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Radiation-enhanced diffusion in amorphous Ni-Zr studied by *in situ* electron irradiation in a transmission electron microscope

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Radiation-enhanced diffusion (RED) in a Ni-Zr metallic glass has been studied by high-energy electron irradiation performed *in situ* in a transmission electron microscope. Irradiations have been carried out on thin foil cross-sectional specimens obtained from Ni-Zr bulk diffusion couples. The diffusivity under electron irradiation has been derived from the growth rate of a thin Ni-Zr amorphous film present at the Ni-Zr interface. Experimental results show that the Ni is the most mobile species in these experimental conditions and that radiation damage occurs in glassy metals at a lower electron energy relative to the corresponding crystalline compound. Moreover, the dose-rate sensitivity of RED appears to depend also on the energy of the electron beam. To explain this effect, the process of radiation displacement in metallic glasses has been modeled within the framework of the free-volume theory of the structure of metallic glasses. The results of this simple model can qualitatively explain our results as well as those relative to RED induced by high-energy ion irradiation.

INTRODUCTION

Atomic transport properties of metallic glasses are the subject of an ongoing debate, mainly related to the nature of point defects in the disordered structure and their relevance to the diffusion process and mechanical properties.[1,2] In fact, while both diffusivity and viscosity are strongly dependent on minor structural modifications as shown by structural relaxation experiments,[3] the modern methods used to probe point defects in crystals are not well suited for amorphous alloys.

Most of the experimental results concerning structural relaxation, diffusion, and mechanical properties have been successfully interpreted within the framework of the free-volume (FV) approach originally introduced by Cohen and Turnbull[4,5] and further developed by Spaepen.[6] According to this theory, the excess FV per atom in the structure relative to a perfectly relaxed glass, the average value of which is v_f, is distributed among the atomic sites following a maximum-entropy criterion. Thus

$$F(v)dv=\left[\frac{\gamma}{v_f}\right]\exp(-\gamma v/v_f)dv \ , \tag{1}$$

where $F(v)$ is the distribution function of FV among the atomic cages and γ is a parameter close to unity accounting for the free-volume overlap among adjacent sites. In this framework the vacancylike defects able to support an atomic jump are identified as those FV fluctuations larger than the hard-sphere volume of the atoms, v^*. Van den Beukel[7] has been able to show that the concentration of defects active in diffusion processes is given by the square root of the density of these vacancylike defects. At constant temperature, therefore,

$$D=D^0\left[\int_{v^*}F(v)dv\right]^{1/2}=D^0\exp(-\gamma v^*/2v_f) \ , \tag{2}$$

where D^0 contains both the usual frequency factor and the Arrhenius-like temperature dependence of the diffusion coefficient. It is important to notice that, in the FV model, cooperative mechanisms able to redistribute any FV variation according to Eq. (1) are considered to be operative, at least in sufficiently relaxed structures, so that any change in the average FV is suddenly redistributed entropically through the sample. This assumption has been recently confirmed by molecular dynamics simulations,[8] which showed how a vacancylike defect, artificially created by removing an atom from the structure, remains localized for times of the order of 10^{-11} s and the excess FV introduced in this way is redistributed through the whole sample for longer times. The atomic mobility, in this picture, is then assisted by collective displacement mechanisms, in which the atomic transport is the final result of a wider atomic redistribution.

It has been pointed out by Spaepen[6] that the FV is chosen often as the order parameter in the metallic glass since it makes several problems mathematically tractable; the results should be quite generally valid to the extent that the FV is equivalent to other possible order parameters.

