

STRUCTURAL ANISOTROPY OF AMORPHOUS ALLOYS INDUCED BY MECHANICAL CREEP DEFORMATION

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Résumé - L'anisotropie structurale d'un alliage amorphe due au fluage a été observée pour la première fois par diffraction X (méthode de la dispersion en énergie). Cette anisotropie vient uniquement de la polarisation inélastique et disparaît par recuit sous tension nulle. L'écoulement plastique ne fait apparaître aucune anisotropie de structure.

Abstract - We present the first evidence of the structural anisotropy of an amorphous alloy due to creep deformation, detected by energy dispersive x-ray diffraction. The anisotropy is due solely to the anelastic polarization and disappears after zero-stress annealing. The plastic flow leaves no structural anisotropy behind.

I - INTRODUCTION

Amorphous alloys show considerable anelasticity and plasticity [1], but the microscopic mechanism of deformation still remains poorly understood. One of the reasons for this difficulty is that the traditional methods to observe microstructure of solids, such as transmission electron microscopy (TEM), are nearly powerless for amorphous materials. However, microscopic structural changes due to mechanical deformation could be detected by other more macroscopic probes as long as the probe has a high accuracy. In our study we used the energy dispersive x-ray diffraction (EDXD) technique [2] to detect the structural anisotropy induced by mechanical creep deformation.

II - EXPERIMENTAL METHODS

The samples used in this study are amorphous $\text{Fe}_{40}\text{Ni}_{40}\text{Mo}_3\text{Si}_{12}\text{B}_5$ (Vitrovac-4040) produced by Vacuumschmelze and provided by Dr. H. R. Hilzinger. The dimension of the sample was 35 μm x 15 mm x 70 mm. The sample was annealed under the tensile stress of 800 MPa, which is about 1/4 to 1/3 of the fracture stress, at 300°C for 24 hrs in vacuum of 10^{-5} torr. Usually another piece of ribbon was annealed simultaneously but without the applied stress, and was used as the control sample. The total elongation of the sample is about 3%, one third of which is anelastic.

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The EDXD method is a spectroscopic technique of structure determination utilizing a white x-ray source and a semiconductor energy sensitive detector [2]. We used a rotating anode x-ray generator with a Mo target and an intrinsic Ge detector with the resolution of about 250eV at 20kV. The major advantage of this method is its high statistical accuracy because of high photon count rates and parallel photon counting without a scanning. For the present study we accumulated typically 5×10^6 total photon counts for each run. This ensures the statistical accuracy, for instance better than .5% for the height of the first peak of the structure factor.

In the EDXD method the diffraction angle θ is kept constant during the measurement, and the diffraction vector \vec{q} is given by

$$|\vec{q}| = 2/hc \cdot E \cdot \sin \theta = 1.014 \cdot E \cdot \sin \theta \quad (1)$$

where q is in \AA^{-1} , an E is the photon energy given in keV. Thus the spectrum of the diffracted beam gives the structure factor of the sample, after corrections for inelastic scattering, multiple scattering, air scattering, detector efficiency and escape peaks and normalization by the incident beam spectrum[2]. The measurements were

