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Influence of the ion incidence angle of alumina surface cleanness, roughness and topography

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Polycrystalline ceramic specimens of 96% sintered alumina and mechanically polished 99.5% alumina were bombarded by Ar^+ ions at an applied voltage of 7 kV at various incident angles from 0 to 1.4 rad (0 to 80°) and at a beam current of about 70 μA. The influence of the incidence angle on alumina surface cleanness, roughness and topography was investigated.

1. Introduction

The ion bombardment of solids can cause erosion (or ion-etching) in which atoms are ejected from the solid surface as a result of the collision processes [1]. A variety of factors affect the rate at which material is removed from the surface and include the ion mass [2–6] and energy [7, 8] and its angle of incidence [9, 10] onto the specimen surface. As a result, ion bombardment may lead to a selective etching of the surface and reveal underlying structural features [11]. Reports in the literature have indicated that the process of ion etching can be applied to reveal subsurface features of biological tissues, the technique being of potential value in combination with scanning electron microscopy [12–15]. However, contrary to the initial optimistic opinions expressed by Stuart *et al.* [12], Hodges *et al.* [16] concluded that the ion etching was unlikely to be of much value with soft biological tissues. Ion etching has recently been utilised in attempts to modify the surface morphology and chemistry of biocompatible materials, such as metals, alloys, polymers and ceramics [17–21]. These modifications may influence the usefulness and biocompatibility of the materials. The chief purpose of this paper is to investigate how the surface cleanness, roughness and topography of alumina ceramics are changed during Ar^+ irradiation from the hollow-anode ion gun [22–25].

2. Experimental procedures

Polycrystalline ceramic specimens of 96% sintered alumina and mechanically polished 99.5% alumina, with the material properties listed in Table I, were bombarded by Ar^+ ions at an applied voltage of 7 kV at various incident angles from 0 to 1.4 rad (0 to 80°) and at an ion current, measured by Faraday cage situated in the place of the specimen, of about 70 μA. Alumina ceramics are commonly used as a substrate material for thick film circuits. Recently it has appeared that they could be a satisfactory biological implant material. It is well known that under ionic bombardment, alumina specimens tend to become positively charged resulting in the deflection of other ions with decreased erosion of the charged area. This effect can be reduced by the use of the glow-discharge ion gun with a hollow anode, which is the source of a neutralized ion-beam [25]. The

TABLE I Material properties of alumina ceramics

Property	Material: Alumina, 96%*	Alumina, 99.5%†
Softening point (K)	1923	–
Thermal conductivity at 298 K (watt cm^{-1} K^{-1})	0.35	0.37
Thermal expansion coefficient (ppm K^{-1})	6.4	–
Density (g cm^{-3})	3.7	3.86
Log volume resistivity (Ω cm) at (a) 298 K	–	16
(b) 573 K	10	–
(c) 773 K	7.9	–
Dielectric constant	9.3/1 MHz	9.3/10 GHz
Loss tangent	0.0028/1 MHz	0.0002/10 GHz

*See [26].
†Made in Poland – No data available.

ion-beam irradiation was performed in an experimental apparatus, similar to that described elsewhere [27].

A scanning electron microscope, (SEM), the Stereoscan 180 equipped with an X-ray detector was used to determine the amounts of Fe, Cr and Ni, from the cathode of the ion gun, which were deposited on the alumina surface. Another scanning electron microscope, a JEOL JSM-35 was employed to document the microstructure formation (topography). Another area of interest was the roughness of alumina surface. It was calculated from the shape of a surface profile, which was recorded by a profilograph, the ME 10 (supplied by VEB Carl Zeiss, Jena).

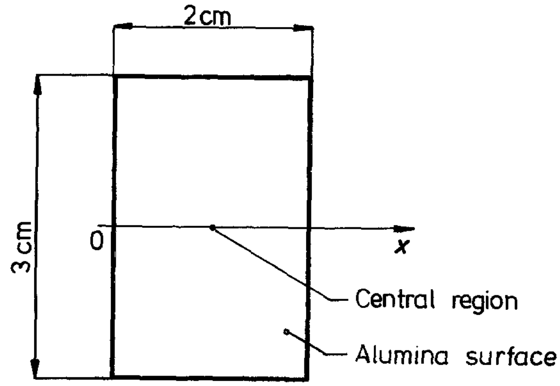

Figure 1 The alumina sample used in the experiment, showing the central region, the region where etching rate was maximal and x, the direction of the cleanness investigation.