To get further information on the nature and on the kinetics of diffusion-carrying defects, several measurements of radiation-enhanced diffusion (RED) have been performed, mainly by high-energy ion irradiation of thin foil diffusion couples made of elements which can form an amorphous alloy by low-temperature interdiffusion, like Ni and Zr.[9,10] Moreover, it has been shown how irradiation can affect structural properties of metallic glasses like bulk diffusivity[11] and crystallization temperature.[12]

At temperatures low enough to neglect the concentration of point defects in thermodynamical equilibrium relative to the Frenkel pairs produced by the irradiation, RED in crystals can be schematically described by the following equations:[13]

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$$D_{\mathrm{RED}} = D_i C_i + D_v C_v \ , \tag{3}$$

where D_{RED} is the radiation-enhanced diffusion coefficient, and D_j are the diffusivities of point defects whose concentrations are C_i for interstitials and C_v for vacancies, respectively, and which show an Arrhenius dependence on the temperature with activation energy ΔH_j. During irradiation with a particle flux ϕ, the instantaneous concentration of defects is given by the following coupled differential equations:

$$\begin{aligned} dC_i/dt &= \sigma\phi - K_{iv} C_i C_v - K_{is} C_i C_s \ , \\ dC_v/dt &= \sigma\phi - K_{iv} C_v C_i - K_{vs} C_v C_s \ , \end{aligned} \tag{4}$$

where σ is the cross section for Frenkel-pair production, K_{iv} is the rate constant for mutual defect recombination, while K_{js} is for annihilation at fixed sinks whose concentration is C_s. In steady state, when the right-hand sides of Eq. (4) are both equal to zero, two regimes are of interest. It is easy to show that, when defects are removed mainly by mutual recombination, the RED coefficient depends at constant temperature on $\phi^{1/2}$, while it shows a linear dependence if defect annihilation occurs mainly at fixed sinks.

RED IN METALLIC GLASSES

The purpose of this paper is to study RED induced by relatively high-energy electrons, since in this case the absence of collisional cascades and the smaller energy transferred to the target atoms can allow a more detailed study of the defect dynamics. The cross sections for Frenkel-pair production by electron irradiation, σ, can be expressed for light elements by the McKinley and Feshbach expression[14]

$$\sigma_{\mathrm{MF}} = \left[\frac{\pi Z^2 e^4 (1-\beta^2)}{m^2 c^4 \beta^4} \right] \times \left[\frac{T_m}{T_d} + 2\pi\alpha\beta \left[\frac{T_m}{T_d} \right]^{1/2} - (\beta^2 + \pi\alpha\beta) \ln \left[\frac{T_m}{T_d} \right] - (1 + 2\pi\alpha\beta) \right] , \tag{5}$$

where Z is the atomic number, e the electron charge, m the electron mass, β the ratio between velocity of particles and that of light c, and $\alpha = Z/137$. T_m is the maximum recoil energy which depends on the particle energy and on the mass of the target atom, while T_d is the energy threshold for defect production. This expression is derived under the hypothesis that the atoms in a crystal are bound to their sites by an isotropic square-well potential of depth T_d, so that any energy transfer larger than this value has a unit probability of displacing the atom. Later in this paper we shall report experimental RED measurements in an amorphous alloy made of Ni and Zr, which are both too heavy to fulfill the requirements for the application of Eq. (5). However, at the electron energy we used, we can suppose that only the light element Ni can be displaced; moreover, since our purpose is to derive a first-approximation analytical approach to the problem, we have approximated the displacement cross section for Ni with the McKinley and Feshbach expression owing to its analytical formulation.

The application of an expression like Eq. (5) to metallic glasses is not straightforward, owing to the fact that every atom in the disordered structure is in its own potential configuration, so that we cannot assume that a single or a discrete number of energy thresholds describe the whole structure. In the framework of the FV model we can suppose that the local value of T_d is correlated with the amount of FV in the atomic cage, so that we expect a lower threshold and a higher cross section for less densely packed atoms than for high-local-density configurations. In practice, T_d and consequently σ become functions of the amount of local FV, so that we can write $T_d(v)$ and $\sigma(v)$. Moreover, once we accept the correlation between the size of the FV fluctuations and the local displacement threshold, we expect that the total damage rate will depend also on the average FV in the structure, since this parameter controls the fraction of potential damage sites on the basis of Eq. (1). In fact, if we classify the atomic sites in the glass according to their local configuration and we use the local free volume as the corresponding parameter, we have to suppose that the total displacement rate depends both on the relationship between the size of the FV fluctuation and the displacement threshold T_d, which defines the effective cross section for each configuration class, and on the population of each class, which is related to the average FV in the structure. The problem is further complicated by the possible effect of the defects produced by the irradiation on the average FV in the structure; in fact, owing to the cooperative redistribution of FV, localized damage events change the average value of the FV during the irradiation process, thus affecting the total displacement rate with a feedback mechanism.

