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EXPERIMENTAL STUDIES OF CASCADE PHENOMENA IN METALS*

M. L. Jenkins[1], M. A. Kirk[2] and W. J. Phythian[3]

[1]Department of Materials, University of Oxford, OX1 3PH, UK
[2]Materials Science Division, Argonne National Laboratory, Argonne, IL 60439
[3]Radiation Damage Department, AEA Technology, Harwell Laboratory, OX11 ORA, UK

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M L Jenkins and M A Kirk[1]

Department of Materials, University of Oxford, OX1 3PH

and W J Phythian

Radiation Damage Department, AEA Technology,
Harwell Laboratory, OX11 0RA

We review recent ion-irradiation experiments which have been performed to investigate the collapse of displacement cascades to dislocation loops in a range of metals and alloys. Many of the results including the dependencies of the collapse probabilities on irradiation temperature, and ion dose, energy and mass, can be explained within the framework of a thermal spike/cascade melting model which has been suggested by computer molecular dynamics simulations. Other aspects, such as the dependence of collapse probabilities on the crystal structure and the effects of alloying and impurities, are less well understood.

1. INTRODUCTION

Under fast neutron irradiation of metals the majority of point-defects are created within displacement cascades initiated by primary knock-ons of energy >5keV. The subsequent fate of these point-defects is crucial for most microstructural changes induced by the irradiation, such as growth, creep and swelling, segregation and precipitation, and alloy mixing and enhanced diffusion. Cascades are important also in ion-beam mixing and ion implantation. For these reasons cascade phenomena have been extensively researched over the past three decades. A common and successful approach to study the vacancy component of the damage has been to examine in the transmission electron microscope thin foils which have typically been irradiated with low doses ($< 10^{16}$ions m^{-2}) of heavy ions with energies in the range 10-100 keV. The ions simulate the primary knock-ons produced in neutron irradiations. Vacancy loops produced at individual cascade sites by "cascade collapse" are imaged, and information obtained on their morphology, sizes and number densities. The irradiation conditions - the type and energy of the ions, the dose and dose rate, and the irradiation temperature - can be closely controlled and allow the materials and irradiation parameters affecting cascade collapse to be explored. The efficacy of cascade collapse is usually described quantitatively by two parameters, the defect yield Y and the cascade efficiency ε. Y is defined as $Y = N_L^v/N_c$ where N_L^v is the number of visible vacancy loops per unit area

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[1]On leave from the Materials Science Division, Argonne National Laboratory, Argonne, IL60439

and N_e is the ion dose, ie the number of cascades generated per unit area. Y therefore measures the proportion of cascades which collapse to visible loops. The cascade efficiency ε is given by $N_v(obs)/N_v(th)$ where $N_v(obs)$ is the number of vacancies retained in a loop of average area and $N_v(th)$ is the calculated number of vacancies generated in the cascade. Absolute values of Y and ε should be treated with caution, particularly in comparing the results of different workers, because of different loop imaging, counting and measuring procedures, the necessity in some cases to make allowance for loss of glissile loops to the foil surface, ion dose errors, and assumptions made in estimating N_v. However they are useful for identifying general trends.

In this review we shall concentrate largely on recent progress in understanding cascade mechanisms made using this approach. We take as a starting point the review by English and Jenkins [1]. The position at the time of this earlier review can be summarised as follows.

(i) Cascade collapse had been observed in room-temperature heavy-ion irradiations in a variety of pure materials (eg Cu [2-3], Ag[4-5], Au [6], Ni [7-9], Mo [1], Fe [10], W [11], Co [12], Ru [13], Cu_3Au [14-16]). It had been found that in most fcc metals collapse is efficient with high values of Y and ε. In bcc metals the defect yields and cascade efficiencies are on average lower for equivalent irradiation conditions even after corrections due to loop loss to the surfaces in these materials. Insufficient research had been carried out in hcp metals to be sure of trends although in the experiments which had been performed the yields were low. In general, the defect yield increased and the cascade efficiency decreased with increasing ion energy. Quantitative data extracted from refs [2-13] illustrating these trends are shown in table I of ref[1] which has been published again as table III in ref[17].

(ii) The cascade deposited energy density and the related vacancy supersaturation at the end of the displacement phase had been proposed as important parameters in determining collapse probabilities. This was deduced from the observation of increasing values of defect yield Y with increasing ion mass at constant ion energy as well as systematic experiments using molecular-ion irradiations (see table II of ref [1]).

(iii) Usually faulted loops nucleate on close-packed planes; these may subsequently unfault to perfect loops, although in fcc metals partial or complete dissociation to stacking-fault tetrahedra is also possible [4]. Generally the majority of loops were of the types expected on the criterion of lowest elastic strain energy. However less energetically favoured types were found in high energy-density cascades in Fe [10] and Mo [1] ($\underline{b}$ = a <100> rather than $\underline{b}$ = a/2 <110>) and in Ru [11] , where the proportion of $\underline{c}$- component loops to the expected faulted and prismatic types was considerably higher for W^+ ion irradiations than for self-ion irradiations. This was interpreted as another energy density effect.

(iv) Cascade collapse was found to occur at low (~30K) irradiation temperatures in a number of materials (Cu_3Au [16] , Cu, Ni, Fe [8]). Since at this temperature no thermal migration of vacancies is possible, this was taken as evidence that the collapse process is athermal and probably occurs in the thermal spike phase of the cascade. The collapse probabilities however were considerably smaller at 30K than at room temperature, although the data for materials other than Cu_3Au were limited. In Cu_3Au no further collapse occurred on warming to room temperature.

(v) In elevated temperature irradiations measured defect yields (after cooling to room temperature) in Cu, Ni and Mo decreased [18,7,19]. In Cu and Ni the decrease was abrupt above a certain threshold temperature (for Cu, 500K and for Ni, 750K). This can be accounted for by an athermal cascade collapse process followed by loop shrinkage due to thermal emission of vacancies. In Mo, by contrast, vacancy loop numbers in self-ion irradiations decreased more gradually at temperatures 420-470K which are too low for thermal emission of vacancies in this material. In irradiations with Xe^+ ions however no fall-off in defect yields were found at temperatures up to 670K [1]. These results suggests that the collapse probability in Mo is itself temperature dependent and again highlight the role of the cascade energy density.

(vi) Rather limited data existed on the influence of alloying and impurities on the collapse process (see table IV of ref[17] for a summary) and the results which had been reported did not seem open to simple explanations. In copper alloys, Stathopoulos et al [20] found that the defect yields increased with alloying in all cases, irrespective of the strength of the solute-point-defect binding. A correlation between the magnitude of the solute-atom size factor and the increase in defect yield led these authors to suggest that the dominant factor in determining this trend was dechannelling of incident ions and channelled knock-ons. This however could not be established with confidence, and the explanation does not seem entirely consistent with the trends seen at higher ion energy [20]. The situation with interstitial impurities was even less clear. Very small quantities of N in Mo appeared to have a dramatic effect on the yield although no really plausible explanation of why could be offered[19]. Yields were also generally low in stainless steels compared with base alloys, but again this was not well understood [7,21].

English and Jenkins [1] discussed the results summarised above in terms of cascade models current at that time. They concluded that defect migration occurring during thermal spikes and the rate of cooling of the spike region were central to an understanding of the collapse process. They suggested also that the initial vacancy configuration and concentration were important parameters controlling loop formation. They postulated the existence in bcc and hcp metals of a barrier to loop

nucleation possibly determined in part by the stacking-fault energy. Over the past five years several experiments have been carried out in order to explore these ideas further and to resolve some of the outstanding issues. These experiments are described in section 3 below.

