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Experimental investigation of the sputter-topographic evaluation of a cylindrical surface

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An experimental method is used to develop the evolutionary contours of circular surface erosion produced by ion bombardment. Silica glass fibres are bombarded from one direction, normal to the fibre axis, with Ar^+ ions at energies from 2 to 16 keV, and the fibre cross-section profile variations are observed as a function of ion fluence. The results indicate that two equilibrium optimum sputtering rate angles obtain, one on each side of the convex hemispherical fibre surface, and the slopes defined by these angles move parallel to themselves into the surface and eventually intersect to form an isosceles triangle with rounded base. Sequential profile variations show close agreement with theoretical predictions, and it is evident that the apex angle varies as a function of energy for the range investigated.

1. Introduction

Numerous publications on the subject of surface topography resulting from ion bombardment have appeared in recent years. It seems that the interest in these efforts are directed towards applications, and to an attempt to understand the basic mechanisms of the sputtering process as well as to learn more about the structure of the surfaces being etched. Much of the work has been experimental, and even though there exist extensive publications, a very limited amount of quantitative data are available. Theoretical investigations have recently predicted changes in specific surface geometries as a function of ion dose, i.e. surface removal depth and, therefore, it appears appropriate to provide quantitative experimental results for the purpose of corroborating or questioning these predictions.

In reviewing the pertinent literature, Ducommun *et al.* [1] and Barber *et al.* [2] have analytically investigated, among other geometries, the surface contour developed by the erosion of a spherical profile. Carter and coworkers [3-6], considered curved surfaces and formulated theories on the development of topographical features based on the differential motion of a point on a surface, and compared their findings to Stewart and Thompson [7] who applied a theory of rectilinear motion of a pair of intersecting plane surfaces. Most of these authors relate to the work by Frank [8] who, in 1958, resolved the problem of dissolution by chemical etching. Witcomb [9] has investigated the cone apex angle formed at the top of protruding surface structures of crystalline targets and reported mostly low energy experimental results. He considered several possible calculations to predict the angle. Sigmund [10, 11] investigated the topographies of ion-bombarded surfaces, relating them to collision theory and atomic displacement, and predicted that the apex angle depends only weakly on the energy of the ion.

There exist many experimental studies of surface topography too numerous to mention here. The work by Tsong and Barber [12] on silica is related to our studies, as well as yield data on silica glass as a function of ion-impingement angle and energy by Bach [13] and Edwin [14]. Bayly [15] has also sputtered silica glass and analytically predicts evolving structure in sputter-topographies as a function of initial surface geometries. Wehner [16] and Wehner and Hajicek [17] have published results of the

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sharpening of round wires and spheres produced by the influence of angle of incidence sputtering and reported some low energy apex angle values.

In this paper, some initial findings of the erosion of cylindrical cross-sections of silica glass (amorphous) by Ar^+-ion bombardment are reported. The evolution of the changing surface contour is examined as a function of ion dose, the "equilibrium optimum sputtering rate angle" (EOSRA) is obtained, and we report how this angle varies with ion energy.

2. Experimental method

The experimental method used in this investigation consists of ion bombardment of silica glass fibres at right angles to the axis of the fibre. The eroded fibres are then severed at various points along their length, and the cross-sectional profiles are observed by metallograph.

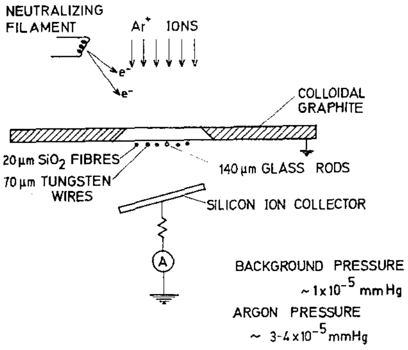

Figure 1 Experimental apparatus.

