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A detailed investigation of surface modification in metallic glasses subjected to 130 MeV ^{28}Si ion irradiation

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Abstract

Surface modification induced in four metallic glasses by 4.64 MeV/u ^{28}Si ion irradiation has been investigated in detail using optical microscopy, SEM and STM. Results obtained in two separate runs have been presented here. The effects of ion fluence (ϕ) and tilt angle (θ) on surface modification have been studied both qualitatively and quantitatively. It has been found that for S_e values smaller than that for track formation, swift heavy ion irradiation leads to smoothing of the irradiated surfaces. The smoothing is evident from decreasing mean roughness R_q and reduction in height of the 'hills' and filling up of the 'valleys' in the SEM and STM pictures. The observations have been explained on the basis of the theory of shear flow within the framework of the viscoelastic model.

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Keywords: Metallic glasses; Ion irradiation effects; Surface modification; Electron microscopy

1. Introduction

Ions of all energies incident on a solid influence its material properties and are used in different ways in research and technology. If the energy of the incident ion is less than the binding energy of the target atoms, the ion may get deposited over the surface. Low energy ions (≈10–100 eV/amu) come to rest at or near the surface of a solid, possibly growing into an epitaxial layer. A 1-keV/amu heavy ion beam is the essential component in the sputtering process. A large fraction of the incident energy is transferred to the atoms of the solid, resulting in the ejection of surface atoms into the vacuum. The bulk of the target is modified at higher energies, ≈100–300 keV/amu, as the ions penetrate the target surface. High energy ions are used as source of atoms to modify materials by ion-implantation. In the case of few MeV/amu energies, the incident ions may get deeply implanted or even penetrate through thin samples, thereby modifying the properties of the target [1]. Thus, the surface of the target is most likely to get modified by the impact of incident ions. The

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type of modification depends on the incident ions' energy.

Swift (kinetic energy $\geqslant 1$ MeV/amu) heavy ions (SHI) lose their energy in the target material in two independent ways: elastic collisions with the nuclei of the target giving the nuclear stopping power $S_n = (dE/dx)_n$ (nuclear energy loss) and inelastic collisions with electrons of the material (electronic energy loss) $S_e = (dE/dx)_e$ [2]. If the incident ion is heavy, S_e dominates by a factor of about 10^3 over S_n [3]. As a result, a large number of atoms in the target are set into motion even at very low temperatures, by the electronic excitation in the wake of SHI. The atomic mobility set in the bulk leads to some well known effects, such as the anisotropic growth and increase in electrical resistivity. On the other hand, atomic mobility of the near surface atoms results in modification of the irradiated surface without detectable mass loss [4]. Therefore, the effects induced by SHI irradiation depend on the amount of electronically deposited energy. Although this research area is now more than two decades old, there is no general consensus about the physical processes responsible for the observed effects. It is well established that these effects result from the atomic motion induced by electronic energy loss S_e of the incident ions in the target material. But the mechanism involved in the conversion of electronically deposited energy to the motion of target atoms is under discussion without any general agreement so far. There is still a need of a theory that could explain all these effects within a single framework.

At such a high ion energy when S_e is few keV/nm, the surface of the solid target is modified in interesting ways. It is known that for a given ion, SHI irradiation induced surface modification in amorphous solids depends on ion fluence ϕ (number of ions incident per unit area of the sample surface) and tilt angle θ (angle of incidence of ions with respect to the normal to the sample surface) [4,5]. For normal incidence of the fast heavy ion beam ($\theta = 0°$), surface smoothing results. This smoothing process dominates for $\theta < 15°$ (off-normal incidences). For $\theta \geqslant 20°$, first the smoothing reduces to smaller length scales, then appearance of regular undulations takes over at higher fluences. The wavelengths of these undulations have been found to be independent of the type of ions, their incidence angle, starting surface topography of the target and the irradiation temperature. At still higher fluences (about 10^{15} ions/cm^2), surface roughening takes place [4,5]. Thus, there are three steps of surface modification already known: smoothing, wave-formation and roughening. These ϕ and θ dependent surface modifications have been accounted for by an ion beam induced shear flow mechanism [4,5,8,9].

In this paper we report the surface modification results of 4.64 MeV/u (130 MeV) ^{28}Si ions irradiated metallic glasses. The experiments have been carried out in two separate runs. The ion–energy combination was selected after several calculations with Monte-Carlo TRIM computer code [3] and keeping in view (i) the easy availability of the beam from the pelletron accelerator at Nuclear Science Centre (NSC), New Delhi and (ii) corresponding S_e smaller than the threshold value for track formation. The second point was taken into consideration because this investigation was intended to study surface modification as an independent effect without having caused severe damage (like tracks) in the bulk. In the first run, we observed the surface modification induced by normal irradiation. Whereas, in the second run, two especially planned experiments were performed and the effects of fluence ϕ and tilt angle θ on surface modification were investigated. Such a detailed study of surface modification as an independent effect in four metallic glasses is being reported for the first time in this paper.