3. Results

3.1. Cleanness of the alumina surface after Ar^+ irradiation

After Ar^+ irradiation the alumina surfaces in the form of plates 2 cm x 3 cm were examined by the SEM equipped with an X-ray detector to determine the amount of cathode material impurities on the surface. The cathode of the gun used in the experiment was made from Type 1H18N9T stainless steel: i.e., Fe–20 wt% Cr–11 wt% Ni with Mn, Si, Ti and C as minor constituents. During ion etching of a sample, the cathode material, in this case Fe, Cr and Ni, was deposited on the surface of the sample due to erosion of the cathode hole. Simultaneously this material was sputtered, with different sputtering ratios for Fe, Cr and Ni, due to ion bombardment of the alumina surface. In the central region of the plate, where the sputtering rate was maximal, the levels of metal contamination were on the limits of detection by SEM/EDS. A cleanness investigation was performed* from point to point of the surface in x-direction, see Fig. 1, from the edge of the alumina plate to the central region, where etching rate was maximal. The typical experimental results for perpendicular bombardment of the surface shown in Table II indicated little change in the surface cleanness in the vicinity of the central region as a result of ion etching process. The amount of cathode material on the alumina surface decreased with increasing distance x. A minimum of cathode impurities were measured in central region ($x = 8$ mm) of the alumina substrate. Similar results were obtained for oblique ion incidence.

3.2. Changes of the surface roughness

The surface roughness is a very important parameter, which must be considered in preparation of thin foils of solids for TEM and biological implant materials. The parameter mainly depends on the ion incidence angle on surface of ion bombarded material. The roughness of the 99.5% alumina surface was calculated from the shape of the surface profile recorded by the profilograph. The calculation method is shown in Fig. 2. The main purpose of the investigation was firstly to estimate the changes of alumina surface roughness as a result of the ion etching process and secondly to determine the influence of the ion incidence angle, θ, on alumina surface roughness. Introducing a coefficient $K = R_A/R_B$, where R_A is the mean roughness after Ar^+ irradiation of alumina surface and R_B is the mean roughness before ion bombardment, shows the changes of the roughness. The $K(\theta)$ function against angle of incidence, θ, for

TABLE II Amount of cathode material on the 96% alumina surface after Ar^+ irradiation

Element	Amount of impurity (%) at a distance x (mm)									
	0.1	1	2	3	4	4.5	5	6	7	8
Fe	2.7	3.7	4	4	2.6	1.7	0.7	0.2	0.08	0.01
Cr	0.3	0.9	1.1	1	0.8	0.6	0.2	0.07	–	0
Ni	0.15	0.5	0.4	0.4	0.3	0.2	0.07	0.03	0.03	0

*The investigation was performed in Laboratory of Electron Microscopy, Technical University of Wrocław.

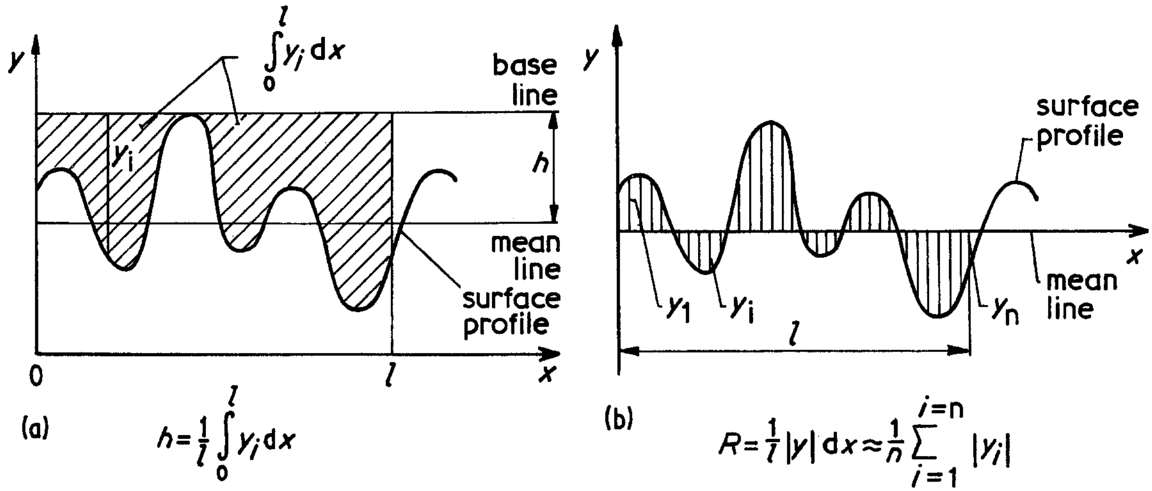

Figure 2 The method of surface roughness calculation showing determination of (a) the mean distance, h, of the base line from the surface profile and (b) the mean distance R of the surface profile from the mean line.

Ar^+ ions on 99.5% alumina is depicted in Fig. 3. A substantial increase of surface roughness ($K > 1$) is seen for perpendicular bombardment, i.e., $\theta = 0$ rad. For very oblique ion incidence the roughness is greatly reduced.