The experimental arrangement is shown in Fig. 1. Ar^+ ions are generated in a magnetron source with a hot cathode and are extracted and aligned using Pierce lenses. Energies of 2 to 16 keV are used and the current densities varied from 40 to 240 μA cm^{-2}. Thermal electrons are introduced into the ion beam near the target to reduce the positive charge that accumulates at the dielectric surface. The vacuum is attained using an oil diffusion pumped system, and the argon pressure during beam operation was approximately 3 to 4 × 10^{-5} mm Hg. The near parallel flux of ions move in a field-free region and some of the ions impinge at right angles onto the fibres mounted on a target holder ring. The major part of the ion beam continues on to the collector plate where the ion-beam density is measured with adjustment for electron emission. The beam width is ~ 3 cm diameter, and the ion density is highest in the centre of the beam and becomes much lower towards the beam edge, resulting in a variation of ion dose with position along each fibre. This property is used to obtain varying fibre profiles as a function of the amount of erosion. The time required for a sputter run is from 1.5 to 5 h, and a total maximum ion fluence of 1.5 × 10^{19} ions cm^{-2} is used. This heavy dose results in an appreciable amount of the fibre cross-section being removed.

The targets bombarded for these investigations were vitreous silica fibres of 20 μm diameter. Vitreous silica is amorphous and believed to be homogeneous in composition. X-ray diffraction patterns were made of the fibres to substantiate this structure.

Following the heavy bombardment of the target fibres, each fibre is removed from the target holder and mounted so that the end of the fibre could be viewed in a metallograph. A Reichart metallograph was used at magnifications of about × 675, and reflected light, as well as dark-field and transmitted light were used. After photographing each cross-section, the fibre is subsequently severed at about 0.5 mm intervals and the new profile observed. It was not always possible to obtain a clean break of the fibre, and a shattered or slanting fracture across the fibre prevented a good outline of the fibre profile to be photographed. This was especially apparent when the fibre was sputtered to a very thin cross-section.

Fig. 2 shows two fibre cross-sections; one was sputtered at 6 keV and the other was not sputtered. The profiles show the original round fibre geometry and the resulting geometry following ion bombardment from one direction as noted by the arrow. The profile of the sputtered fibre shows the two dimensional "cone"

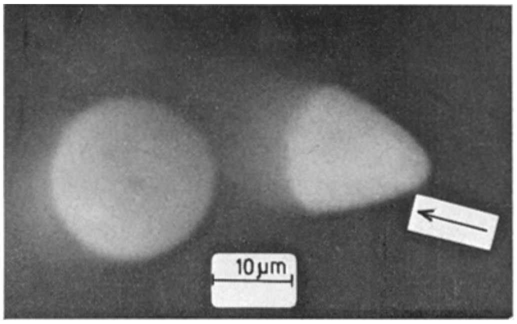

Figure 2 Two silica fibre profiles; one unsputtered, the other sputtered at 6 keV.

form predicted, and the original rounded unsputtered far side of the fibre.

The profiles are recorded in microphotographs where the negative can be enlarged for refined angle and geometry measurements. Angle and other dimension measurements were also made using a rotatable and measuring eyepiece on the metallograph. The apex angle measurement represents an average fit of a straight edge along this slightly curved slope. The accuracy of these measurements is within 2° when the full apex angle is measured. This error is reduced to 1° when the values of the ion impingement angle is calculated by taking the complement of half the apex angle.

3. Results

Numerous micrographs were taken of the cross-sections of silica glass fibres bombarded at energies of 2, 3, 6, 9, 12 and 16 keV. Variation in ion dose along the fibre produced heavily eroded profile geometries at the centre of the fibre length, while the ends of the fibres were only slightly sputtered. Some of the micrographs are included here to illustrate the variation in the profile geometry as a function of ion dose and energy.

Fig. 3 shows the cross-sectional profile of a fibre sputtered at 9 keV and with low ion dose. The foremost surface (that surface directly impacted by the ions) is rounded slightly more than the original fibre surface and the sloping sides are beginning to form on each side of the cylindrical surface. The back or unsputtered surface of the fibre remains rounded with the radius of the original fibre. Fig. 4 shows a 9 keV bombarded fibre that has evolved a subsequent contour following additional bombardment. The radius at the front has become smaller, and the sides longer and straighter. The flat surface structure, planar with the photograph's surface, reveals only the fractured face of the fibre and does not contribute to our information; the profile outline is the important feature with which we are concerned in this study. (It may be, however, that the microstructure very near the bombarded surface, i.e. within ion penetration and damage depth, is significant.)

