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The use of scanning electron microscopy to study the ion beam sputter modification of the surface topography of biological implants

ZBIGNIEW W. KOWALSKI
Technical University of Wrocław, 50-370 Wrocław, Poland

One factor which affects the biological tissue response to an implant material is the surface topography of the material. Ion beam sputtering, as a potentially useful roughening technique, has recently been used in attempts to modify the surface topography of biocompatible materials, such as metals, alloys, polymers, and ceramics. The ion-beam sputter modification of the surface topography of three different materials presently used or under consideration for implant devices were studied. A scanning electron microscope was used to examine all the materials tested.

1. Introduction

A new area of potential application for ion sputtering process is in the field of implantology. The ion bombardment of implants can cause erosion in which atoms are ejected from the surface and, as a result, may lead to modifications of the surface morphology and chemistry of implant materials. In order to develop clinically acceptable materials, the influence of material parameters on the biological response must be understood.

Different problems, such as:

(a) changes in the healing process that result from the presence of an implant;

(b) a tissue inflamatory and/or foreign body response in the tissue surrounding the implant;

(c) a firm attachment of the surrounding tissue to the implant material;

(d) a firm attachment of the thrombus to the vascular implant (to avoid embolization);

must be considered depending whether use is in soft or hard tissue or in contact with blood. The implant materials may therefore be categorized biologically into two groups [1]:

(a) soft tissue implants – vascular prostheses, artificial heart pump diaphragms, pacemaker fixations, percutaneous connectors,

(b) hard tissue implants – orthopaedic prosthesis fixations, dental implants.

In the last five years the ion sputtering technique has become widely used in attempts to modify the surface morphology and chemistry of biocompatible materials, such as metals, alloys, polymers, and ceramics [1–3]. Many experiments were performed to investigate the influence of the ion sputtering of implants on surface composition and mechanical properties of implant materials, surface topography of implants, and tissue response in the tissue surrounding the sputtered implant. Energy dispersion spectrometry results obtained by Weigand *et al.* [2] indicated very little change in the composition near the surface of ion beam sputtered surgical implant alloys. However, X-ray photoelectron spectroscopic examination of ion sputtered polymers [3] indicated an increase of oxygen (polyethylene samples) or carbon (segmented polyurethane, carbon-impregnated polyolefin, silicone rubber) on the surface. Examination of the effects of an ion sputtered surface on the mechanical properties (the ultimate strength, yield strength, and fatigue strength) of representative biological implant materials revealed very little degradation of the properties [2, 3]. The average ultimate strength and average yield strength of cobalt–chromium–tungsten alloy (Haynes 25) and 316 stainless steel tensile samples were unchanged (within several

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per cent) after ion sputtering. Also fatigue specimens of titanium–aluminium–vanadium alloy (Ti-6, 4) and 316 stainless steel that were ion sputtered showed no change in fatigue strength when compared to unsputtered samples.

Ion beam sputtering is potentially useful in the study of the effect of surface morphology (topography) on the biological response because of the ability of this technique to control the surface roughness [4]. Metals and ceramics generally can be sputtered at high ion beam energies, high current densities, and high surface temperatures. Polymers generally require low ion beam energies, current densities, and surface temperatures, to obtain the desired surface topography. To obtain larger surface roughness a sputter resistant material (seed material) supplying the surfaces during ion sputtering can be used [5], or a screen mesh may be superimposed on the biomaterial during sputtering [3]. The screen will prevent the erosion of material directly beneath it, resulting in a surface with an array of pores of constant dimension. An optimum implant surface roughness and/or texture can be deduced from *in vivo* tests of implants with controlled, precise surface topographies. Preliminary tissue response data of ion sputtered samples have been obtained and described by several authors. Babbush [6] testing xenon-ion-sputtered titanium and cobalt–chromium alloy (MP 35N) dental implants in beagles has stated that there was close adaptation of interfacial tissue with implant surface and a minimal tissue inflamatory or foreign body response. Banks *et al.* [1] have sputtered segmented polyurethane (Biomer) vascular implants using an electron bombardment ion thruster as an ion source. After argon ion sputtering Biomer samples were implanted into canine arteries. The initial thrombus growth (after 1 h) was accelerated when compared to the growth on unsputtered samples. However, the thrombus thickness after 4 days was the same for both ion sputtered and unsputtered samples. Gibbons [6] has tested flat implants made of PTFE, alumina, Haynes 25, Ti-6, 4, and 316 stainless steel for mechanical attachment of soft tissue to the implant material. After 6 weeks of implantation in dorsal subcutaneous soft tissue of rats the implants (1 cm x 3 cm) were tested for mechanical attachment by means of a "pull out" test. Results obtained by Gibbons indicate an increase in the tissue attachment to ion sputtered implants compared to unsputtered samples. There was no evidence of an inflamatory cell response in the tissue surrounding the implants.

