Creep and electrical resistivity of metallic glass $Ni_{78}B_{14}Si_8$ under proton irradiation

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Plastic deformation and electrical resisitivity of the metallic glass $Ni_{78}B_{14}Si_8$ were measured during 6.3 MeV proton irradiation at ≈420 K under tensile stresses up to 430 MPa. Irradiation creep rate depended linearly on stress σ and particle flux Φ (or displacement rate K), giving $\epsilon^*/\sigma\Phi \approx 4.5 \times 10^{-33}\,m^2/Pa$ ($\epsilon^*/\sigma K \approx 5 \times 10^{-9}\,Pa^{-1}\,dpa^{-1}$). After recrystallization this value was reduced by a factor of ≈25, falling into the range of polycrystalline pure nickel and dilute Ni alloys. The electrical resisitivity in the amorphous state was decreasing under irradiation, while the initially lower resistivity of recrystallized material strongly increased. © *1999 American Institute of Physics.* [S0021-8979(99)07321-1]

INTRODUCTION

Metallic glasses are supposed to be resistant against displacive irradiation due to their inherent disordered structure.[1,2] For this reason and due to some favourable mechanical properties (strength, etc.),[3] these materials are potential candidates for applications in irradiation environments (fusion, spallation sources, etc.). The stability of the amorphous state under irradiation is of paramount interest and therefore was previously investigated under irradiation by various particles. While electron irradiation at room temperatures to a dose of $2 \times 10^{25}/m^2$ had no effect on the crystallization temperature of amorphous $Ni_{45}Co_{20}B_{16}Cr_{10}Mo_4Fe_5$,[4] some structural relaxation was observed in amorphous $Ni_{42}Fe_{30}B_{15}Cr_6Si_5Mo_2$, using Mößbauer spectroscopy and Curie temperature measurements after irradiation to a thermal neutron dose of $\approx 10^{23}/m^2$ around ambient temperature.[5] On the other hand, these changes recovered well below the recrystallisation temperature as indicated by annealing studies. A dose of $6.5 \times 10^{23}/m^2$ (26 dpa) thermal neutrons at 380 K decreased the crystallization temperature of $Fe_{40}Ni_{40}B_{20}$, while $3 \times 10^{23}/m^2$ did not.[6] Irradiation with 3 MeV Ni ions to doses of $1.5 \times 10^{20}/m^2$ (≈20 dpa) at room temperature showed no effect on crystallization, phase change, or swelling of amorphous $Ni_{60}Nb_{40}$.[7] Also $6 \times 10^{20}/m^2$ of 0.5 MeV Ni ions had no effect on the amorphous structure of $Fe_{40}Ni_{38}B_{18}Mo_4$ at room temperature, while irradiation at 470 K caused some crystallization, i.e., at a temperature which is 370–420 K lower than in unirradiated material.[8] Finally, $9 \times 10^{20}/m^2$ (≈86 dpa) of 60 MeV Ni ions caused significant swelling in amorphous $Fe_{40}Ni_{40}P_{14}B_6$ already at 293 K.[9] From these results, stability of the amorphous state under irradiation at room temperature can be tentatively assumed up to doses of ≈50±30 dpa, depending on the material and irradiation conditions.

Comparison of irradiation behavior and defect properties in the amorphous and crystalline state showed that in $Fe_{75}B_{25}$ displacement energies of Fe atoms (22±3 eV) are identical,[10] while the resistivity per unit concentration of Frenkel defects in the amorphous material ($\rho_{F(Fe)} = 3 \pm 1\ \mu\Omega$ m/u.c.) is reduced by a factor of about 7,[11] and the recombination volume ($v_r = 36 \pm 18$ at vol.) is a factor of ≈3 smaller[12] than in crystalline material, assuming identical displacement cross sections σ_d. (This means that instead of a lower ρ_F also a lower σ_d could account for the experimental results. For an estimation of σ_d in amorphous material, information on displacement process, and defect stability would be needed, as well as on defect annealing at the irradiation temperature.) Lower defect resisitivity ($\rho_F = 4.3\ \mu\Omega$ m/u.c.) and smaller recombination volume ($v_r = 88$ at. vol.) have also been found in $Pd_{80}Si_{20}$ (Ref. 13) when compared to crystalline Pd ($\rho_F = 9.\ \mu\Omega$ m/u.c., $v_r = 633$ at. vol.).[14]

