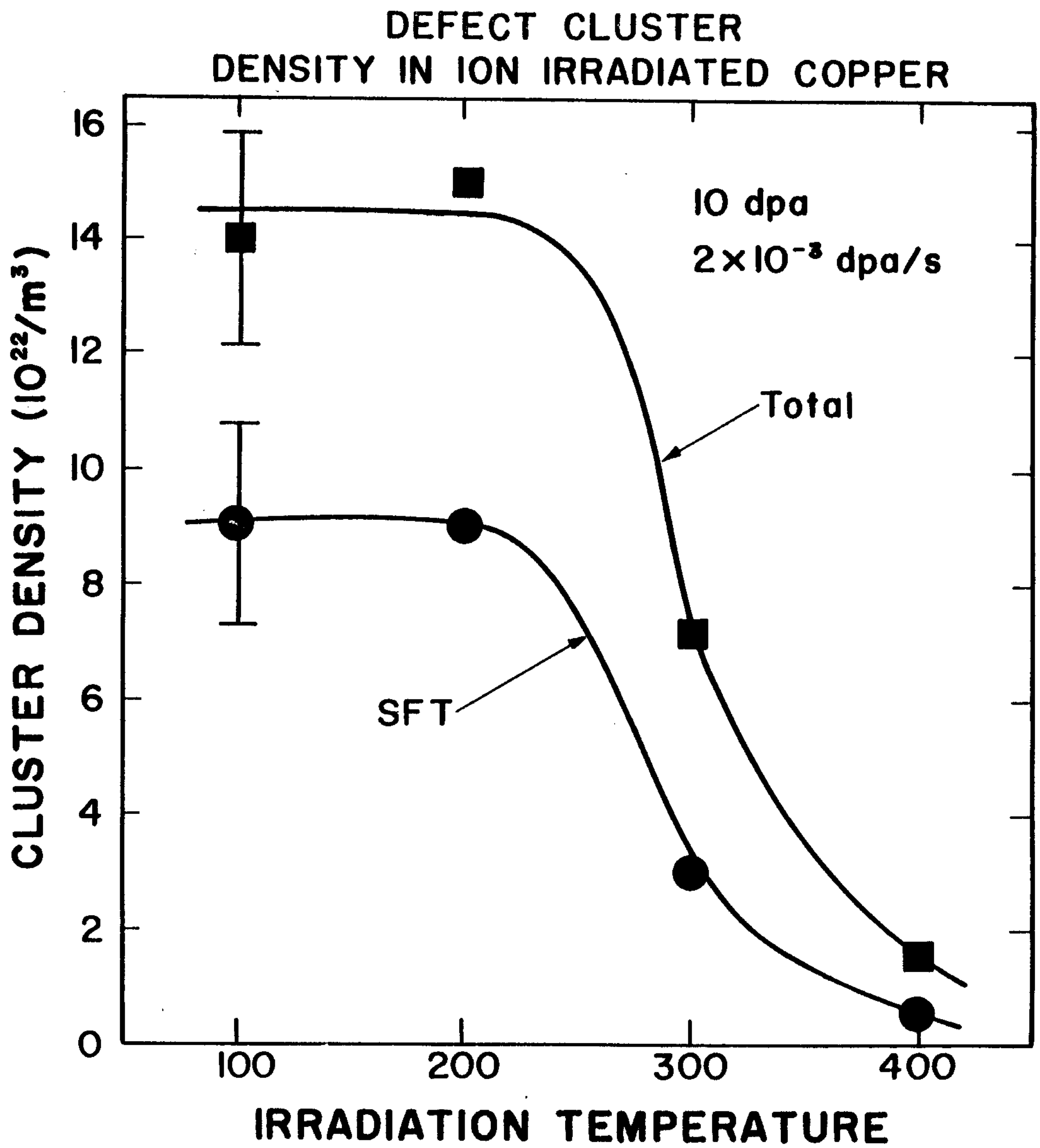

Figure 7. Stacking fault tetrahedra and total defect cluster density in ion-irradiated copper versus irradiation temperature.

