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THE EFFECTS OF TEMPERATURE AND IMPURITIES ON THE ATOMIC DISPLACEMENT ENERGY DURING ELECTRON IRRADIATION

Robert Michael Drosd
(M. S. thesis)

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THE EFFECTS OF TEMPERATURE AND IMPURITIES ON THE ATOMIC DISPLACEMENT ENERGY DURING ELECTRON IRRADIATION

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ABSTRACT

The threshold energy for Frenkel pair production (E_d) is a parameter of importance when theoretical calculations of displacement damage rates are needed to predict long term radiation effects in materials. Hence it is desirable to understand how the threshold energy may be affected by important environmental factors such as temperature and impurities.

Copper and Cu-Al alloys were studied as model pure metal and metal-impurity systems. Irradiations were performed in a high voltage electron microscope from room temperature up to 300°C.

Impurities lighter than the solvent atoms were found to lower the critical value of electron accelerating voltage required to cause atomic displacement. This result arises from the fact that momentum from the incident electrons is more efficiently transferred to the lighter impurity atoms.

Increasing the temperature in the range of room temperature to 300°C caused a decrease in E_d from 18 to 12 eV. Thermally activated

escape from mutual annihilation of closely spaced vacancies and interstitials is thought to be responsible for this effect.[2] The above mentioned decrease in E_d is of importance since it results in a 300% increase in the displacement rate during 650 keV electron irradiation.

I. INTRODUCTION

When two perfectly elastic particles of unequal mass suffer a head-on collision, something less than 100% of the kinetic energy of the first particle will be transferred to the second. The phenomenon is described by the following equation:

$$E_2 = E_1 \cdot \frac{4M_1M_2}{(M_1+M_2)^2}$$

where E_2 is the kinetic energy of the target particle after the collision, E_1 the kinetic energy of the incident particle before the collision, and M_1 and M_2 are the appropriate masses of the incident and target particles respectively. If $M_1=M_2$ then $E_1=E_2$ and thus the transfer of kinetic energy is 100% efficient.

When high energy electrons collide with atoms this effect is especially pronounced due to the large differences in mass of the two particles involved. In numbers this means that electrons accelerated to several hundred thousand electron volts of energy can only transfer on the order of twenty eV to an atom in a head-on collision (see Fig. 1). An important result of this effect is that in a lattice containing two different atomic species each type of atom will be absorbing different amounts of momentum from the same incident electrons.

The true threshold energy, E_d, is by definition[1] the minimum energy needed to create Frenkel pairs by a displacement process that

escape correlated recombination. E_d may be broken into three components, that is the formation energies of a vacancy and interstitial plus the energy required to separate them to a distance where the chances of correlated recombination (mutual annihilation) is small. Strictly speaking the value of E_d by this definition would be very close to the formation of energies of the point defects since there is always some finite probability that thermally assisted escape of the two point defects from one another can occur, even at very small separation. Practically speaking, however, there will be a fairly distinct value of energy needed to create stable Frenkel pairs that will be much larger than the formation energies of the isolated point defects, especially at low temperatures. The apparent distinctness of the threshold energy will depend on the sensitivity of the experimental apparatus used. Hence what is measured experimentally is an effective threshold for observable effects of atomic displacements and can be taken only as an upper limit to the true value of E_d. It is expected therefore, that the measured value of E_d from dislocation pinning experiments will yield a lower value than that obtained by measuring dislocation loop growth rates in the HVEM due to the higher sensitivity of the former technique.[2]

Since the escape of a vacancy from an interstitial can be aided by thermal vibrations[3] it is reasonable to expect that temperature will affect the value of E_d. Roth et al.[2] have proposed that as the

temperature is increased the value of E_d will drop since a smaller separation distance is required to yield a reasonable chance of escape from correlated recombination. This occurs since thermal vibrations, if energetic enough, may overcome the recombining force due to the overlapping of the point defect strain fields.

Corresponding to E_d there is a minimum energy, E_c, that the electrons must possess to be able to transfer E_d to the atoms. In pure copper undergoing electron irradiation at room temperature and below E_d=19 eV[4,5] while E_c=390 keV, for aluminum E_D=16 eV[6,7] and E_c=165keV. E_c is substantially lower for aluminum mainly because aluminum atoms have less than half the mass of copper atoms and are, therefore, closer in mass to the incident electrons. Hence kinetic energy transfer is more efficient between electrons and aluminum atoms than for those of copper (see Fig. 1).

In a pure metal undergoing electron irradiation two regions of electron energy are found. The subthreshold region corresponds to an energy of the incident electrons below E_c where displacement damage is almost absent. In the above-threshold region the electrons are of energy E_c or greater and atomic displacements will occur.

In a binary alloy undergoing electron irradiation the situation becomes more complex. Three regions of incident electron energy exist; they are defined as being above, between, and below the values of E_c for <u>both</u> atomic species. These regions will be called the above

threshold, selective knock-on, and subthreshold regions respectively. In the subthreshold region atomic displacements do not occur in significant numbers. In the selective knock-on region only the lighter of the two atomic species can absorb enough energy from the incident electrons to result in displacement damage (see Fig. 2). In the above threshold region both atomic species will suffer direct displacements. For electron irradiation of a copper aluminum alloy at cyrogenic temperatures the boundaries between the three regions of accelerating voltage are 165 and 390 keV, corresponding to the values of E_c for Al and Cu. As a result of this it can be seen that due to the presence of lighter solute atoms the minimum electron accelerating voltage required for the onset of atomic displacements can be drastically reduced in Cu.

Bauer and Sosin[8] first proposed that the presence of light impurity atoms might be the cause of subthreshold damage that they observed in supposedly pure metals. It was thought that light impurities could absorb enough kinetic energy from the incident electrons to initiate displacements while the matrix atoms could not.

To substantiate their theory Bauer and Sosin purposely alloyed two metals, largely different in mass, and irradiated them in the selective knock-on region. By doing this they hoped for agreement between their experimental results and theoretical estimates of the damage cross section. Agreement was to be taken as confirmation of the

proposed damage mechanism on which their calculated cross section was based. Several possible damage mechanisms were considered. Experimental results most closely matched the model where the small size of the solute atom, rather than its small mass, made subthreshold damage possible. In the copper-beryllium system, their choice of alloy, the solute beryllium atoms are not only lighter but smaller than those of copper. Hence the possibility still existed that the mass effect could have been partly responsible for subthreshold damage along with the atomic size effect.

In this investigation a further look is taken at the contribution light solute atoms may make to subthreshold damage. Specifically the mass related effect on energy transfer efficiency has been studied. To isolate this effect the copper-aluminum system is used because while the solute aluminum atoms are lighter than those of copper they are about ten per cent larger in size. Hence selective knock-on of aluminum atoms cannot be due to their size since their misfit would only tend to increase the energy needed to displace them.

The basic approach however, is similar to that taken by Bauer and Sosin. An experimental as well as theoretical measure of the damage rate is obtained. The theoretical estimate depends on our assumption of the selective knock-on damage mechanism. Agreement of the theoretical and experimental damage cross section confirms the proposed damage mechanism.

The goals of this research are twofold, to study the effects of temperature and also impurities on the threshold energy. The temperature dependence that has been observed is of direct engineering importance since it indicates that there will be an accelerated damage rate at the temperatures at which materials must operate in nuclear reactors. The impurity effects on E_d are only of practical importance to electron irradiation experiments to determine the value of the threshold energy. It is important to note that impurities do not actually change E_d, but rather the experimentally observed value of E_c. E_d is usually obtained from calculations based on this value E_c.

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II. EXPERIMENTAL PROCEDURE

Electron irradiations were performed on three Cu-Al alloys (1,3, and 7 wt% aluminum) as well as pure copper in an HVEM (see Fig. 3, 4). All materials were initially 99.999% pure, the oxygen content of the copper being 4ppm. The Cu, and Cu-Al alloys were grown as single crystals by the Bridgeman technique and then sliced into foils with (110) surfaces. The foils were then prepared by the standard jet polishing technique.

A temperature of approximately 325° was required during irradiation to obtain nucleation and growth of interstitial dislocation loops of a low enough density such that individual loop growth rates were easily measured. The temperature was monitored by a thermocouple which made contact with the tip of the specimen holder inside the microscope. The irradiating flux was measured by a Faraday cup mounted beneath the viewing screen.

To perform an irradiation the electron beam was first defocussed such that the beam intensity was uniform to within 5% over the area of examination. The specimen was then moved under the beam and micrographs were taken at appropriate time intervals to monitor the rate of increase of the size of the resulting dislocation loops.

In an experiment of this type there are many factors which must remain constant to obtain acceptable precision and accuracy. The foil

thickness and temperature are the most important parameters but there are other less obvious conditions whose effects must be considered.

One source of experimental error is the angular dependence of the damage rate upon the incident irradiation. Several studies[1,9,10,11,12] have been made of this effect and it has been found that there are two totally independent mechanisms by which the damage rate may depend on the angle between the incident electrons and the crystal lattice.

Doyle,[9] Thomas,[10] and Makin[11] have studied the effect of electron channeling on the damage rate and found that thin foils as well as low order diffracting planes serve to enhance the channeling effect. Under "worst case" conditions electron channeling can account for a two fold change in the displacement damage rate. To avoid this effect all irradiations were conducted at the same diffracting conditions.

The second type of angular dependence is related to the presence of focussed replacement collisions (FRC). FRC's are a damage mechanism whereby atomic displacements are restricted to the close packed direction of the lattice. Hence only when the electron beam is similarly aligned will FRC's operate with peak efficiency. Jung[12] has shown that angular changes of about five degrees are needed to produce significant changes in the damage rate. To avoid complications from this effect all irradiations were performed at the same angle, approximately five degrees from the (110) pole.

Another possible source of systematic experimental error is thin foil effects. It is well documented[13] that the concentration of point defects, created by electron bombardment, will not be uniform throughout the thickness of a thin foil. The degree to which it is not is a function of temperature, foil thickness, and the damage rate as well as many material parameters. Following the work of Forman[13] and Lam et al.[14] point defect concentrations have been calculated for the conditions expected to be encountered in this experiemnt. In most cases, especially during low energy irradiations which result in low damage rates, the steady state point defect concentrations do not match those of bulk conditions, nor are they uniform throughout the foil thickness. Because of this the dislocation loop growth rate will be a sensitive function of foil thickness, hence great care has been taken to assure that a constant specimen thickness was used from one experiment to the next. Since the concentration of point defects will vary through the thickness of the foil, loops at different depths will grow at different rates. Hence, the growth rates of many loops have been averaged to assure a sampling of all depth positions in order to obtain more consistent results.

Another thin foil effect that should be considered is the finite amount of time that is necessary for the point defect concentration profile to reach steady state. Under conditions such as thick foils, low temperatures, or low damage rates the time to reach steady state may be on the order of an hour. Following the work of Rothman[15] this

transient time has been calculated for what can be considered "worst case" conditions in these experiments and was found to be on the order of one minute. This short transient offer has no serious complications since it takes usually several minutes before dislocation loops can be grown to visible sizes.

To get measurable growth rates during low energy electron irradiations used during studies of selective knock-on displacements, the highest possible beam currents are used. This is not necessary for the higher energy irradiations. This leads to the complication that different amounts of beam heating[15,17] will exist during irradiations at different energies. It is absolutely essential that if dislocation loop growth rates from different irradiations are to be compared that the temperature at which they take place is constant. Hence, to compensate for the differences in beam heating small corrections were made to the power applied to the heating stage in the microscope, thus resulting in an approximately constant temperature for all the irradiations performed.

A final experimental factor of importance is the presence of gaseous impurity atoms which may contribute to selective knock-on displacements in much the same manner as the aluminum atoms. To clearly measure the selective knock-on damage rate due to Al atoms, or to determine the threshold energy in pure copper the level of gaseous impurities must be kept low. To accomplish this the initial materials used contained

only 4 ppm oxygen, the most probable gaseous impurity. After the alloys were formed and grown as single crystals the oxygen content was found the 160 atomic ppm, still an acceptable level. It has been suggested[4] that during jet polishing the foils could pick up large amounts of hydrogen due to the electro-chemical reaction. If this were the case it is hard to understand how the interstitial H atoms would remain in a thin foil (5000Å) for any length of time since their diffusion coefficient is large, especially at elevated temperatures used in this experiment. In any event, to test for the presence of significant amounts of H a high purity Cu specimen was irradiated after being annealed to 700°C in the HVEM vacuum to drive off any gaseous impurities. The resulting damage rate was essentially the same as that in an unannealed specimen. From this it was concluded that there was no significant contribution to the measured value of the displacement damage rate due to gaseous impurities.

III. RESULTS

A. The Displacement Energy Threshold in Copper

The threshold energy was determined at several temperatures by irradiating copper specimens at successively lower electron accelerating voltages to a point where Frank dislocation loops ceased to nucleate. Between approximately 150°C and 375°C dislocation loops grew to large enough sizes such that enclosed stacking fault fringes could be seen (see Fig. 5). Above this temperature range no loops could be nucleated, although one could nucleate loops at a lower temperature and then grow them at a higher temperature. At still higher temperature the tendancy for Frank loops to shrink at elevated temperatures (see Fig. 5) will prevail. Between about 150°C and just above room temperature the dislocation loops grew only to small sizes and appeared as small dots. Below approximately 50°C no loops were found.

The displacement threshold energy was 12 and 15 eV at 300 and 200°C respectively (see Fig. 6). At 300°C the behavior of the dislocation loop growth rate could be observed as the accelerating voltage was decreased (see Fig. 7,8), this was not possible at 200°C since the dislocation loops did not grow to sufficient sizes for growth rate measurements.

The 200°C threshold value must be determined carefully since the dislocation loops at this temperature and below often are seen as very small (<300Å) black dots (see Fig. 5). These images may be easily confused with the small defects resulting from ion damage (ion damage is due to stray ions in the HVEM gun being accelerated, like electrons, down to the specimen). To avoid this possibility the area of irradiation was always observed at a low magnification to ascertain if the suspected electron damage was confined to the area under the electron beam. Ion damage was found to be of uniform density, covering the entire upper surface of the specimen and hence is easily distinguished from defects caused by the focussed electron beam.