This picture outlines a situation which is physically different from what happens in crystalline compounds, whose structure and thus the displacement cross section are practically unaffected by the nonequilibrium concentration of defects introduced by the irradiation, and where every atomic position in the sample is a potential damage site. On the contrary in glassy metals, at least when they can be described according to the previous considerations, we can expect a noticeable effect of the structural modifications induced by the irradiation on the displacement behavior.

To try to quantify, at least to a first approximation, the effect of electron irradiation on the diffusivity in metallic glasses, we follow an approach similar to that used by Spaepen to account for the mechanical properties of metallic glasses, with the purpose of computing the steady-state value of v_f under irradiation in order to evaluate the RED coefficient by Eq. (2). We consider that the elementary damage process is to force an atom with a hard-sphere volume v^* into a spherical hole of volume v and we identify this last with the size of the FV fluctuation in the corresponding atomic environment. The energy required by such an operation, the energy threshold $T_d(v)$, can be evaluated, at this level of approximation, according to the continuous mechanics approach of Eshelby.[15] The displacement probability is then given by the corresponding cross section σ_{MF} multiplied by the population of the corresponding configuration class. The net FV increase of this kind of event is given by (v^*-v). Considering the atoms with a FV between v and $v+dv$, we can express the rate of FV generation for this class as

$$\frac{dv_f^+(v,v+dv)}{dt}=\sigma(T_d(v))\phi\frac{\gamma}{v_f}\left[\exp\left[-\frac{\gamma v}{v_f}\right]\right]\times(v^*-v)dv\;. \tag{6}$$

The total rate of FV generation can be obtained by integration over the range of volumes active in the process,

$$\frac{dv_f^+}{dt}=\int_{v_{\min}}^{v^*}\sigma(T_d(v))\phi\frac{\gamma}{v_f}\left[\exp\left[-\frac{\gamma v}{v_f}\right]\right](v^*-v)dv\;, \tag{7}$$

where $v_{\min}$ is the minimum hole size into which an atom can be forced, corresponding to the maximum transferred energy T_m, while the upper integration limit is the larger hole leading to FV increase in a displacement event. According to Eshelby,[15] T_d can be expressed as

$$T_d=\frac{2}{3}\mu\left[\frac{1+\nu}{1-\nu}\right]\frac{(v^*-v)^2}{v}\;, \tag{8}$$

where μ is the shear modulus and ν the Poisson ratio. Analytical integration of Eq. (7) is not easy. However, the product $\sigma(T_d(v))(v^*-v)$, reported in Fig. 1(a), versus v can be approximated with a linear relationship in the range of v where the function under integration, shown in Fig. 1(b), is not vanishing, provided that v_f/v^* is less than 0.05, a value which appears reasonable on the basis of the available experimental data.[7] We can thus approximate this function as

$$\sigma(T_d(v))(v^*-v)=\alpha(v-v_{\min})\;, \tag{9}$$

where α is a constant depending on the electron energy, which is considered independent of the average FV owing to the fact that the FV enhancement induced by the irradiation is expected to be sufficiently small.

After integration of Eq. (7) and neglecting v_f relative to v^*, we have

$$\frac{dv_f^+}{dt}=\phi\alpha[(v_{\min}-v^*)\exp(-\gamma v^*/v_f)+(v_f/\gamma)\exp(-\gamma v_{\min}/v_f)]\;. \tag{10}$$

Considering that $v_{\min}/v^*$ for Ni atoms irradiated with 400-keV electrons is about 0.1, we can also neglect the first term relative to the second.