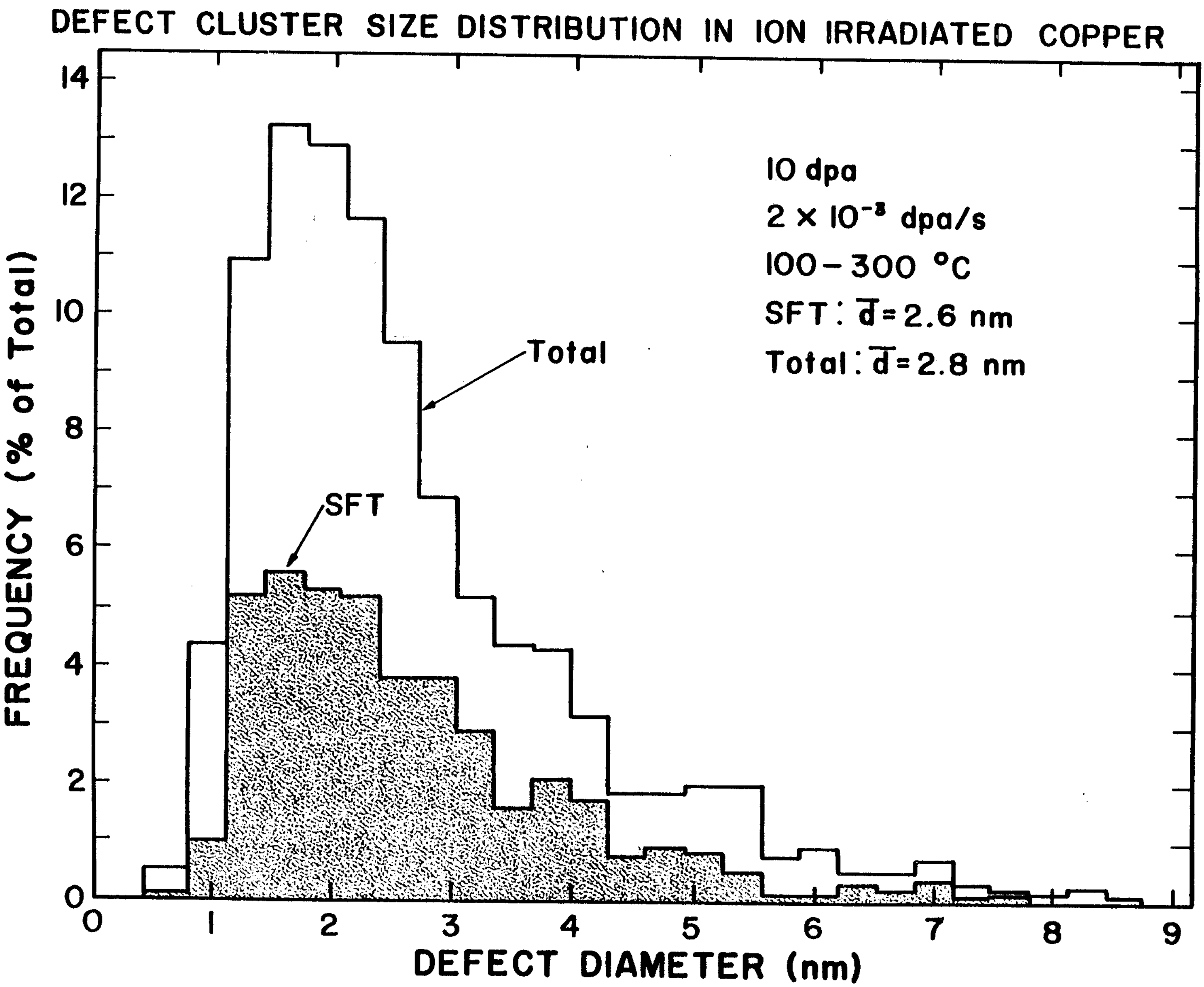

Figure 8. Size distribution of stacking fault tetrahedra and total defect clusters in ion irradiated copper.