Metallic glasses[15] like virtually all amorphous materials show under irradiation at sufficiently high electronic stopping power and sufficiently low temperatures after some incubation fluence a stationary, volume conserving, anisotropic plastic deformation, with contraction along the particle direction and dilatation perpendicular to it. This ''hammer'' effect has been quantitatively described by a visco-elastic model.[16] ''Thermal'' creep of metallic glasses, i.e., plastic deformation under external stress without irradiation, follows a hyperbolic sine law stress dependence as for crystalline metals,[17] but with the range of viscous flow, i.e., linear stress dependence, extending to much higher stresses and lower temperatures.[3,17] This viscous flow[18,19] has been described theoretically on the basis of the free volume model[20–22] for various metallic glasses, cf. Refs. 23 and 24. On the other hand, irradiation-induced creep of metallic glasses has so far only been studied with heavy ions in the GeV range at temperatures up to ambient, employing resistivity measurement under tensile stress,[25] and bending measurements under an undefined compressive stress state.[26] Both experiments gave no quantitative results on creep behavior.

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Therefore, the present work was aimed to investigate irradiation creep of a Ni-base metallic glass under well-defined experimental conditions and to compare it to that in the recrystallized state and to that of pure polycrystalline nickel and Ni alloys.[27,28] The study was furthermore stimulated by a basic interest in the behavior of vitreous materials under irradiation, mainly coming from recent findings of strongly enhanced plastic deformation of silica glasses and glassy carbon.[29–31] Irradiation particle, energy, and temperature were chosen such that the hammer effect and thermal creep were negligible and doses ($\leqslant$0.1 dpa) remained far below the above estimated stability limit of the amorphous state.

EXPERIMENTAL DETAILS

For the present investigation, a Ni-based metallic glass with 14 wt% B and 8 wt% Si ($Ni_{78}B_{14}Si_8$) was chosen mainly for its relatively high transition temperature, giving some safety margin during irradiation for beam excursions or inhomogeneous temperature distribution. The material was supplied by Goodfellows GmbH in the form of 25 mm wide ribbons of 40 μm thickness. The supplier quotes density $\Theta = 8.0\ \mathrm{g/cm^3}$, electrical resistivity $\rho = 0.9\ \mu\Omega\,\mathrm{m}$, crystallization temperature $T_{a\rightarrow c} = 720\ \mathrm{K}$, elastic modulus $E = 150\ \mathrm{GPa}$, tensile strength $\sigma_m = 1.5-2.0\ \mathrm{GPa}$, and a maximum application temperature of 470 K. Our measurements on virgin specimens gave at room temperature $\rho \approx 0.91\ \mu\Omega\,\mathrm{m}$, slightly increasing to a value of 0.98 $\mu\Omega\,\mathrm{m}$ at 720 K and then sharply dropping to 0.74 $\mu\Omega\,\mathrm{m}$ with the temperature dependence of resistivity after annealing above 720 K was much stronger than in the amorphous state. Cooling to room temperature gave $\rho \approx 0.44\ \mu\Omega\,\mathrm{m}$, i.e., the room temperature resistivity was reduced by about 50%. Annealing for $\frac{1}{2}$ h at temperatures from 730 to 1020 K caused contractions ($-\Delta l/l_0$) of about 0.55%, while contraction was about 2.1% after $\frac{1}{2}$ h annealing at 1220 K. Resistivity drop and contraction are clear indications of recrystallization, but nevertheless x-ray analysis after annealing even up to 1220 K gave only diffuse diffraction, probably due to small grain size and large internal strains. Those were also indicated by severe embrittlement after recrystallization, probably with contribution from segregation.[32] Only specimens annealed for $\frac{1}{2}$ h at 730 K could be mounted in the creep apparatus, while $\frac{1}{2}$ h at 820 K caused extreme fragility, which was only slightly reduced by heating to 1020 K.