2. Experimental

Metallic glass (MG) samples were procured from Allied Signals, New Jersey, USA and Good-Fellow Cambridge Inc., England in the form of 25 μm thick foils. All of these MGs were prepared by rapid quenching of the molten alloys at a rate of 10^6 K/s. These metallic glasses were chosen for investigation because they have large deformation yield A_0 corresponding to the selected ion–energy combination. Hence, they were expected to give easily detectable effects under irradiation. The compositions of various MGs used in the present work are given in Table 1.

Table 1
Material parameters of metallic glasses used in the present investigation

Sample[a]	Composition[b]	R_p (μm)[c]	S_e (keV/nm)[c]	α (10^{-5} K^{-1})[b]	ρ (gm/cm^3)[b]	C (J/kg K)[d]	A_0 (10^{-15} cm^2)[e]
MG2204	$Ti_{50}Be_{40}Zr_{10}$	33.28	3.22	3.30	4.13	680	2.60
MG2605SC	$Fe_{81}B_{13.5}Si_{3.5}C_2$	19.91	5.50	0.59	7.32	519	2.00
MG2705M	$Co_{69}B_{12}Si_{12}Fe_4Mo_2Ni_1$	19.15	5.75	1.21	7.80	496	4.21
MG2826MB	$Fe_{40}Ni_{38}B_{18}Mo_4$	18.95	5.91	1.17	7.90	492	4.16

[a] Sample names are registered trademarks of Allied Signals Inc., USA.
[b] Sample compositions and values of thermal expansion coefficient (α) and mass density (ρ) have been provided by the supplier (GoodFellow Cambridge Ltd., England).
[c] Projected range of ions (R_p) and electronic energy loss (S_e) have been calculated using TRIM computer code, version 97.09 [3], for 130 MeV ^{28}Si ion beam.
[d] Specific heat (C) has been calculated by the method given in Section 3.3.
[e] Deformation yield (A_0) has been calculated using Eq. (2) in [9].

During the first run, all metallic glass samples were mounted side by side on a target ladder in four sets and irradiated up to two fluence values of 3.765×10^{14} and 1.154×10^{16} ions/cm^2 at about 80 K.

In the second run, samples were mounted according to special plans to see the effects of fluence and tilt angle. The details about special arrangements done regarding sample mounting etc. is given below.

2.1. Variable fluence experiment

In this experiment we made use of the beam scanner facility available in the material science beam line at NSC. During normal irradiation experiments this facility provides uniform irradiation by scanning a beam spot of 1.2 mm $\times$ 1.2 mm over a desired area.

Two sets of two samples of dimensions about 10 mm $\times$ 5 mm were mounted behind a thick aluminium foil mask with about 8 mm $\times$ 8 mm window (Fig. 1(a)). Irradiation was started through the window of the mask, with a beam scanning over an area of 10 mm $\times$ 10 mm. After a pre-calculated time, so that a fluence of about 10^{13} ions/cm^2 was reached, the length of the scanned area was reduced to half keeping the same width. Irradiation was continued for another fixed time calculated for an overall fluence of about 10^{14} ions/cm^2. The length

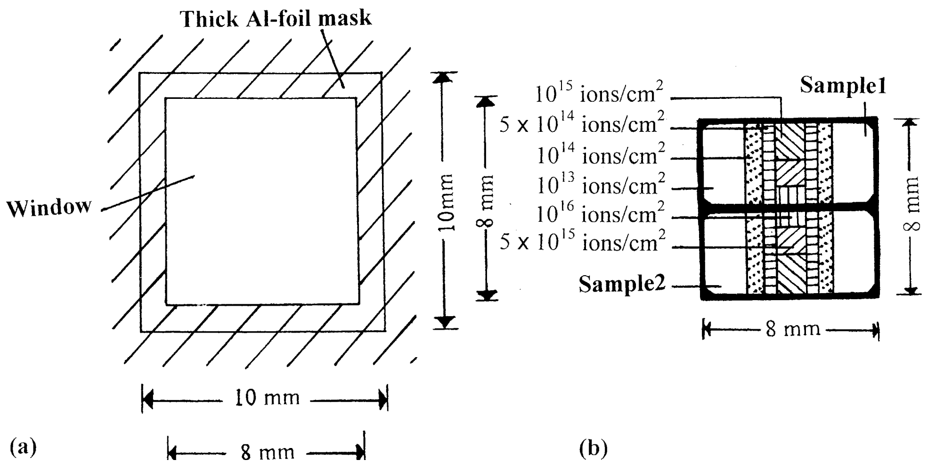

Fig. 1. Irradiation plan in variable ϕ experiment. (a) Schematic diagram showing the thick aluminium foil mask and window arrangement used during irradiation. (b) Schematic diagram showing the area of the samples exposed to the ion beam through the window. The doses given to different regions are also shown.