Results presented above show the beneficial effect of ion sputtering on tissue response (initial thrombus growth, tissue inflamation, mechanical attachment of soft tissue to the implant material and/or thrombus to the vascular implant). The most important factor which affects the biological tissue response to an implant material is the surface roughness of the material. The surface topography resulting from the ion sputtering of implant materials also must be considered in the preparation of the biological implants because of the large influence this can have on surface roughness.

The aim of this paper is to discuss the results of ion beam sputter modification of the surface topography of three different biological implant materials.

2. Experimental procedure

2.1. Specimens

Three different biological implant materials presently used or under consideration for implant devices were investigated: (a) polyester, an elastic polymer used for vascular prostheses; (b) chrome–nickel stainless steel used for orthopaedic implants; and (c) alumina ceramic.

Samples of polyester material were cut from the vascular implant, Symbol 10 (made in Poland). Chrome–nickel stainless steel samples 16 mm wide and 30 mm long were cut from the wide plate type 63018 (Catalogue of OSTEO AG, Selzach products, 1976). This steel (chromium 17.5%, nickel 12.5%, molybdenum 3%, and carbon 0.03% max.) corresponds to the American standard AISI 316LC or the German material number 4435.

Alumina ceramic is commonly used as a substrate material for thick film circuits, but recently it appeared that it could be a satisfactory biomaterial. Therefore polycrystalline ceramic specimens of 96% fine-grained sintered alumina in the form of plates 2 cm x 3 cm were investigated.

2.2. Irradiation

The glow discharge ion gun with hollow anode was used as a neutralized ion-beam sputtering source [7]. The ion irradiations were performed in an experimental apparatus, similar to that described elsewhere [8]. All three materials investigated were bombarded at normal incidence by Ar^+ ions at an applied voltage of 7 kV and at

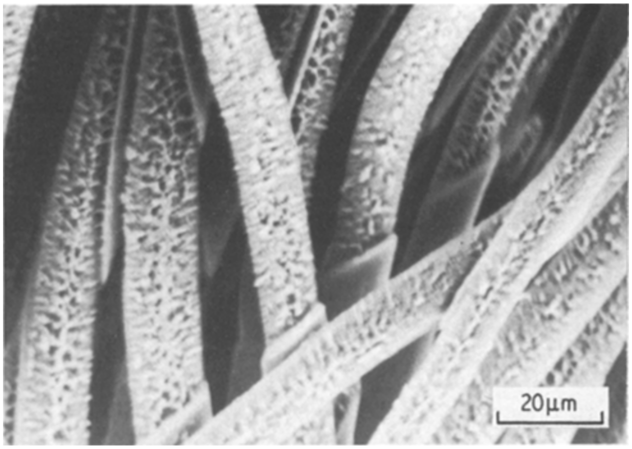

Figure 1 SEM image of polyester material after 90 min of ion-beam sputtering.

an ion current, measured by a Faraday cage situated in the place of the specimen, of about 70 μA. Stainless steel and alumina ceramic specimens were also sputtered with a sputter resistant material (seed material). During all sputtering processes the operating pressure was between 2.6×10^{-3} and 3.9×10^{-3} Pa (2×10^{-5} and 3×10^{-5} Torr). All the samples were positioned and sputtered for 90 to 200 min at an ion source–sample distance of about 1.5 cm.

2.3. Surface topography observations

The ion beam sputter modification of the surface topography of all specimens were examined by a Japan Electron Optics Laboratory, model JSM-35, scanning electron microscope. Before SEM observations non-conductive samples (polyester, alumina ceramic) were coated with thin films of chromium.

3. Results and discussion

Polyester, an elastic polymer used for vascular implants consists of –COOR groups, where C is carbon, O is oxygen, and R is the organic radical. The bombardment region of the polyester sample had a matt black appearance to the eye, probably due to an increase of carbon on the sputtered surface.