As far as the annihilation of free volume is concerned we have to remark that, whatever local mechanism we can consider for atomic rearrangement, the average glass density can be increased only by a permanent transfer of FV at the glass free surface. As will be described later, in this work, as in some other experiments on RED in metallic glasses,[9,10] we have experimentally studied the behavior of diffusion couples, where a few-nm-thick amorphous layer is bounded by the parent crystalline phases so that no free surface of the amorphous phase is present in the specimens. Since our purpose is also the interpretation of our experiments, we can consider that the elementary annihilation event is an atomic jump across the glass-crystal interface. Consequently, we can hypothesize that only FV fluctuations larger than v^* are active in the annihilation process and that the rate of FV removal is proportional to the concentration of such defects through the relative reaction constant K. This framework corresponds to a first-order annihilation kinetics; however, in a more general way we can leave the order of the kinetics as a free parameter n so that the rate of free-volume removal is given by

$$\frac{dv_f^-}{dt}=K\exp(-n\gamma v^*/v_f)\;. \tag{11}$$

In steady state, where the creation and the annihilation rates are equal, the amount of FV is given by the equation

$$K\exp(-n\gamma v^*/v_f)=\phi\alpha(v_f/\gamma)\exp(-\gamma v_{\min}/v_f)\;. \tag{12}$$

Neglecting the linear variation of v_f relative to the ex-

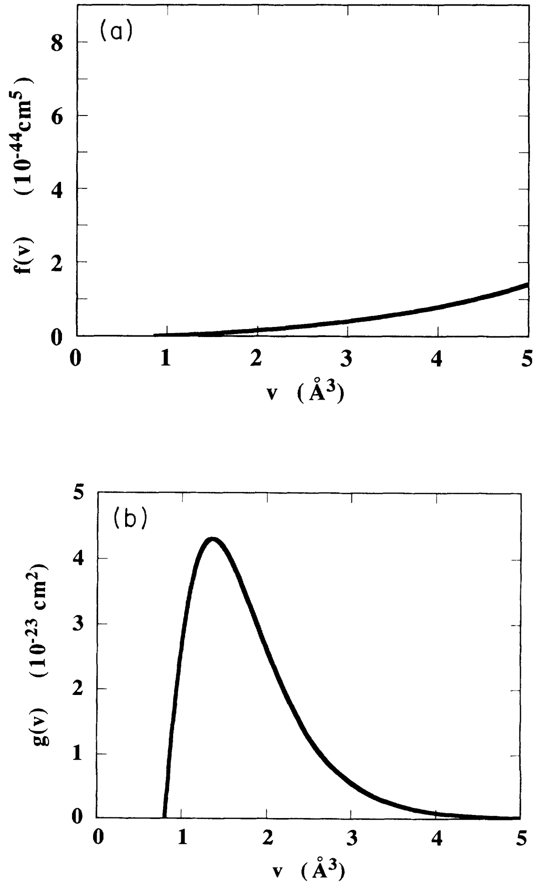

FIG. 1. (a) Plot of the function $\sigma(T_d(v))(v^*-v)$ versus v. (b) Plot of the function $\sigma(T_d(v))\phi(\gamma/v_f)[\exp(-\gamma v/v_f)](v^*-v)$ versus v.

ponential one, so that v_f becomes v_f^0 in the linear term, we obtain

$$\frac{\gamma}{v_f}=\frac{-\ln[\phi\alpha(v_f^0/\gamma K)]}{(nv^*-v_{\min})} . \tag{13}$$

Substituting this result into Eq. (2) we can finally write the expected value of the diffusion coefficient under steady-state irradiation:

$$D=D^0\exp\left[\frac{v^*\ln[\phi\alpha(v_f^0/\gamma K)]}{2(nv^*-v_{\min})}\right] \tag{14}$$

and

$$\ln D=\ln D^0+\frac{v^*}{2(nv^*-v_{\min})}\ln(v_f^0\alpha/\gamma K)+\frac{v^*}{2(nv^*-v_{\min})}\ln\phi . \tag{15}$$

