

EFFECTS OF THE PRIMARY RECOIL SPECTRUM ON MICROSTRUCTURAL EVOLUTION*

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MASTER

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Abstract

For quantitative predictions and comparisons of microstructures that evolve during exposure to different radiation environments at elevated temperature one needs to develop methods that go beyond those based on the number of displacements per atom. The number of freely migrating defects that contribute to the microstructural development is far less than the total number of defects produced, as has been recognized for some time from measurements of radiation-induced segregation and of radiation-enhanced diffusion. One major reason for the small amount of defects available for long range migration is the high concentration and close spatial correlation of vacancies and, to a somewhat lesser degree, of interstitials in cascades produced by high energy knock-ons. As a consequence, many defects either recombine or form immobile defect clusters during the defect formation and cooling phases of the cascades. After doses exceeding a few tenths of a displacement per atom, the residue of small clusters and dislocation loops of vacancy type remaining in the central portions of energetic cascades and subcascades, is the second major reason for the reduction of the mean free path of defects between creation and annihilation. Defect production in various neutron and ion irradiation environments is discussed in light of these facts. A method to calculate the fraction of freely migrating defects from the cluster size distribution of defects produced in cascades is suggested. The results are in good agreement with available data.

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1. INTRODUCTION

Microstructural changes occur as a consequence of irradiation with energetic particles in a number of distinct ways. Although the underlying processes can be defined conceptually quite well, several of them almost invariably are active simultaneously under experimental conditions. It has become customary to distinguish the following processes: (1) displacement mixing, (2) recoil dissolution, (3) sink nucleation (vacancy and interstitial loops, void embryos), (4) void and dislocation network growth, (5) radiation-enhanced diffusion, (6) radiation-induced segregation, (7) radiation-induced precipitation and (8) radiation-induced phase changes. The major effects of the primary recoil spectrum on these processes and, hence, on the microstructural evolution during irradiation, are related to the initial defect distribution produced in displacement events. Atomic rearrangements within the cascade volume during the thermal spike or cooling phase are of importance to such processes as amorphization, defect clustering, and cascade collapse. However, many of the major microstructural changes, such as the development of voids, dislocation networks and radiation-induced precipitation, that do occur at temperatures at which point defect mobility is significant, depend on long range defect migration, i.e., over distances large compared to typical cascade dimensions of a few tens of nanometers.

Certain phase changes, i.e., order-to-disorder and crystalline-to-amorphous transformations, are primarily induced by irradiations at relatively low temperatures and with limited defect mobility. Therefore, the primary

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recoil spectrum can have significant effects on these transformations. This has been most strikingly illustrated by Seidman et al. on silicon.¹ Si can be amorphized easily by neutrons as well as by energetic ions at room temperature or somewhat elevated temperatures with doses less than a few tenths of a displacement per atom (dpa). However, it can not be amorphized by electron irradiations at temperatures as low as 10 K to doses as high as 14 dpa. Seidman et al. have shown that silicon retains its crystallinity under 1 MeV Kr⁺ ion bombardment if it is simultaneously irradiated with 1 MeV electrons at fluxes that generate a displacement rate at least half of that produced by the Kr ions.¹ Thus, the additional Frenkel pairs, randomly produced by the electrons, "heal" the damage structure induced by the displacement cascades sufficiently to prevent the transformation to the amorphous state. This provides direct evidence that the primary recoil spectrum affects the microstructural development; in this case, the addition of low energy primary recoils at sufficient rate prevents amorphization that normally would be induced by the predominantly large cascades resulting from 1 Mev Kr⁺ irradiation.

Whereas the first two processes mentioned above, i.e., displacement mixing and recoil dissolution, are clearly linked to the cascade event and, therefore, to the primary recoil energy and the density of energy deposition, sink nucleation (vacancy and interstitial loops, void embryos) may occur either during an individual cascade event or as a consequence of the general supersaturation of mobile point defects, possibly, in conjunction with impurities or transmutation products. The development of the sink structure, non-equilibrium redistribution of alloying elements, radiation-induced precipitation and the acceleration of precipitation of stable or metastable phases are related to the presence of excess mobile point defects and their fluxes. These, in turn, are determined by the rate at which mobile defects are released from cascades into the general crystalline background and the rate of loss by the recombination of freely migrating interstitials and vacancies, and their annihilation at defect sinks.

Clearly, a complete and accurate description of microstructural development during irradiation would require a simultaneous treatment, in time and space, of the defect production process, the nucleation and growth of

defect sinks, the spatial redistribution of alloying elements within the matrix and precipitated phases, and the elimination of mobile defects by recombination, and by annihilation at defect clusters and other sinks. Such comprehensive treatment of microstructural evolution has not been attempted and seems to be beyond the present state of art. Therefore, void swelling and other microstructural processes, such as radiation-enhanced creep or precipitate coarsening, have been treated by using simplifying assumptions, most commonly those of rate theory. The key assumption is the establishment of a quasi-steady-state in the medium with regard to defect concentrations, determined by the balance between production of defects by irradiation and their loss by recombination and at sinks. The microstructural process of interest is then treated as occurring in this medium. For short reviews of the method and its applications see, e.g., Bullough and Wood², and Wiedersich³.