of the scan was further reduced by half, keeping the width unchanged, and an overall fluence of about 5×10^{14} ions/cm^2 was achieved. The process was followed until the length of the scanned area was reduced to 1.2 mm, which is the minimum possible value (y-scanner off condition). Now, to achieve higher fluence values, the width of the scanned area was reduced keeping the length fixed at its minimum possible value of 1.2 mm. Width was reduced in similar gradual steps until the minimum possible value of 1.2 mm was reached (x-scanner off condition). Thus, the final beam scan area was 1.2 mm $\times$ 1.2 mm. The fluence of 10^{16} ions/cm^2 was reached for this area (Fig. 1(b)).

With this meticulous method we were able to irradiate a single sample to fluence values of about 10^{13}, 10^{14}, 5×10^{14}, 10^{15}, 5×10^{15} and 10^{16} ions/cm^2 in different regions (Fig. 1(b)). The advantage of this method is that it saved a lot of time. Otherwise, a lot of running time (i.e. beam time shifts), would have been required, had we started each run from the beginning (zero fluence value).

2.2. Variable tilt angle experiment

Two sets of two samples each were mounted on especially designed curved surface mounts (Fig. 2(a) and (b)) which provided a variation in tilt angle of the beam from 0° to about 45° at the same time. This curved surface mount was fabricated using a copper cylinder (of diameter $\approx$7.5 cm). Its diameter was reduced to about 6.5 cm on a lathe machine. Then one side of it was cut parallel to its diameter at a distance of about 5 mm from the surface so that the area of the flat surface was nearly 2 cm $\times$ 2 cm. This copper piece was polished using 25 μm finish sand-paper and two grooves parallel to its width were made on the flat side for tying wires. This curved surface mount was affixed to the target ladder using GE varnish, and samples were mounted over it on the curved surface (Fig. 2(a)). Two pieces of thin copper wires were tied across this mount which supported the samples, as well as acted as masks. The masked regions were used for later comparisons. It is noteworthy that the surface of the samples in this experiment were polished using Minimet 1000 machine (from Buehler Inc., USA) before mounting in order to minimize the local variations in tilt angle of the beam, which results from the roughness of the pristine sample surface. Irradiation was done at about 80 K and the final dose given to the samples was about 10^{15} ions/cm^2. Considering the maximum thickness of the curved mount the difference of temperature between two edges of the samples was not more than 2 K.

2.3. Irradiation

All the irradiation experiments have been performed in the high vacuum chamber (HVC) at the material science beam line at NSC, New Delhi.

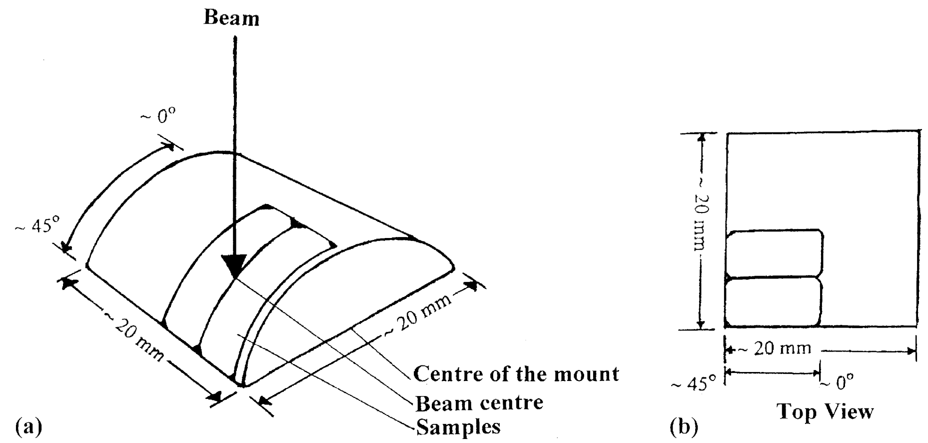

Fig. 2. Irradiation plan in variable θ experiment. (a) Schematic diagram of the curved surface mount. (b) Top view of the curved surface mount showing the position of samples and the effective values of θ at two ends.

4.64 MeV/amu ^{28}Si ions (charge state, $q = +9$) beam was used for irradiation with a flux ranging from 6.25×10^9 ions/cm^2/s to 2.5×10^{10} particles/cm^2/s (1–4 pnA beam current). Taking into account the possible beam divergence, beam intensity and accumulated flux, the fluence at the position of the samples were accurate within ±10%.

2.4. Analysis

The changes in surface morphology of the glassy samples have been examined by light microscopy, scanning electron microscopy and scanning tunneling microscopy performed before and after irradiation.