The elastic modulus of the present foils in the amorphous state, as derived from length measurements at different stresses ($E = \Delta\sigma \cdot l_0/\Delta l$), amounted $\approx$100 GPa (A somewhat lower elastic modulus compared to bulk material is also observed for other thin foil materials.) From $1/E$ and from the concurrently measured initial change of relative resistance ($\Delta R/R_0$) with tensile stress ($\Delta R/R_0/\Delta\sigma = 1.6 \pm 0.1 \times 10^{-11}/\mathrm{Pa}$), the stress dependence of electrical resistivity ρ can be derived by

$$\frac{\Delta\rho}{\rho_0} = \frac{\Delta R}{R_0} - \frac{\Delta l}{l_0}(1 + 2\cdot\nu). \tag{1}$$

This gives within experimental error $\Delta\rho/\rho/\Delta\sigma = -0.1 \pm 0.1 \times 10^{-11}\ \mathrm{Pa^{-1}}$, using Poisson's ratio $\nu \approx 0.35$.[33] The independence of electrical resisitivity of the metallic glass on stress is in contrast to results for $Fe_{80}B_{10}Si_{10}$ where a finite stress dependence and furthermore a strong hysteresis was found during the initial stress cycles at stresses above $\approx$500 MPa.[34] The reason for this finite stress dependence of resistivity may be that the authors used literature values of the elastic modulus derived from acoustic wave velocity measurements which, as in the present case, may be higher than the actual $\Delta\sigma/\epsilon$ values. After recrystallization (1/2 h at 730 K) E increased to 125 GPa and ρ was reduced by about 50% (see above), with the stress dependence of ρ remaining negligible. The relative increase of the elastic modulus by a factor of 1.25 falls into the range (1.24–1.33) observed for other metallic glasses.[33,35] All measurements showed a small but unambiguous hysteresis, i.e., strain and resistance at a given stress were slightly higher when a higher stresses had been applied before. This effect was not studied in detail. It occurred before and after irradiation, was seemingly larger for the recrystallized material, and is probably related to reversible structural relaxation.[36]

The specimens were mounted in an irradiation creep apparatus, which was described in detail elsewhere.[37,38] Uniaxial tensile load was applied by a spring. The specimens were heated by dc current to a temperature of 420 ± 15 K which was below the maximum application temperature but was sufficiently high to allow proton (6.3 MeV) fluxes Φ up to about $1.1 \times 10^{18}\ \mathrm{p/m^2\,s}$. Cooling was supplied by purified helium gas and temperature was measured and controlled by an infrared pyrometer. Strain and resistance measurements were performed during irradiation and for higher precision and better stability also intermittently during beam shutdowns. Proton doses were converted to displacement damage using $\sigma_d = 9 \times 10^{-25}\ \mathrm{m^2}$ from pure Ni.[37] This value which may be appropriate for the recrystallized material was also used for the amorphous state (see note in the preceding paragraph) and gives displacement rates $K = \sigma_d \cdot \Phi$ at maximum Φ of about 1×10^{-6} dpa/s.

RESULTS

The strain of glassy $Ni_{78}B_{14}Si_8$ under proton irradiation at 50 MPa uniaxial tensile stress is shown as a function of dose in Fig. 1. Without any transient a straight increase with a slope of $1.1 \times 10^{-25}\ \mathrm{m^2/p}$ (0.12/dpa) is obtained. Almost exactly the same slope is obtained for relative electrical resistance change. Elastic modulus and stress dependence of resistance were practically not changed by irradiation. Reduction of dose rate by a factor of 2 gave within experimental error a linear dose rate dependence of the irradiation creep rate (exponent $= 0.95 \pm 0.15$). This allows a plot of creep rates ϵ^* normalized to displacement rate K as a function of stress σ in Fig. 2. Linear stress dependencies (dashed lines) were observed with apparent proportionality factors $\epsilon^*/\sigma\Phi = 1.8 \times 10^{-33}$ and $4.5 \times 10^{-33}\ \mathrm{m^2/Pa}$ ($\epsilon^*/\sigma K = 2 \times 10^{-9}$ and $5 \times 10^{-9}\ \mathrm{Pa^{-1}\,dpa^{-1}}$) below 100 and above 200 MPa, respectively.