3.3. Topography of ion-etched alumina surfaces

In addition to the changes of the alumina surface roughness, the development of surface topography was observed during ion-bombardment. At normal ion incidence, $\theta = 0$ rad, the initially rough topography changes with a tendency to overall smoothing, but simultaneously some roughening occurs due to the preferential etching of the pore walls, see Fig. 4. Both these processes determine the final state of the surface roughness (Fig. 3) and topography (Fig. 4), whichever material is irradiated.

To compare the bombarded alumina surface topography with the initial one, to produce a cross-section through the material and to measure the removed material thickness, the alumina surface was partially screened by an Mo foil [28]. The sharp slope between screened surface- and ion-etched area is shown in Fig. 5. At oblique ion incidence a tendency to smoothing of the alumina surface is seen (Fig. 6). On the other hand a "scale-shaped" structure also could be observed. To explain the "scale-shaped" structure one should use the mechanism suggested by Teodorescu and Vasiliu [29] which takes into consideration the initial geometry of the sample surface and the strongly angular dependence of the sputtering yield.

4. Concluding remarks

The most notable aspects of the Ar^+ ion irradiation of alumina surfaces are (a) the fact that the cleanness of the surface is rather independent of ion incidence angle and (b) the fact that the surface roughness and topography depend strongly on incidence angle. Therefore, the required surface roughness and topography could be obtained by choosing a suitable angle of incidence.

The surface roughness is a very important parameter, which must be considered in preparation of specimens for TEM and biological implant materials. The roughness of the specimens for TEM must be reduced, as much as possible. For that reason ion thinning process should be performed on an extremely oblique ion incidence angle. This situation reduces the depth of radiation damage [30] but also results in very low rates of ion etching. The best solution seems to be to

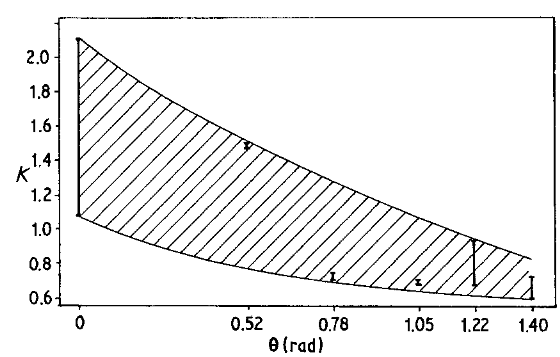

Figure 3 Angular dependence of the K function for Ar^+ irradiation of 99.5% alumina surface.