2.4.1. Light microscopy

Light microscopy has been carried out using optical microscope models Leitz–Diaplan from Leitz–Wetzlar, Germany, with Wild MPS 52 camera from Wild Leitz, Switzerland and model Olympus SZH10 with Olympus PM-10AK camera attachment from Olympus, Japan. Optical micrographs were taken at 100× and 200× magnifications.

2.4.2. Scanning electron microscopy

Scanning electron microscopy has been performed at room temperature using the instrument model JSM-35CF supplied by Jeol Ltd., Tokyo, Japan. Scanning electron micrographs (SEM) have been taken at various magnifications from 100× to 1500×.

2.4.3. Scanning tunneling microscopy

Scanning tunneling microscopy has been carried out in air at room temperature using a NanoScope II workstation from Digital Instruments Inc., USA. Scanning tunneling micrographs (STM) have been taken for 10 nm × 10 nm, 25 nm × 25 nm, 100 nm × 100 nm and 500 nm × 500 nm scan areas.

2.4.4. Roughness parameter

The roughness parameter R_q was also measured for the unirradiated and irradiated sample surfaces using the STM workstation. The roughness parameter R_q is the mean roughness and it is given by

$$R_q = \frac{1}{L}\int_0^L |f(x)|\,dx,$$

where, $f(x)$ is the roughness curve relative to the central line and L is the length of the roughness curve.

3. Results and discussion

3.1. Results of the first run

Scanning electron microscopy was carried out at different magnifications before and after irradiation for MG2705M, MG2605SC and MG2204 samples. It was clear from all the SEM pictures that the surface irregularities have smoothened due to irradiation. The heights of the ‘hills’ have decreased and ‘valleys’ have been filled giving an effect of smoothing to the irradiated surface.

Scanning tunneling microscopy carried out on the unirradiated and irradiated samples show surface modifications at nanometer length scales. The STM results for MG2705M samples are shown in Fig. 3(a) and (b) for unirradiated and irradiated samples, respectively. All STM pictures showed that at nanometer length scales, the smoothing due to heavy ion irradiation of the sample is observed as a removal or reduction in size of the sharp surface irregularities.

3.2. Results of the second run

3.2.1. Results of variable fluence experiment

All the optical micrographs, SEM and STM pictures taken before and after irradiation revealed SHI irradiation induced smoothing of the surface. It was evident from these micrographs that surface modification depends on fluence. A clear distinction between masked and irradiated regions was observed in all the micrographs for fluence values higher than about 10^{14} ions/cm^2. Typical STM pictures are shown in Fig. 4(a) and (b) for MG2705M samples for $\phi = 5 \times 10^{14}$ and 10^{16} ions/cm^2, respectively. A comparison between the STM pictures of unirradiated and irradiated samples for all MGs showed same kind of smoothing as observed in the SEM pictures. The

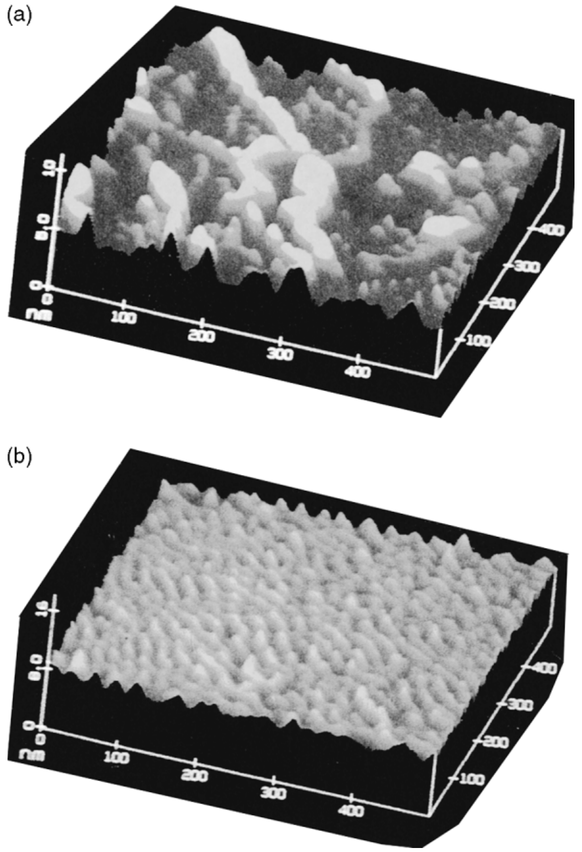

Fig. 3. STM pictures of the surfaces of metglass 2705M: (a) unirradiated, (b) irradiated with 130 MeV ^{28}Si ions ($\phi = 1.154 \times 10^{16}$ ions/cm^2).