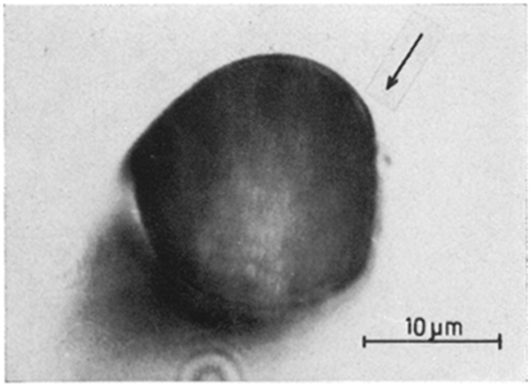

Figure 3 Low dose eroded fibre, 9 keV.

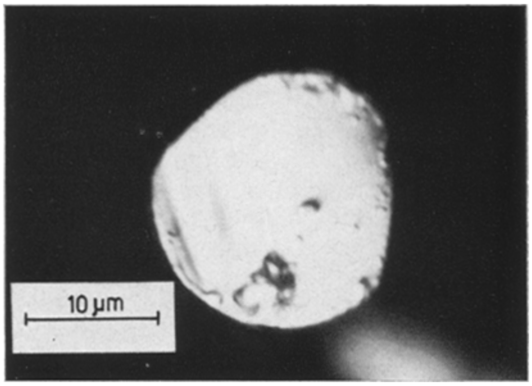

Figure 4 Reflected light micrograph of a 9 keV bombarded fibre.

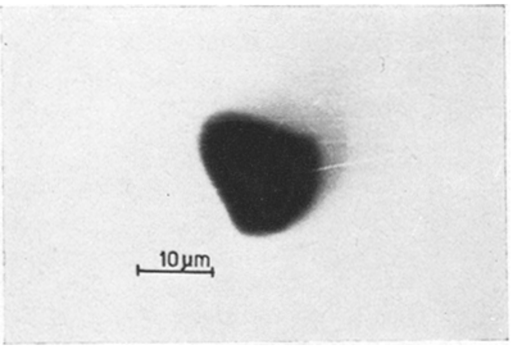

Figure 5 Medium dose eroded fibre, 6 keV.

Fig. 5 shows how the profile geometry evolves with further erosion. The fibre shown is a 6 keV bombarded fibre where it is evident that the sides have become quite long and flat and the cone apex radius has been considerably reduced. The 6 keV bombarded fibre shown in Fig. 6 has received very high ion dose. The sloping sides have now intersected and have become almost flat.

When looking at the next set of micrographs, in the first, (Fig. 7a, a 3 keV bombarded fibre) we can see that the apex angle of the cone is

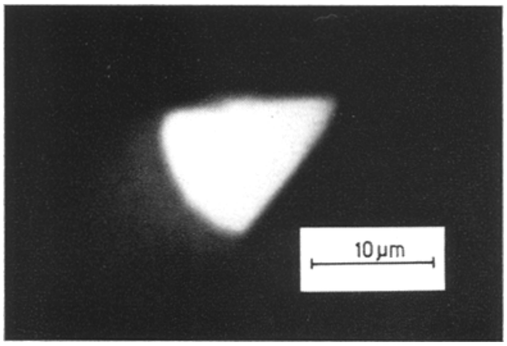

Figure 6 Heavily eroded fibre bombarded at 6 keV.

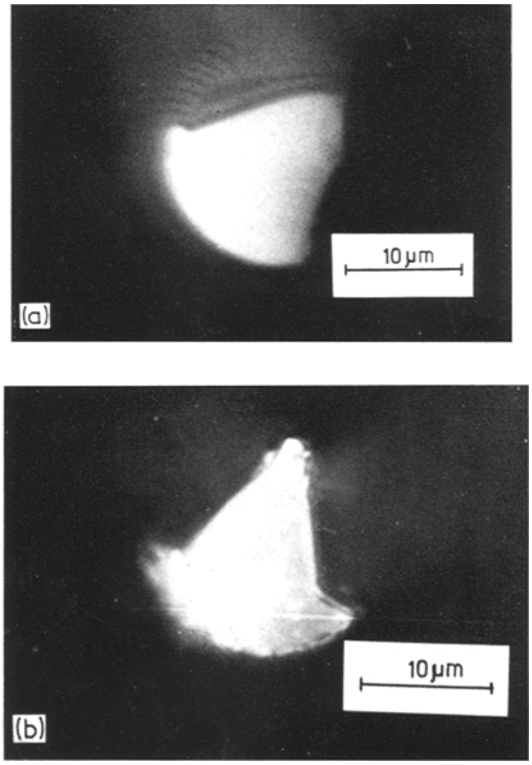

Figure 7 (a) 3 keV bombarded fibre showing ledges or steps; (b) enlargement of ledge size as erosion continues.