On the basis of Eq. (15) we can expect that the dose-rate sensitivity of RED in metallic glasses can depend both on the particle energy, since $v_{\min}$ depends on this parameter through the maximum recoil energy T_m, as well as on the order of the annihilation kinetics n, while only the last controls the behavior of crystalline compounds. In the case of high-energy ion irradiation, when $v_{\min}$ is vanishingly small owing to the large mass of the irradiating particles, experimental results[10] showed a $\phi^{1/2}$ dependence, which has been interpreted on the basis of the behavior of crystalline compounds as indicative of a second-order annihilation kinetics. However, the authors noticed that their experimental points do not display the expected extrapolation at infinite flux, thus showing that a straightforward interpretation of RED in metallic glasses does not explain all the experimental features. Considering our formulation, this result is instead consistent with a first-order kinetics, which appears more realistic in a thin amorphous layer bounded by the crystalline parent phases, where the interface can easily act as a powerful defect sink. However, this interpretation is directly connected with the results of Van den Beukel[1] about the concentration of diffusion-carrying defects. In fact, the factor 2 appearing in the last term of Eq. (15) is directly connected to the conclusion that the concentration of defects active in diffusion is given by the square root of the concentration of elementary defects. This interpretation of the ion-induced RED is then a further support to the conclusions of Van den Beukel. Alternatively, it is possible to explain the experiments by considering that single vacancylike defects are effective in the diffusion process and that the annihilation kinetics is of second order. We think that the first framework can be considered more realistic since it is coherent with other results[7] and accounts for the presence of a high density of sinks in the relevant experimental situation.

EXPERIMENTAL METHOD

From the experimental point of view we have irradiated, at room temperature in a transmission electron microscope (TEM) with a maximum accelerating voltage of 400 keV, thin foil cross-sectional specimens prepared from bulk Ni-Zr diffusion couples having an interdiffusion amorphous layer (hereafter a-Ni-Zr) about 5-nm thick. The method used to prepare the diffusion couples and the TEM samples is described elsewhere.[16] The resulting specimens have the amorphous-crystal interfaces parallel to the electron beam so that the thickness of the amorphous film can be easily measured. A typical example of the Ni-Zr interface before the irradiation is reported in Fig. 2(a). Each experiment consisted in the measurement of the growth rate of the a-Ni-Zr under constant-current-density irradiation. Pictures of the irradiated area were taken every 10 or 20 min. At the same time the current density was monitored by a Faraday cage. Irradiation experiments have been carried out at 300 and at 400 keV with current densities in the range from 5 to 500 A/cm^2 and with a beam-spot diameter of about 0.5 μm.

Owing to the strong effect of the actual microscope magnification on the measurement of both the growth rate of the a-Ni-Zr layer and the current density, which is measured at the level of the viewing screen of the microscope, this parameter has been experimentally determined after each run with a catalase crystal. The temperature increase due to beam heating can play an important role in the experimental results. Unfortunately, on the basis of the previous considerations, we cannot set a sharp energy threshold for the onset of radiation damage in metallic glasses, so that we cannot use irradiation un-