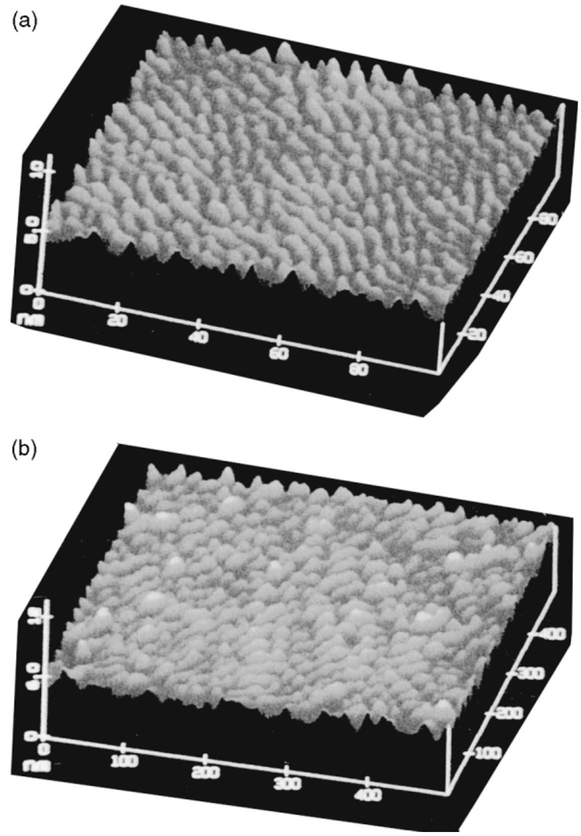

Fig. 4. STM pictures of 130 MeV ^{28}Si ions irradiated surface of metglass 2705M: (a) $\phi =$ about 5×10^{14} ions/cm^2, (b) $\phi =$ about 10^{16} ions/cm^2.

variation of mean roughness R_q with fluence for all the four MGs is shown in Fig. 5. The values of R_q for pristine samples were 6.02 ± 0.45, 5.72 ± 0.72, 5.95 ± 0.57 and 5.81 ± 0.64 μm, respectively, for MG2705M, MG2826MB, MG2605SC and MG2204 (points not shown in Fig. 5). It is evident from the figure that in the investigated fluence range R_q decreases logarithmically with ϕ indicating smoothing of the surfaces with increasing dose.

3.2.2. Results of variable tilt angle experiment

Optical micrographs for the MG samples showed tilt angle dependence of SHI induced surface modification. It was also evident from the micrographs of MG2705M and MG2605SC that surface modification occurred in the form of orientation of surface irregularities in the direction of shear flow. Grain size of the surface irregularities has also been modified due to irradiation. Surface modification was more distinct for $\theta = 45°$ irradiation than that for $\theta = 0°$ irradiation, in all of these micrographs.

A clear distinction between the masked (unirradiated) and exposed (irradiated) regions was visible in all of the SEM micrographs and smoothing of the surface due to irradiation was evident. However, the modifications towards $\theta = 0°$ side were less significant than those appearing towards $\theta = 45°$ side. The STM pictures are shown in Fig. 6(a) and (b) for MG2705M sample for $\theta \approx 0°$ and 45 °, respectively. Surface smoothing as observed in the SEM pictures, was evident in these pictures also. The mean roughness R_q decreases linearly

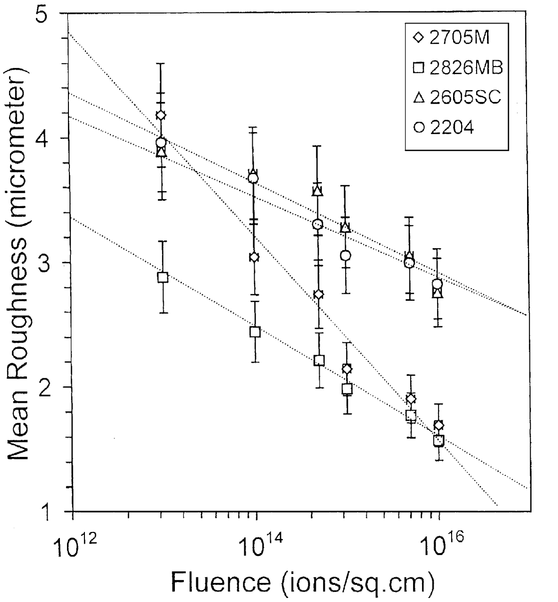

Fig. 5. Variation of mean roughness with fluence for 130 MeV ^{28}Si ions irradiated metallic glasses. The straight lines are shown just to guide the eyes.