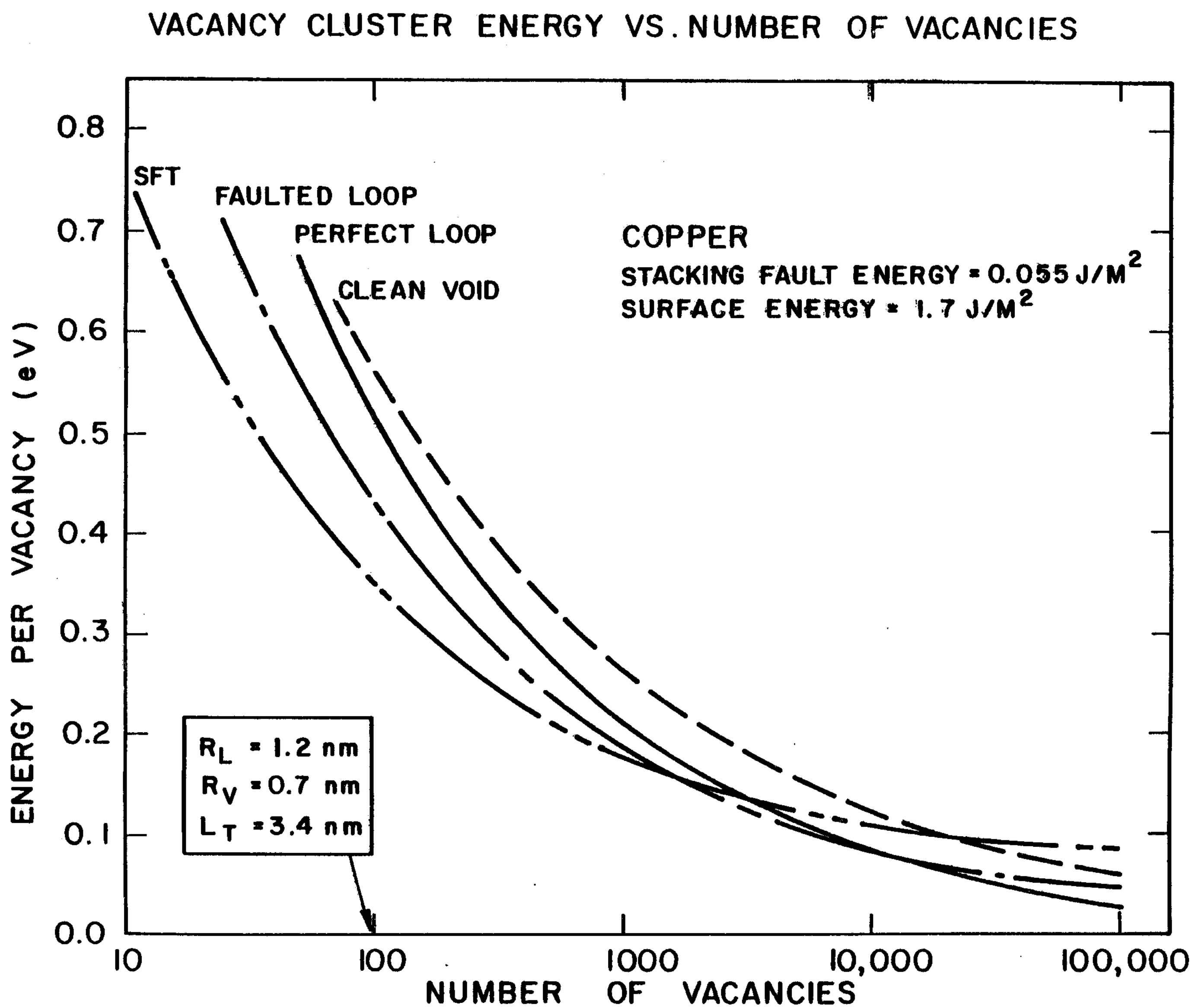

Figure 9. Calculated energy per vacancy of various vacancy cluster morphologies in pure copper [30].