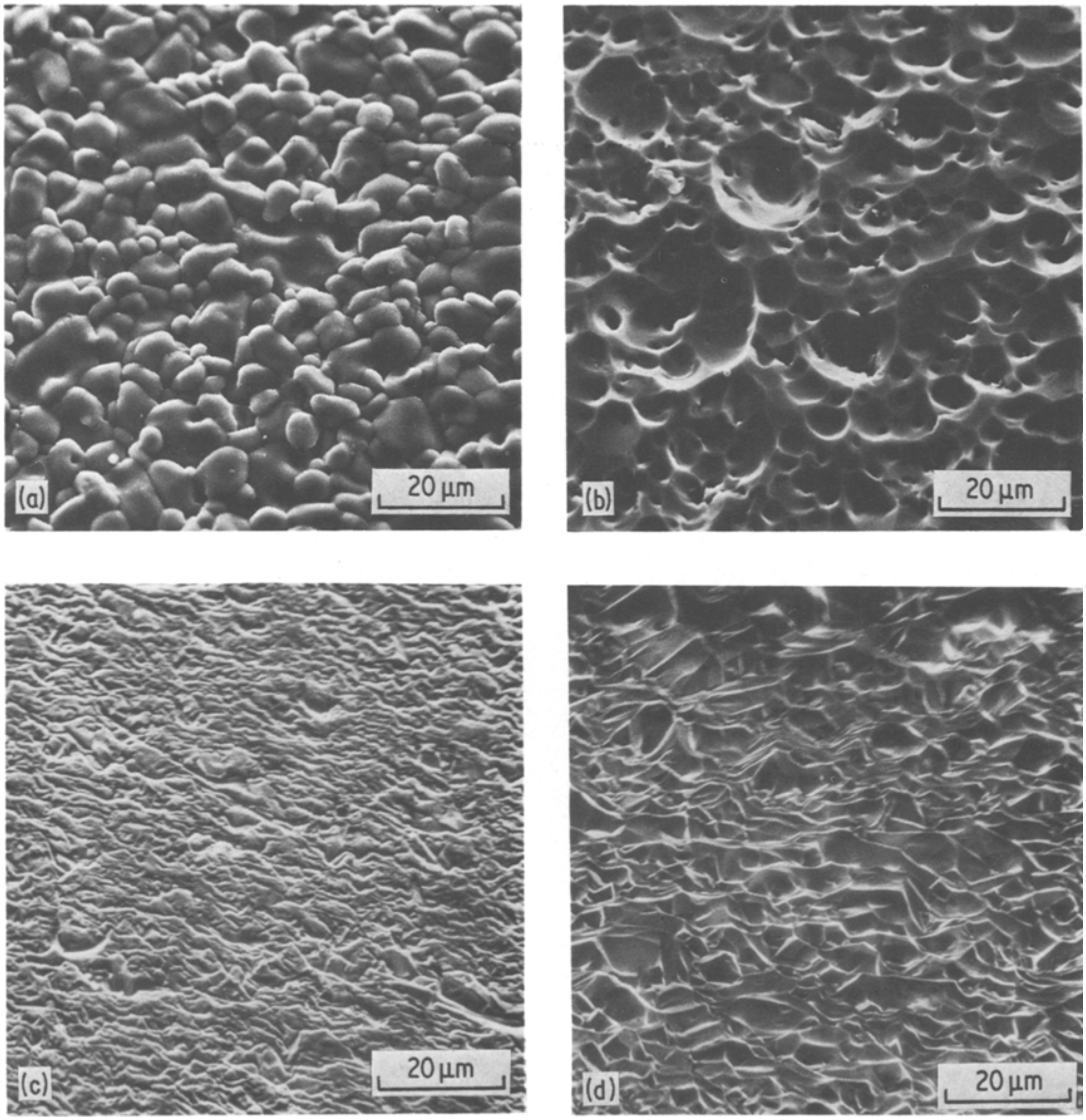

Figure 4 SEM images of the alumina surface, showing (a) 96% alumina before ion-etching, tilt 0 rad, (b) 96% alumina after Ar^+ irradiation at normal incidence, tilt 0 rad, (c) 99.5% alumina before ion bombardment, tilt 1 rad and (d) 99.5% alumina after ion-etching process, $\theta = 0$ rad, tilt 1 rad.

perform most of the thinning at an angle near θ_m, the angle of maximum erosion, and then change to the angle which produces further smoothing. The surface roughness is a factor which also affects the biological tissue response to an implant material. Increasing the surface roughness of an implant by ion etching of the surface increases tissue adherence. Numerous studies have shown the beneficial effect of surface roughness on tissue in-growth [19]. Ion-etched surfaces may also enhance thrombus attachment to implant surfaces [17].

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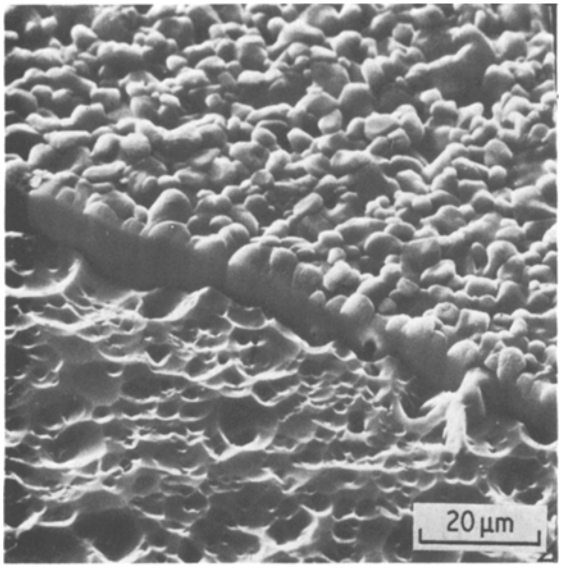

Figure 5 Sharp slope between screened surface of 96% alumina (upper right) and an area Ar^+ ion-etched at normal incidence (lower left).

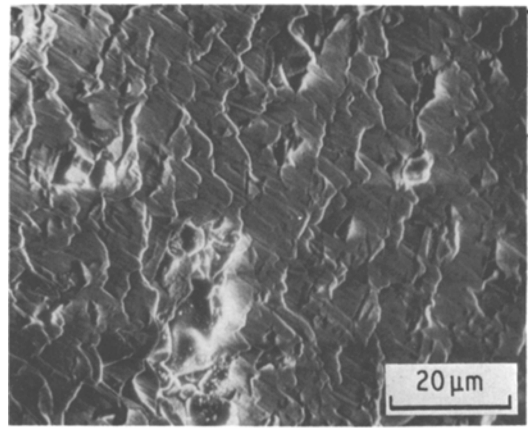

Figure 6 SEM image of 99.5% alumina surface after very oblique ion-irradiation, $\theta = 1.4$ rad, tilt 1 rad.

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