The effects of the primary recoil spectrum on the microstructural developments at elevated temperatures are majorly related to its effect on the production of freely migrating defects, although spectral effects may also influence the nucleation of the sink structure, e.g. by creation or dissolution of void embryos in large cascades. Since the latter effects are likely to be small and have been little explored, we will mainly concentrate here on the effects of the primary recoil spectrum on the production of freely migrating defects. The often used comparison of microstructural effects based on the concept of displacements per atom is not a good approximation because this quantity is calculated from the total energy deposited in nuclear collisions and a modified Kinchin and Pease expression, thus ignoring the differences in spatial distribution of the defect production entirely. We will use the next better approximation, i. e., include an efficiency factor for freely migrating defects based on the cluster size distribution produced by the primary recoil spectrum.

In the following section we discuss at first the cascade characteristics which determine the spatial distribution of defects after the cascade event. In section three, the concept of the cluster size distribution and the weighted primary recoil spectrum will be outlined. The present state of

knowledge of the production of freely migrating defects will be reviewed in the subsequent section followed by some general conclusions.

2. CASCADE CHARACTERISTICS

The major effect of the primary recoil spectrum on the initial defect distribution can be described as follows. At primary knock-on energies between about the threshold displacement energy, E_d , and $2.5 E_d$, isolated Frenkel pairs are created. As the energy increases, multiple interstitial atoms and vacancies are produced, generally with the vacancies in closer proximity to the location of the primary knock-on than the interstitials. Also, more intra-cascade rearrangements of defects occur during the "cooling phase" as the energy increases from a few tenths to a few tens keV.^{4,5} This leads to defect recombination as well as to clustering of like defects in the form of dislocation loops, stacking fault tetrahedra or other three-dimensional clusters.

The number of defect pairs produced by a primary recoil atom of energy P , $N(P)$, can be described by the modified Kinchin-Pease expression as given by Norgett et al.⁶ :

$$N(P) = \begin{cases} 0 & \text{for } 0 < P < E_d \\ 1 & \text{for } E_d < P < 2.5 E_d \\ 0.8 E_D(P)/(2 E_d) & \text{for } 2.5 E_d < P < P_{\max} \end{cases} \quad (1)$$

where E_d is the average threshold energy, and $P_{\max} = E(4m_1m_2)/(m_1 + m_2)^2$ is the maximum energy that can be transferred from the incoming ion of energy E and mass m_1 to target atoms of mass m_2 . $E_D(P)$ is the damage energy, i.e., the energy dissipated in nuclear collisions (P less the electronic energy losses).

When the primary recoil energy exceeds about 20 keV (corresponding to about 150 defect pairs), the cascades tend to break up into clusters of sub-cascades as first pointed out by Merkle.⁷ These defect structures, visible by electron microscopy, are fairly stable up to temperatures approaching significant self-diffusion. English and Jenkins⁸ have recently reviewed

cascade formation and structures induced by ion and neutron irradiation. The number of observable defect clusters in Cu and Mo, e.g., are essentially independent of the irradiation temperature up to about 500 and 450K, respectively, and then drops more or less quickly to zero at higher temperatures for both ion and neutron irradiations, thus indicating that defect clusters produced by cascades are unstable with respect to thermal decomposition at higher temperatures.

Kiritani reported on extensive investigations of defect clusters produced by 14 MeV neutrons from RTNS II in a series of metals and alloys.⁹ Some important observations are: The size distribution of disordered zones in Cu₃Au extends from about 2 to 20 nm in diameter with a maximum at 5 nm. Kiritani relates the sizes of the disordered zones to the energy of the primary knock-on atom responsible for the cascade with the zone size increasing from below 2 nm for PKA energies of a few keV to 20 nm for the maximum PKA energy of about 900 keV. The defect cluster size distribution, as contrasted to the disordered zone size, extends only to about 6 nm and peaks at about 4 nm. This observation is consistent with a break-up into subcascades contained within the larger disordered zones. Kiritani and coworkers (see Ref. 9) have studied the break-up of cascades in 14 MeV neutron irradiated Au in some detail and come to the conclusion that the energy of highly energetic PKAs is distributed over an increasingly larger number of subcascades with an average energy of 17 keV within a subcascade cluster.

Kiritani and coworkers have estimated from measurements of the size distributions of stacking fault tetrahedra in Au that only about 13 to 25% of the defects created in the cascades survive recombination during the cascade lifetime.⁹ This fraction appears to decrease at irradiation temperatures above 150° C (423K). The number of visible defect clusters after irradiations at 290° C (563K), e.g., is markedly lower, indicating increased in-cascade recombination. However, the visible defect cluster population also tends towards saturation with increasing dose, or is less than linearly proportional to the dose, which shows that interstitials from subsequent cascades are at least partially responsible for the decreased defect survival.

The cluster formation of defects in cascades has also been studied by computer calculations, e. g., by Muroga and Ishino ¹⁰ and by Heinisch and Mann ^{11,12}. The methods are to calculate the initial defect distributions in cascades by the binary collision code MARLOWE followed by short term annealing during and after the cooling phase. With sets of reasonable input assumptions results consistent with experimentally observed cluster characteristics were obtained.

3. Primary Recoil Spectrum

As pointed out earlier, the fraction of defects which escapes at elevated temperature from individual cascades will depend on the distribution of defects within the cascade and, therefore, on the energy of the primary recoil atom which created the cascade. The fraction of primary recoil atoms (PKAs) or, therefore, the fraction of defect clusters, produced by the bombarding ion (or neutron) of energy E , with PKA energies below a recoil energy P , can be represented as (see, e. g., Averback et al.¹³) :

$$F(P, E) = [1 / M_t(E)] \int_{E_d}^P (ds(E, P') / dP') dP' \quad (2)$$

where

$$M_t(E) = \int_{E_d}^{P_{\max}} (d s(E, P') / dP') dP' \quad (3)$$

Here, $ds(E, P') / dP'$ is the differential cross section for an incoming particle of energy E to produce recoils with energies between P' and $P' + dP'$. The integral in eq. (2) gives the number of PKAs with energy $< P$ created by the bombarding particle, whereas eq. (3) gives the total number of PKAs produced by the incoming projectile.