B. Subthreshold Damage in Copper-Aluminum Alloys

Perfect prismatic dislocation loops ($b = 1/2 < 110 >$) were found to grow at relatively high rates (approximately 1.0Å/sec at a beam current of $\phi = 1.0$ amp/cm^2) during 300kV electron irradiation (see Fig. 9). In pure copper under the same irradiation conditions the dislocation loop growth rate was found to be more than an order of magnitude slower (see Fig. 10).

In order to arrive at quantitative results as to the effect of the lighter solute atoms on the displacement mechanism, the displacement cross section σ can be easily determined if the displacement rate (K) is known since $\sigma = K/\phi$. Unfortunately it is not possible at present to measure K in the electron microscope. Dislocation loop growth rates

are easily measured in the HVEM but are difficult to accurately relate to K, especially when dislocation jog nucleation is rate controlling as it may be in low stacking fault energy metals such as Cu-Al alloys.[18] This problem can be surmounted by the following mathematical procedure. First consider that the dislocation loop growth rate (G) is equal to some unknown function of the damage rate (K). Therefore,

$$G = f(K) \qquad (1)$$

The damage rate is in turn equal to the electron flux (ϕ) times σ.[1] It is σ that is sensitive to the damage mechanism. It follows then that;

$$G = f(\phi \cdot \sigma) \qquad (2)$$

If the two irradiations are performed, one in the above threshold region (AT) and the other in the selective knock-on region (SKO) of electron accelerating voltage, while the respective electron fluxes are adjusted to give equal values of the dislocation loop growth rates then then the following ratio can be written;

$$1 = \frac{\text{G above threshold}}{\text{G selective knock-on}} = \frac{f(\phi_{AT} \cdot \sigma_{AT})}{f(\phi_{SKO} \cdot \sigma_{SKO})} \qquad (3)$$

It is crucial to point out that the functional dependence of G upon K should be independent of the energy of the irradiating electrons from which the damage arises. In other words the dislocation loops respond uniquely to a given supersaturation of point defects regardless of the manner in which those defects were created. Hence the functions in the numerator and denominator are identical. It follows that if the

two growth rates are equal then the arguments of the functions must also be equal. Therefore;

$$\phi_{AT} \cdot \sigma_{AT} = \phi_{SKO} \cdot \sigma_{SKO} \, , \qquad (4)$$

or by rearranging terms,

$$\frac{\phi_{AT}}{\phi_{SKO}} = \frac{\sigma_{SKO}}{\sigma_{AT}} \qquad \text{IF } G_{SKO} = G_{AT} \qquad (5)$$

The left hand side of equation (5) serves as an experimental measure of the ratio of the displacement cross sections for the alloy. The value of this ratio of experimentally determined electron fluxes has been plotted against the solute concentration in Fig. 11 (see also Fig. 12). A linear relationship is found which closely follows that of the theoretically determined value of the ratio of the displacement cross section, this is discussed later in more detail.

Although the above mentioned functional dependence of G upon K will not depend on the accelerating voltage it will be very sensitive to the foil thickness and the specimen temperature, hence these parameters must be held constant for both irradiations. The foil thickness determines to a large extent what fraction of the point defects reach the dislocation loops since the foil surface is a major sink. The temperature strongly affects the point defect diffusivity and the dislocation loop nucleation density (see Fig. 13), among other factors, which in turn affect the loop growth rate.

IV. DISCUSSION

A. The Temperature Dependence of the Threshold Energy in Pure Copper

Experimental evidence presented here indicates that the threshold energy for displacement damage is temperature dependent. This effect has been previously reported for a lower temperature range by Roth et al.[2] Their work was performed using dislocation pinning measurements. A higher temperature range, lying between approximately room temperature and 300°C, can be studied by using the high voltage electron microscope. The temperature restriction for HVEM studies arises from the fact that dislocation loops will only be found in this narrow range of temperature for copper. Employing this technique the value of E_d was determined by irradiating at successively lower values of accelerating voltage until dislocation loops ceased to nucleate. At 275 kV and 300°C Frank dislocation loops could still, with some difficulty, be nucleated and grown to visible sizes (at a beam current of 1 amp/cm^2 the growth rate was .2Å/sec) see also Fig. 14. The nucleation step is more difficult than subsequent growth so it is quite reasonable to expect that dislocation loops, initially nucleated at a higher voltage, would be found to grow at even lower accelerating energies, thus yielding a still lower value of E_d. Hence all values of E_d reported here should be taken as upper limits. The measured threshold energy will vary considerably depending on the sensitivity of the experimental equipment. This is true because the whole concept of a sharp value of E_d is only an approximation. In reality there is only a probability

function for atomic displacements that starts near zero when the electron energy is equal to the vacancy and interstitial formation energy and approaches unity at high electron energies. Hence, as experimental sensitivity is increased the incident electron energy required for measurable levels of atomic displacements will decrease until the limit imposed by the formation energies of the constituent point defects is reached. Because of this the measured value of the threshold energy is not a precise fundamental parameter. This fact may explain some of the difficulty encountered[4] during attempts to theoretically correlate it with other material properties such as the sublimation energy or the lattice parameter.

Since 275 kV is considerably below the commonly accepted value of about 390kV for E_c it will be worthwhile to address the possibility that this large discrepancy is due to some anomalous subthreshold mechanism rather than a result of temperature.

"Soft spots", such as grain boundaries, dislocations, and stacking faults have been suggested by Bauer and Sosin[8] as regions where the threshold energy is lower than in a defect free lattice. The presence of these defects could account for some amount of subthreshold damage but specimens used in this work were single crystals with no dislocations or other large faults initially in the area of irradiation.

Impurity atoms of lower mass will cause subthreshold damage by the selective knock-on mechanism discussed earlier for Al atoms.

Several precautons were taken to maintain high purity as is discussed in the experimental section. As a check for significant quantities of light impurities one can examine the dislocation growth rate versus electron accelerating voltage plot. If the observed displacement rate in the range of 275 to 325 kV resulted from selective knock-on of light impurity atoms such as oxygen, carbon, or hydrogen then one would expect only a minor change in the dislocation loop growth rate over this 50 kV change in accelerating voltage. This follows from the behavior of the selective knock-on damage cross section for these light impurities in copper which is essentially constant over this energy range (see Fig. 7). From this figure it is seen however, that the dislocation loop growth rate is rising sharply. Hence light impurities cannot be responsible for these low energy atomic displacements.

Neither of the above mentioned subthreshold damage mechanisms seems capable of explaining the drastic reduction in E_d. The high temperature at which the experiment was conducted seems the most likely explanation and is in agreement with results of other studies.[2]

Roth et al.[2] have proposed an explanation for the temperature dependence of E_d based on the idea of thermally activated escape of point defects from correlated recombination. If Frenkel pairs that are separated to a sufficient distance, 2 or 3 atomic spacings, where spontaneous recombination (recombination of a vacancy and interstitial that takes place in the absence of thermal activation

due to mechanical instability) does not occur[1] but correlated recombination, via diffusion, of the point defects is still dominant, then an increase in thermal energy can aid in increasing the fraction of Frenkel pairs that manage to escape correlated recombination. The process of spontaneous recombination cannot be affected by thermal vibrations since it occurs in a time interval shorter than that of thermal oscillations. According to the above discussion one would expect E_d to decrease with increasing temperature and then reach a limiting value imposed by spontaneous recombination.

An alternative explanation to the temperature dependence of E_d may lie in the fact the lattice parameter and elastic constants change by about 0.5 and 20 per cent respectively in copper for an increase from room temperature to 300°C. From a detailed analysis of the focussed replacement collision damage mechanism[1] it can be seen that these temperature induced changes in material properties should result in a smoothly decreasing value of E_d as the temperature is increased. Thermal vibrations will have a defocussing effect on FRC's and hence will tend to increase the value of E_d.

Sosin[19] has pointed out that thermal vibrations may increase the efficiency of momentum transfer between the incident electrons and atoms if the thermal velocity of the atom is aligned with the displacement direction at the time of collision. However, E_d will only decrease by about 2 eV at most due to this effect.

The observed temperature dependence of E_d could be attributed to any of the above mentioned explanations. Combinations of these effects are possible as well. Conclusions concerning the relative importance of each one are difficult to make at this time.

B. Selective Knock-on Displacements in Copper-Aluminum Alloys

1. The Damage Mechanism

The results of low energy electron irradiations performed on the Cu-Al alloys of this study indicate that light solute atoms can cause selective knock-on displacements. Before discussing the possible mechanisms by which the aluminum atoms contribute to displacement damage in the alloy it will be helpful to first look at the case of a pure metal.

If E_m, the kinetic energy transferred to an atom from a head-on collision with an electron, is not too much greater than E_d the resulting displacements will take place, for the most part, via focused replacement collisions (FRC).[1,24,21,22] An FRC occurs when an atom, struck by an electron, is displaced not directly into an interstitial position, but rather replaces one of its neighboring atoms. The replaced adjacent atom then repeats this sequence until enough energy has been lost so that further replacements are not possible. The last atom to be replaced then falls into the lowest energy interstitial position. This sequence of replaced atoms is found to occur most easily along the close packed directions of the crystal lattice.

FRC's are the predominant damage mechanism at low values of E_m because the replacement process is the most efficient method of separating the vacancy and interstitial to a distance where the chance of correlated recombination is small. Hence at low values of E_m FRC's will result in freely migrating point defects. At higher values of E_m FRC's are not as likely and a more random displacement mechanism is found that is not restricted to occur along any particular crystallographic direction. FRC's cease to function at high values of E_m because as the kinetic energy of the colliding atoms increases, their distance of closest approach decreases. Hence in the hard sphere approximation the effective radius of the atoms decreases and they become effectively less close packed (see Fig. 5). The atoms involved in the FRC are then more easily "defocussed" since the atoms can be displaced away from the close packed direction through the relatively large spaces now separating them.

One of the most important differences between the high energy damage mechanism and that of FRC's is the identity of the resultant interstitial. The interstitial formed by an FRC is not the atom initially struck by the electron, as may be the case by the high energy displacement process, but rather it is the last atom in the chain to be replaced. As will be seen shortly this may have important effects in an alloy where all the atoms are not identical.

In a substitutional binary alloy if the two atomic species are not too dissimilar in size or mass FRC's will probably be the damage mechanism at low electron energies. If Cu-Al alloy is irradiated in the selective knock-on region where essentially only the aluminum atoms can absorb enough energy to suffer displacements by electrons, an interesting situation results (see Fig. 2). If FRC's are the displacement mechanism then only FRC's where the first atom in the chain is Al will be possible. The Al atom, once struck by the electron, can then transfer more energy to a neighboring copper atom than would have been possible from direct electron copper atom collision. Even though the aluminum atom was initially struck by the electron it does not become the resultant interstitial. Rather, the last atom in the FRC chain to be displaced will form the final point defect. This last atom may be either copper or aluminum, the probability of each is simply their respective mole fractions, if there is no bias as to which type of atom that the FRC terminates on. If this bias exists (see Fig. 15) then the composition of the interstitials initially produced will differ from the alloy composition.

In an alloy there are several factors, however, by which the presence of two types of atoms might inhibit the occurrence of FRC's. In this particular case of a Cu-Al alloy there is a ten per cent size difference between the two atoms. This variation in atomic size will, to some extent, discourage the momentum from focussing by disrupting the geometric regularity of the lattice (see Fig. 15). In addition

the FRC's will not be as effective in an alloy because the kinetic energy transfer along the FRC chain will take place between atoms of unequal mass and hence will not be 100% efficient as it is in a pure metal (neglecting "frictional" losses). Since M_{cu}=64 and M_{Al}=26 only 84% of the kinetic energy can be transferred in a collision of two such atoms. Another consideration is whether the impurity atom lies on an interstitial or substitutional site. If it is substitutional, as is the case for a Cu-Al alloy, then the foreign atom is in the correct position to take part in an FRC. If it is interstitial, as are many light impurity atoms, it will have a disrupting effect in the focussing process.

If FRC's are not possible in these Cu-Al alloys, or their effectiveness is severely reduced, then radiation damage will be more difficult to produce than is pure copper. Any alternative displacement mechanism will be less efficient and hence a higher value of E_d, accompanied by a lower damage rate, will result. One can only speculate whether FRC's are operative in these alloys since even though radiation damage was readily obtained, it is not possible in our experiments to compare the damage rate in the alloy with that in pure copper. The growth rates of dislocation loops in Cu-Al and Cu cannot simply be compared since the diffusion coefficients of the self interstitials may be different (accurate self interstitial diffusion data is not available). Hence even if the efficiency of FRC's in copper and the alloy are the same the resultant dislocation loops

will grow at different rates according to different diffusion coefficients. In addition to this, Frank dislocation loops, ($b=\frac{1}{3} < 111 >$) are found in pure copper while perfect dislocation loops ($b=\frac{1}{2} < 110 >$) result in the alloys studied here. It is likely that these two types of dislocation loops will not respond identically to the same point defect supersaturation even if it existed in the two different materials.

To briefly summarize, although FRC's are accepted as the low energy damage mechanism in a pure metal there are several reasons to believe that in a substitutional alloy their effectiveness may be reduced substantially. If this is true an alternative mechanism may be operating, possibly consisting of many short FRC's sequentially traveling in different directions. Experimental work performed by Becker et al.[3] indicates that in some alloys FRC's are operative but for Cu-Al alloys no conclusive proof exists to clarify the situation.

During irradiation in the selective knock-on region radiation damage is readily observed in the alloys while being almost in pure copper at the same accelerating voltage. This observation supports the theory that the light solute atoms are responsible for displacements in the selective knock-on region. It is most likely that electron-aluminum atom collisions are responsible for these displacements although the details of the atomic motion which follow are not clear.

2. Interstitial Migration and Identity

Upon creation of a Frenkel pair the point defects, given favorable conditions diffuse through the lattice form dislocation loops. In a binary alloy the possibility exists that one atomic species will be favored in the interstitial configuration (see Fig. 16). This could have profound effects on nucleation and growth of the radiation induced dislocation loops since it would mean that they would be receiving predominantly one or the other type of atom.