First however it is appropiate to describe briefly the advances in cascade simulations made in the last five years which have considerably changed our perception of cascade processes and provide a framework for much of the interpretation of recent results.

2. Recent advances in computer simulations of cascades

Computer simulation experiments have advanced considerably in recent years with not only better hardware but also more realistic simulations. These have been reviewed recently by de la Rubia and Phythian [22] and Guinan [23]

The largest activity in the area of cascade simulations has been with the use of molecular dynamics. A large proportion of the work has been conducted for "Cu", and to a lesser extent "Ni" and "Fe"[24-32]. Known work is currently underway on "Ti", "Ag", "Au" and alloys such as "Ni-Al" and "Cu-Au". We put "Cu" and "Ni" etc in quotation marks here to remind us that these are simulations, and the detailed processes found may well depend on the choice of potentials and other computational details. To date, the highest primary knock-on energy simulated is one 25keV event, although many cascades of energy 10 kev and below have been examined. With these low energies (by experimental standards) and the differences in detail brought about by simulation methods etc. a degree of caution should be used in comparing data; however, the simulations do provide interesting insight into what may be happening in reality.

In the context of this review, the most important observation of the computer simulations was the identification that local 'melting' of the cascade core had occurred. This was achieved by comparison of the densities, temperatures and pair-correlation functions with the corresponding quantities in the liquid phase [24]. In this simulation of "Cu", the local melting persisted for several picoseconds, a time long enough for several lattice vibrations to occur and for vacancies located within the melt to lose their individual identities. At this stage the vacancies were represented as a generally lower density within the melt, typically ~ 85% that of crystalline "Cu". As the cascade melt cooled it was observed to recrystallised coherently at the cooler peripheries. As recrystallisation proceeded density fluctuations within the melt increased and eventually "froze out" as vacancies. The final number of vacancies was dependent on the number of replacement collision sequences extending beyond the melt boundaries whilst their final configuration depended on the speed of the solidification front. The sweeping of vacancies into the cascade centre by this process is similar to zone refining. These earlier simulations with pair potentials have

since been repeated with more sophisticated many body potentials that show essentially the same conclusion that a liquid like cascade core does exist with a density ~80% that of a liquid at similar temperature [32]. However, no well defined solidification front has been observed in the simulations with $E \leq 5$ keV [29,22]

A further question which has been addressed by molecular dynamics is the mechanism whereby vacancies and interstitials are separated in the cascade. All the simulations have concluded that replacement collision sequences do play a role in the separation process, but the degree is temperature sensitive. In studies of cascades conducted at 100K the numbers and average lengths are small [32]: eg, in 2 keV cascades in "Cu" an average of about 7 focussons and replacement collision sequences, the latter of average length 6 ± 2 replacements, were emitted per cascade. They were responsible for transporting only a small fraction, ~2%, of the total cascade energy. The short average lengths of collision sequences is consistent with experiments by Kirk et al [33] and Bullough et al [34]. Kirk et al [33] for example found a very low fraction of <110> replacement events extending away from the average neutron cascade (30 keV recoil event) in Ni_3Mn at 5K. No more than 4% of the total replacements in the average cascade could be placed into these replacement collision sequences, indicating that the spatial distribution of interstitials is rather compact around the defect cascade. This result, however, does not reveal the extent of focussons emanating from the defect cascade because they do not produce replacements.

In 0K and 4K simulations, using a simpler pair potential [24], replacement collision sequences play a more significant role, with typical lengths of ~20 replacements. An interesting feature of all simulations is the very narrow time window towards the end of the displacement phase when the collision sequences appear, suggesting that their propagation is very sensitive to the conditions pertaining at the cascade peripheries, and these conditions may be satisfied for only a short time.

A second mechanism, that of "ballistic clustering" was found to be increasingly important in "Cu" as the cascade energy increased [29]. In this mechanism ballistic ejection of interstitials from the cascade core was followed by interstitial clustering at the cascade periphery. These small interstitial clusters invariably had Burgers vectors tangential to the cascade periphery allowing them to escape from the cascade region by thermally-assisted glide. This mechanism was considered to be critical in preventing recombination and so enhancing vacancy survival. A higher-energy manifestation of this process has been observed by de la Rubia and Guinan in the highest energy cascade so far simulated [28]. Here the high compressive forces generated in the collisional phase lead to the punching of a 17 interstitial atom platelet from the cascade periphery.

The mechanisms described above lead to reduced recombination at the cascade core. If these same phenomena hold true for cascades

generated experimentally with higher energy, one could expect an increase in vacancy loop production. It should however be noted that of all the simulations conducted to date, very little evidence for vacancy loop production has been reported. The formation of a vacancy loop has however been followed with molecular dynamics by creating areas of high vacancy concentration, and following the subsequent motion at high temperature [eg 35,36].

3. Recent experimental studies

Most of the experiments reported in the last few years have been designed to explore systematically the parameters identified as controlling cascade collapse. For convenience we shall consider first experiments in pure metals, then in alloys.

3.1 Pure metals

Selected values of defect yields and cascade efficiencies for recent experiments in pure metals are shown in table 1.

3.1.1 Face-centred cubic metals

Several studies have been published in the past few years, with particular emphasis on Ni and Au and on the use of in-situ irradiations and observations.

Nickel and copper Robertson et al [37] completed their detailed study of cascade collapse in Ni following irradiation with 50 and 100 keV Ni^+ and 50 keV Kr^+ ions in the HVEM-Accelerator Facility at Argonne National Laboratory. Vacancy loop formation was followed as a function of both the irradiation temperature (30K and room-temperature) and irradiation dose (10^{15}-10^{17} ions m^{-2}). The major conclusions were as follows:

(i) Dislocation loops were produced in the low-temperature irradiations but the probability of collapse at 30K was only about half of the room-temperature value (see table 1). These observations are consistent with previous experiments in Cu_3Au [16] and the preliminary results in Cu, Fe and Ni [8] which were reviewed by English and Jenkins [1]. Vacancy loop formation at temperatures where the vacancies are immobile implies a thermal-spike mechanism of collapse. Lower yields at 30K were also found in the earlier experiments and attributed to more effective quenching of the thermal spike.

(ii) A new population of loops appeared when foils irradiated at 30K were subsequently warmed to room temperature. This new observation was attributed to a population of sub-microscopic clusters which grow to a visible size when there is at least limited vacancy mobility. It is interesting to note that new loops do not form at 30K under the influence of the electron beam

(by a mechanism of radiation-induced migration) in any of the materials so far studied.

(iii) At cascade overlap doses a new phenomenon of "cascade dissolution" was observed. With successive incremental doses a fraction of the loops disappeared (fig.1). Others changed their positions or their Burgers vectors or appeared to coalesce with neighbouring loops. Similar effects have been seen in Cu[38] and Ni-1%Al[39,40]. They were believed to originate in existing loops being engulfed by new cascades. The plausibility of this is demonstrated by recent computer molecular dynamics simulations of Foreman and co-workers [41,42], such as the example shown in fig.2 where a pre-existing vacancy loop is effectively annihilated by the new cascade event. In other simulations, not shown, new loops reformed at the cascade site. These annihilation/reformation effects are capable of explaining the trend of decreasing defect yield with increasing dose found in the experiments.

(iv) Defect yield values (table 1) were confirmed to be lower than for Cu irradiated under similar conditions.