Since the average number of defects produced by a primary recoil atom is a unique function of P for a given target material (see eq. (1)) we can convert $F(P, E)$ into $F(N, E)$, i.e., the fraction of clusters of all sizes produced below

size N . $F(N)$ is shown as function of cluster size in Fig. 1 for nickel irradiated by several different bombarding ions of energy $E=2$ MeV; note that N is plotted logarithmically on the abscissa. An average threshold displacement energy of 40 eV was used. The calculations for all curves for the ions shown were performed with the computer code PINTO written by R. Benedek using the modified Kinchin-Pease damage function, eq.(1), Lindhard electronic stopping and Thomas-Fermi-Lindhard nuclear stopping. A description of the methods used can be found in the paper by Averback et al.¹³ Included in Fig.1 are also the integrated cluster size distributions, $F(N)$, for several neutron spectra, appropriately averaged over the corresponding neutron energy distributions.* The calculations for the neutron spectra were kindly provided by Greenwood using the computer code SPECTER.¹² A description of the methods used in the damage parameter calculations was given by Kirk and Greenwood.¹⁵

As the mass and, to a lesser extent, the energy (not shown) of the incoming ion increases, the primary recoil spectrum becomes harder, i. e., an increasing fraction of the defects is produced in large cascades and subcascades by high-energy recoils. The distributions of recoil energies for the neutron spectra used for Fig. 1 are harder than those for the ions and, hence, even a larger fraction of defects is generated in large clusters. Especially noteworthy is the hardness of the recoil spectrum for 14 MeV neutrons which produces approximately half of the defect clusters in sizes of more than 1000 Frenkel pairs. It should be kept in mind, however, that large cascades tend to break up into clusters of smaller subcascades.

To characterize the primary defect state, i. e., the state that would prevail in the material immediately after the cascade event (including the cascade cooling phase), but before any significant long range thermal migration takes place, Averback et al.¹³ introduced the concept of the primary recoil spectrum weighted by the total number of defects produced by recoils of

*The choice of the integrated quantity eliminates much of the fluctuations in the corresponding distribution function caused by nuclear reactions in the case of neutrons or high energy ions.

energy, P . This quantity is the fraction of defects, $W(P, E)$, produced by all primary recoil atoms with energies less than P . $W(P, E)$ is given by

$$W(P) = [1/N_t(E)] \int_{E_d}^P N(P') (ds(E, P')/dP') dP' \quad (4)$$

where

$$N_t(E) = \int_{E_d}^{P_{\max}} N(P') (ds(E, P')/dP') dP' \quad (5)$$

Here, $N(P')$ is the number of defect pairs resulting from a PKA with recoil of energy P' , eq.(1), and $ds(E, P')/dP'$ is the differential cross section as before. The integral in eq.(4) gives the number of Frenkel pairs created by all primary recoils with energies $< P$, whereas eq.(5) gives the total number of Frenkel pairs produced by the incoming projectile. Again we convert $W(P)$ to $W(N)$, the fraction of defects produced in clusters of sizes $< N$.

$W(N)$ is shown as a function of cluster size in Fig. 2 for nickel bombarded by several ion species of 2 MeV energy and by neutrons of several energy spectra. This figure illustrates that only a rather small fraction of defects is created in small clusters, e.g., below $N \sim 30$, for neutron and heavy ion irradiations whereas the majority of defects formed during light ion bombardment are generated in small clusters. The fraction of defects generated by ions in small clusters decreases initially rapidly as the mass of the bombarding ion increases but decreases rather slowly when the mass of the incident ion exceeds that of the target ion (compare the change from H to Li with that from Ni to Au). It is also clear from Fig. 2 that fewer defects are produced in small clusters for all the neutron spectra used than for any of the ion irradiations illustrated.

It should be noted that details of the spectral distribution of PKA energies can be important as becomes apparent from comparison of the curves for EBRII and for Starfire in Figs. 1 and 2: although many more small clusters would be produced in a spectrum anticipated for a fusion reactor (Starfire) than in EBRII, the fraction of defects created in large clusters would be

much larger in Starfire than in EBRII. This is a consequence of large components of low as well as high energy neutrons in the spectrum for Starfire compared to that of EBRII.

4. Long Range Migrating Defects

It has been long recognized that only a fraction of the defects initially produced in cascades survive even during irradiation at low temperatures. For example, Averback et al.^{13,16} have found from resistivity measurements that the fraction of surviving defects in ion bombarded specimens decreases rapidly as the mass of the projectile increases. The defect production efficiency in copper, normalized to that of H, decreases to about 30% as the mass is increased from that of H to that of Ar, and then remains at this level to ion masses as large as that of Bi. Similar results have been found for other materials. Rehn et al.¹⁷ and Hashimoto et al.¹⁸ have determined the efficiency for production of defects undergoing long range migration during elevated temperature irradiations from measurements of the kinetics of radiation-induced segregation in Ni-Si and Cu-Au alloys, respectively. Qualitatively, the results for the efficiency during elevated temperature irradiation are rather similar to those for low temperature irradiation except that the magnitude of the efficiency decrease is significantly larger at the higher temperatures. Relative to H, the efficiency decreases to only a few percent for Ne, Ni or Kr ions.