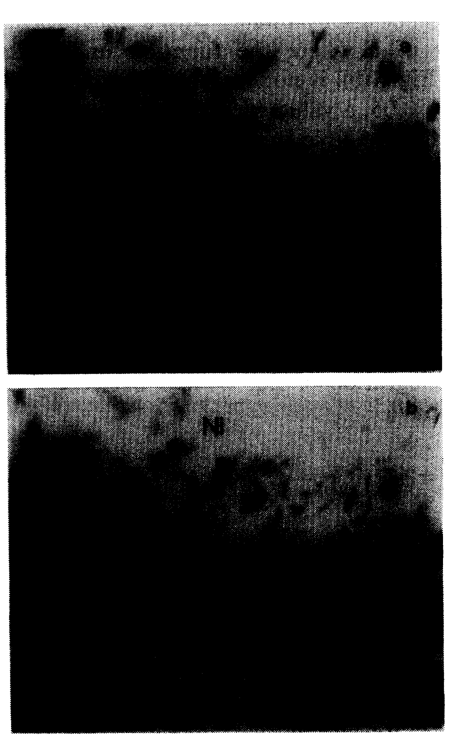

FIG. 2. (a) TEM micrograph of the Ni-Zr interface before the irradiation, showing the presence of an a-Ni-Zr layer about 5-nm thick. (b) Same area after 25 min of irradiation with 400-keV electrons at a current density of 464 A/cm^2.

der the threshold to rule out any thermal contribution to the growth process.

In a flat, circular, homogeneous thin sample of radius R, with parallel surfaces and in good thermal contact at its periphery, the temperature increase ΔT at the center of an irradiating circular beam of radius r_0 and current density J is given by[17]

$$\Delta T=\frac{J\rho}{2e\lambda}\frac{\Delta Q}{\Delta x}r_0^2\ln\frac{R}{r_0}\,, \tag{16}$$

where ρ is the sample density, e the electron charge, λ the thermal conductivity, and $\Delta Q/\Delta x$ the stopping power. This equation, derived under the hypothesis that the specimen is homogeneous, cannot be directly applied to our specimens which are made of Ni for one half and of Zr for the other half, owing to the strong differences between the two elements, mainly for what concerns both the stopping power and the thermal conductivity. However, we can consider that the trends displayed by Eq. (16) are still valid by considering effective values for the material-dependent parameters. In particular, we can notice that the beam heating of the specimen is expected to depend more on the total beam current $\pi J r_0^2$ than on the current density, at least in the reduced range of spot sizes used in this work, which is typical of the focused beam of the TEM. Considering that the RED effect instead depends only on the current density, we have measured the growth rate of the amorphous phase at the same current density and with different spot sizes, thus changing the total beam current and the heat input to the specimen. From this set of measurements it has been observed that the growth rate of the glassy interlayer does not depend on the spot size, even at the maximum current density, up to values larger than 0.5 μm. All the experimental data have been collected with this spot size, thus allowing us to neglect any thermal contribution to the growth of the *a*-Ni-Zr.

EXPERIMENTAL RESULTS AND DISCUSSION

Figure 2(b) shows the interface layer after irradiation at 400 keV with a current density of 464 A/cm^2 for 25 min. It is possible to observe how the irradiation-induced growth occurs mainly by displacement of the *a*-Ni-Zr/Zr interface, showing that, in agreement with thermally induced growth,[18] Ni is the more mobile species even under electron irradiation. In fact the growth process appears to be diffusion controlled since, as reported in Fig. 3, the square of the glassy layer thickness depends linearly on the irradiation time.[19] This fact allows us to use this experimental method to study RED in this system. Moreover, the thickness of the amorphous layer, for a fixed total irradiation time, does not depend on the duration of the irradiation interval, showing that the time required to set up a steady-state situation after the onset of the irradiation process is short relative to the relevant experimental times.

The main results of this investigation, having the purpose of an experimental verification of the trends displayed by Eq. (15), are reported in Figs. 4(a) and 4(b). Two main features can be noticed. At 400 keV of electron energy the curve in Fig. 4(a) shows two regimes with a discontinuity at about 27 A/cm^2. The slope is 0.74 in the high-current-density range while it is approaching 1 in the lower one. In this last regime the experimental errors connected with the measurement of such low diffusivities prevent an accurate determination. At 300 keV the slope is 0.85 while the lower value of the RED