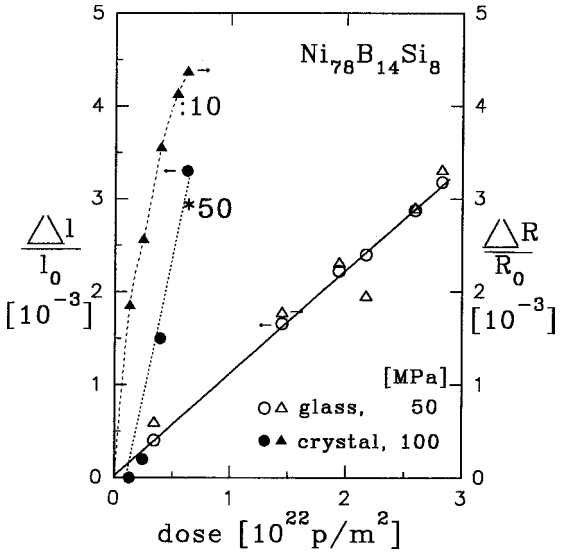

FIG. 1. Uniaxial strain (circles) and relative change of electrical resistance (triangles) as a function of proton dose of $Ni_{78}B_{14}Si_8$, irradiated at 420 K under uniaxial tensile stress in the amorphous state (50 MPa, ○,△) and recrystallized (100 MPa, tensile data ●, multiplied by 50; resistance data, ▲, divided by 10). Lines are included to guide the eye.

Strain rate of a recrystallized specimen (1/2 h at 730 K) gave $\epsilon^*/\Phi = 7.2\times10^{-27}\,\mathrm{m^2/p}$ ($\epsilon^*/K = 8\times10^{-3}$/dpa) at 100 MPa (Fig. 2). Its resistance ($\rho_0 \approx 0.74\,\mu\Omega\,\mathrm{m}$) increased strongly during irradiation with an initial slope $\Delta R/R_0/\Delta\Phi t \approx 1.5\times10^{-23}\,\mathrm{m^2/p}$ ($\Delta R/R_0/\Delta K t \approx 16$ dpa) which slightly decreased with dose.

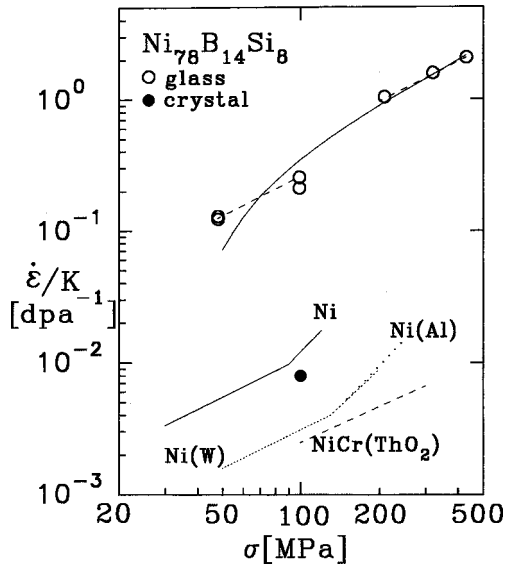

FIG. 2. Irradiation-induced strain rate per atomic displacement rate K as a function of tensile stress of $Ni_{78}B_{14}Si_8$ in the amorphous (○) and recrystallized (●) state, compared to 20% cold worked pure nickel, Ni-4% W, Ni(Al) alloys with 4–6 wt % Al,[27] and a dispersion hardened $Ni_{20}Cr$-1%ThO_2 alloy (see Ref. 28). The dashed lines in the upper part of the figure indicate linear stress dependence, while the solid line corresponds to a fit of Eq. (2).