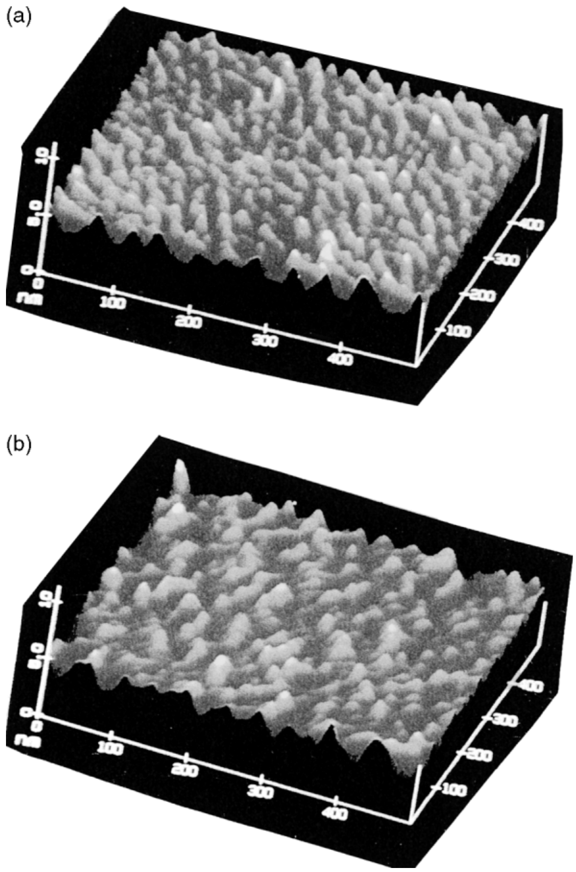

Fig. 6. STM picture of 130 MeV ^{28}Si ions irradiated surface of metglass 2705M: (a) $\theta = 0°$, (b) $\theta = 45°$; dose = 10^{15} ions/cm^2.

with θ for a given dose, as shown in Fig. 7. The starting roughness parameter R_q of the polished unirradiated surfaces were determined to be 3.05 ± 0.52, 3.28 ± 0.49, 3.30 ± 0.38 and 3.00 ± 0.54 μm, respectively, for MG2705M, MG2826MB, MG2605SC and MG2204 (not shown in Fig. 7).

3.3. Discussion

Materials subjected to SHI irradiation behave like viscoelastic fluid [6,7]. The surface smoothing observed in our samples during first and second runs can be qualitatively explained on the basis of shear flow mechanism within the framework of viscoelastic model [6–9]. According to the theory of shear flow, fast ions incident on the target set a large number of near surface atoms into motion. During subsequent relaxation, a net shear flow, depending on the tilt angles θ (angle of the direction of ion-beam with the z-axis) and ψ (angle between projection of the beam direction on x–y plane (Fig. 8), sample surface is assumed to be in this plane, and the x-axis), and fluence of the beam, is induced on the irradiated surface. These shear velocities lead to a net flow of mass that gives the surface modification.

Starting with the expression for local rate of deformation [8],

$$\frac{\partial v_x}{\partial z} + \frac{\partial v_z}{\partial x} = 6A\varphi \sin\theta \cos\theta, \tag{1}$$

where v_x and v_z are the shear velocities in x and z directions, respectively; A is the deformation yield of the sample corresponding to the S_e at surface and ϕ is the ion flux.

Assuming that the local coordinate system is oriented in such a way that $\psi = 0$ holds and the $z = 0$ at the surface. The implantation depth is then $z = -R_p \cos\theta$, where R_p is the projected range of the ions. For an ideally planar surface

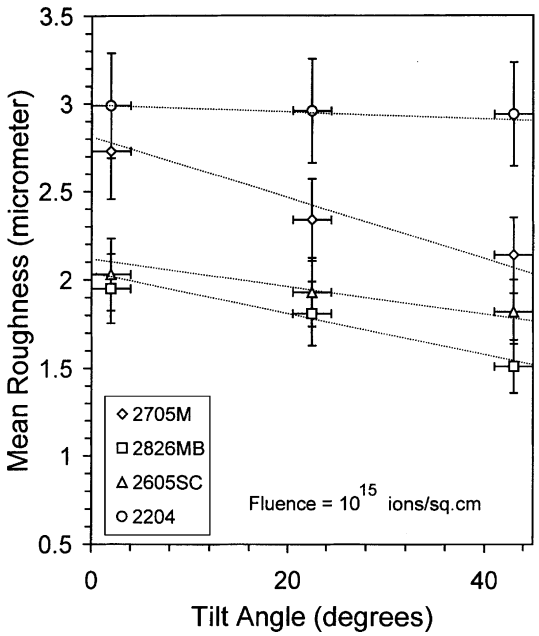

Fig. 7. Variation of mean roughness with tilt angle of the beam for 130 MeV ^{28}Si ions irradiated metallic glasses. The straight lines are shown just to guide the eyes.