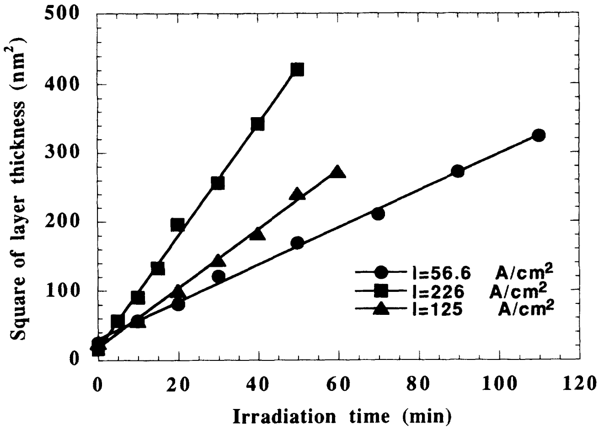

FIG. 3. Square of the *a*-Ni-Zr layer thickness versus the irradiation time for different values of the current density for 400-keV irradiation.

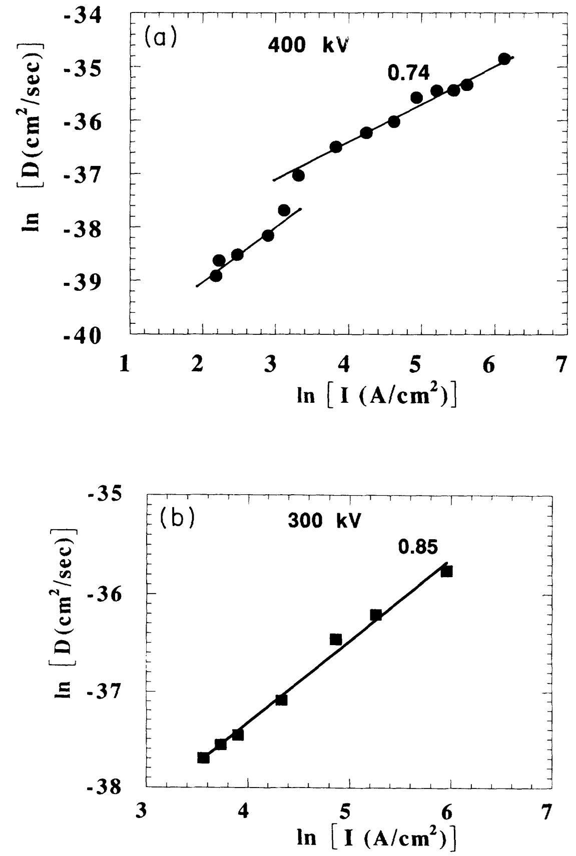

FIG. 4. (a) Dose-rate sensitivity of RED induced by (a) 400-keV irradiation. (b) 300-keV irradiation.