A reasonable interpretation of the decreases in efficiencies with increasing mass of the projectile can be based on the defect cluster size distributions resulting from the irradiations. Recombination of defects within cascades should increase rapidly with increasing in cluster size. At low temperature, recombination and clustering of like defects is limited to the "lifetime" of the cascade ($\sim 10^{-11}$ s) and the remaining defect density can be relatively high. At elevated temperatures, continuing thermal migration under mutual interactions among closely spaced defects will lead to additional recombination within the cascade. In addition, defects will annihilate at defect clusters remaining from other cascades.

In order to describe the efficiency of defect production empirically in a simple quantitative way, we will assume that the probability, $p(N)$, of a defect surviving recombination within a cluster decays exponentially with cluster size:

$$p(N) = (1 - p_\infty) \exp[-(N-1)/N_d] + p_\infty \quad (6)$$

where N is the number of Frenkel pairs produced in the cluster, p_∞ is the fraction of defects that survives from a large cluster (typically that of an average subcascade) and N_d is a decay size or length. We expect that this size is larger for low temperature than for high temperature irradiations since the defect distribution within the cluster becomes frozen-in at low temperature after the kinetic energy liberated in the cascade is dissipated in the surrounding crystal. Equation (6) is just a crude approximation to describe with two parameters the general features of defect survival one expects. It assigns a survival probability of 1 to Frenkel pairs that are produced isolated in low energy events. The probability decreases to a finite value as the number of defect pairs created in close proximity increases. This reflects the fact that the cascades from high energy recoils break up into a number of spatially somewhat separated subcascades.^{7,9} This break-up occurs as the cluster size exceeds approximately 100 to 200 Frenkel pairs, but has not been taken into account in the calculations of the primary and weighted primary recoil spectra. Equation (6) has a simpler form than the relations suggested by Doran et al.¹⁹ from fitting cascade annealing results obtained by computer simulations.

The decay length has been chosen as 5 defect pairs in a cluster and p_∞ to be 0.5% in order to be consistent with the experimental results of a few percent surviving defects during high temperature irradiations with heavy ions. Figure 3 illustrates that eq. (6), with the chosen values for $p_\infty=0.005$ and $N_d=5$, can give a fair representation of the experimental results obtained by Rehn et al. and by Hashimoto et al.^{17,18} from radiation-induced segregation in Ni-Si and Cu-Au alloys. The drop-off of the free fraction is somewhat steeper than calculated, indicating perhaps that the decay size should be chosen even smaller than 5. Macht et al. have derived fractions of freely migrating defects from measurements of radiation enhanced diffusion in nickel and in a fcc Fe-20Cr-20Ni alloy that are similarly low, 1.5 to 2.5%.^{20,21}

Figure 4 shows the calculated fraction of defects that migrate long range in nickel bombarded with a number of different 2 MeV ions and several neutron spectra, using $p_{\infty}=0.005$ and $N_d=5$. The fraction of freely migrating defects decreases from about 75% for H, via 8% for Ni to 4.5% for Bi with average cluster sizes of 1.6, 17.4 and 31.9 Frenkel pairs, respectively. Although the surviving defect fraction decreases rather smoothly with increasing average cluster size, or similarly with average, or weighted average recoil energy or any other single quantity reflecting the "hardness" of the particular recoil spectrum, none of these quantities is a very good measure for the total effect of the recoil spectrum. This shows, e.g., in Fig 3 for the points for EBRII and Starfire, reflecting details in the characteristic spectra.

The low overall survival rate of defects for long range migration is not likely to result solely from correlated annihilation of defect within the cascades of their origin. The low temperature, low dose results by Averback et al. indicate approximately 30% of the defects survive in-cascade recombination. Simulation of annealing of isolated cascades by Heinisch, e.g., indicate that at least 14% of the interstitials leave the cascade region as single interstitials escaping recombination or interstitial clustering.^{11,12} Thus, significant defect loss must be occurring as a result of inter-cascade losses.

A simple model can be developed by realizing that the vacancy clusters which are observed to form directly within cascades are sinks for defects regardless of the location of their creation. As a consequence of the stronger clustering of vacancies than interstitials in the formation of cascades, an excess of free interstitials arrives at these vacancy clusters which leads to the dissolution of the clusters even in the absence of thermal vacancy emission. Hence, these clusters have a finite life time, and a quasi-steady state will develop which includes a distribution of vacancy clusters in addition to the excess concentration of mobile point defects. Because of the finite cluster life time, no lasting microstructural changes, such as radiation-induced precipitation will persist at any individual cluster location. Using the effective medium approach developed by Brailsford and Bullough¹⁸ the life time of the clusters can be calculated

from standard rate theory. It can be shown that the life time of a cluster, t_{cl} , is given by

$$t_{cl} = (2/3 \pi^2)^{1/3} n_{v0}^{2/3} p/K(F_i - F_v) \quad (6)$$

neglecting thermal evaporation. Here n_{v0} is the number of vacancies in the cluster as formed in the cascade, p the total sink strength, including the clusters, K the defect production rate, and F_i and F_v the average fraction of interstitials and vacancies, respectively, that escape intra-cascade recombination and clustering. The life time of vacancy clusters is shown as a function of cluster size in Fig. 5. We note that life time is inversely proportional to the defect production rate. Since the cluster production rate is proportional to K for a given spectrum of irradiating particles, a flux-independent steady state cluster density results for a fixed sink density. F_i and F_v are by nature characteristic for the spectrum but flux independent. Cluster densities of $\sim 5 \cdot 10^{-6}$ (in atom fractions) and annihilation of $\approx 25\%$ of the defects on clusters are estimated for an average cluster size of 25 vacancies, indicating that the difference in the survival fraction of defects at low and high temperature can be rationalized in terms of intercascade annihilation at vacancy clusters.