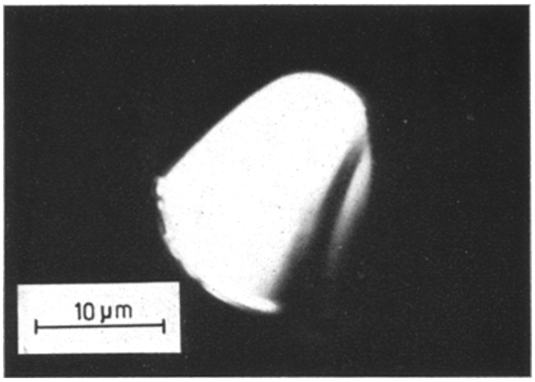

Figure 8 16 keV sputtered fibre, low ion dose.

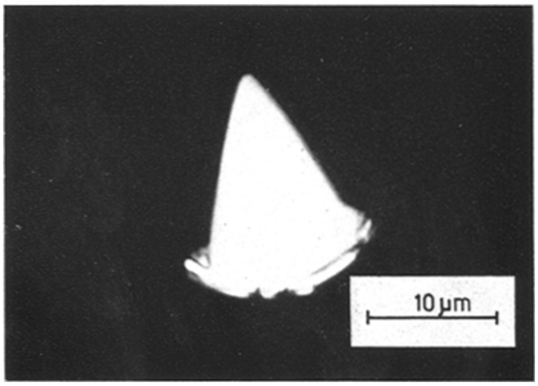

Figure 9 Medium dose 16 keV sputtered fibre.

larger than that for the 6 and 9 keV cases, and steps or ledges are also noticeable at the base of the sloping sides. These ledges are usually symmetrical on both sides of the fibre, and, as the sputtering continues, the ledges become wider as the cone is reduced by the more efficient erosion at oblique angles. The ledges recede downwards but at a slower rate than the cone sides. This contour change is shown in Fig. 7b. Fig. 8 shows a fibre sputtered at 16 keV. Careful inspection reveals the first evidence of ledge formation on the low ion dose profile. The step can be observed only on one side because the fibre did not break cleanly. Again the ledges become larger as sputtering continues (Fig. 9).

Ledges were always observed on fibres bombarded at 2, 3, and 16 keV. On fibres bombarded at 6, 9, and 12 keV, however, the ledges were never observed. A discussion of possible explanations for the formation of these structures is given below.

4. Discussion

The experimental method used in this investigation to study macrostructure does not directly examine the microstructure of surface topography produced by ion bombardment. However, we believe the macrostructural contours, e.g. in the 10 to 20 µm range, in general reproduce the same apex angle and geometrical evolutionary sequence found in the microstructure. (Of course, this does not include the atomic dimension structures as treated by Sigmund [11] and Wilson [18].)

When round surfaces such as the fibres are bombarded, it is obvious that the initial sputtering takes place over all impingement angles, from normal incidence to very oblique angles. Slopes form on both sides of the rounded surface and become almost straight upon further erosion. These two equilibrium angle surfaces once formed, generally move inwards simultaneously. Fig. 10 shows four projected fibre profiles produced by varying ion doses at 16 keV superimposed. Their slopes never become completely flat until the planes intersect, at which time a sharp apex angle forms. Fig. 10 also includes a theoretical set of profiles obtained by using Edwin's [14] values for Ar$^+$ ions on silica at 16 keV, and the erosion slowness curve method developed by Barber *et al.* [2]. The relative geometrical fit of the theoretical and experimental results is shown.

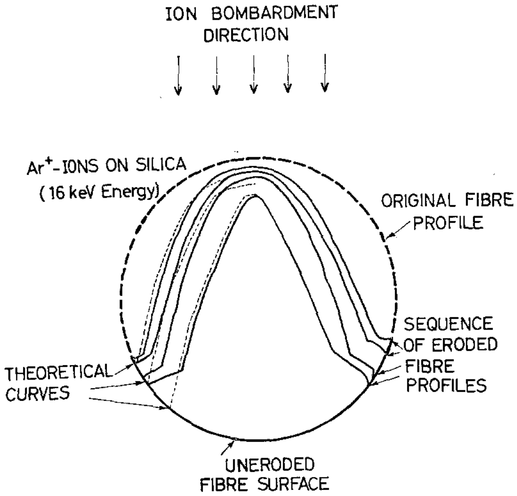

Figure 10 Overlay of sequentially eroded fibre profiles (solid lines); erosion slowness curve (dashed lines).