DISCUSSION

According to Eq. (1) $\Delta R/R_0$ and $\Delta l/l_0$ of glassy $Ni_{78}B_{14}Si_8$ (Fig. 1) yield $\Delta\rho/\rho/\Phi t = -1.1\times10^{-25}\,\mathrm{m^2/Pa}$ ($\Delta\rho/\rho/\Delta K t \approx -0.12$/dpa), when $\nu = 0.5$ is used for volume conserving creep deformation, while $\Delta\rho/\rho/\Delta K t$ of the recrystallized material, due to negligible straining, is equal to $\Delta R/R_0/\Delta K t \approx +16$/dpa. On the other hand, a relative increase of resistivity $\Delta\rho/\rho/\Delta K t \approx +0.9$/dpa, [assuming $\sigma_d = 3\times10^{-24}\,\mathrm{m^2}$ (Ref. 39)] was measured at 4 K in amorphous $Cu_{50}Ti_{50}$ after irradiation at 300 K with $10^{21}/\mathrm{m^2}$ protons of 2 MeV, with $\Delta\rho/\rho/\Delta K t$ still increasing during annealing.[40] Also amorphous as well as crystalline $Fe_{75}B_{25}$ showed extrapolated initial increases of relative resistivity of $\Delta\rho/\rho/\Delta K t \approx +0.9$/dpa ($\sigma_d = 9.8\times10^{-27}\,\mathrm{m^2}$) under 2.4 MeV electron irradiation at 21 K.[11] A similar increase of +0.9/dpa was obtained for dispersion hardened $Ni_{20}Cr-1\%ThO_2$,[28] while pure nickel and stable austenitic alloys show negligible changes of resistivity under proton irradiation above ambient temperature. Only in Ni(Al) alloys a decrease of resistivity was found with an initial slope of about −0.2 dpa, caused by irradiation-induced precipitation of Ni_3Al.[41] For comparison, disordered Cu_3Au irradiated in a moderated reactor at 353 K yielded even higher initial decreases of $\Delta\rho/\rho/\Delta K t \approx -200$/dpa (tentatively using $\sigma_d = 2\times10^{-27}\,\mathrm{m^2}$), while the relative resistivity of the ordered alloy increased by +200/dpa.[42] In general, such irradiation-induced resistivity changes can only be considered as sensitive but unspecific indications of structural changes. Therefore, the relatively small change of the present material in the amorphous state can probably not be taken as an quantitative measure of higher resistance against radiation damage.

Pure and alloyed crystalline metals show transient strains under irradiation which are commonly ascribed to microstructural changes towards a new, irradiation-induced structure, including dislocations, loops, precipitates, etc. Therefore, the missing transient of $\Delta l/l_0$ in $Ni_{78}B_{14}Si_8$ (Fig. 1) may indicate a stability of the amorphous phase against irradiation-induced structural changes.

The two, slightly different proportionality regimes of normalized strain rates in Fig. 2 (dashed lines) can be reconciled by including a stress-independent compaction, as for example, found in vitreous silica:[29]

$$\epsilon^*/K = C_v/3 + C_c\sigma, \tag{2}$$

where C_v is a parameter describing the relative volume change per unit displacement dose and C_c is the so-called irradiation creep compliance $\epsilon_c^*/\sigma K$. A reasonable fit to the data (solid line in Fig. 2) is obtained with $C_v = -0.15$/dpa ($1.4\times10^{-25}\,\mathrm{m^2}$) and $C_c = +5\times10^{-9}\,\mathrm{Pa^{-1}\,dpa^{-1}}$ ($4.5\times10^{-33}\,\mathrm{m^2/Pa}$). In the low stress regime, the apparent irradiation creep compliance of amorphous $Ni_{78}B_{14}Si_8$ is by a factor of about 25 larger than that of the recrystallized specimen (Fig. 2), but still by a factor of about 12 smaller than the respective value for vitreous silica.[29] The compliance of the recrystallized specimen is in the range of polycrystalline nickel and Ni alloys,[27,28] which in turn is about a factor of 3 above values of austenitic stainless steels. Considering the missing x-ray reflexions, this means that irradiation creep

TABLE I. Comparison of normalized experimental strain rates $\epsilon^*/\sigma\Phi$ of amorphous materials under irradiation to the model (see Ref. 16) described by Eq. (3), with Poisson's ratio ν, elastic modulus E, mass density Θ, specific heat C per mass, nuclear stopping power S_n, giving the temperature rise in a cascade ΔT as fitting parameter.