5. Conclusions

Microstructural changes that occur during irradiation of materials at elevated temperatures are predominantly caused by long range migration of defects produced by the irradiating particles. Under most neutron and ion irradiation conditions a significant fraction of the defects is generated in cascades and clusters of subcascades. The high density of defects following their production by primary knock-on atoms leads to large defect losses within cascades. Therefore, predictions of microstructural developments can not be made from those determined in a different radiation environment simply on the basis of calculated doses in terms of displacements per atom. An improved way of calculating the fraction of defects that migrate freely has been suggested. The method is based on a probability that defects escape from the defect cluster in which they are produced or from annihilation at residual vacancy clusters that decreases exponentially with the

number of defects in the cluster to a small residual value. The defect cluster size distribution can be calculated for neutron spectra and ion irradiations with standard computer codes. The predictions of the suggested method agree well with available experimental determinations of freely migrating defect fraction.

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Figure Captions

Fig. 1. Fraction of primary knock-on atoms producing defect clusters in Ni of sizes smaller than N as function of cluster size for several different incident ions of 2 MeV energy and for several neutron spectra.

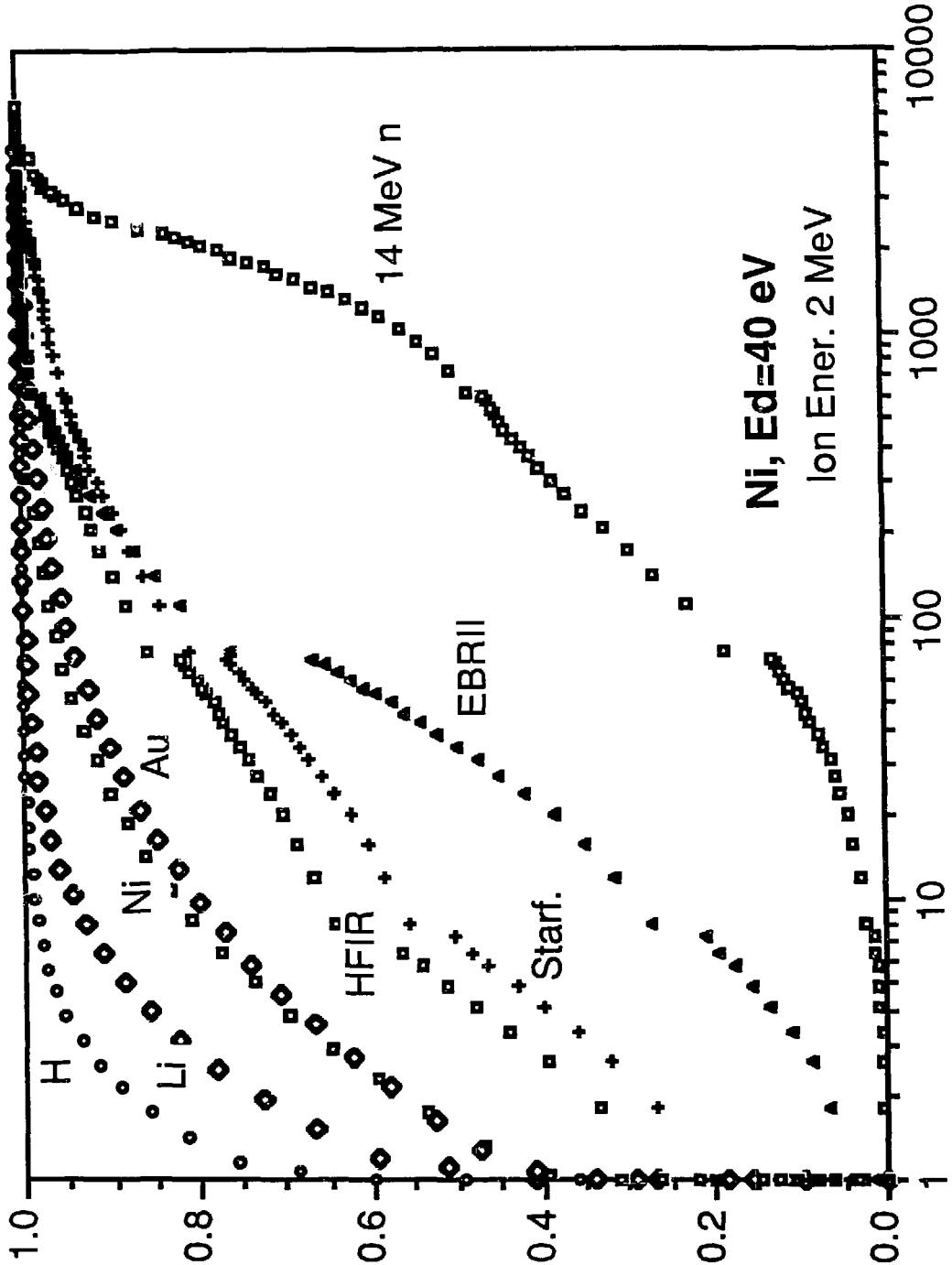
Fig. 2. Fraction of defects produced in Ni in clusters smaller than N by 2 MeV H, Li, Ni and Au ions or by neutrons of several different energy spectra. A threshold displacement energy of 40 eV was used in the calculations.