The vascular implant is made of knitted polyester material. It is composed of synthetic fibres or threads which are shown in Fig. 1 after ion beam sputtering. Fig. 2 shows scanning electron photomicrographs of polyester fibres before (Fig. 2a) and after (Fig. 2b) ion irradiation. The unsputtered surface was found to be almost completely smooth. After ion bombardment a dense mass of whiskers, very irregular in shape, could be observed on the surface of the fibres. It is worth noticing that during ion irradiation some fibres were partially shadowed by others. As a result of shadowing, parts of the fibres were not sputtered and remained smooth. It is difficult to say anything definite to explain the mechanism of sputtering of polyester material. Possible mechanisms could be considered:

(a) ion sputtering of –COOR molecules;

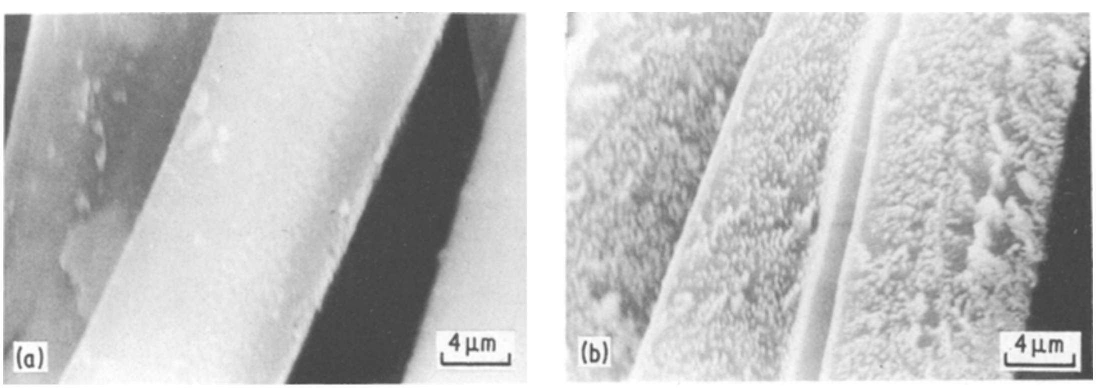

Figure 2 Scanning electron photomicrographs of polyester fibres, (a) before ion irradiation, (b) after ion-beam sputtering.