coefficient does not allow an extension of the explored range toward lower current densities. Comparing the results in the high-flux regime where, owing to the higher atomic mobility, we can consider that the cooperative redistribution of FV is operative, we notice that the experimental results confirm the trends of the theoretical calculations; in fact, the slope at 300 keV is higher than at 400 keV. The drastic approximations used in the derivation of Eq. (15) do not allow a quantitative comparison of theoretical and experimental results. However, we want to stress that the difference in the slope is well outside the experimental errors and that the two curves cover the same range of diffusivities, so that the concentration of defects active in the diffusion process should be the same in the two cases. This fact excludes the possibility that the difference in the dose-rate sensitivity can be ascribed to different kinetics of the defects induced by irradiation and shows that a straightforward interpretation of the experimental results on the basis of the behavior of crystalline compounds does not fit well the whole set of data. This fact is further supported by the presence of the discontinuity and by the change in slope displayed in Fig. 4(a). As pointed out by Spaepen and Turnbull,[2] cooperative redistribution of FV can occur only if the atoms in the glass have sufficient mobility to rearrange their relative positions. For lower mobility the metallic glass has to be considered in an isoconfigurational state where atomic positions are frozen into the structure in a way similar to a crystal even if they do not cover a regular lattice. Consequently, by reducing the electron flux and so the steady-state value of the free volume, we enter into a regime where the defects created by the irradiation remain localized for a time comparable with the experimental ones and behave like vacancies in a crystal. Consequently, the rate of defect production is reduced relative to a relaxed configuration since the feedback mechanism enhancing the concentration of sites where displacement can occur, through an increase of the average FV, is not operative. This can explain the drop of the RED coefficient while crossing this threshold. Moreover, the dose-rate sensitivity is determined only by the defect annihilation kinetics in a way similar to crystalline compounds and we experimentally recover a slope close to unity as we expect for a thin metallic glass film where the interfaces with the bounding phases can easily act as a high concentration of fixed sinks. The behavior we observed shows some similarities with the interpretation given by Spaepen and Turnbull[2] in the framework of the free-volume approach of some discrepancies about the activation volume for diffusion in metallic glasses. In fact, studying the effect of external pressure on diffusivity, Limoge[20] found an activation volume of the order of the atomic size, thus supporting the hypothesis of diffusion of vacancylike defects, while Faupel, Huppe, and Ratzke[21] reported a negligible effect of the external pressure on the diffusivity in their specimens. Considering the differences in the two experimental approaches, Spaepen and Turnbull have been able to show how in the second a cooperative mechanism is expected to govern the atomic diffusion, while in the first case localized vacancylike defects can control the atomic mobility. The main difference between the two experiments is in the degree of relaxation of the metallic glasses and so in the ability to redistribute the change in the average free volume introduced by the external pressure, in a way similar to what we have used to explain the behavior of the RED coefficient below and above the threshold. If this interpretation is correct, so that the slope below the threshold is really indicative of a first-order annihilation kinetics of the defects introduced by the irradation, and if we can consider this behavior to be quite general, the $\phi^{1/2}$ dependence reported by Averback and Hahn,[10] interpreted with our Eq. (15), is a strong support to the findings of Van den Beukel[1] about the concentration of diffusion-carrying defects in metallic glasses.

As a final point we want to mention that radiation damage occurs in metallic glasses at lower threshold energies than in the corresponding crystalline compounds, showing the presence of atomic configurations where atoms are less bound to their sites. In fact the maximum energy transferred by a 300-keV electron to a Ni atom is about 15 eV, while the displacement threshold for crystalline Ni-Zr has been computed to be about 20 eV.[22]

CONCLUSIONS

In conclusion, we have shown that the dose-rate sensitivity of the RED coefficient during electron irradiation of metallic glasses is dependent not only on the defect annihilation kinetics, but also on the energy of the irradiating particles, in qualitative agreement with a simple model developed in the framework of the free-volume theory. The cooperative redistribution of the FV created by atomic displacement can explain this phenomenon, accounting, even if in a qualitative way, for our experimental data as well as for results obtained by high-energy ion irradiation.

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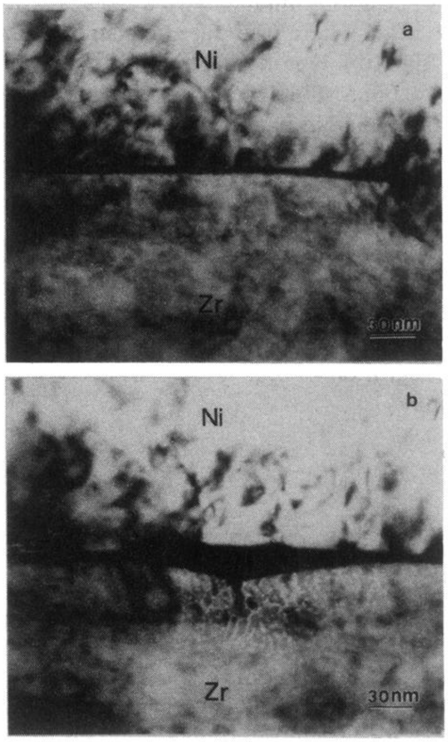

FIG. 2. (a) TEM micrograph of the Ni-Zr interface before the irradiation, showing the presence of an *a*-Ni-Zr layer about 5-nm thick. (b) Same area after 25 min of irradiation with 400-keV electrons at a current density of 464 A/cm^2.