Gold Jäger and Merkle[43] and Calder et al[44] have examined energy-density effects by studying loop formation produced by low-energy ions: Jäger and Merkle employed 10-20 keV Bi^+ and 20-40 keV Bi_2^+ molecular ions at room-temperature , whilst Calder et al irradiated with 5-20keV Bi^+ ions at both 4 K and 285 K. These low-energy irradiations result in very compact cascades of high energy density close to the foil surface. The latter authors compared their results with self-ion irradiations of Cu of the same total energy but an order of magnitude lower energy density.

Jäger and Merkle concluded that there was a higher probability for loop formation and a more efficient vacancy-interstitial separation in dense cascades. It is interesting to note however that the defect yields for 20keV monatonic Bi^+ and diatomic Bi_2^+ ions, ie the same total energy, were in fact very similar (table 1). It was only when cascades were compared on an energy per atom basis that significant differences in yield were seen. Molecular ions are expected to dissociate on impact and produce strongly overlapping cascades with enhanced energy densities and vacancy concentrations compared with single ions of the same total energy. In Sb^+ molecular-ion irradiations of Mo (see English and Jenkins[1], table II) the defect yield increased with the number of atoms in the ion at the same total energy indicating a stronger energy density effect than seen in Jäger and Merkle's experiments in Au.

A relatively weak energy-density effect was also found in Calder et al's experiments. Their data for 10keV ions (table 1) showed that the defect yields for Bi^+ -> Au and Cu^+ -> Cu were very similar at both 4 K and 285 K despite the markedly different energy densities. As in other experiments, the yields at 4 K were lower than at room temperature. The cascade efficiency ε was

however larger in Au than in copper. In Au the efficiency was higher at 4 K than at 285 K whilst in Cu this trend was reversed.

Elevated temperature irradiations of Au ,Cu and Ni Sekimura et al [45] have performed in-situ Xe^+ ion irradiations of Au at elevated temperatures up to 770 K using a 400kV ion-accelerator linked to a 200kV TEM. Vacancy loops and stacking-fault tetrahedra were observed to form during irradiation, and then shrank and disappeared, consistent with the model based on athermal collapse and thermal shrinkage by vacancy emission deduced from the ex-situ experiments on Cu and Ni described in section 1. The lifetimes of the defects under irradiation were however much shorter than under isothermal annealing at the same temperature without irradiation and were strongly dependent on the ion dose rate (which was varied from 1.3×10^{13} to 1.3×10^{15} ions m^{-2} s^{-1}). At 470 K, when vacancy clusters are thermally stable, loops were still seen to shrink in a step-like manner. These observations suggest that interstitials produced in subsequent nearby cascades play a role in the loop annihilation process. Cascade overlap effects such as sudden size increases of vacancy loops, and a tendency to larger loop sizes at higher dose rates were also identified. Sub-cascade formation was found to be less likely at higher irradiation temperatures.

Sakaida et al, [46] have performed in-situ 400 kev Xe^+ irradiations of Ni and Cu at elevated temperatures 670-770 K. Using video techniques, two defect lifetimes were observed in thicker foil, which they suggested was due to populations of both vacancy and interstitial loops. They suggest the longer lifetime component, virtually stable at 770 K, was due to interstitial loops. This analysis is in disagreement with several other experiments in Ni, eg [7]. A possible alternative possibility is that the different vacancy cluster morphologies expected to be present- Frank loops and stacking-fault tetrahedra- have different stabilities. The question of interstitial loop formation at individual cascade sites is discussed in section 4.

Aluminium Results of Bui [47] et al in Al are shown in table 3 and are discussed briefly in section 3.2.4 below. In this light metal the yields are very low.

3.1.2 Body-centred cubic metals

Iron Early experiments in Fe had established that cascade collapse does not occur under self-ion irradiation to low doses, $<10^{18}$ ions m^{-2} [10]. Kirk and co-workers [8,39] have shown however that collapse does take place at high overlap doses ($>10^{19}$ ions m^{-2}). The yield is very low ($\sim$0.001) for both 50keV and 100keV self-ion irradiations (table 1). No difference in yield between irradiations at 30K and 300 K could be detected.

Comparisons of loop production as a function of dose in Fe, Ni and Cu at 300K and in Fe and Ni at 30K are shown in figs. 3 and 4 respectively. The linear behaviour with n=1 seen for Cu and Ni at 300K and Ni at 30K indicates a loop production rate within individual cascades which is constant with dose at low doses. The deviation of the slopes towards n<1 at high doses in Cu and Ni is due to overlap effects such as loop coalescence, see section 3.1.1. The slope of n=0.7 in Fe at 300K (fig.3) may also be due to saturation type behaviour. The slope of n=1.5 in Fe at 30K (fig.4) may be the net result of a low probability for isolated cascade collapse (n=1) combined with a quadratic loop production rate (n=2) expected from simple overlap of two cascades resulting in one dislocation loop.

Vanadium Preliminary results on cascade collapse in vanadium under 80keV W^+ ion-irradiation at room temperature have been reported by Phythian et al [48]. A population of small vacancy loops of edge character, with $\underline{b}$ = 1/2<111> was produced. The defect yield and cascade efficiency values of Y = 0.115 $\pm$ 0.01 and ε = 0.20 $\pm$ 0.05 respectively are comparible with other bcc metals.

3.1.3 Hexagonal metals

Preliminary results by Phythian and co-workers on cascade collapse in Ru were reviewed by English and Jenkins [1]. This group has now completed a systematic investigation of cascade collapse at room temperature in a range of hexagonal metals [13,49,50]. In Ru [13,49] irradiations were performed under a wide range of irradiation conditions (ion energies from 10 to 100 keV and ion masses from 84(Kr^+) to 184(W^+)). A further series of comparative experiments was carried out in Ti, Co, Re, Ru and Mg, chosen to encompass a wide range of materials properties, using Sb^+ ions of energy 100keV and 150keV [50]. The main results were:

(i) Vacancy dislocation loops were found in all cases except for 100keV Sb^+ -> Mg. For irradiation directions close to [0001] the predominant defect geometries were prism loops. Cascades collapsed initially onto {10$\bar{1}$0} planes to form loops with Burgers vectors 1/2 <1$\bar{1}$00> some of which subsequently unfaulted to form perfect 1/3<11$\bar{2}$0> type loops. In Ru, for beam directions in the basal plane, a small minority of loops collapsed onto the basal planes to form faulted $\underline{b}$ = 1/2[0001] loops which can transform to lower energy faulted loops with $\underline{b}$ = 1/6<20$\bar{2}$3>.

(ii)The defect yields Y varied widely from material to material. Cascade efficiencies were less variable (table 1). Within a given metal the yield Y and efficiency ε values showed similar trends to cubic metals ; these values were comparable with those found in bcc metals and in general were lower than in fcc metals.

(iii) In Ru, the defect yield Y increased with ion energy but had

a complex dependence on ion mass. At 50kV or lower the yield increased with increasing ion mass; at 100kV this trend was reversed (fig.5).

This last result is at first puzzling but may be understood in terms of the formation and spatial arrangement of sub-cascades at higher cascade energies. Attempts however to correlate the overall materials trends with obvious materials parameters (the c/a ratio, stacking-fault energy, atomic mass and melting temperature) were not successful. The authors concluded that the complex behaviour could be controlled by many parameters, with interpretation made more difficult by phase changes in Ti and Co.

Results of Yellen et al [51] on two different purities of Ti are shown in table 3 and are described briefly in section 3.2.5 below.

3.2 Alloys

Recent experiments on alloys have fallen into two categories, with separate objectives. In the first type of experiment alloys such as Cu-Ni are chosen which exhibit complete solute solubility over the entire composition range. Since the atomic masses of the constituents are similar, the cascade energy density remains almost constant in alloys of different composition. The melting temperature varies with composition. This enables the role of this parameter (which is suggested by cascade simulations to be important, see below) to be explored. In the second type of experiment the affect of dilute alloying or impurity elements on the collapse process is of interest.