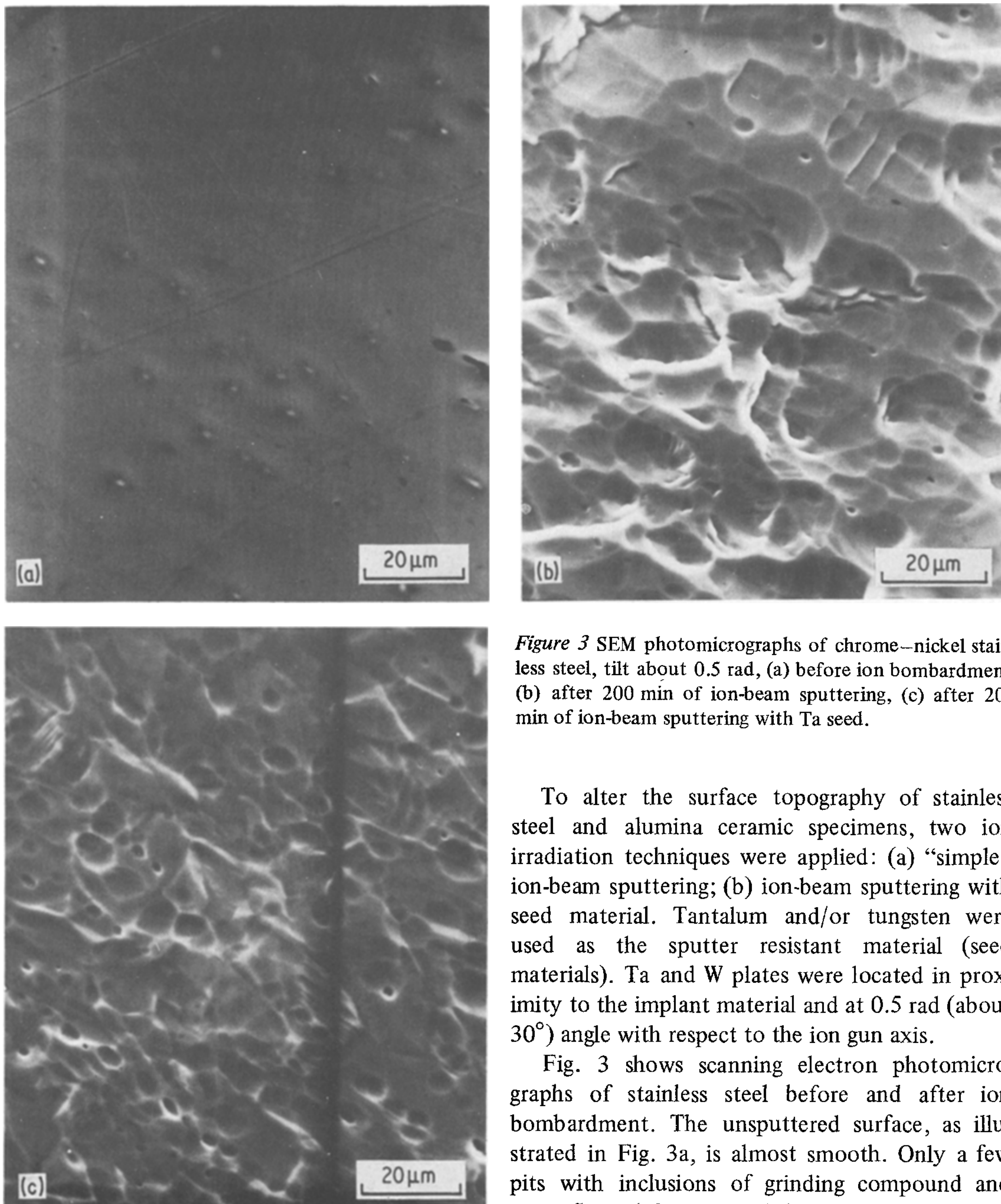

Figure 3 SEM photomicrographs of chrome–nickel stainless steel, tilt about 0.5 rad, (a) before ion bombardment, (b) after 200 min of ion-beam sputtering, (c) after 200 min of ion-beam sputtering with Ta seed.

(b) ion sputtering of polyester material, with different sputtering ratios for C, O, and R;

(c) ion sputtering of polyester material and/or chemical decomposition of the material.

The observed surface topography of the ion beam sputtered polyester sample is probably due to the differences between the sputtering yields of carbon, which has an extremely low sputtering yield, and the rest of the chemical elements, of the polymer.

To alter the surface topography of stainless steel and alumina ceramic specimens, two ion irradiation techniques were applied: (a) "simple" ion-beam sputtering; (b) ion-beam sputtering with seed material. Tantalum and/or tungsten were used as the sputter resistant material (seed materials). Ta and W plates were located in proximity to the implant material and at 0.5 rad (about 30°) angle with respect to the ion gun axis.

Fig. 3 shows scanning electron photomicrographs of stainless steel before and after ion bombardment. The unsputtered surface, as illustrated in Fig. 3a, is almost smooth. Only a few pits with inclusions of grinding compound and some flaws (after the polishing process) could be observed. The ion sputtered surfaces are shown both made without any seed material (Fig. 3b) and with tantalum seed (Fig. 3c). The surface, smooth before ion irradiation, changes with a tendency towards roughening, independent of the ion sputtering technique used in the experiment. However, the surface topography of stainless steel sputtered with Ta seed is less pronounced than when sputtered without any seed. The results of ion-beam sputtering of alumina specimens are illustrated in Fig. 4. Figure 4a shows an SEM