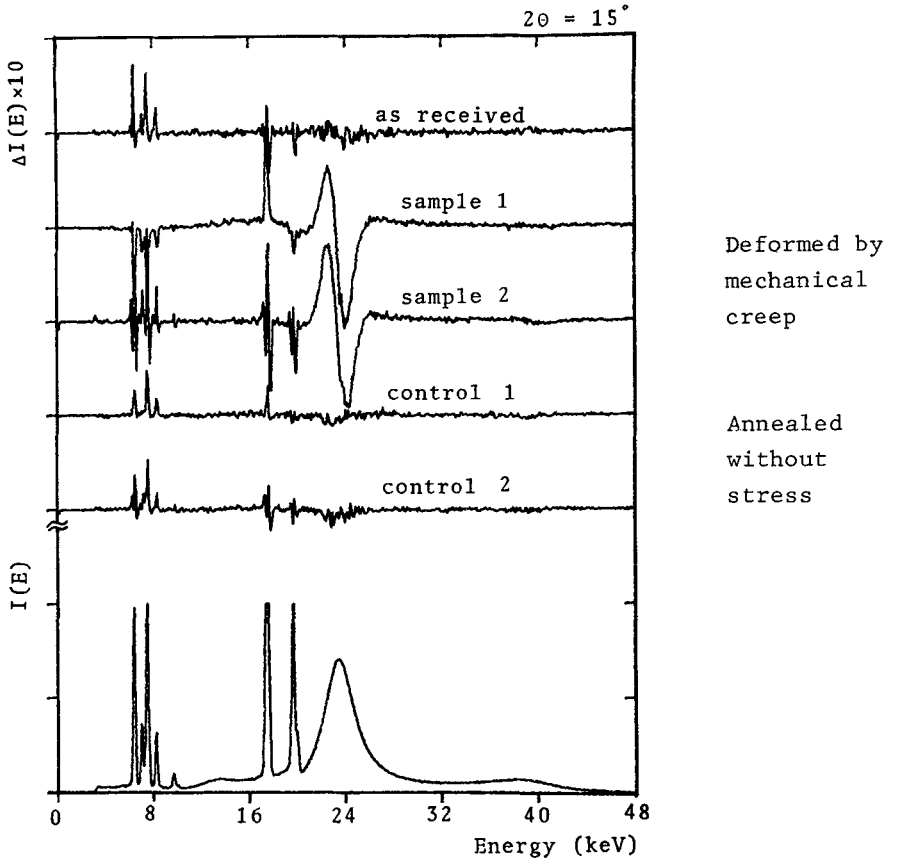


Fig. 1 The EDXD diffraction spectrum of amorphous $\text{Fe}_{40}\text{Ni}_{40}\text{Mo}_3\text{Si}_{12}\text{B}_5$ at $2\theta = 15^\circ$ (bottom), and the difference spectra, $I(//) - I(\perp)$, for the samples as-received, mechanically crept, and annealed without stress (top).

carried out with the diffraction vector parallel and perpendicular to the axis of the applied stress, \vec{a} , in order to determine the structural anisotropy. A transmission geometry was chosen to minimize the effect of geometrical anisotropy of the sample surface; the surface of the sample was macroscopically uniform, but usually there are very small pockets due to trapped gas during the production of the alloy by melt spinning, which are elongated along the direction of the ribbon. Also in order to avoid the effect of drifts in the electronics, the measurements were done alternatingly in two sample directions, \vec{a}/\vec{q} and $\vec{a} \perp \vec{q}$, typically 1 hour each, and the spectra were accumulated separately.

III - RESULTS

Detailed study of structural anisotropy and the effect of annealing recovery was carried out at the diffraction angles of $2\theta = 15^\circ$ and 25° . The diffraction spectrum and the difference between the spectra with a parallel and perpendicular to q , $\Delta I = I(//) - I(\perp)$, are given in Fig. 1. At this diffraction angle ($2\theta = 15^\circ$) the photon energy E is related to q by $q = 0.132 \cdot E$. Thus the peak at around 23 keV corresponds to the first peak of the structure factor at 3.1 \AA^{-1} . Two sharp peaks at 17.4 and 19.6 keV are due to fluorescent radiation from Mo atoms, while smaller peaks at $6 \sim 9 \text{ keV}$ are the Fe and Ni fluorescent peaks.

The difference spectrum ΔI for the as-received sample shows this particular sample is nearly isotropic. This, however, is not always true. Other samples of similar composition often showed small anisotropy. The control samples annealed without the applied stress remained isotropic after annealing, but two samples deformed by mechanical creep showed significant anisotropy as illustrated in Fig. 1; the magnitude of ΔI is as large as 10% of I itself. The similarity of the two spectra for different samples shows a good reproducibility of the results. The difference spectrum resembles the derivative of the original spectrum. This means that the first peak shifted toward smaller values of q for the parallel (\vec{q}/\vec{a}) direction whereas for the perpendicular direction it shifted to larger values of q . Such a shift is expected when the sample is uniformly elastically deformed; atomic distances in the direction of the stress increase while those perpendicular to the stress decrease. The equivalent elastic strain which would produce such a shift is about 0.5%. This, however, does not imply that there is a real macroscopic elastic strain in the system, since the measurement was done in a zero stress condition. It was found that the anisotropy in the structure factor at the position of the second peak does not resemble the derivative of the structure factor, but it resembles the structure factor itself. Therefore we conclude that there is a long range uniform internal shear strain of 0.5% which is balanced by the short range (mostly nearest neighbour) atomic rearrangements. In other words the apparent long range strain is produced by the local transformation of the atomic clusters which act like shear transformed inclusions.

It was found that this structural anisotropy is gradually decreased by the subsequent annealing without an applied stress, as shown in Fig. 2. The logarithmic plot, Fig. 3, shows that the relaxation of the anisotropy is logarithmic with time, as is often seen for relaxation phenomena in glasses. It is important, however, that this logarithmic decay appears to extrapolate to zero. In other words the structural anisotropy is essentially recoverable, therefore it is almost purely due to anelasticity. The total strain of 3% induced by this creep experiment includes a large fraction ($\sim 2/3$) of unrecoverable plastic strain due to viscous flow, but apparently such a plastic deformation leaves no structural anisotropy behind.