3.2.1 Cu-Ni and Ag-Pd alloys .

The experiments of Smalinskas et al [52] fall into the first of the above categories and are described in detail in another paper in this issue . In summary, Smalinskas et al found that vacancy loop production rates and vacancy retention in the Ag-Pd alloys scaled with melting temperature over the whole range of compositions: the higher the melting temperature the lower the defect yield and cascade efficiency. In the Cu-Ni alloys the defect yield also decreased as the melting temperature increased in the range Cu-30%Ni to Cu70%Ni. These trends are in agreement with the predictions of computer simulations, as discussed in section 4. However in the Cu-Ni system the behaviour for small (< 30%) or high Ni contents was more complex and was not dependent on melting temperature.

3.2.2 Dilute Ni alloys

The experiments of Vetrano et al [40,53] fall into the second category and are also described in another paper in this issue. These authors explored the effect on cascade collapse of small

additions of Si (0.6% and 4%) and Al (0.5%, 5% and 7%) to nickel. Irradiations were with 50 keV Kr^+ ions at room-temperature and at 30K. The presence of these substitutional alloying elements was found to affect strongly the collapse probabilities and loop sizes. At 30K the defect yield showed an initial increase followed by a decrease with increasing solute content. Experiments at 300K showed the same trends (table 2). Mean loop sizes were measured only in the room-temperature ex-situ experiments and showed the loops to be larger in the more concentrated alloys. The yields were considerably lower at 30K than for the corresponding alloy at 300K. The trends with alloying additions should be compared with the results of Stathopoulos et al [20] on dilute Cu alloys where again complex effects were seen (see section 1). Vetrano et al explain their results by invoking various impurity-caused changes to both the collisional and thermal-spike phases of the cascade. The specific mechanisms considered included defocussing of focusons by impurities to change the energy density profile of the cascade and interstitial trapping to reduce recombination during the thermal-spike phase.

3.2.3 Fe-Ni-Cr alloys

Tappin [54] has examined cascade collapse in a number of high-purity Fe-Ni-Cr alloys of varying Ni content (15-70at%). The Cr content was held constant at 15at%. Irradiations were at both low- and room-temperature with low-energy Bi^+ and 80KeV Au^+, W^+ or Xe^+ ions. Some defect yield and cascade efficiency values for the 15at%Fe15at%Cr70at%Ni alloy are shown in table 2 (see also ref[55]). They show trends with temperature and ion mass similar to pure materials. The more interesting aspect of this work- the dependence on the Ni content - has not yet been fully analysed.

3.2.4 Al-H alloys

Bui et al [47] have investigated the effect of small quantities of hydrogen (900 and 1300appm) on cascade collapse in Al under 50 keV Kr^+ ion irradiation. The results are shown in table 3. The presence of the hydrogen was found to decrease the defect yield markedly, although loop sizes and geometries were unaffected. It was speculated that the effect is due to the formation of hydrogen-vacancy complexes during the lifetime of the cascade.

3.2.5 Ti of different purities

Yellen et al [51] examined Ti of two purities, a high-purity iodide material containing 300ppm oxygen, 30ppm C, with <10ppm of other impurities, and a less pure commercial material containing 1100ppm oxygen, 600ppm C, 500ppm Al, plus smaller quantiies of other impurities. Irradiations were with Sb, Sb_2, and Sb_3 ions of energy 50-150 keV. The results are also shown in table 3. In contrast to Bui et al's results, yields were somewhat

higher for the less pure material. Other trends were as expected.

4. Discussion

Attempts have been made to interpret most of the experiments described above in terms of mechanisms seen in molecular dynamics simulations. The "cascade melting" model in particular has several attractive features which suggest that it might occur in actual materials, and so has been invoked to explain several of the trends found in the experiments described in sections 1 and 3:

(i) It can explain the large differences in defect yields and cascade efficiencies between materials such as Cu and Ni which have similar structures and atomic weights and where the ballistic phases of cascades are therefore similar. In the simulations for "Ni" the solidification front moved much more rapidly than in "Cu" as a result of its higher melting temperature and this led to less effective sweeping of vacancies into the cascade centre [24].

(ii) It provides a mechanism for athermal cascade collapse at low temperatures. The lower yields found at low temperatures are then explained as a consequence of the more rapid quenching of the thermal spike due to the larger temperature gradients. This has been shown explicitly in simulations [27].

(iii) It provides a natural mechanism for the very effective disordering found at the cores of cascade sites in ordered alloys such as Cu_3Au. In their study of cascades in Cu_3Au produced by Cu^+ ions Jenkins and Wilkens [14] estimated that the ratio of replacements to displacements is at least 10 and could be far higher. A large ratio is easily explained by local melting followed by recystallisation at a rate too fast to allow reordering. Other mechanisms of disordering may be operating at the cascade peripheries however.

(iv) Energy density effects are also explained naturally. In general more efficient vacancy clustering would be expected in more compact cascades as is usually observed. Molten zones would be less likely to form in the more diffuse cascades in light materials such as Al, Mg and Ti, and this is consistent with the low yields in these materials. The rather weak dependence of defect yields on the energy density in very compact high energy-density cascades in Au described in section 3.1.2 may be understood as a consequence of the size of the molten region exceeding the initial cascade volume. In this case the size of the molten region is governed by the total cascade energy. Rather direct evidence for this was presented by Jenkins and English [15] for high energy-density cascades in Cu_3Au produced by molecular Sb ions where the size of disordered zones was determined by the total ion energy rather than the energy per atom.

(v) The model may be capable of explaining at least qualitatively some of the impurity/alloying effects. Vetrano et al [40,53] have argued that disruption of focusson chains by impurity atoms will reduce the effective cascade volume leading to an increase in the extended cascade energy density. The resulting increase in temperature of the material immediately surrounding the molten zone decreases the solidification rate and so increases the effectiveness of the vacancy sweeping mechanism. In discussing the plausibility of this mechanism Vetrano et al assumed fairly arbitrarily an average focusson length of 30 atom chains. In current molecular dynamics simulations (which are however for much lower energy cascades) focussons appear to play a rather minor role and their average lengths are much shorter, so this mechanism must be treated with some caution.

(vi) Cascade overlap effects - annihilation of existing loops, loops apparently changing Burgers vectors, loop coalescence- are explained naturally. Loop dissolution is to be expected if an existing loop is engulfed by the molten zone of a new cascade [38].

(vii) Cascade melting also explains well the differences in atomic mixing between Cu and Ni which are observed experimentally[56].

The cascade melting model suggests that the melting temperature may be an important parameter in determining cascade collapse, and this is consistent with the experiments of Smalinskas et al [52]. This effect may have been exaggerated in the simulations because the potentials used predict a bigger difference in melting temperature than is in fact the case. In practice also, another effect not included in the simulations may be important. This is energy tranfer to the electronic system, via electron-phonon coupling, which can lead to effective "quenching" of the thermal spike. This has been the subject of some theoretical attention over the past few years [57-60] Flynn and Averbach [57] developed a model for treating ion-electron interactions in cascades and demonstrated that the effects could be significant. Calculations based on this model by Finnis et al and Caro and Victoria [59,60] found large differences in the cooling rates of cascades for different strengths of electron-phonon coupling. Cascades in Ni quench more rapidly because of the more effective coupling due to the higher density of states at the Fermi level. When the coupling was incorporated into molecular dynamics simulations the net effect was to inhibit defect production in low-energy cascades (fig 6). In the Cu-Ni alloy experiments the fact that the yield does not follow the melting temperature at high Ni content suggests that electron-phonon coupling may be playing a role.