Fig. 3. Comparison of calculated ($p_\infty=0.005$, $N_d=5$) and experimentally determined fractions of defects undergoing long range migration. The experimental data were normalized to coincide with the calculated values for H. The average threshold displacement energies used in the calculations for the Ni and Cu alloys were 40 and 29 eV, respectively. Experimental data are from Refs. 17 and 18.

Fig. 4. Calculated fraction of defects undergoing long range migration during elevated temperature irradiation with different 2 MeV ions or with neutrons of several energy spectra. A threshold displacement energy of 40 eV was used for the nickel target. $p_\infty=0.005$ and $N_d=5$.

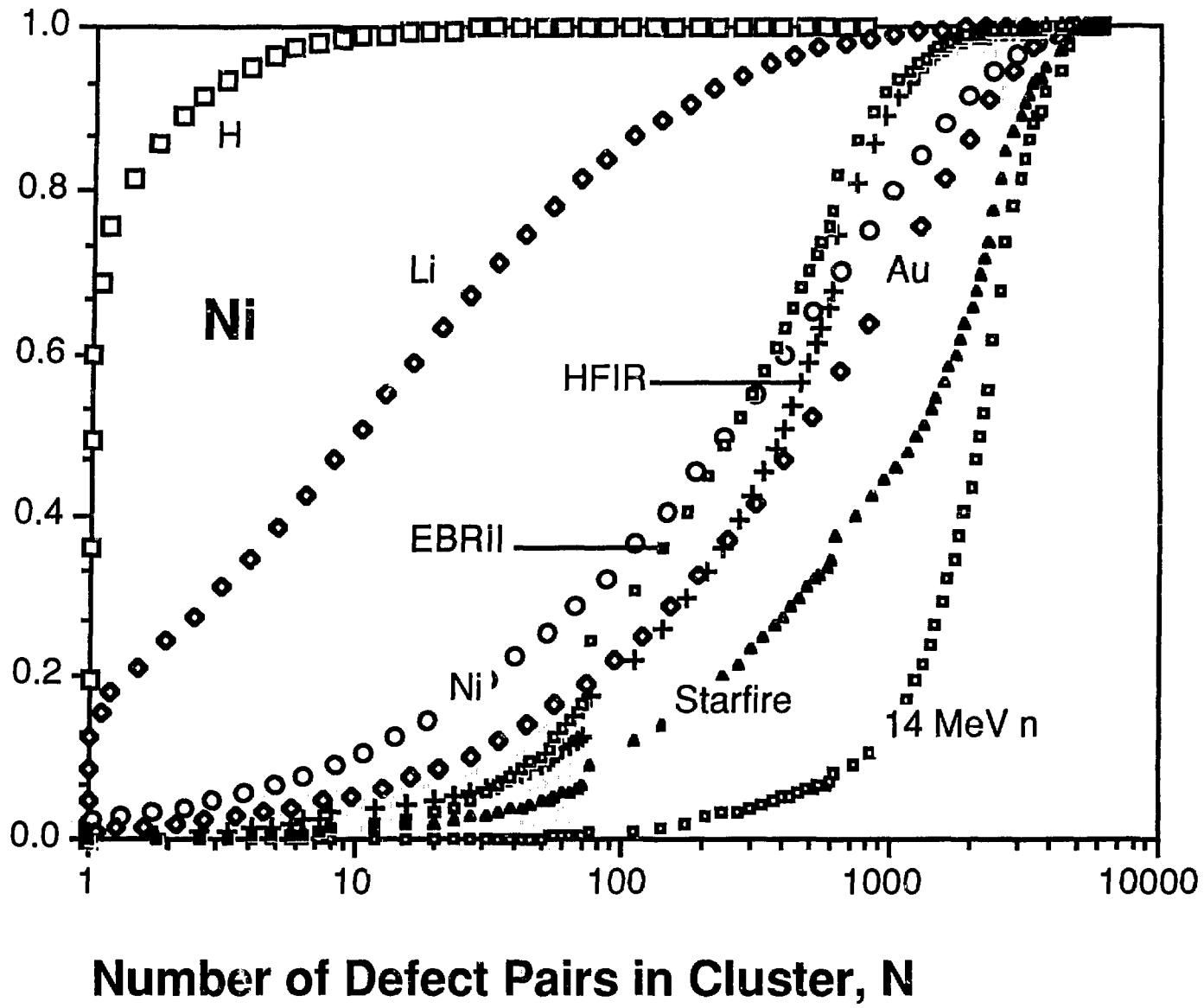
Fig. 5. Life time of clusters of size n_v resulting from absorption of excess interstitials. Displacement rate $K=0.001$ dpa/s, sink strength $p=0.0001$, i. e., $(p/K=0.1)$, $F_i=1$, $F_v=0.3$ and 0.7.