Material	$\epsilon^*/\sigma\Phi$ (10^{-33} m^2/Pa)	ν (1)	E (GPa)	$\Theta \cdot C$ (10^6 J/m^3 K)	S_n [10^{-13} J/m]	ΔT [K]	Ref.
ν-$Ni_{78}B_{14}Si_8$	4.5	0.35	150	3.4	9.6	355	this work
Glassy C	1.0	0.3	35	3.0	1.62	1098	30
ν-SiO_2	22.	0.18	75	2.8	2.82	49	29

behavior changes already at a very early stage of crystallization. Further light on this point could be shed by studies on nanograin materials. The relative enhancement of irradiation creep rate in the amorphous versus crystalline state of $Ni_{78}B_{14}Si_8$ by above one order of magnitude is in the same range as for glassy carbon versus graphite.[30] For ceramic glasses, e.g., ν-SiO_2, such a comparison is not possible as in quartz or other crystalline ceramics irradiation creep is masked by dilatations due to structural changes and defect accumulation.[31,43] Figure 2 shows that the viscous regime, i.e., linear stress dependence, of irradiation creep extends for the metallic glass to much higher stresses than for the nickel alloys. Considering the much higher strength of the glass, this is in qualitative agreement with a relation between the extension of the linear stress regime and yield strength, derived for nickel alloys and austenitic stainless steels.[44]

In Table I, the creep compliances $\epsilon^*/\sigma\Phi$, including glassy carbon[30] and vitreous SiO_2,[29] are compared to a visco-elastic model which describes the viscosity ($=\sigma/3\epsilon^*$) under cascade damage.[16] This model is based on the assumption that in an amorphous solid under external stress, some strain which will be frozen-in after relaxation of the stresses in the molten core of a displacement cascade, while in a crystal only transient straining appears which almost completely recovers during subsequent recrystallization. The normalized strain rate in the amorphous solid is described by

$$\epsilon^*/\sigma\Phi = \frac{10(1-\nu^2)}{(7-5\nu)} S_n \sqrt{6/\pi e^3}/(E\Theta C\Delta T) \qquad (3)$$

with particle flux Φ, specific heat C, and nuclear stopping power S_n which is derived from Monte Carlo type calculations (TRIM95).[45] ΔT is a parameter which describes the increase in temperature within the cascade, necessary to allow sufficient flow for plastic deformation. This value was used as a free parameter to fit the model to the experimental values. The resulting ΔT values allow to estimate, which contribution to the observed irradiation-induced creep comes from the above process. An upper limit of ΔT can be estimated from the difference between melting and irradiation temperature, but also somewhat lower values are conceivable due to the presence of irradiation defects. On the other hand, in the above model only those cascades contribute which reach sufficient temperatures in the core to allow viscous flow. If for example only cascades above 1 keV are considered, in the present case of light ion irradiation S_n and consequently also ΔT would be reduced by a factor of 3. From these considerations, the ΔT values derived for glassy carbon and metallic glass may be regarded sufficiently high to ascribe a significant amount of the observed irradiation-induced creep in these materials to cascade damage according to the above model. On the other hand, the very low value for vitreous silica indicates that additional mechanisms are contributing which may be related to ionization induced defects, e.g., broken bonds, which are stable in insulators but not in electrically conducting materials such as carbon or metals.

SUMMARY AND CONCLUSIONS

(1) Irradiation-induced creep and stress effects on resistivity changes of a metallic glass have been directly determined by strain measurements under uniaxial tensile stresses.

(2) Length and resistivitity, both in the amorphous and recrystallized state, increase without any significant transients almost linearly with dose, but at very different rates.

(3) The irradiation creep rate in a metallic glass is by about a factor of 25 higher than in comparable crystalline materials.

(4) The high irradiation creep rates may be beneficial in reducing irradiation-induced internal stresses, but in most cases are probably disadvantageous in terms of dimensional stability for the application of metallic classes in irradiation environments.

(5) Resistivity changes are sensitive but unspecific indication of structural changes in metallic glasses under irradiation.

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