Prior to discussion of the interstitial behavior in an alloy it will be helpful to first look at the case of a pure metal, where there is a long-standing controversy[23] over what is the configuration of the self interstitial. Two theories have been proposed, they are the "one interstitial"[20,24,25,26,27] and "two interstitial"[28,29,30] models.

The simpler, one interstitial, model is more commonly accepted and will be the one used in this work. Here it is proposed that at all temperatures the interstitial takes the form of a < 100 > dumbell (split interstitial) where two atoms share one lattice site, a line connecting the two would be in the < 100 > direction. This type of interstitial migrates by passing one of the two atoms towards a nearest neighbor atom, thus recreating the < 100 > dumbell at a position one atomic spacing away. The other atom of the original split interstitial is left behind, now in the normal unshared lattice position. It is interesting to note that once an atom which is

ejected out of its lattice site by the displacement mechanism and finds itself in a split interstitial configuration it is then indistinguishable from the atom it is sharing the interstitial site with. Either one may then be passed on to the next interstitial position. Hence the chances are great that after just a few jumps the atom originally displaced will be left behind in a substitutional site. This has particular significance in alloys as is discussed below.

In an alloy the question arises as to whether there is some bias for which atomic species will be found in the < 100 > dumbell configuration. Any such bias will strongly affect the ratio of solvent to solute atoms that a dislocation loop will receive. Intuitively one might expect that if the solute is oversized as compared to the solvent atoms then the mixed dumbell (a dumbell interstitial containing a solvent and solute atom) would have a higher energy than one containing two solvent atoms. Early work[31,32,33] done on this consideration employed the principles of continuum elasticity to calculate this energy difference. It was found for a ten per cent oversized atom contained in a split interstitial that the formation energy would be 5 eV higher than an unmixed < 100 > dumbell. Although continuum elasticity calculations are not applicable on the atomic level they do serve to indicate that significant energy increases may result from incorporation of an oversized solute atom into the interstitial configuration. The

electronic redistribution energy may be equally important to elastic energy but is much more difficult to calculate.[31] Hence there is good reason to believe, from theoretical considerations, that a mixed dumbell interstitial may have significant energy differences when compared to a split interstitial composed of two solvent atoms.[34] The possibility of two solute atoms is not considered here since only dilute alloys are of interest.

Experimental attempts to determine the possibility of mixed dumbells have been made in several alloys. The two major techniques used for these experiments are ion backscattering [35,40] and ordering studies.[41,42] Mixed dumbells have been confirmed in Au-Ag,[43,44] Cu-Ni[45] and Cu-Zn.[46] However in Cu-Al conflicting reports have been given,[35,41,42,47,48] hence a confident conclusion is difficult to draw at the present time. If Al atoms are excluded from migrating interstitially then the dislocation loops will be receiving only Cu atoms.

This discussion of interstitial identity bears an interesting relationship to the earlier discussion of the damage mechanism. No definite conclusion could be drawn about the exact nature of the displacement process, which is what determines the identity of the resultant interstitial. As far as the dislocation loops are concerned, however, it makes no difference which atomic species is placed in the interstitial position by the electron-atom collision. The

interstitial diffusion mechanism will select, during migration, which of the atomic species will reach the dislocation loops. Hence the dislocation loops will not reflect the original identity of the interstitial atom, therefore no conclusions could be drawn about the damage mechanism from studying the atomic species that will comprise the extra half plane of the dislocation loop.

3. The Displacement Cross Section for an Alloy

Since during 300 KV electron irradiation of Cu-Al alloys the dislocation loop growth rate was found to decrease rapidly with Al concentration (see Fig. 14), as well as being almost absent in pure copper, it seems reasonable to attribute these displacements to electron-aluminum atom collisions. If this is the case then to construct the displacement cross section for the alloy one may take an average of the cross sections of the pure components (see Fig. 17), weighted by their respective mole fractions.[8] Hence;

$$\sigma_{cu \cdot Al} = X_{Al}\sigma_{Al} + X_{cu}\sigma_{cu} \qquad (6)$$

Where X_{cu}, X_{Al} are the mole fractions and σ_{cu}, σ_{Al} are the damage cross sections for pure copper and aluminum (see Fig. 18). The cross sections for the pure components may be used for the alloy in the above fashion since they basically describe the displacement probability for electrons of a given energy impinging upon an atom of a given mass, where an energy E_d is required for an atomic displacement.[49] Only the value of E_d reflects the environment of

of the atom. Therefore if the correct value of E_d is chosen for the alloy then the displacement cross sections for the pure components can be used in equation (6).

As mentioned in the results section, ratios of the displacement cross sections, one at an above threshold and the other at a selective knock-on voltage, are what is actually measured experimentally. Hence from equation (6) above the following expression can be written:

$$\frac{(\sigma_{cu-Al})_{AT}}{(\sigma_{cu-al})_{SKO}} = \frac{[X_{Al}\sigma_{Al} + X_{cu}\sigma_{cu}]_{AT}}{[X_{Al}\sigma_{Al} + X_{cu}\sigma_{cu}]_{SKO}} \tag{7}$$

where AT and SKO refer to the accelerating voltages mentioned above. Simplifications can be made to equation (7) since $(\sigma_{cu})_{SKO} = 0$ and also for the range of accelerating voltages and Al concentrations used in this experiment $(X_{Al}\sigma_{Al} + X_{cu}\sigma_{cu})_{AT} \approx (\sigma_{cu})_{AT}$, therefore:

$$\frac{(\sigma_{cu-Al})_{AT}}{(\sigma_{cu-Al})_{SKO}} = X_{Al}\frac{(\sigma_{cu})_{AT}}{(\sigma_{Al})_{SKO}} \tag{8}$$

As previously mentioned the value of E_d is required for the calculation of σ. Unfortuantely the displacement energy is not well known for Cu-Al alloys. This difficulty can be avoided by noting the fact the value of the ratio of the cross sections is relatively insensitive to E_d provided the proper values of the

AT and SKO voltage are chosen (see Fig. 19,20,21). It is for this reason that 300 and 575 kV were chosen for the SKO and AT voltages respectively. Conversely, if one is confident of experimental accuracy, one could exploit this behavior to determine the value of E_d by choosing values of AT and SKO such that the ratio of the cross sections is sensitive to the displacement energy. Then from figures 19,20 and 21 the value of E_d could be determined if the experimental value of the ratio is found. The values of σ for Cu and Al were taken from the computer calculations done by Oen.[50]

The linear relationship between the ratio of the cross sections and the solute concentration, as predicted in equation (8), was verified experimentally as seen in Fig. 11. Hence it is confirmed that the displacement damage cross section, for the alloys studied here, is a weighted average of the cross sections of the pure components.

V. CONCLUSIONS

A. The Temperature Dependence of the Threshold Energy in Copper

The significant reduction in the measured value of E_d can be confidently attributed to a temperature dependent displacement energy threshold rather than to the several subthreshold mechanisms mentioned. The origin of this temperature dependence is not as certain however. Thermally activated escape from correlated recombination seems to be the most likely explanation, but the effects of temperature induced changes of the lattice spacing and elastic constants on the process of focused replacement collisions may also be important.

Since the damage rate is a sensitive function of E_d, the temperature dependence of the displacement energy should be considered when calculations of K are made.

B. Subthreshold Displacement Damage in Cu-Al Alloys

Al atoms are responsible for the significant damage rates observed during 300 kV irradiation of the Cu-Al alloys. Since experimental agreement was found with the theoretical value of the ratio of the damage cross section for these alloys it is verified as well that the displacement cross section for an alloy can be written as the mole fraction weighted average of the cross sections of the components. In addition it seems the only reasonable displacement mechanism for this low energy radiation damage is that, due to their low mass, the Al atoms absorb more energy from

the incident electrons and may initiate displacements while the copper atoms cannot, hence this has been termed "selective knock-on" displacement damage. These types of displacements, involving hydrogen impurities, have long been suggested[4,8] as being responsible for low energy damage. However for very light impurities such as H the inefficiency of momentum transfer between the impurity and solvent atom becomes a dominant factor. In an Al to Cu atom collision 84% of the energy may be transferred, but only 6% is possible for the collision of a H and Cu atom. Hence a hydrogen atom must possess about 300 eV to be able to transfer the displacement energy of 20 eV to a copper atom. Therefore the <u>effective</u> value of E_d for the selective knock-on damage mechanism involving hydrogen in copper is 300 eV which yields a negligibly small value for σ hydrogen and insignificant amounts of atomic displacements are expected.

0 0 0 0 4 7 0 6 7 5 8

ACKNOWLEDGEMENTS

I would like to express my gratitude to my friends who have helped me through the ordeal of preparing this work.

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FIGURE CAPTIONS

Fig. 1. a. Graphical relationship between the electron accelerating voltage, atomic mass, and maximum energy in eV(E_m) imparted to a target atom (taken from ref. 11).

b. Graphical relationship between E_m and the electron accelerating voltage specifically for Cu and Al.

Fig. 2. Schematic representation of selective knock-on and above threshold displacement damage in a substitutional alloy via focused replacement collisions. Note that during SKO displacements only the lighter solute atoms can initiate displacements, although the resultant interstitial may contain both types of atoms.

Fig. 3. a,b,c,d Electron micrograph growth sequence of Frank dislocation loops in pure Cu at 650 kV, 600°K. The electron flux was .066 amps/cm^2 which resulted in a growth rate of 1.2Å/sec; < 110 > orientation.

Fig. 4. a,b,c,d Electron micrograph growth sequence of perfect prismatic dislocation loops in Cu+15 at %Al at 650 kV, 600°K. The electron flux was .016 amps/cm^2 which resulted in a growth rate of .55Å/sec, < 110 > orientation.

Fig. 5. a. Frank dislocation loops in pure Cu exhibiting rounded corners due to shrinkage after irradiation has ceased at 300°C.

b. Typical Frank dislocation loop density at 300°C in Cu.

c. Typical Frank dislocation loop density at 200°C in Cu.

Fig. 6. Displacement energy threshold (E_d) versus temperature in pure copper.

Fig. 7. Dislocation loop growth rate versus electron accelerating voltage in pure Cu at 300°C as compared to displacement cross sections for pure copper and copper with hydrogen as an impurity. Note that G and σ_{cu} increase at nearly the same rate while σ_H is almost constant. This is a good indication that atomic displacements at these low electron energies are not due to light impurities.

Fig. 8. Values of the displacement cross sections for Cu, Al, and Cu-Al alloys for several values of E_d and electron accelerating voltage (taken from ref. 50 and using equation 6).

Fig. 9. a,b,c Growth sequence of perfect dislocation loops in Cu+15 at % Al at 300kV, 600°K. The electron flux was 53 amps/cm^2 which resulted in a growth rate of 2.2Å/sec, < 110 > orientation.

Fig.10. a,b,c,d Growth sequence of Frank dislocation loops in pure Cu at 275 kV. 600°K. The electron flux was 1.1 amps/cm^2 which resulted in a growth rate of 0.2Å/sec. Note that for the same irradiation conditions in the Cu-Al alloy of Fig. 9 the resultant dislocation loop growth rate is more than an order of magnitude higher.

Fig. 11. Theoretical and experimental values of the ratio of the 300 to 575 kV displacement cross sections versus the Al concentration.

Fig. 12. Tabulated values of the electron fluxes required at 300 and 575 kV to obtain identical dislocation loop growth rates. Note the good agreement between the values of ϕ_{575}/ϕ_{300} and $\sigma_{300}/\sigma_{575}$ for the three alloys. This is predicted by equation 5.

Fig. 13. a. Typical perfect dislocation loop density at 325°C in Cu+15 at% Al.

b. Typical perfect dislocation loop density at 275°C in Cu+15 at% Al.

c. Typical perfect dislocation loop density at 220°C in Cu+15 at% Al.

Fig. 14. Tabulated values of the dislocation loop growth rates found in Cu and Cu-Al alloys at several accelerating voltages.

Fig. 15. Schematic representation of focused replacement collisions and solute atom interactions.

Fig. 16. Schematic representation of the interstitialcy diffusion mechanism in an FCC substitutional alloy. Note how the solute atom may become incorporated into or expelled from the interstitial position, case 2a and 1a respectively, depending on which is energetically most favorable.

Fig. 17. a. Displacement damage cross sections for aluminum (taken from ref. 50).

b. Displacement damage cross sections for copper (taken from ref. 50).

Fig. 18. a. Displacement damage cross sections for Cu+2 at% Al.

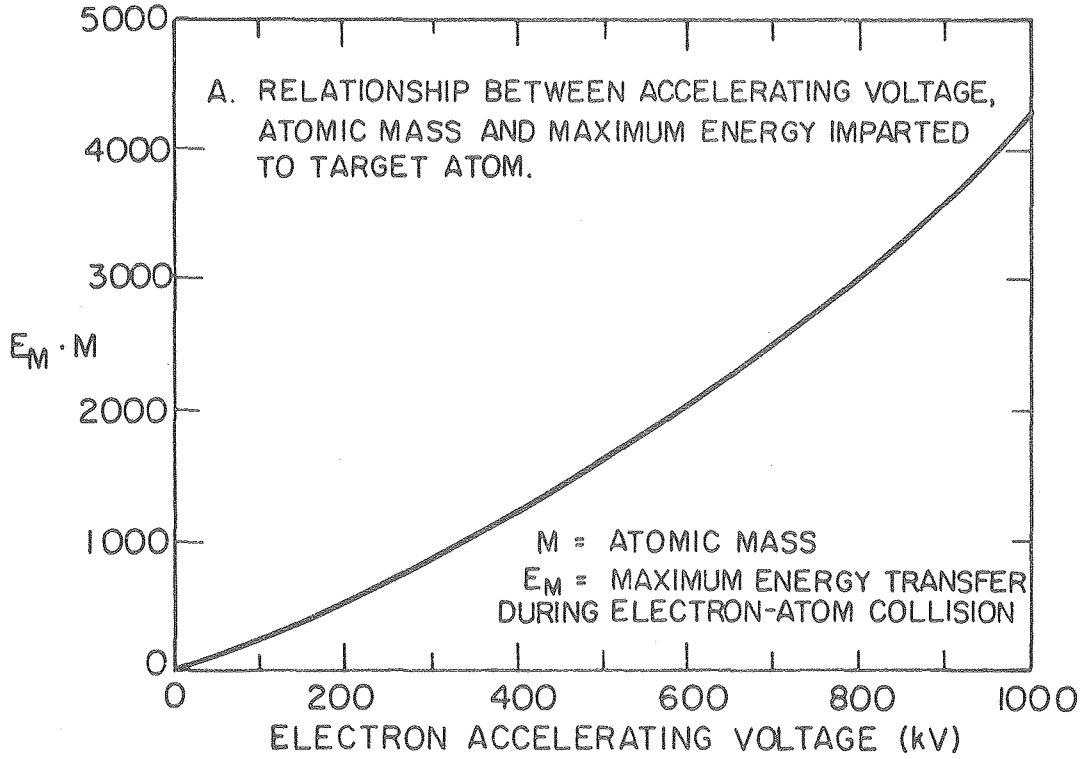
b. Displacement damage cross sections for Cu+6.8 at% Al.

c. Displacement damage cross section for Cu+15 at% Al.