Despite the success of the cascade molecular dynamics simulations in explaining many of the observed trends, it would be wrong at this point to place total reliance on their applicability. The "cascade melting" model is eminently plausible but whether cascade melting is absolutely required to explain the

experimental results is more debatable. As yet, several features of the results are not well understood. These include the low defect yields in bcc and hcp metals and the affect of impurities. The two may be related, because the yields are particulary low in materials where impurities, especially interstitial O, N, and C, are significantly more soluble, suggesting that these impurities may play an important role in governing point-defect survival and cascade collapse. It would be interesting to see if cascade simulations specifically aimed at these problems would support any of the mechanisms put forward to account for these affects. Also valuable would be simulations to yet higher cascade energies to see if the collapse process can be followed to vacancy loop production.

It is perhaps valuable to compare the results of the cascade collapse studies using low-dose ion irradiations described above with certain recent results reported for 14 MeV neutron irradiations to low doses, especially at low temperatures with cryotransfer for TEM purposes. The latter experiments are comparable with the in-situ ion-irradiation studies, also at temperatures near 30 K ; note that in both cases the irradiations are of thin foils. Several differences can be noted. The neutron experiments find defect cluster or loop populations in thin foils of Au and Cu at low temperatures (24 K), and no TEM visible defects in Ni or Al thin foils [61]. The nature of the low-temperature defects in Au and Cu was determined to be interstitial by the 2 1/2 D method. The computer simulations described in section 2 do suggest that interstitial loop generation within individual cascades may be a possibility. However, we have been unable to find any reported low-dose ion-irradiation experiment which claims to find interstitial loops, except those which have employed the 2 1/2 D method [eg 62], and recent work by Fukushima et al [63] which claims to confirm the accuracy of the 2 1/2 D method. These results which find interstitial loops in thin foils at low doses are in striking disagreement with earlier determinations of the loop nature using $\underline{1}$-vector analysis (the black-white contrast change with depth, measured by stereo-microscopy) applied by eg Häussermann [64], Wilson [65] and Stathopoulos [2,25]. In all these cases and many others all defects have been found to be of vacancy type. In other cases, eg the work on Ag and Cu by Jenkins [4], the vacancy nature of the damage was evident from the geometry of the observed defects -which were all partially dissociated Frank loops or stacking-fault tetrahedra. Theoretical work of Gruschel and Wilkens (unfortunately only published in thesis form [66]) indicates that the 2 1/2 technique can give misleading results. In the case of stacking-fault tetrahedra in Ag this has been shown explicitly: tetrahedra identified as interstitial by the 2 1/2 D method have been shown by other methods to be vacancy [67,68]. This causes us to view results based on the 2 1/2 D method with great caution. On the other hand both Ruault et al [62] and Fukushima et al [63] checked their 2 1/2 D results against those obtained from the $\underline{1}$-vector stereo method and report that both techniques gave identical proportions of vacancy and interstitial loops. The question of whether interstitial loops are formed at individual cascade sites therefore remains

controversial.

Other differences found when comparing ion-cascade results with 14 MeV neutrons include observations of loops in Ni under 50 keV self-ion irradiation at 30 K [8,37], but not under 14 MeV neutron irradiation [61]. Also, significant loop formation is observed under subthreshold electron irradiation in low-temperature neutron-irradiated Au, Cu, Ni and Al, while similar effects have not been found in low-temperature ion-irradiated Ni, even though the concentration of uncollapsed defect cascades is quite high in this case. We do not understand the reasons for these discrepancies.

As a final point we note that great care must be taken when employing the weak beam technique to defect microscopy. Robertson et al [37] found significant artifact contrast, which could easily be confused with defect contrast, in ion-irradiated Ni, which was often associated with a weak diffuse ring in the diffraction pattern. This was believed to result from either surface contamination or oxide structure. Interestingly, Shimomura et al [61] found similar "defect" contrast in weak-beam conditions. "Defect" contrast was also seen when a portion of a weak ring pattern was used for imaging in dark field. We believe that the possibility that these "defects" are artifacts cannot be excluded.

5. Conclusions

We have reviewed several recent ion-irradiation experiments which add to our knowledge of cascade collapse effects in metals. New cascade overlap effects have been identified. It has been shown that the observed dependencies of the collapse probabilities on the irradiation temperature, the cascade energy density and the ion dose can be understood within the framework of a local cascade-melting model which has been suggested by molecular dynamics simulations. Further simulations are suggested to try to elucidate the role of alloying elements and impurities in the collapse process, which is presently poorly understood. The formation of interstitial loops at individual cascade sites is still the subject of some controversy and further investigations would be worthwhile.

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Table 1

Recent data on vacancy loop formation in some pure metals

Metal	Ion	Energy (keV)	Irradiation temp. (K)	Defect yield Y	Cascade efficiency ε	Ref.
Ni	Ni^+	50	30	0·05 ± 0·01		[37]
		50	300	0·14 ± 0·01		
		100	300	0·07 ± 0·01		
	Kr^+	50	30	0·12 ± 0·01		
		50	300	0·21 ± 0·02		
Au	Bi^+	10	300	0·25		[43]
		20	300	0·33		
	Bi_2^+	20	300	0·5		
		40	300	0·73		
Au	Bi^+	10	4	0·03	1·55	[44]
		10	285	0·05	1·27	
Cu	Cu^+	10	4	0·01	0·32	
		10	285	0·07	0·59	
Fe	Fe^+	50	30	~ 0·001		[8,39]
		100	30	~ 0·001		
		100	300	~ 0·001		
V	W^+	80	300	0·12 ± 0·01	0·20 ± 0·05	[48]
Ti	Sb^+	100	300	0·07 ± 0·02	0·19 ± 0·02	[50]
		150	300	0·06 ± 0·03	0·08 ± 0·01	
Co	Sb^+	100	300	0·14 ± 0·04	0·51 ± 0·03	
		150	300	0·24 ± 0·03	0·40 ± 0·02	
Ru	Sb^+	100	300	0·36 ± 0·07	0·24 ± 0·01	
		150	300	0·41 ± 0·04	0·23 ± 0·02	
Re	Sb^+	100	300	0·31 ± 0·06	0·18 ± 0·01	
		150	300	0·36 ± 0·07	0·17 ± 0·01	
Mg	Sb^+	150	300	< 0·01		

Table 2

Recent data on vacancy loop formation in some alloys

Alloy	Ion	Energy (keV)	Irradiation temp. (K)	Defect yield Y	Cascade efficiency ε	Ref.
Ni	Kr^+	50	30	0·12 ± 0·01		[53]
			300	0·21 ± 0·02		
Ni0·5at%Al	"	"	30	0·13 ± 0·01		
			300	0·34 ± 0·01		
Ni0·6at%Al	"	"	30	0·09 ± 0·01		
			300	0·23 ± 0·01		
Ni5at%Al	"	"	30	0·07 ± 0·01		
Ni4at%Si	"	"	30	0·06 ± 0·01		
15Fe15Cr70Ni	Bi^+	20	8	0·26 ± 0·05	0·46	[54]
	"	"	300	0·45 ± 0·05	0·52	
	Xe^+	80	300	0·52 ± 0·05	0·14	
	W^+	80	300	0·53 ± 0·05	0·17	

Table 3

Effects of low levels of impurities. Room temperature irradiations.