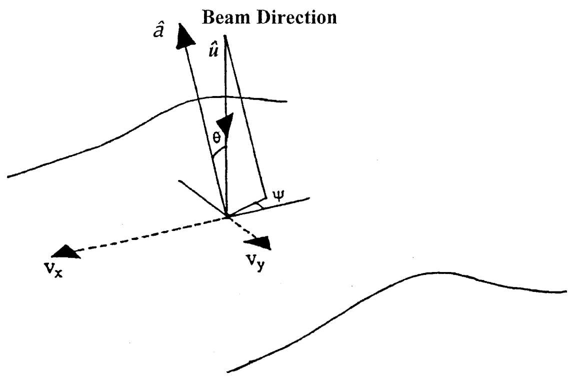

Fig. 8. Schematic diagram showing irradiation of a rough surface by normally incident beam. $\hat{a}$ represents the direction of the normal to the surface. The directions of shear velocities are also shown by broken lines.

a steady-state solution is possible with $v_z = 0$ and the shear velocity at the surface is

$$v_x = 6\varphi \sin\theta \cos\theta \int_{-R_p \cos\theta}^{0} A[S_e(Z)]\mathrm{d}z. \tag{2}$$

The evaluation of the integral requires the knowledge of $A(S_e)$, which is not known for most glasses. However, as a first approach, for 130 MeV ^{28}Si ions, S_e may be taken as constant along the ion range. With this assumption, integrating Eq. (2) and writing φt as ϕ (ion fluence), we get an expression for the shear shift Δx of the near surface atoms (using expression for A from [8]).

$$\Delta x = v_x t = 6\phi A \sin\theta \cos\theta (R_p \cos\theta)$$
$$= 6R_p\phi \frac{1.16(1+v)\alpha S_e}{3e(5-4v)\rho C} \sin\theta \cos^2\theta. \tag{3}$$

Here, v, α, ρ and C are the Poisson number, thermal expansion coefficient, volume density and specific heat capacity of the material, respectively. The value of C is approximately given by $3k_B N_A/M$, where k_B is the Boltzmann constant, N_A is the Avogadro number and M is the molar weight of the material.

If we consider an irradiation experiment in which the ion beam is incident normally on the sample, these tilt angles are actually given by the local variations in the angle of incidence due to the roughness of the surface. It is clear that for slant surface irregularities as in the case of slope of the 'hills', the value of θ is between $-\pi/2$ and $\pi/2$. Hence, the contribution of $(\sin\theta\cos\theta)$ is large compared to the case when θ is near 0 (at the peaks and shoulders of the 'hills') or $\pi/2$ (completely erect irregularities). However, the area covered by the peaks of the 'hills' is very small and completely erect surface irregularities are very less probable. Therefore, most of the irradiated surface area is affected by the shear flow and hence the surface topography modifies. It is also worth mentioning here that even the small regions where the shear flow is very small, do not remain the same during an irradiation process, because their neighbouring areas flow away very soon, leaving them unsupported. As a result, the shear flow gives an overall modification of the irradiated surface.

The whole surface topography keeps changing during a certain irradiation period. This gives continuously changing values of the tilt angles θ and ψ. Therefore, it is not possible to determine the exact values of these angles, and hence their contributions to the shear flow during irradiation.

However, the final contribution of these angles to the shear flow is finite and non-zero, and it depends on the starting surface topography of the target.

Qualitatively, to explain the smoothing observed in our samples irradiated with a normally incident beam up to a fixed fluence, we consider a surface as shown in Fig. 8. For such a surface, since $\psi < 45°$ for most of the regions, the shear velocity v_x would be larger than v_y. This would lead to a larger mass flow in the x direction than that in the y direction. As a result, the "channels" (space between the "hills") would become wider. The height of the "hills" would decrease and the flowing mass would fill the "valleys".

In the variable θ experiment, the effect of tilt angle was investigated. The surfaces of the unirradiated samples in this experiment were polished in order to minimize the local variations in tilt angle of the beam, which results from the roughness of the surface. The tilt angle at a given point on the sample surface was estimated from the geometry of the curved surface mount used in the experiment. Taking into account these facts, the tilt angle used in calculation of shear shift in x direction (Δx) was therefore reliable within $\pm 2°$.

To explain our results of second run quantitatively, we have used the method adopted by Gutzmann and Klaumünzer [5]. The value of Δx was experimentally determined by identifying some distinct irregularities present on the surface of pristine samples, as evident in SEM pictures, and using them as 'markers'. Same region on the surface was carefully spotted after irradiation and the change in length of the space between two 'markers' was measured. This change in length gives the value of Δx. However, shear shifts larger than 500 μm could not be measured experimentally. Occurrence of such large shifts destroys the starting features of the surface completely, making the identification of same regions after irradiation nearly impossible. Mathematically, the shear shift was calculated using the material parameters given in Table 1 and Eq. (3).