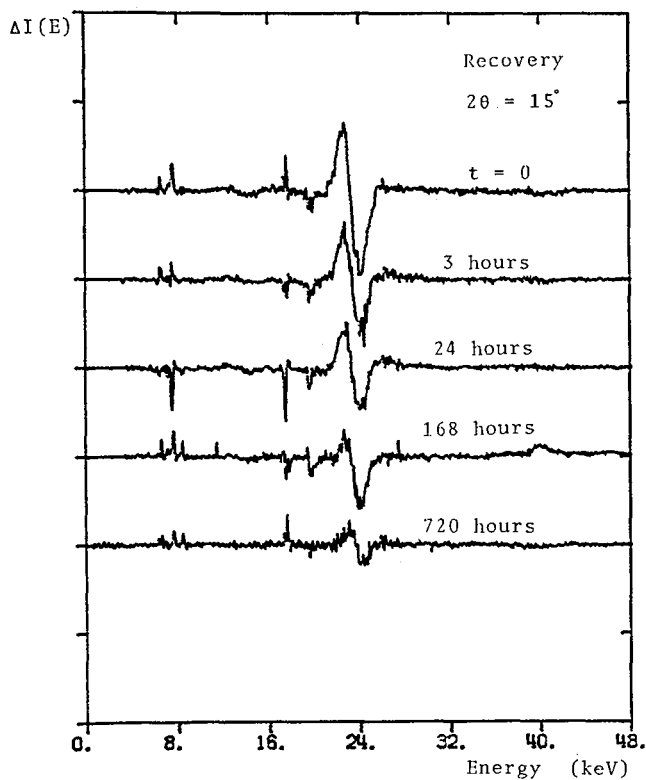


Fig. 2 Difference spectra during the recovery at 300°C for specified lengths of annealing time.

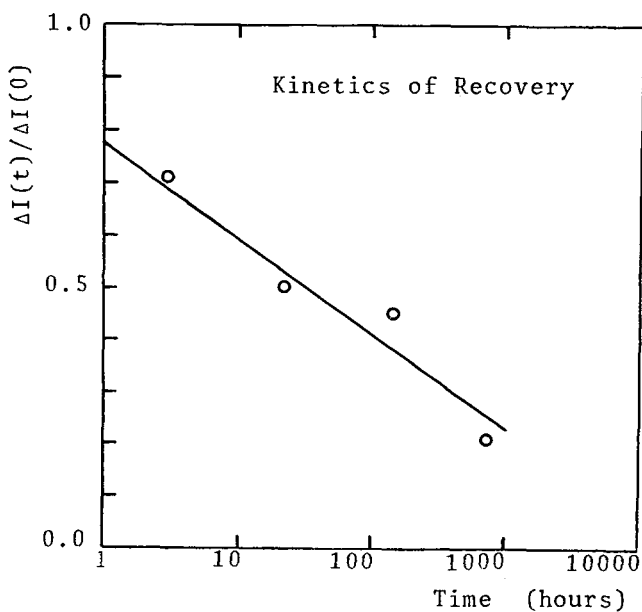


Fig. 3 The amplitude of the difference spectra (the first peak region) in Fig. 2 plotted against the logarithm of the annealing time.

This point was also confirmed by the preannealing experiment. When the sample was preannealed at 300°C for 24 hrs., the total creep strain induced by the same treatment as above was only about 1% which was almost entirely anelastic strain. But the structural anisotropy induced by the creep deformation was only slightly reduced, by about 1/4, compared to the case without preannealing. This result, coupled with the earlier observation that the preannealing greatly reduces the viscous plastic strain but affects only slightly the anelastic strain [3], supports our view that the induced structural anisotropy is due primarily to anelastic local polarization.

IV - DISCUSSION

The present study confirmed for the first time that the anelastic polarization of the amorphous alloy induces structural anisotropy [4]. The experimentally determined anisotropic pair distribution function compared with the computer simulation (Y. Su uki, Thesis, University of Pennsylvania, 1985), which will be published elsewhere in detail, suggests that the structural anisotropy is produced by the atomic bond reorientations from the direction parallel to the stress to the perpendicular direction. The atomic rearrangements occur apparently in very localized regions. The anisotropy is maintained by the elastic stress field around it, so that it could disappear when the density of the polarized regions becomes high enough to start the percolation which would result in plastic deformation, as has been suggested by Argon and Shi [5]. Therefore it is reasonable that the plastic deformation leaves no structural anisotropy behind.

ACKNOWLEDGEMENT

This work was supported by the Office of Naval Research through the contract N00014-80-C-0896. The research was conducted in part using the facilities of the Laboratory for Research on the Structure of Matter, supported by the National Science Foundation by the MRL Grant DMR-82-16718.

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