Fig. 19. The ratio of the selective knock-on to above threshold displacement damage cross section versus the displacement energy for several values of the AT voltage (for Cu+2 at% Al). Note that for the AT voltage of 575 kV the value of the ratio is approximately constant.

Fig. 20. Same as Fig. 19. but for Cu+6.8 at% Al.

Fig. 21. Same as Fig. 19 but for Cu+15 at% Al.



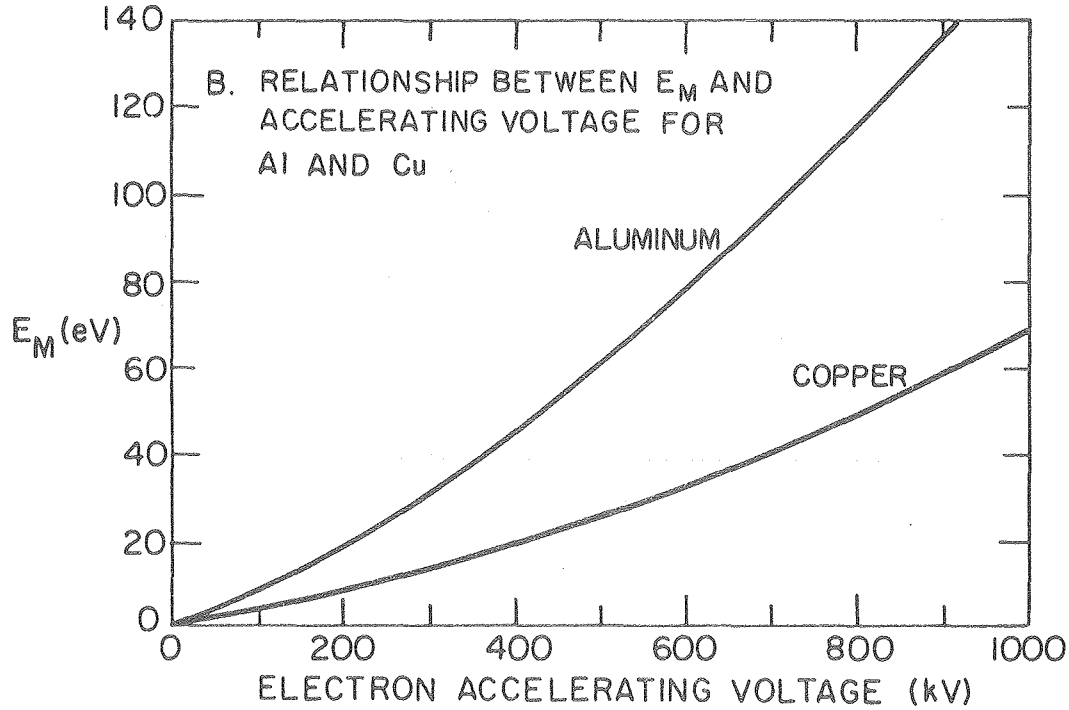


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Fig. 1

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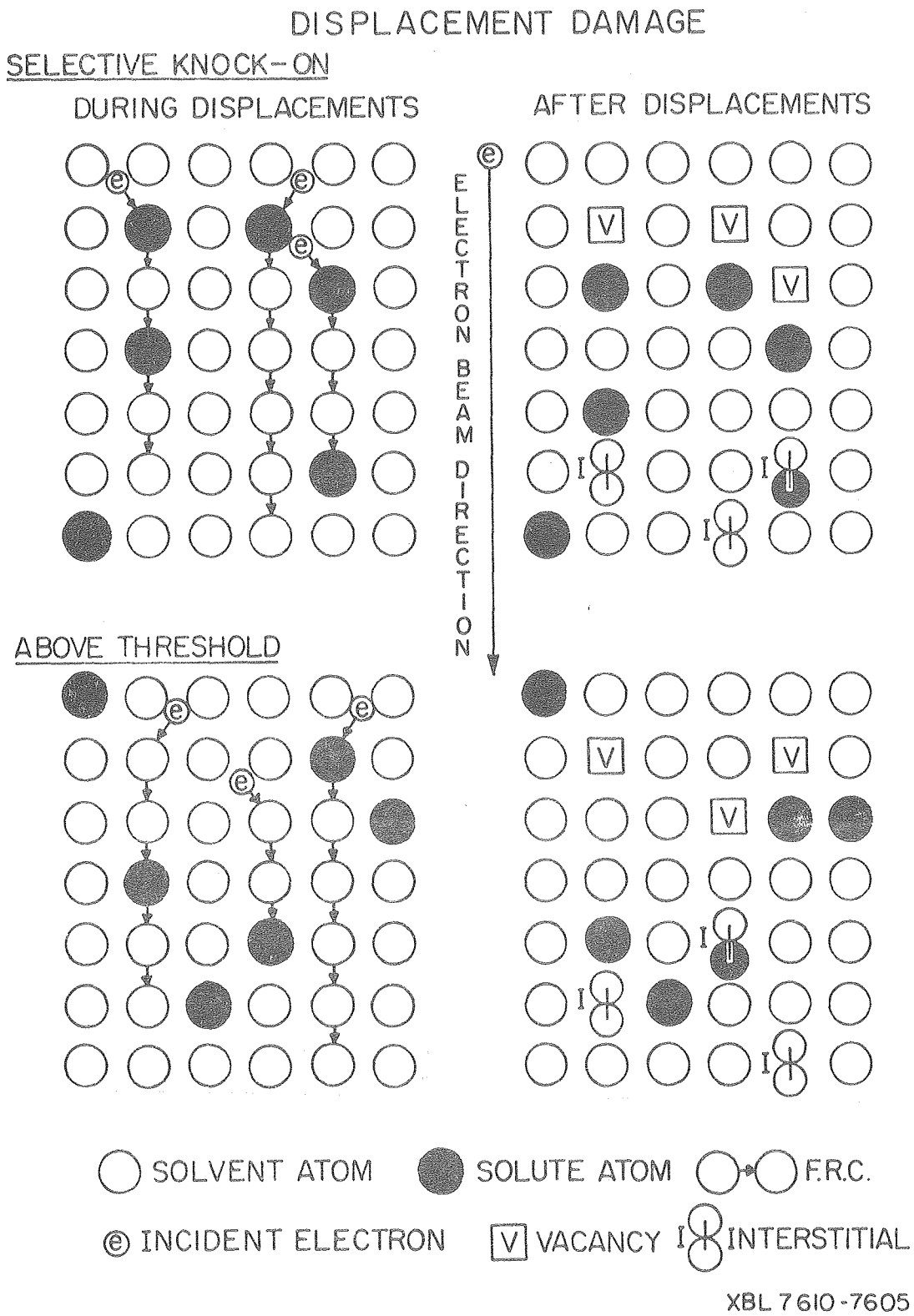
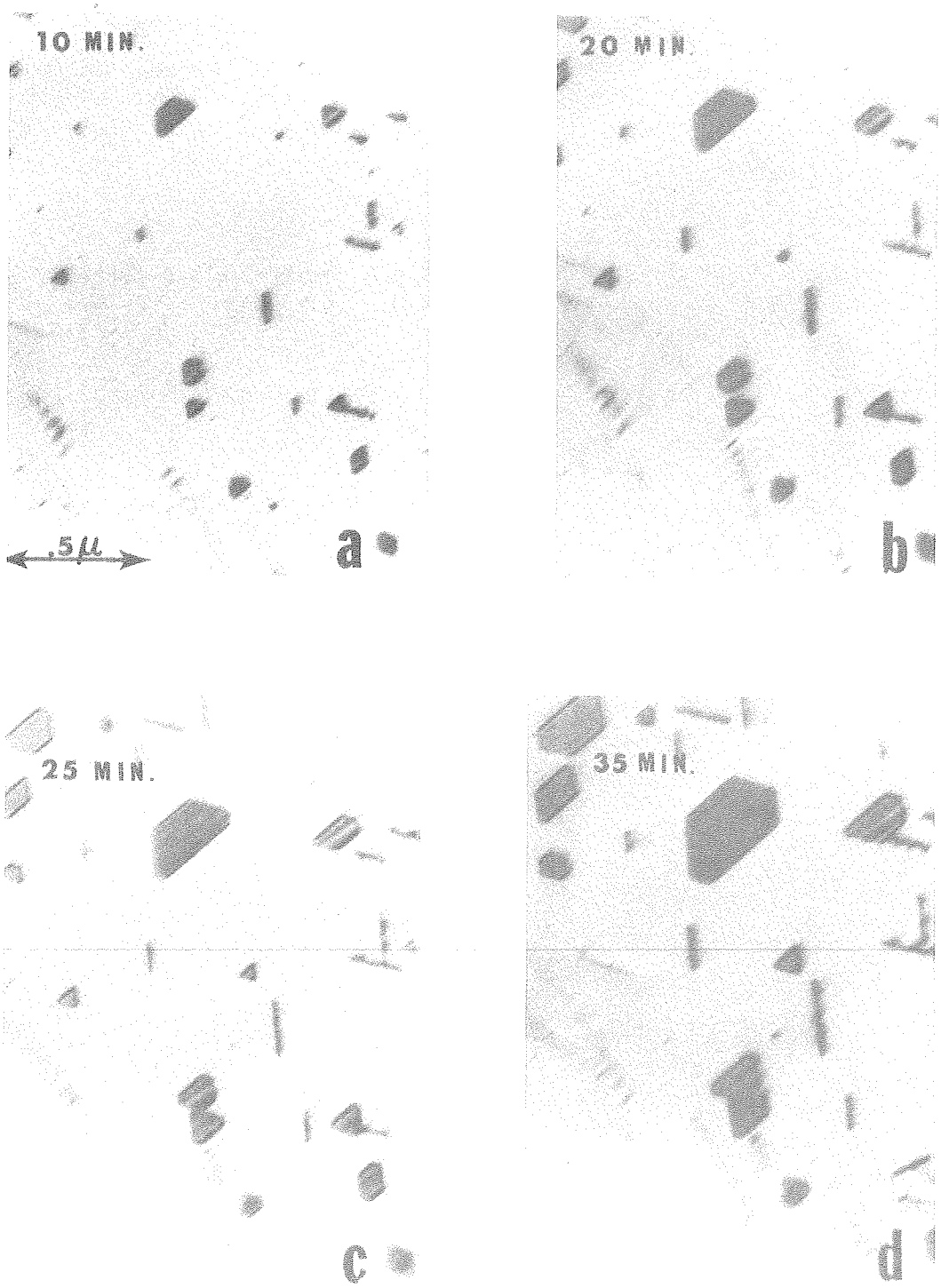