Material	Ion	Energy	Defect yield	Ref.
Al	Kr^+	50	0·06 ± 0·01	[47]
Al-900appmH			0·05 ± 0·01	
Al-1300ppmH			0·04 ± 0·01	
Ti-iodide purity*	Sb^+	50	0	[51]
		100	0·06	
		150	0·04	
	Sb_2^+	50	0·42	
		100	0·43	
		150	0·51	
	Sb_3^+	50	0·66	
		100	0·69	
		150	0·84	
Ti-commercial purity*	Sb^+	50	0	
		100	0·07	
		150	0·06	
	Sb_2^+	150	0·67	
	Sb_3^+	100	0·77	
		150	1·01	

*Orientation S_1, yields corrected for loop loss to surfaces.

Figure captions

Figure 1: "Cascade dissolution". Micrographs of the same area of a Ni specimen (a) irradiated at room-temperature with 100keV self-ions to a dose 3.4 x 10^{16} ions m^{-2}, and (b) after further irradiation to a total dose of 4.5 x 10^{16} ions m^{-2}. Loops marked with arrowheads are visible in (a) but absent in (b). After ref [37].

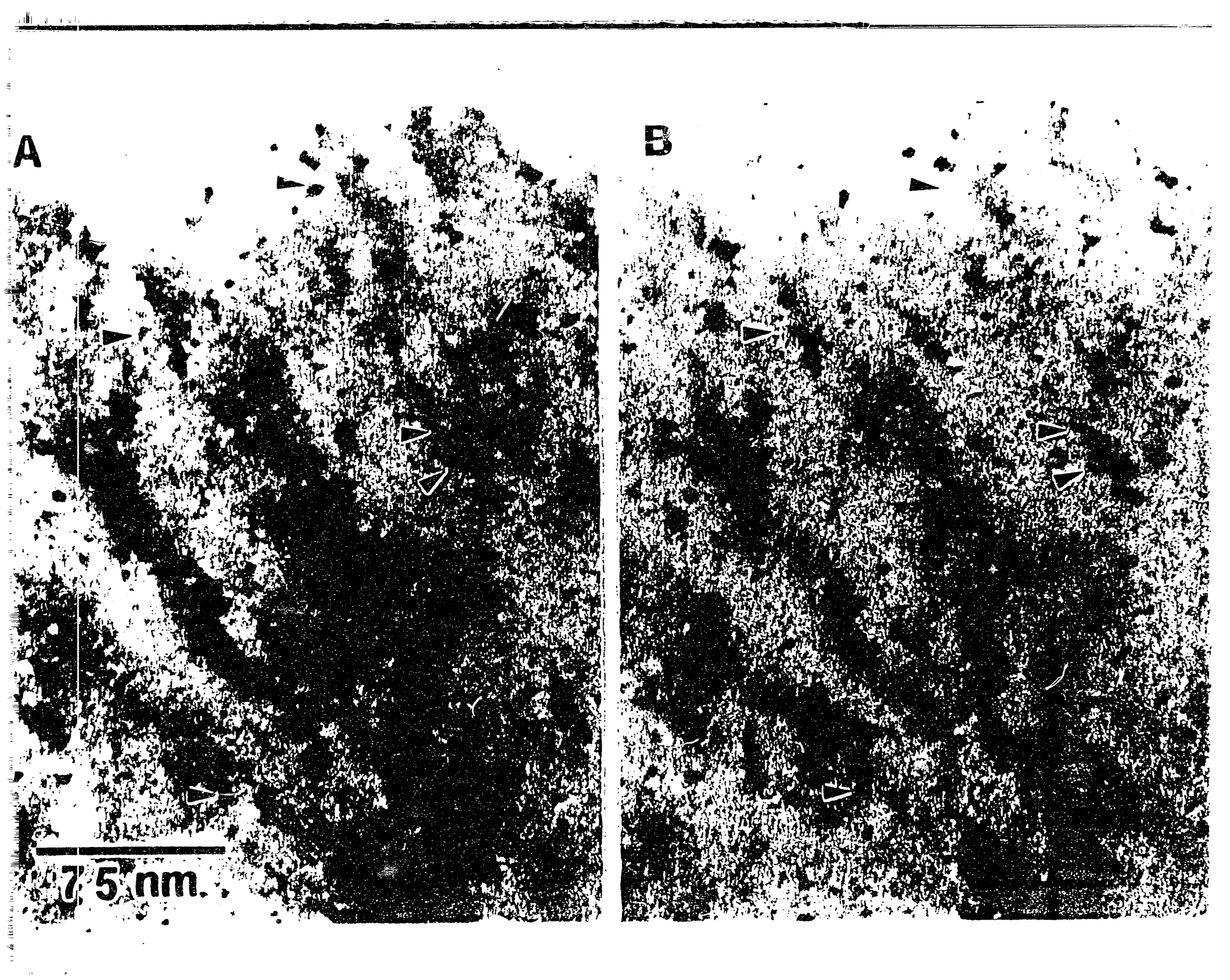
Figure 2: Molecular dynamics simulation of a 1 keV cascade in "Cu" impinging upon a pre-existing 19-vacancy loop. The position of the primary event is marked with an arrowhead. The loop is engulfed and dissolved. A loose cluster of vacancies is produced but no new loop forms. After ref [42].

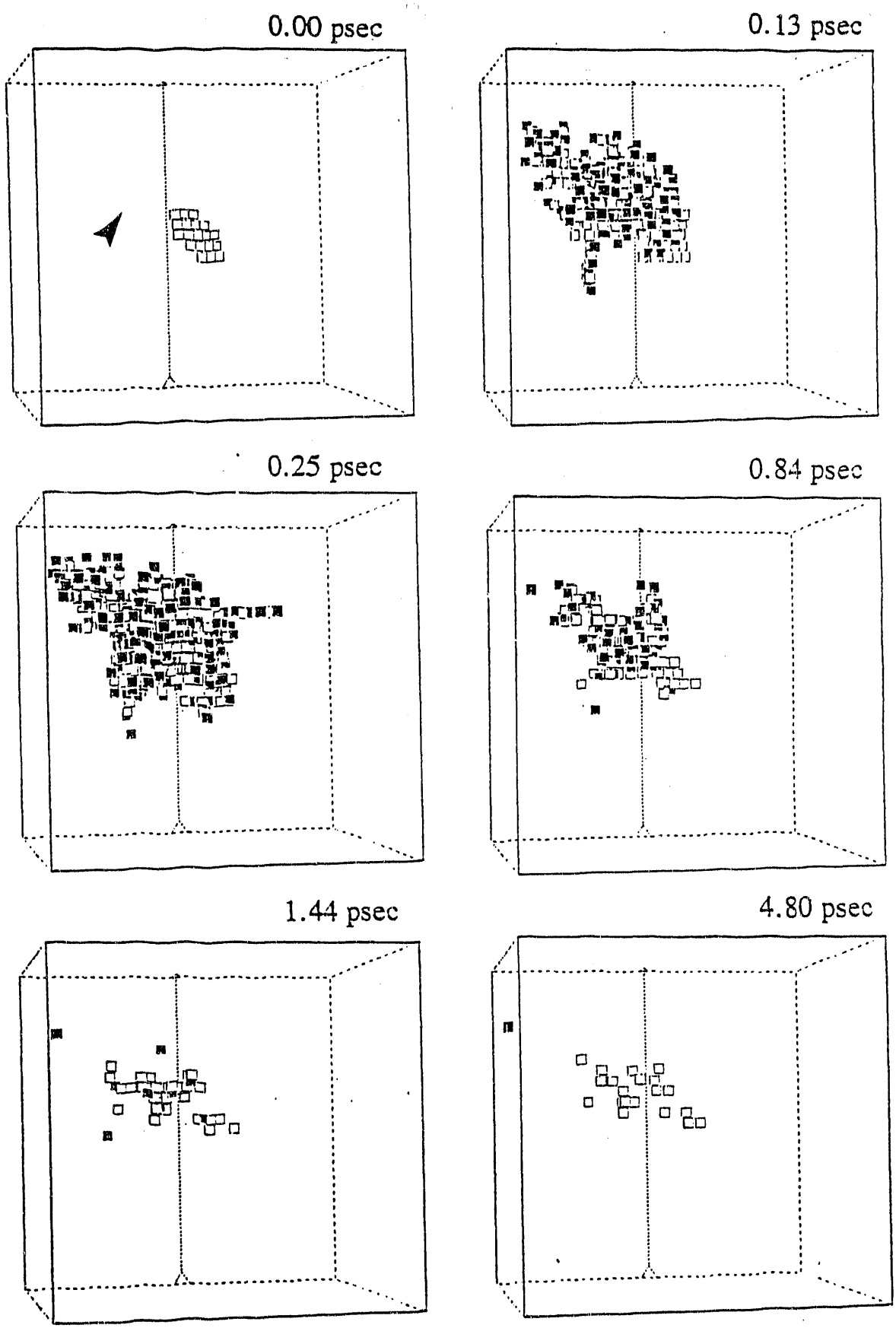
Figure 3: Comparison of loop production as a function of dose in Cu, Ni and Fe during 100 keV self-ion irradiations at room-temperature. The slope of n=1 at low doses in Ni and Cu indicates cascade collapse at individual cacade sites. Note deviation from the n = 1 lines at high doses due to overlap effects. Loops are formed in Fe only at high overlap doses. After ref [8].