The calculated and experimentally determined results of Δx have been plotted in Figs. 9 and 10 for variable ϕ and variable θ experiments, respectively. It is evident from the figures that the experimental values of Δx are in good agreement

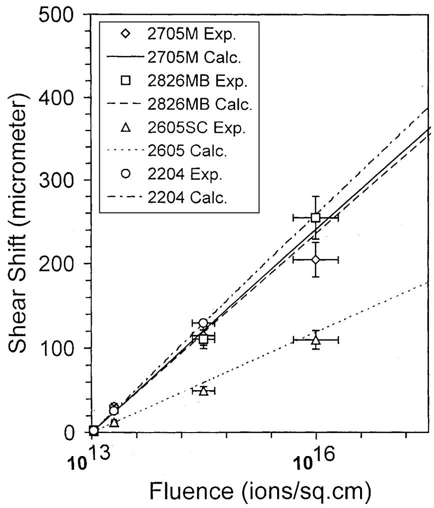

Fig. 9. Variation of shear shift in x-direction with fluence for 130 MeV ^{28}Si ion irradiated metallic glasses.

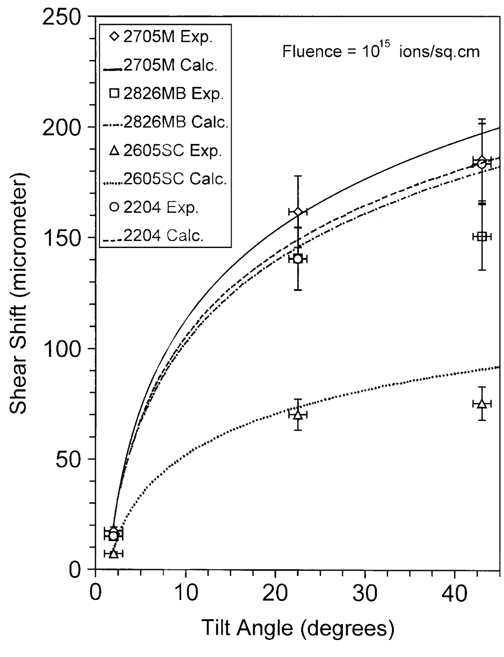

Fig. 10. Variation of shear shift in x-direction with tilt angle for 130 MeV ^{28}Si ion irradiated metallic glasses.

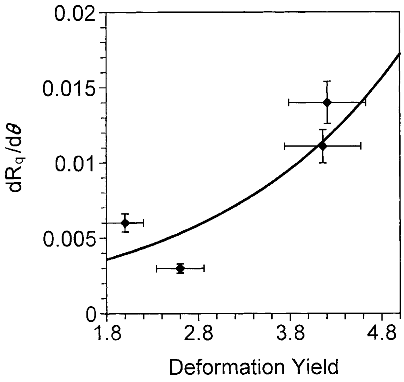

Fig. 11. Variation of rate of decrease of mean roughness with respect to tilt angle ($dR_q/d\theta$); with deformation yield A_0 corresponding to 130 MeV ^{28}Si ion irradiation of metallic glasses.

with the calculated values. Maximum shear shift has been observed for MG2705M samples followed by MG2826MB, MG2204 and MG2605SC samples, respectively. This may be attributed to the fact that MG2705M has largest deformation yield and the other MGs have gradually decreasing values of A_0 in the same sequence. Same argument could be given to explain the rate of decrease of R_q with increasing ϕ and θ. For a comparison we have plotted the $dR_q/d\theta$ calculated from Fig. 10 with deformation yield in Fig. 11.

4. Summary and conclusion

SHI irradiation of glassy metals at a given temperature leads to modification of the irradiated surface. Even if S_e is less than the threshold of track formation, the surfaces get modified substantially. The observed modification depends on the ion fluence and tilt angle for a given S_e in certain material. A detailed investigation of this phenomenon for four MGs submitted to 130 MeV ^{28}Si ions irradiation showed the following observations:

(1) Smoothing of surface is the first process occurring on the irradiated amorphous solids as indicated by logarithmically decreasing mean roughness of the surfaces with increasing dose.

(2) As the tilt angle of the incident beam increases, mean roughness decreases linearly resulting in more smoothing.

The smoothing of the surfaces of amorphous solids by SHI irradiation is attributed to the mass flow resulting from the shear shifts of near surface atoms during subsequent relaxation. It is worth mentioning that the formation of regular undulations or roughening as reported earlier [4] was not observed in our experiments. This may be due to the fact that S_e was less by a factor of about 3.5 in our case as compared to that in the earlier studies. SHI induced effects are governed mainly by S_e and larger S_e is likely to affect a thicker layer near the irradiated surface causing more mass to flow under shear velocity, resulting in 'wave' formation or even roughening. Within the S_e range used in our experiments, the starting surface topography does get destroyed after certain dose of ions, but the affected thickness was too less to provide enough mass to flow and result in regular undulations or roughening.

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