Fig. 2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a
20 MIN.
b
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c
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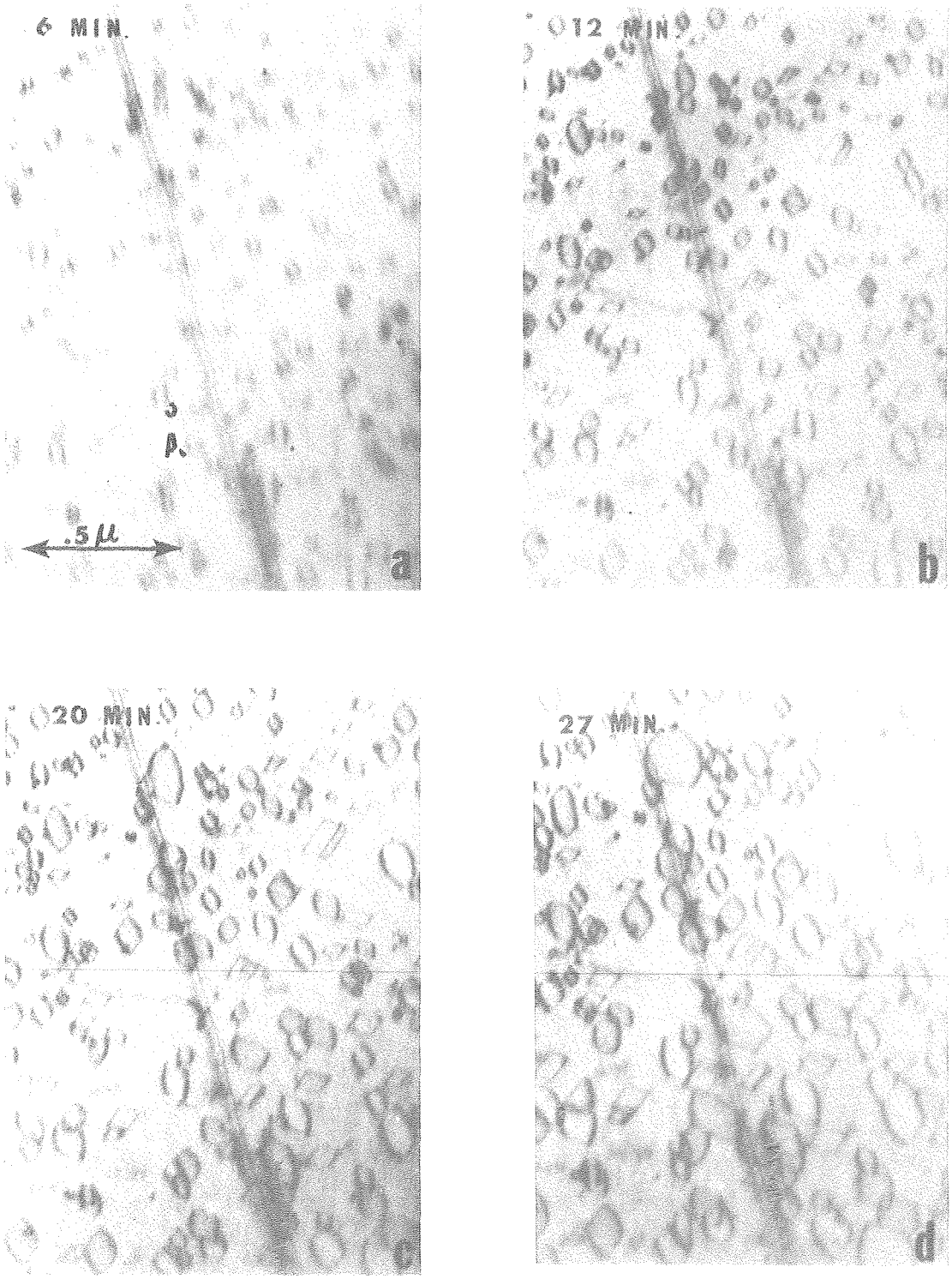
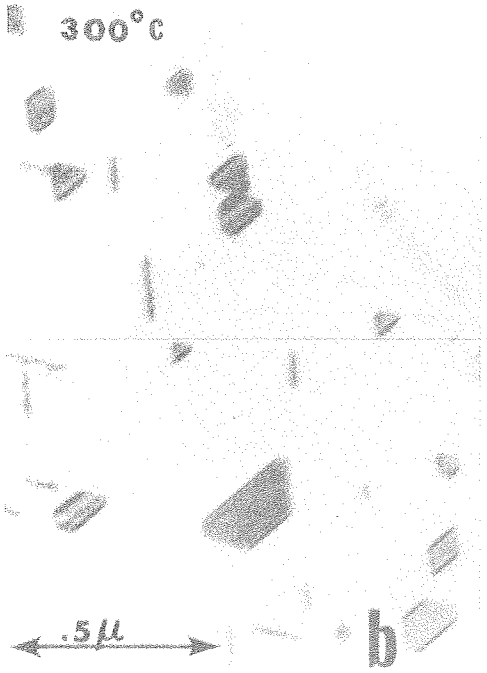


Fig. 4

300°C
a


300°C
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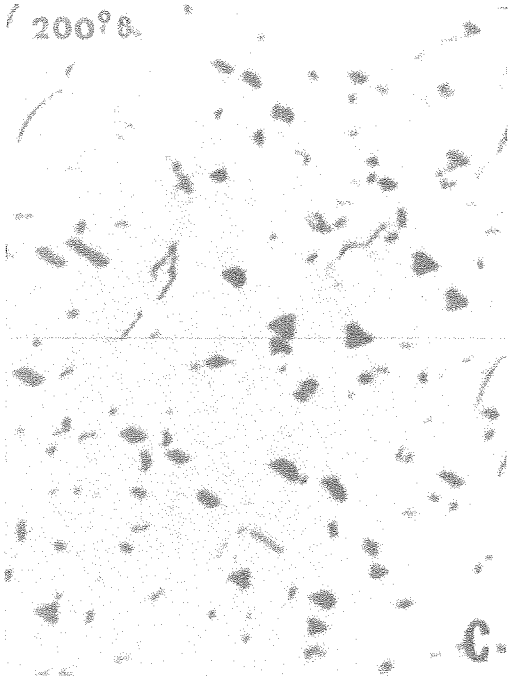
200°C
c

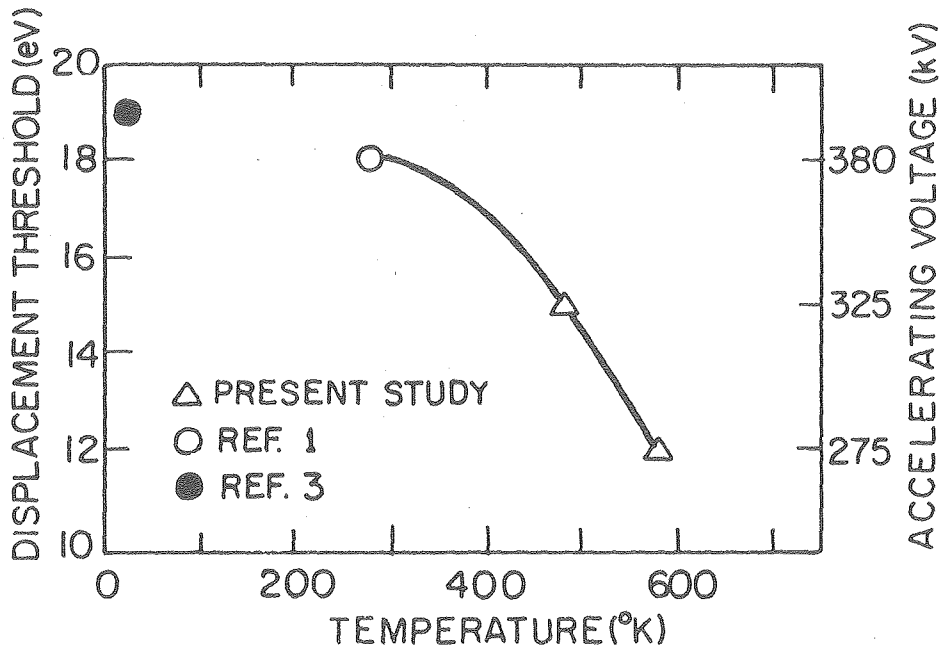

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Fig. 6

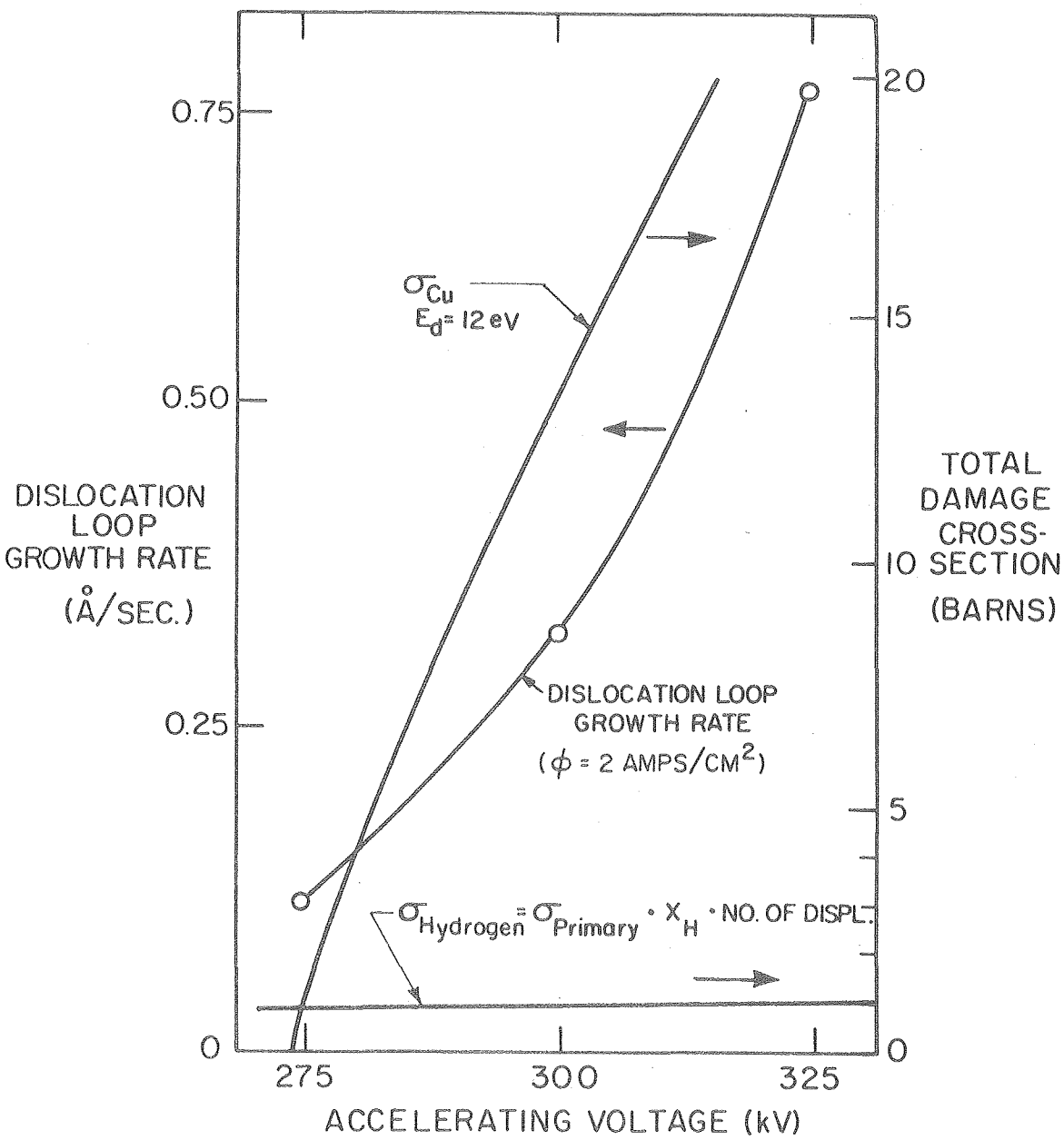

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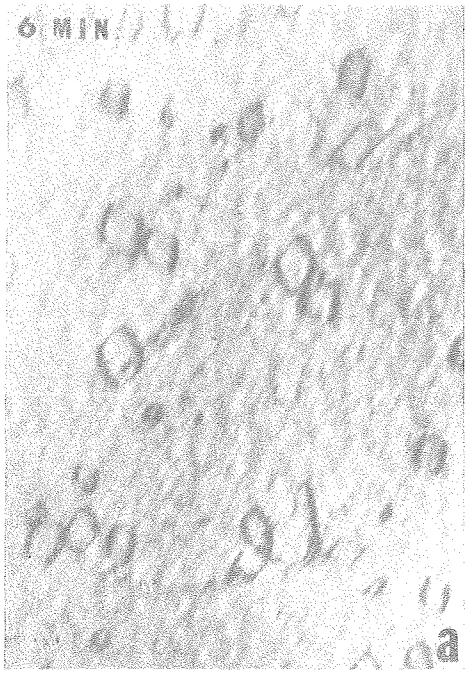
Fig. 7

Figure 8. Damage Cross Sections for Cu, Al, and Cu-Al alloys (in BARNS)