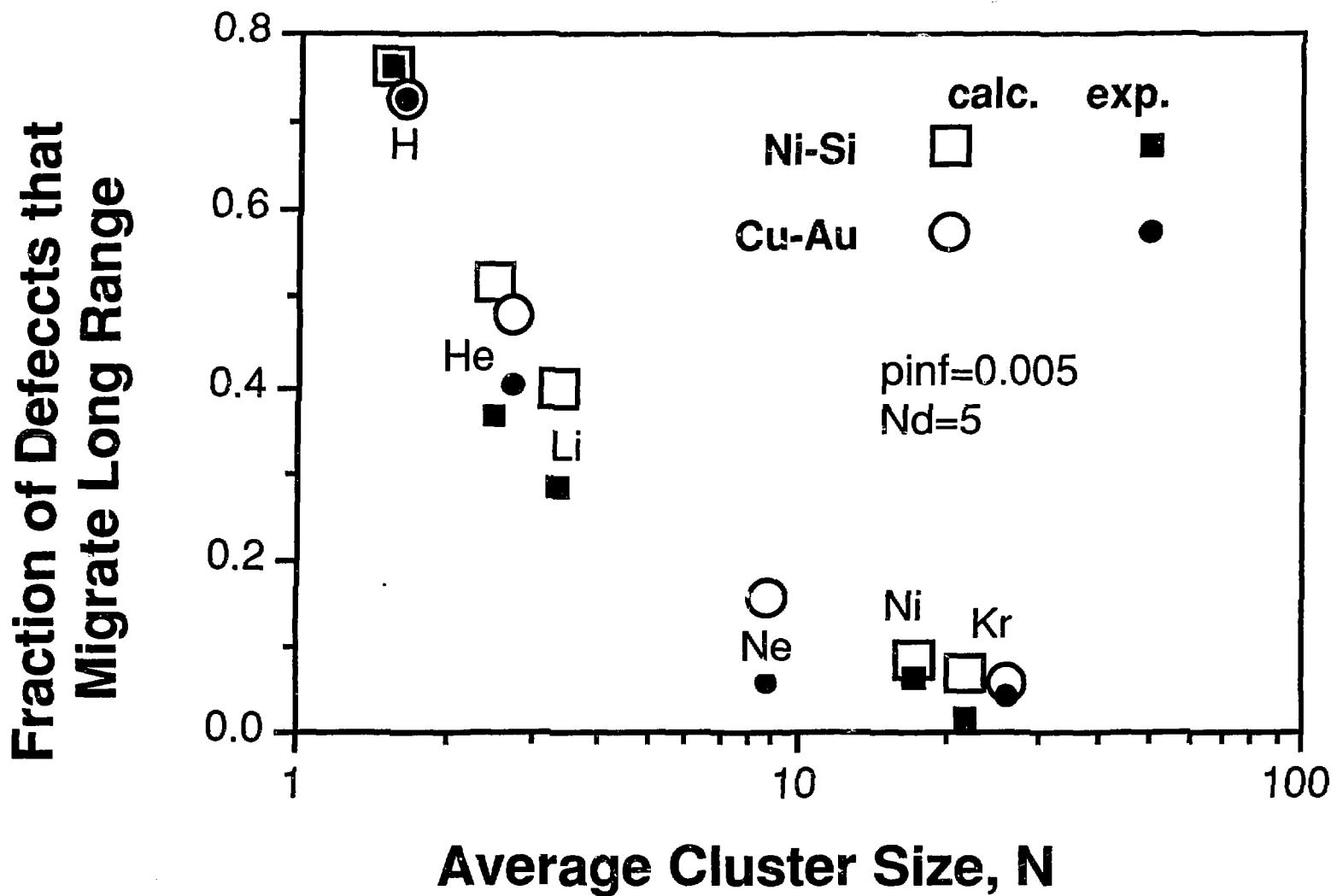
Number of Defect Pairs in Cluster, N



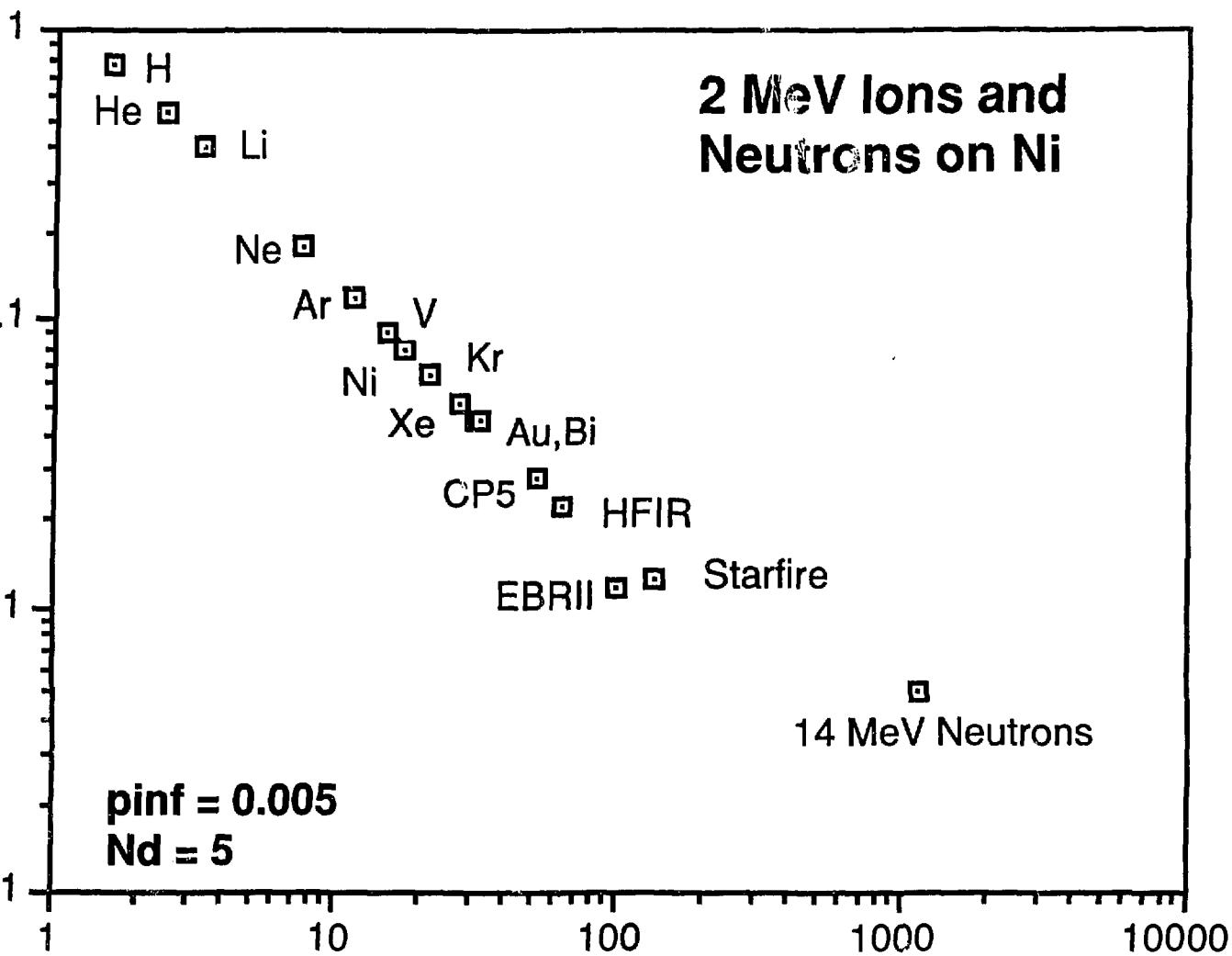
Fraction of Primary Knock-Ons Producing Clusters of Sizes $< N$