Figure 4: Comparison of loop production as a function of dose in Ni and Fe during 50 keV self-ion irradiations at 30K. Note the difference in slopes. After ref [8].

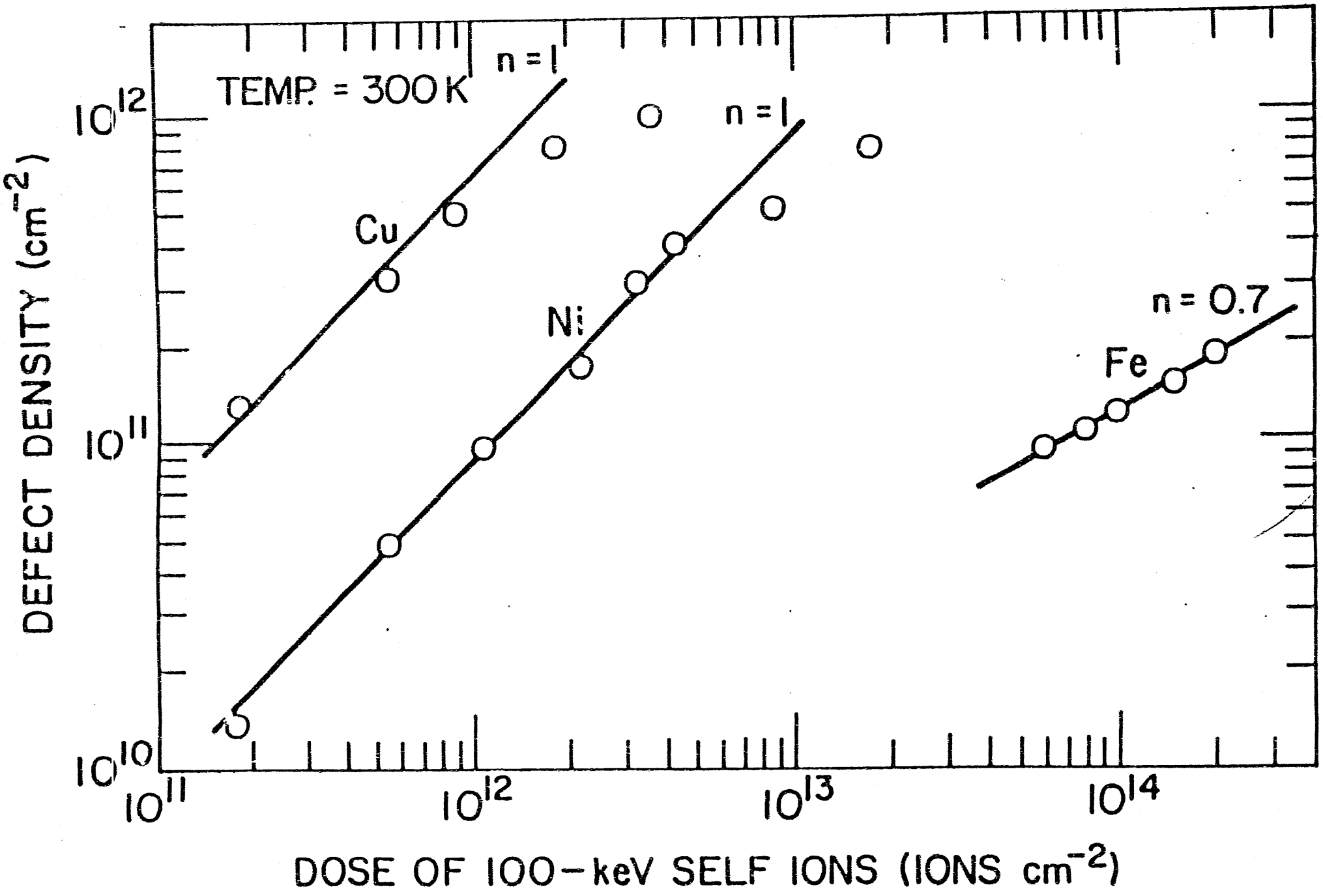
Figure 5: Defect yield in Ru as a function of ion energy for Kr^+ ions (square symbols) and W^+ ions (diamond symbols). For either ion the yield increases with energy. However there is a reversal in the dependence of yield on ion mass on going from low to high ion energy. After ref [49].