Displacement energy (eV)	Electron energy-Material	300 KeV	400 Kev	500 KeV	550 KeV	600 KeV	650 KeV
15	Cu	0	16.4	36.6	46.1	53.3	59.9
	Al	31.3	39.6	46.1	49.1	50.8	52.4
	Cu+2A/OAl	.72	16.9	36.8	46.2	53.4	59.7
	Cu+6.8A/OAl	2.1	17.9	37.4	46.3	53.1	59.3
	Cu+15A/OAl	4.7	19.9	38.0	46.6	53.0	58.9
20	Cu	0	0	11.4	17.8	23.0	28.3
	Al	13.9	20.8	25.5	27.2	29.9	32.0
	Cu+2A/OAl	.32	.48	11.8	18.0	23.2	28.3
	Cu+6.8A/OAl	1.0	1.5	12.4	18.5	23.4	28.6
	Cu+15A/OAl	2.1	3.3	13.5	19.3	24.1	28.9
25	Cu	0	0	1.1	5.0	9.2	13.1
	Al	4.9	11.9	16.4	18.3	19.8	21.5
	Cu+2A/OAl	.11	.27	1.5	5.3	9.4	13.3
	Cu+6.8A/OAl	.35	.84	2.2	5.9	9.9	13.7
	Cu+15A/OAl	.73	1.8	3.4	6.9	10.8	14.4
30	Cu	0	0	0	0	2.5	5.6
	Al	0	6.29	10.2	11.9	13.4	14.8
	Cu+2A/OAl	0	.14	.23	.27	2.8	5.8
	Cu+6.8A/OAl	0	.45	.72	.85	3.3	6.3
	Cu+15A/OAl	0	.94	1.5	1.8	4.2	6.9
35	Cu	0	0	0	0	0	.8
	Al	0	3.2	6.6	8.0	9.2	10.2
	Cu+2A/OAl	0	.07	.15	.18	.21	1.0
	Cu+6.8A/OAl	0	.23	.48	.57	.65	1.5
	Cu+15A/OAl	0	.48	.99	1.2	1.4	2.2
40	Cu	0	0	0	0	0	0
	Al	0	1.2	4.2	5.3	6.4	7.4
	Cu+2A/OAl	0	.03	.10	.12	.15	.17
	Cu+6.8A/OAl	0	.09	.30	.38	.46	.52
	Cu+15A/OAl	0	.18	.63	.80	.96	1.1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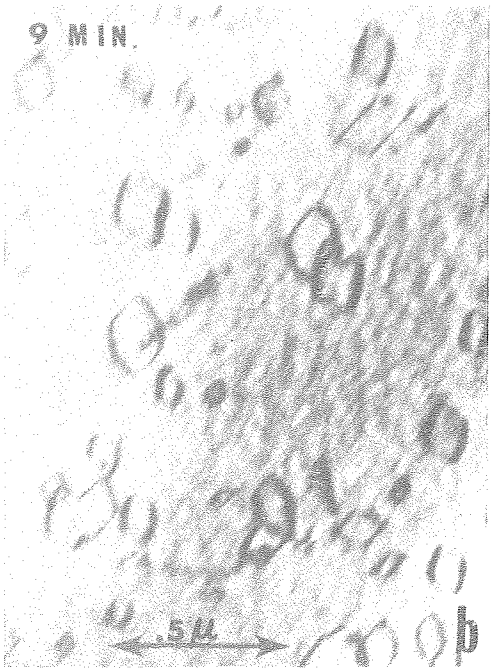
a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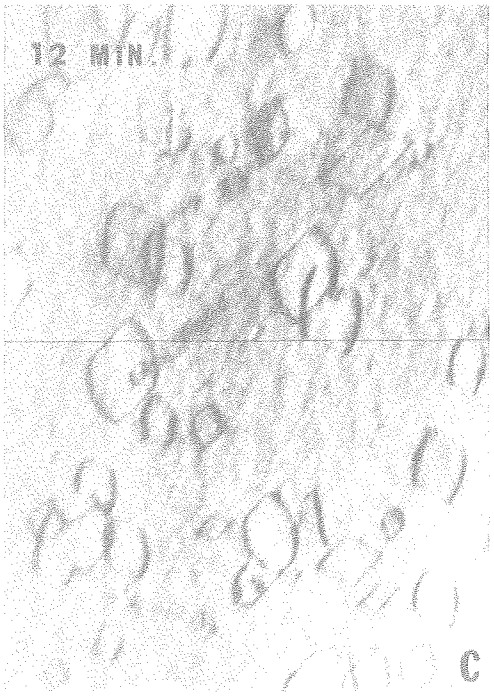
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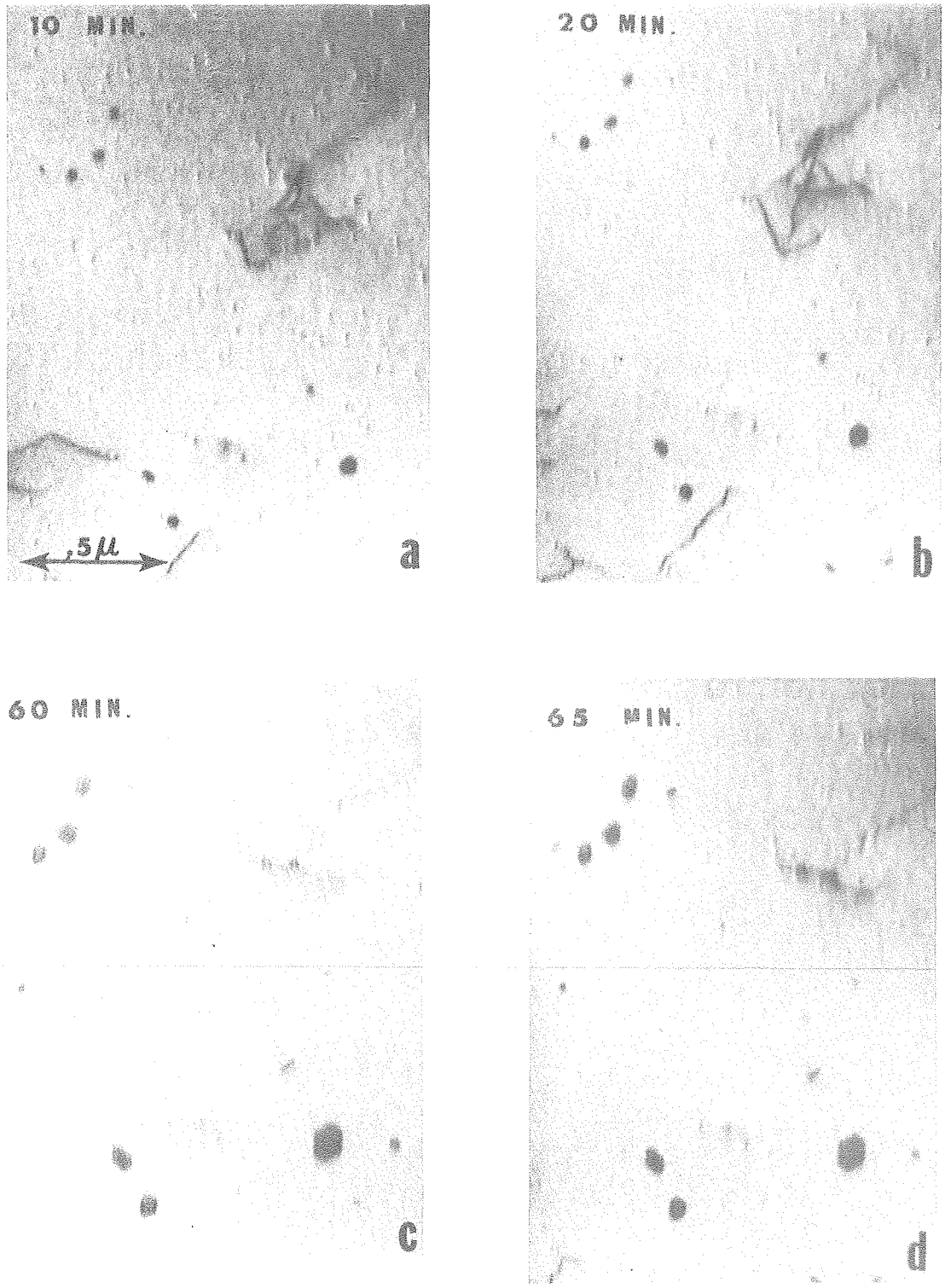


Fig. 10

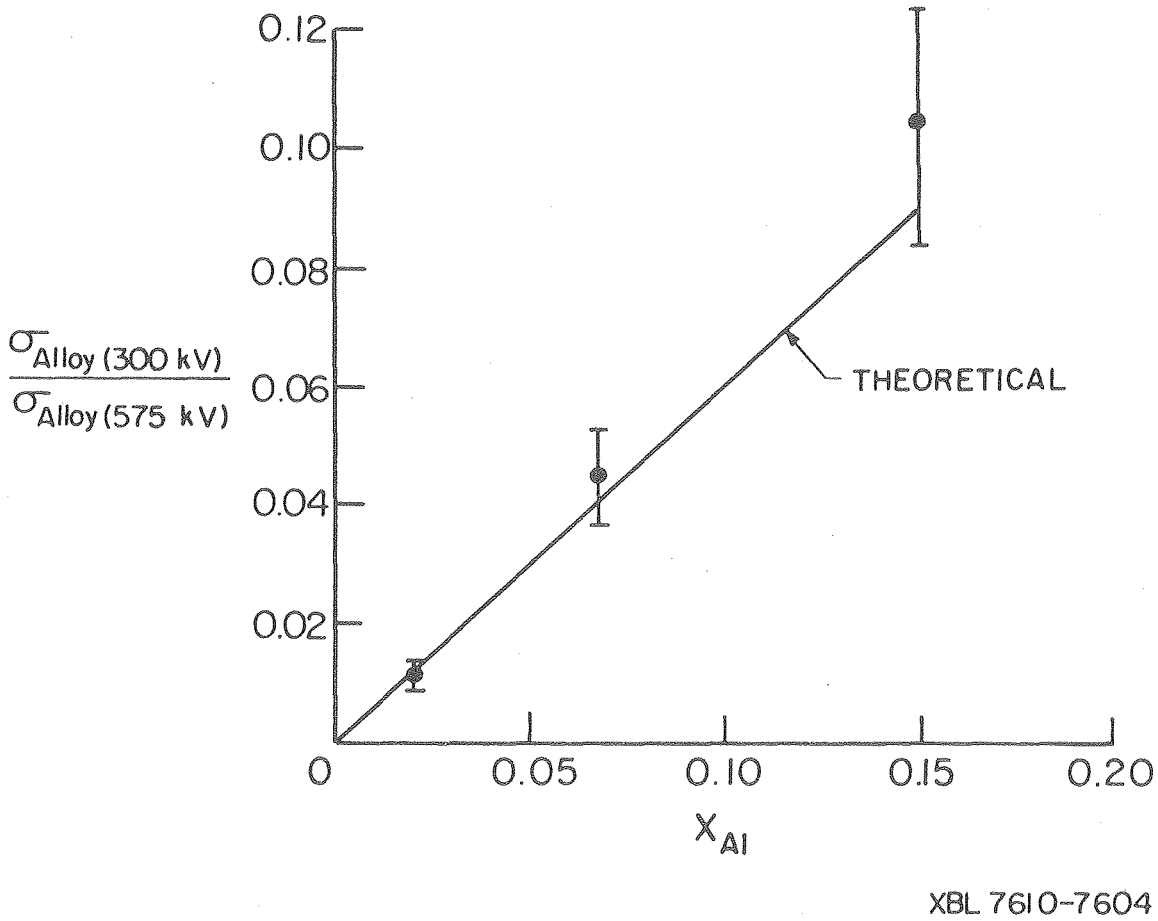
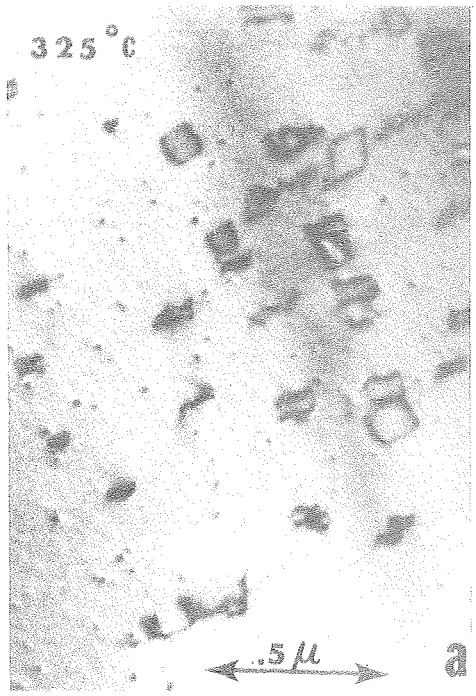

0.12
0.10
0.08
0.06
0.04
0.02
$\frac{\sigma_{Alloy\,(300\,kV)}}{\sigma_{Alloy\,(575\,kV)}}$
THEORETICAL
0
0.05
0.10
0.15
0.20
X_{Al}
XBL 7610-7604

Fig. 11

Figure 12. Ratios of electron beam currents at 300 and 575 Kv

IRRADIATION DATA	ALLOY COMPOSITION		
	Cu+2 at % Al	Cu+6.8 at % Al	Cu+15 at % Al
Dislocation* Loop Growth Rate (Å/sec)	1.0	1.1	1.6
$\phi_{300\ kv}$ (AMPS/cm^2)	1.1	1.1	.46
$\phi_{575\ kv}$ (AMPS/cm^2)	.016	.050	.048
$\phi_{575\ kv}/\phi_{300kv}$	.014	.045	.01
$\sigma_{300\ kv}/\sigma_{575\ kv}$ (theoretical)	.012	.040	.09

*There is no significance to the different values of G for the different alloys since the temperature, which drastically effects G, was not held absolutely constant from one alloy experiment to the next.


325°C
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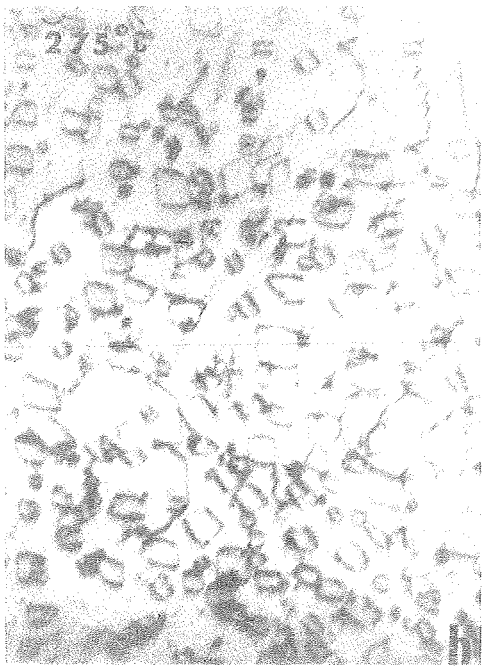
a


275°C
b

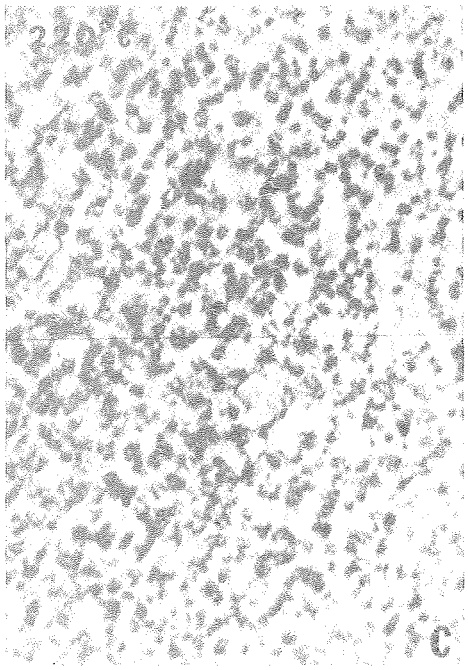
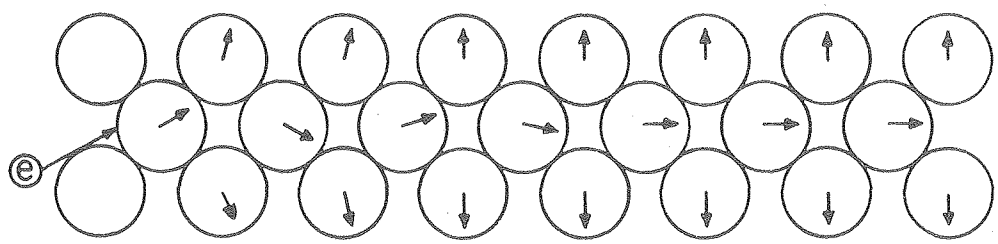

220°C
c

Figure 14. Dislocation loop growth rate data for Pure Cu and Cu-Al alloys. Temperature = 325°C ±5°C*