Fraction of Defects Produced
in Clusters Smaller than N





**Fraction of Defects that
Migrate Long Range**



Average Defect Cluster Size

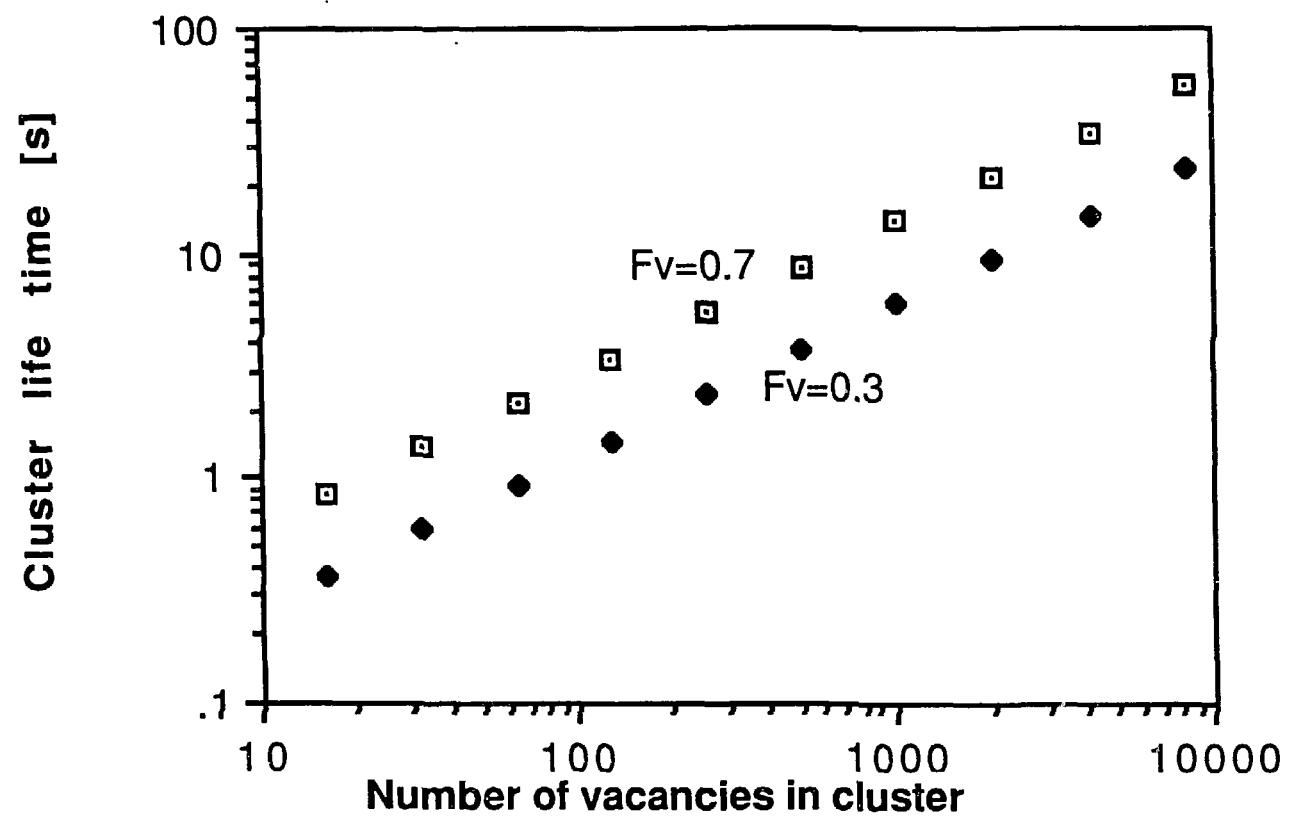


Fig. 5