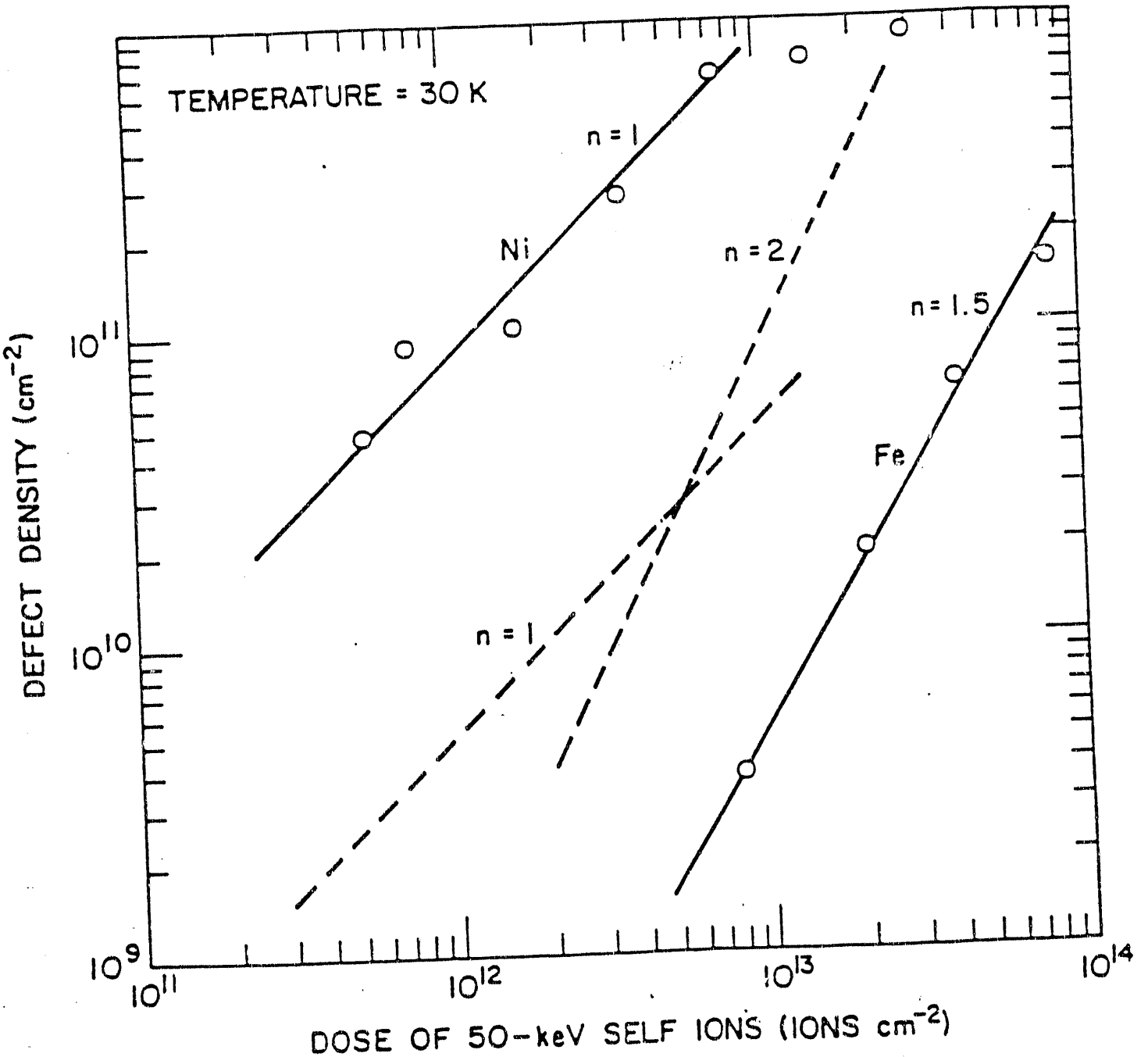
Figure 6: Molecular dynamics simulations showing the effects of different electron-phonon coupling strengths on the evolution of a 500eV cascade in "Cu". Interstitials are shown as full squares and vacancies as open squares. The left-hand column shows the defect configuration 0.2ps after the primary event, whilst the right-hand column is after 7ps. The coupling strengths used correspond to (a) zero coupling,(b)Cu, (c) ten times Cu (or one-third Ni), and (d) Ni. Note the large difference in remnant defects between (a) and (d). After ref [59].

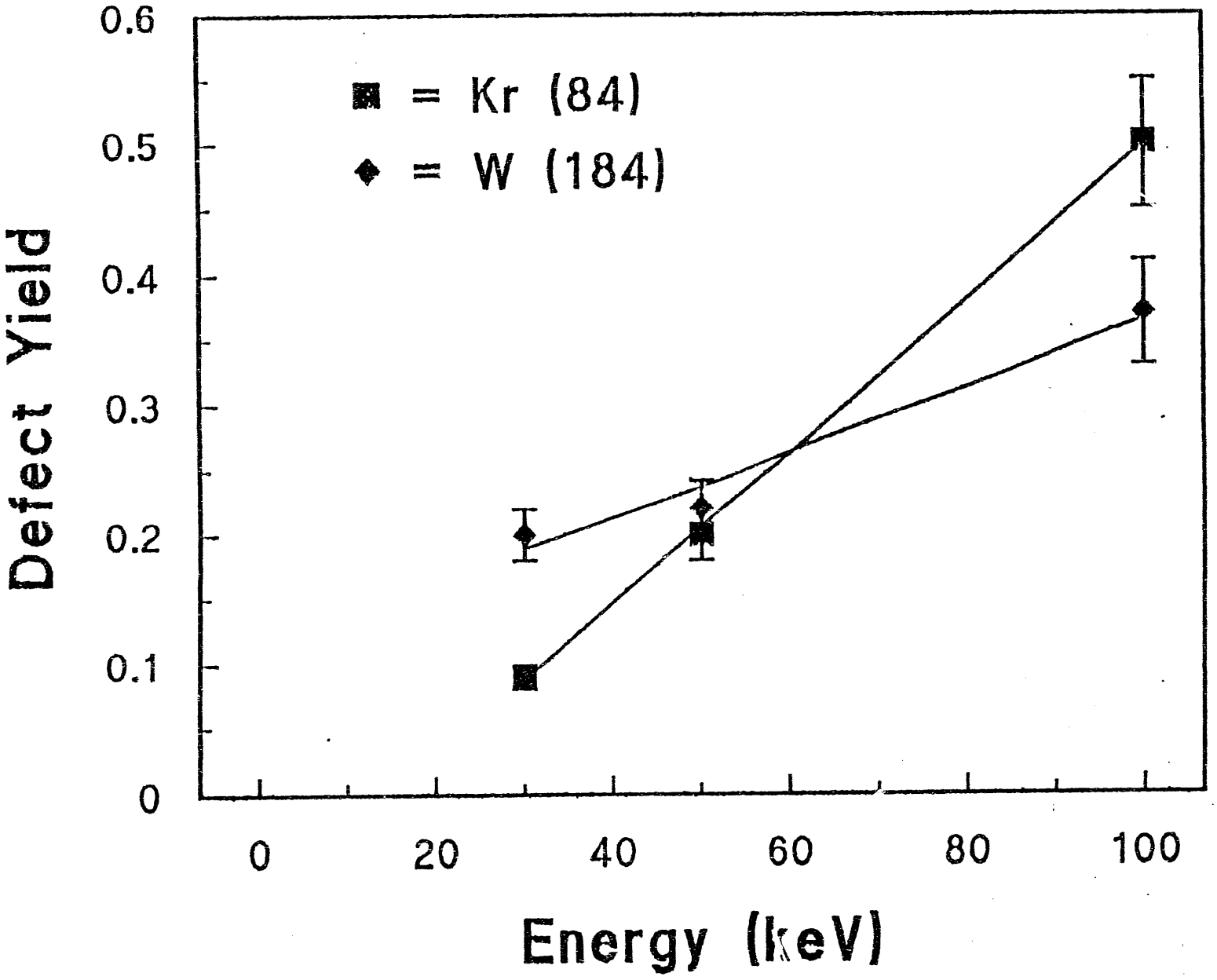

A
B
75 nm



1 keV CASCADE onto VACANCY LOOP


TEMP. = 300 K
n = 1
n = 1
n = 0.7
Cu
Ni
Fe
DEFECT DENSITY (cm^{-2})
10^{12}
10^{11}
10^{10}
10^{11}
10^{12}
10^{13}
10^{14}
DOSE OF 100-keV SELF IONS (IONS cm^{-2})


TEMPERATURE = 30 K
n = 1
Ni
n = 2
n = 1.5
n = 1
Fe
DEFECT DENSITY (cm^{-2})
10^{11}
10^{10}
10^{9}
10^{11}
10^{12}
10^{13}
10^{14}
DOSE OF 50-keV SELF IONS (IONS cm^{-2})


0.6
0.5
0.4
0.3
0.2
0.1
0
Defect Yield
■ = Kr (84)
◆ = W (184)
0
20
40
60
80
100
Energy (keV)

(a)
(b)
(c)
(d)
(a)
(b)
(c)
(d)

END

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