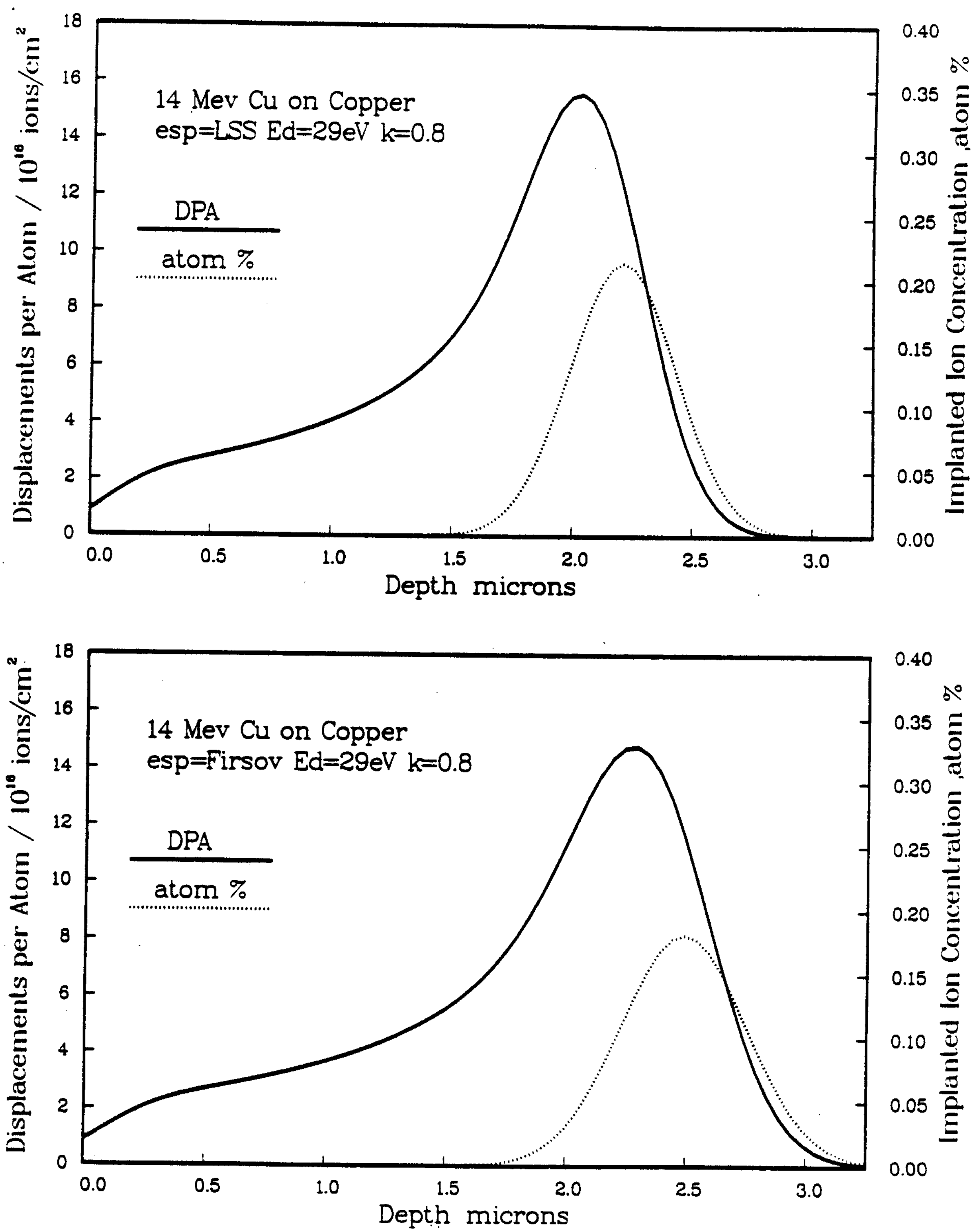

Figure 10. Ion damage profiles using LSS [44] and Firsov [45] electronic stopping power values.