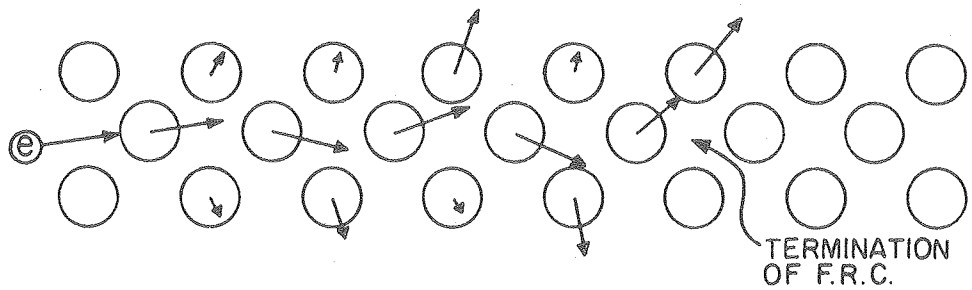
A/o Al	Accelerating Voltage (kv)	Electron Flux (Amps/cm^2)	Dislocation Loop Growth Rate (Å/sec)	Standard Deviation of Growth Rate (Å/sec)	# of Loops counted	95% Confidence Limits on growth rate (Åsec)
2.0	300	1.2	.40	.23	23	±.10
2.0	575	.057	1.8	.44	19	±.20
2.0	575	.037	1.1	.10	6	±.10
6.8	300	1.1	1.1	.28	10	±.18
6.8	575	.09	1.6	.32	16	±.16
6.8	575	.062	1.10	.27	14	±.14
15.0	300	.66	2.3	.60	52	±.20
15.0	575	.084	2.0	.34	26	±.10
15.0	575	.070	1.5	.30	26	±.12
15.0	575	.053	.93	.17	26	±.05
15.0	575	.043	.89	.13	12	±.08
0	275	1.1	.19	.10	13	±.06
0	300	1.5	.45	.22	32	±.08
0	325	1.3	.80	.30	23	±.13
0	500	.22	1.5	.60	21	±.26
0	650	.066	1.2	.60	14	±.32

*Temperature is constant to within ±5°C for each alloy studied but may vary ±50°C from one alloy to the next

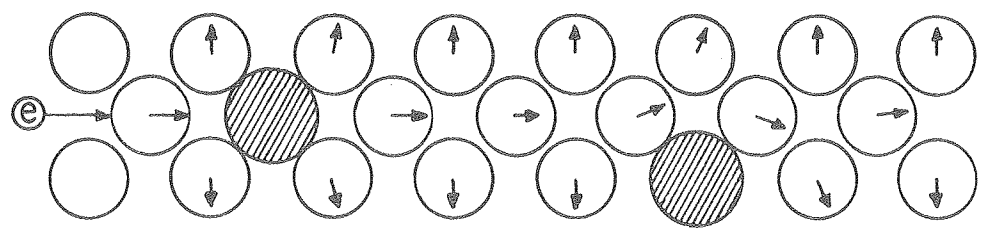
SCHEMATIC REPRESENTATION OF FOCUSSED REPLACEMENT COLLISIONS



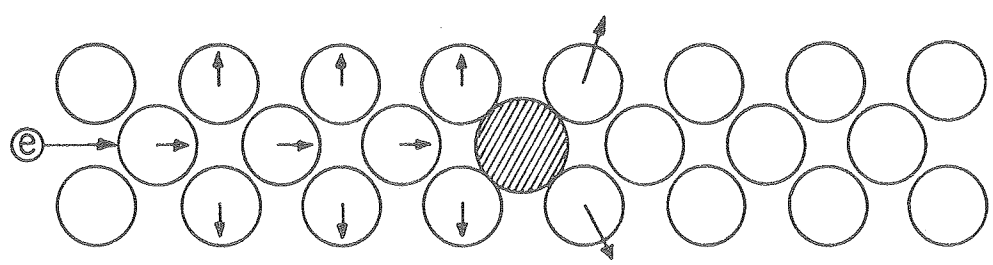
A. FOCUSSING ACTION IN A CLOSE PACKED FCC LATTICE



B. DEFOCUSSING ACTION IN AN OPEN FCC LATTICE



C. SOLUTE ATOM DEFOCUSSING

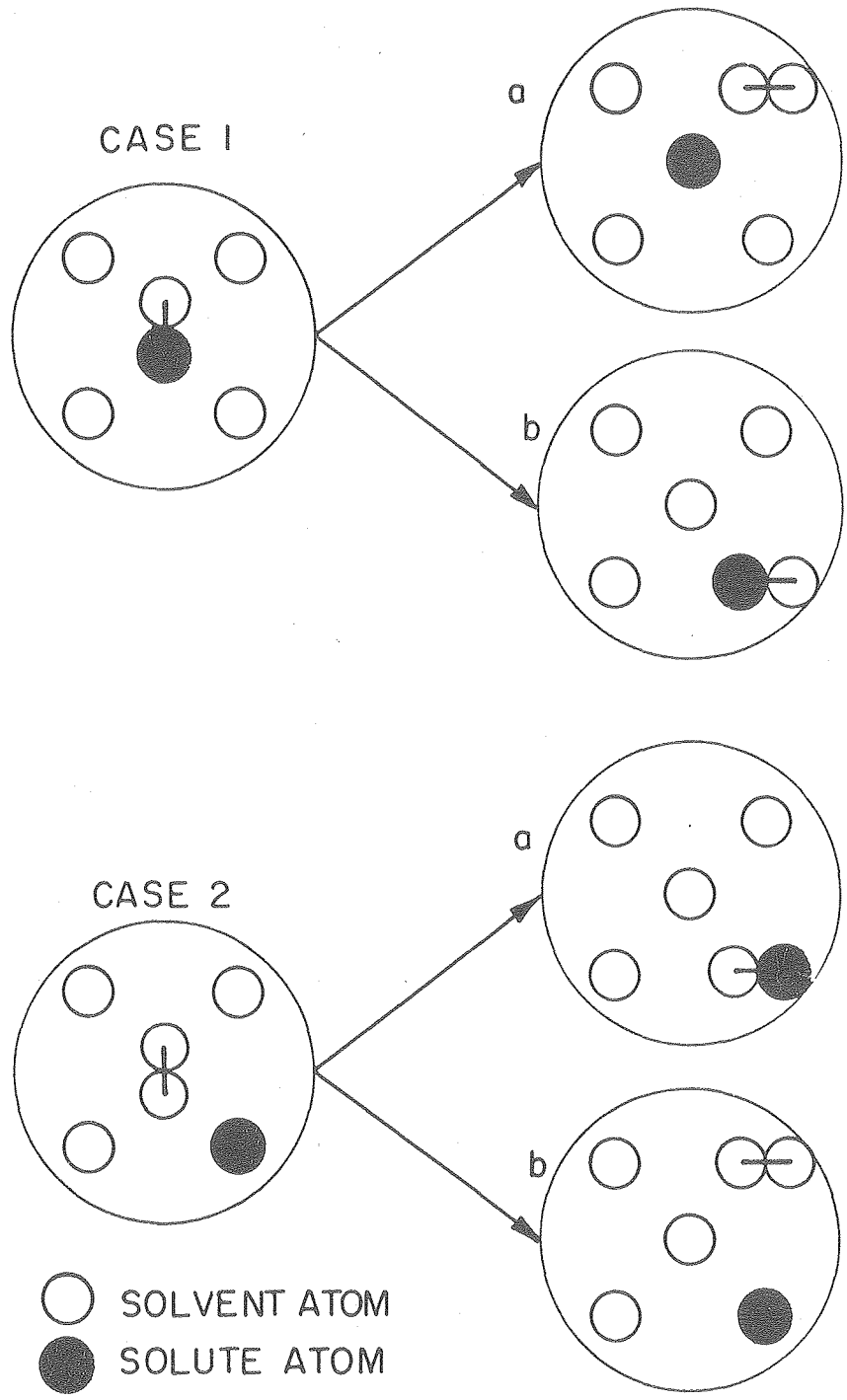


D. PREFERENTIAL F.R.C. TERMINATION ON A SOLUTE ATOM

XBL 7611-7716

Fig. 15

0 0 0 0 4 7 0 0 7 7 0



INTERSTITIALCY DIFFUSION MECHANISMS IN A FCC SUBSTITUTIONAL BINARY ALLOY

XBL 771-4920

Fig. 16

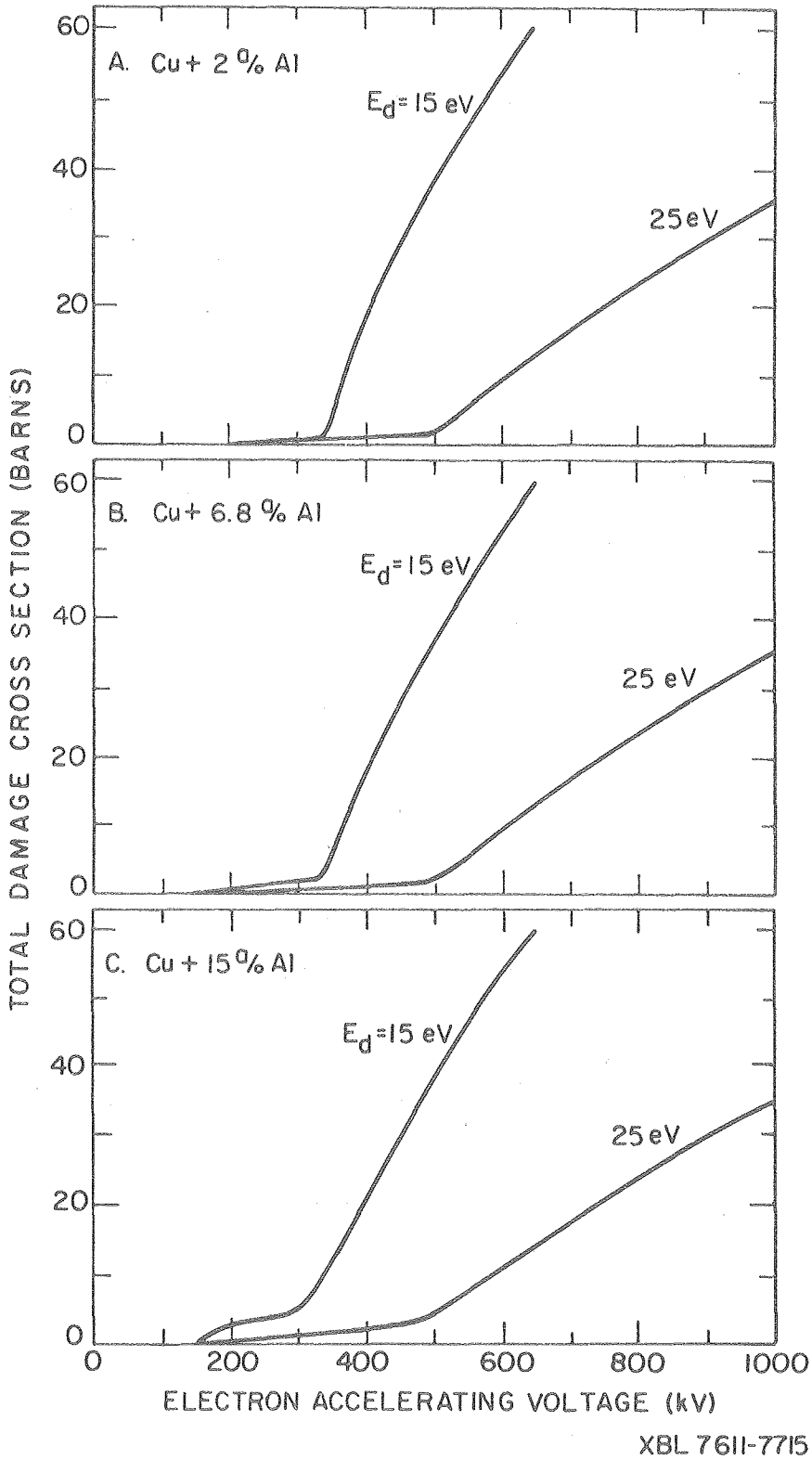
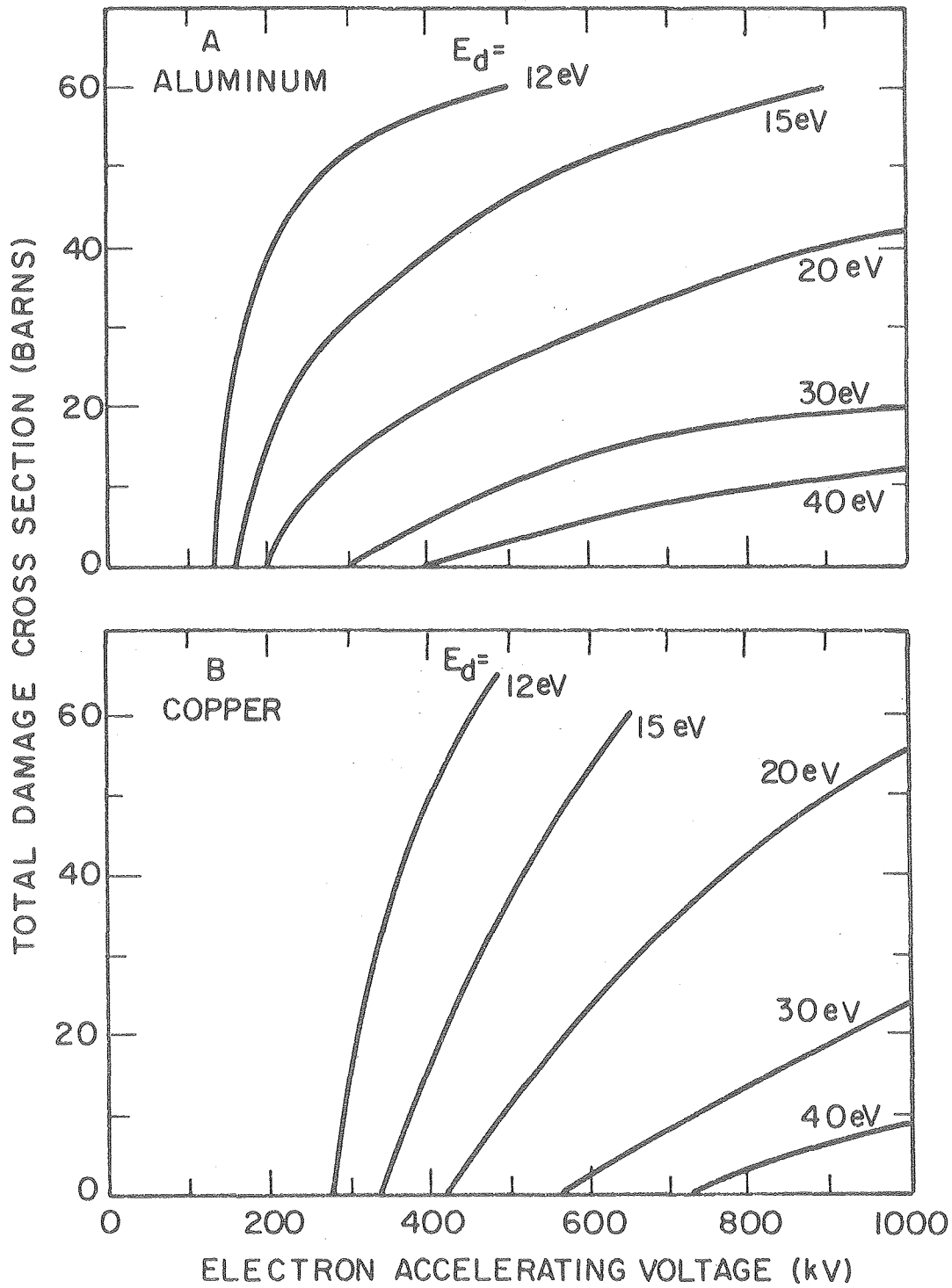