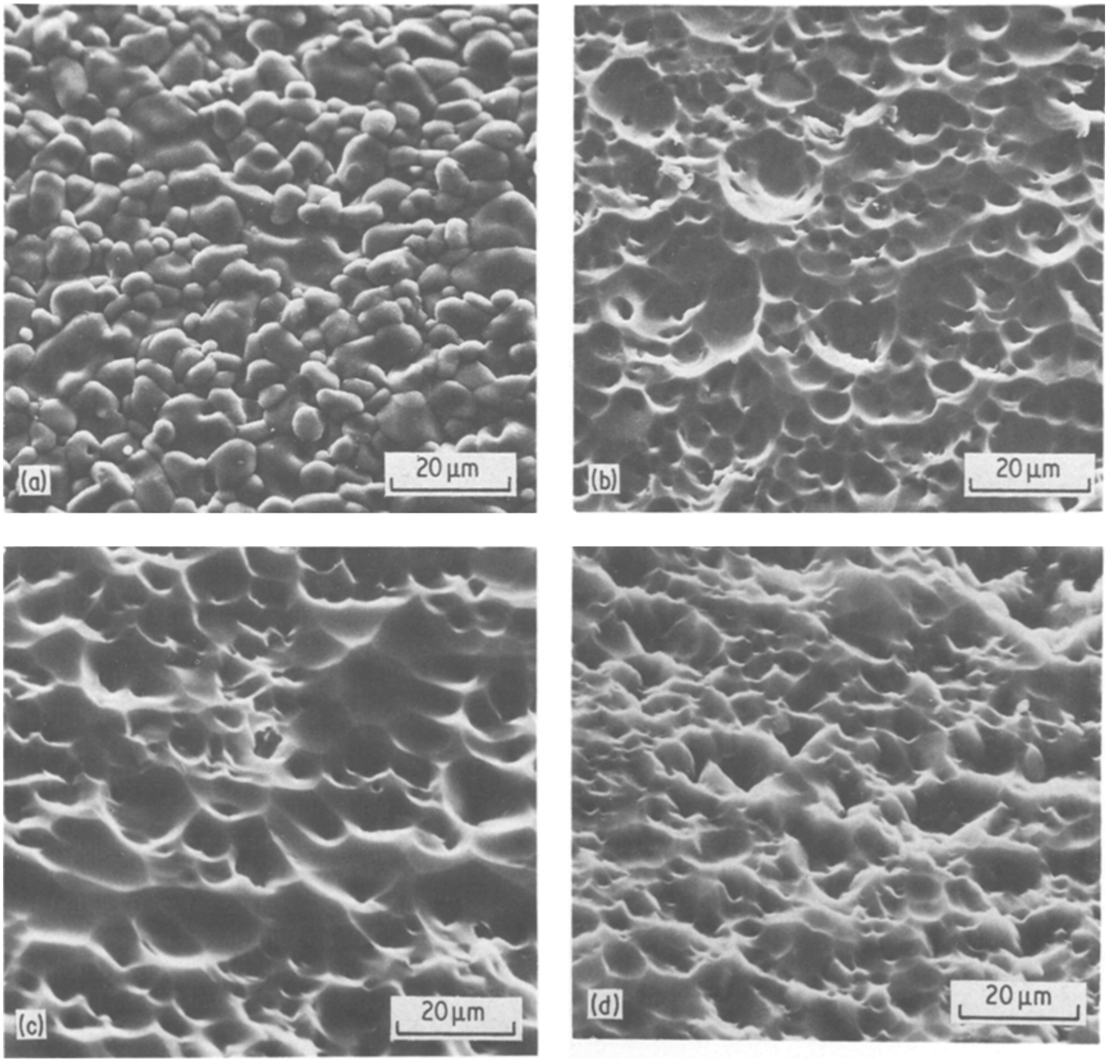

Figure 4 SEM images of 96% alumina surface, (a) before ion irradiation, tilt 0 rad, (b) after 90 min of ion-beam sputtering, tilt 0 rad, (c) after 90 min of ion-beam sputtering with Ta seed, tilt 0.8 rad, (d) after 90 min of ion-beam sputtering with W seed, tile 0.8 rad.

image of the alumina surface before ion irradiation. SEM images of the alumina surface after ion bombardment are shown in Fig. 4b (without any seed), Fig. 4c (with Ta seed), and Fig. 4d (with W seed). The initial topography (Fig. 4a) changes with a tendency towards smoothing, but simultaneous roughening occurs due to the preferential sputtering of the pore walls. These processes together determine the final state of the surface roughness and topography [4]. Grains and grain boundaries observed before ion sputtering are not seen after ion bombardment. On the alumina surface sputtered with tungsten seed material (Fig. 4d) some isolated cones can be seen, especially at the bottom of the pores. Possible nuclei for the formation of the cones, such as debris on the surface and/or inclusions at grain boundaries, could be considered [9].

Although the differences between the surface topographies shown in Figs. 3b, 3c and 4b to d can be observed, it is difficult to interpret the photomicrographs. In our experiment the ion sputtering with seed material was used to obtain large surface roughness (cone or ridge structure). Unfortunately, the cone or ridge structure was not observed, which may be due to the differences between the sputtering ratios of seed materials (Ta, W) and specimen materials (stainless steel, alumina). Tantalum and tungsten have higher sputtering ratios than stainless steel and alumina. On the other hand, Hudson [5] has shown that several elements (C, Si, Ti, Zr) have lower sputtering yields than seed material (Ta), but nevertheless were still textured. As yet, no unambiguous explanation of this problem has been provided.

4. Conclusion

Ion-beam sputtering can microscopically roughen the surfaces of implant materials such as polymers, alloys, and ceramics. The resulting surface topography may potentially be used to improve the biological response to implant materials.

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