The ledges or steps that are apparent at the base of some of the sputtered slopes were not expected and are not easily explained. We have considered the possibility of material differences on the outer surface of the fibre, but do not believe this to be the cause. It is possible, however, that there exists a lack of charge neutralization on the dielectric surface even though care was taken to make sufficient electrons available to neutralize this charge. If such a positive charge exists, it could cause the ions impinging at the very edge of the fibre to be repelled or reflected slightly. This would result in no erosion at this furthest protruding point at the outer edge of the fibre. The ledge that would then form, would have a much slower sputtering rate, i.e. near normal incidence yield, than the oblique incidence rate experienced at the optimum sputtering rate angle. This mechanism does not, however, explain why the ledges only form at certain ion energies. It may be possible that the decrease in the sputtering rate at high impingement angles may play a part in the ledge formation, as discussed by several authors [2, 4]. To our knowledge, however, there is nothing in the theoretical considerations that directly explain this phenomenon.

One benefit ensuing from ledge formation is that when sputtering for long periods and the fibre is heavily eroded, the cone that forms can be observed to recede down towards the relatively flat surface formed by these ledges. In other words, the two-dimensional cone is tending to become level with the surface as predicted in some studies.

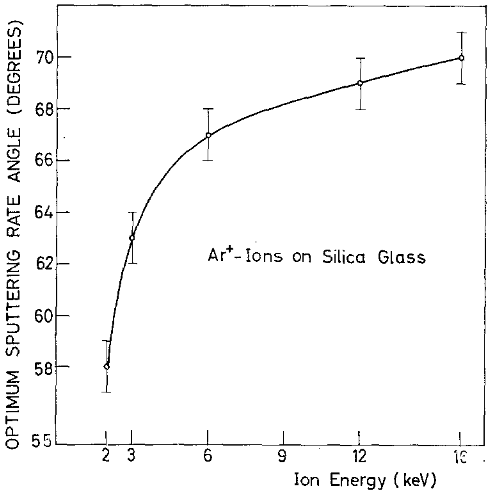

Figure 11 Equilibrium optimum sputtering rate angle versus ion energy.

The apex angle of the amorphous material varies as a function of ion energy. When this angle is reduced to the ion-impingement angle (the angle which the ion beam makes with the normal to the surface) and plotted as a function of energy, we obtain the curve as shown in Fig. 11. These values are accurate to ± 1.0° and range from 70° at 16 keV to 58° at 2 keV. This equilibrium angle which develops early in the erosion sequence is retained as sputtering continues as the theories predict.

The angle which we call the equilibrium optimum sputtering rate angle (EOSRA) is not to be confused with the maximum sputtering yield (i.e. atoms/ion) angle. The maximum sputtering yield angle is usually found at larger angles and does not obtain as the equilibrium angle because the ion flux decrease at these larger angles results in a substantial reduction in the erosion rate of the sloped surface, i.e. reduced by the cosine of the angle.

The variation in the EOSRA (or cone angle) with energy is not in agreement with Sigmund [10]. The angle levels out at 9, 12 and 16 keV, and appears to approach a constant angle as the energy increases further.

5. Conclusion

A technique has been devised for observing the geometry of right cylindrical cross-section fibre profiles under ion bombardment and erosion. We have found slopes, noted as the equilibrium optimum sputtering rate angle, are formed on each side of the hemispherical surface and these slopes move parallel to themselves as predicted by theory. The sides, which sputtered at a rate higher than other impingement angles, finally intersect and form an isosceles triangular geometry with a rounded base. The slopes that are formed, and hence the apex angle, vary as a function of ion energy. Steps or ledges developed at the lower end of the slopes at certain energies; these are being investigated further. As all angles of impingement are used in the bombardment of a cylindrical surface, the method is believed to be useful in determining sputtering yields as a function of impingement angle. This possibility is being studied further using the contour data obtained.

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