A. Cu + 2 % Al
E_d = 15 eV
25 eV
B. Cu + 6.8 % Al
E_d = 15 eV
25 eV
C. Cu + 15 % Al
E_d = 15 eV
25 eV
TOTAL DAMAGE CROSS SECTION (BARNS)
0
20
40
60
0
200
400
600
800
1000
ELECTRON ACCELERATING VOLTAGE (kV)
XBL 7611-7715

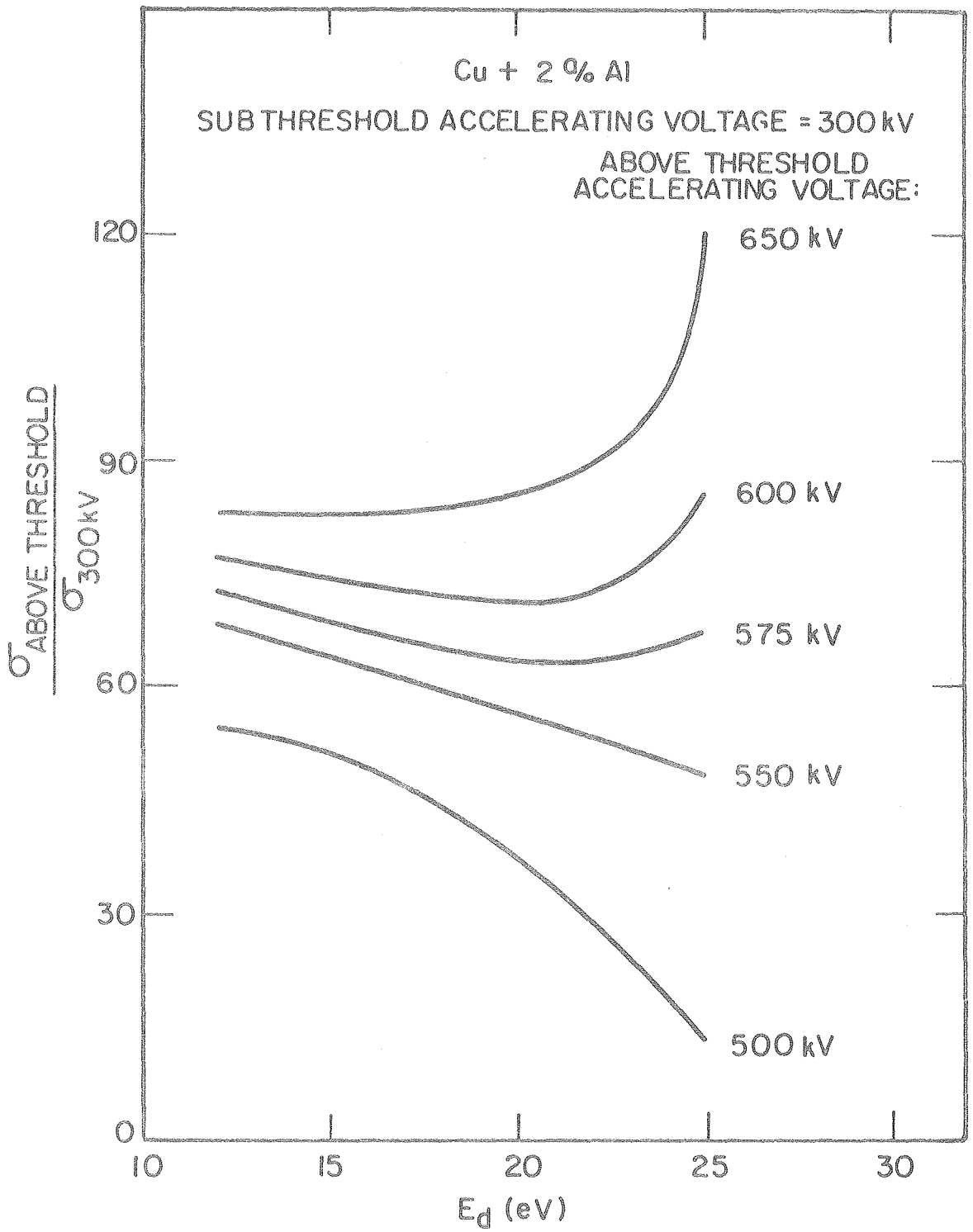
Fig. 17

0 0 0 0 4 7 0 0 7 7 1



XBL 7611-7714

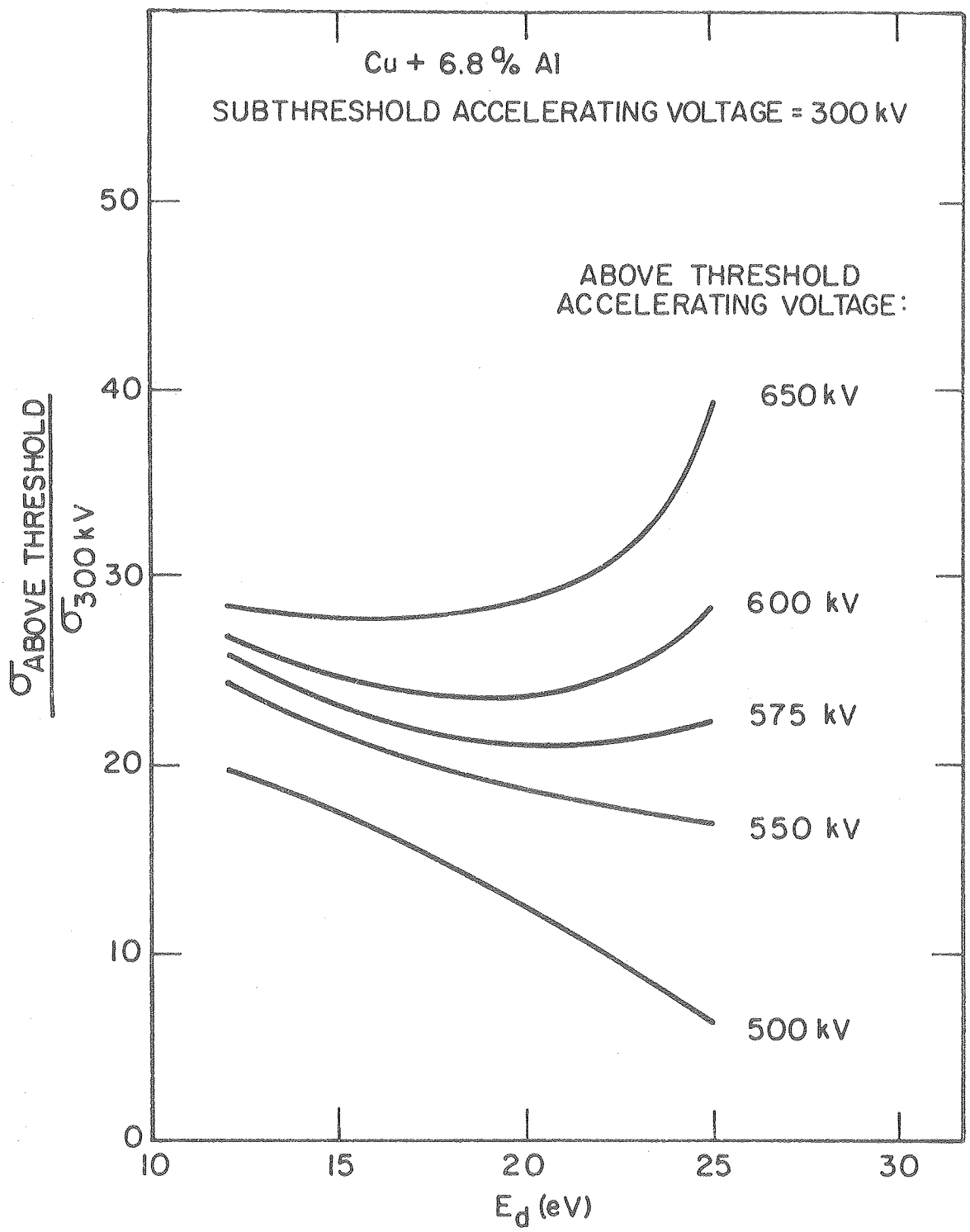
Fig. 18



XBL 7611-7712

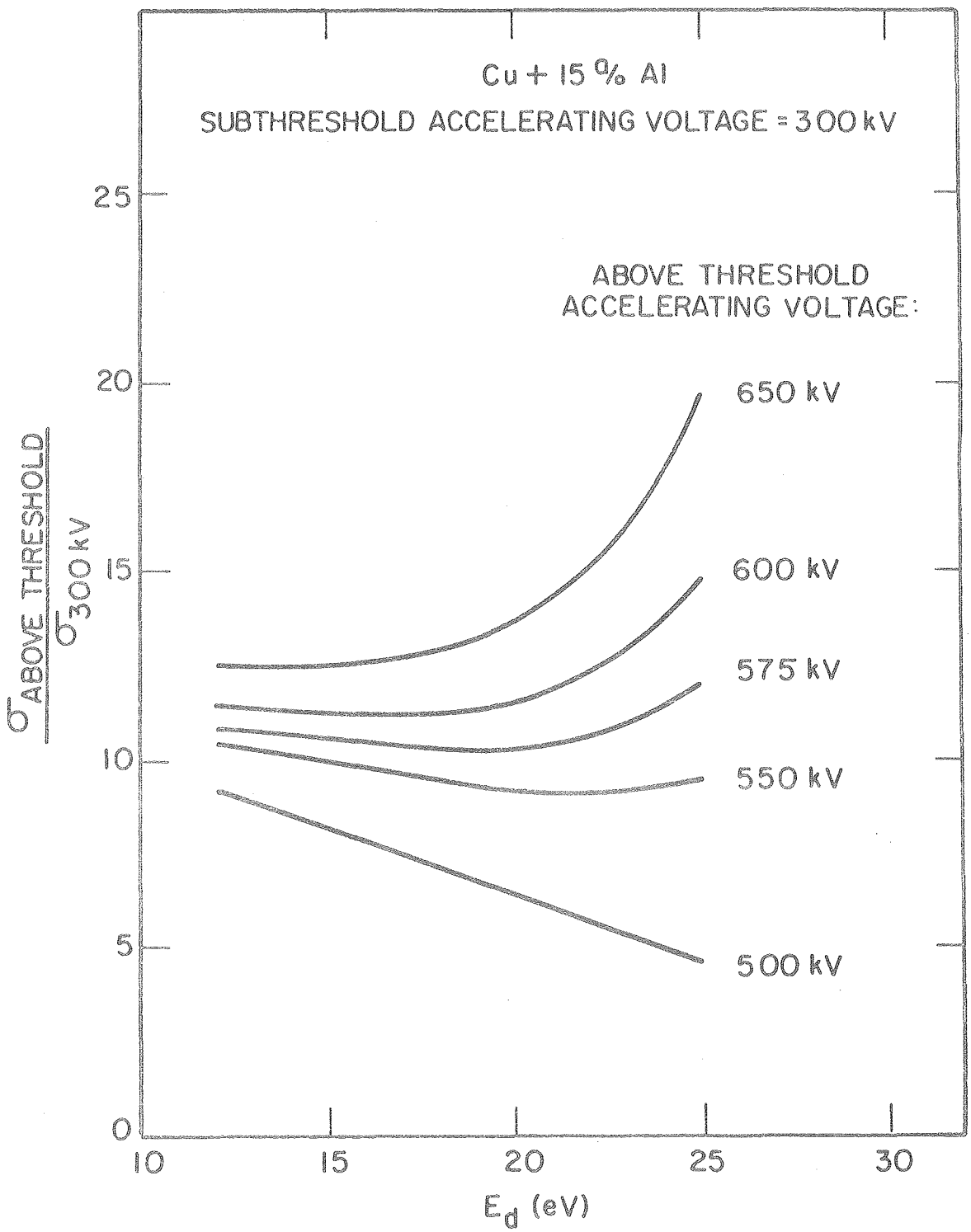
Fig. 19

0 0 0 0 4 7 0 0 7 7 2



XBL 7611-7711

Fig. 20



XBL 7611-7713

Fig. 21

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0 0 0 0 4 5 0 2 2 9 4