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(NASA-TM-X-73468) ION BEAM SPUTTER
MODIFICATION OF THE SURFACE MORPHOLOGY OF
BIOLOGICAL IMPLANTS (NASA) 19 p HC \$3.50

N76-30797

CSCL 06B

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**ION BEAM SPUTTER MODIFICATION OF THE
SURFACE MORPHOLOGY OF BIOLOGICAL IMPLANTS**

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TECHNICAL PAPER to be presented at the
23rd National Vacuum Symposium
sponsored by the American Vacuum Society
Chicago, Illinois, September 21-24, 1976



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ABSTRACT

The surface chemistry and texture of materials used for biological implants may significantly influence their performance and biocompatibility. Recent interest in the microscopic control of implant surface texture has led to the evaluation of ion beam sputtering as a potentially useful surface roughening technique. Ion sources, similar to electron bombardment ion thrusters designed for propulsive applications, are used to roughen the surfaces of various biocompatible alloys or polymer materials. These materials are typically used for dental implants, orthopedic prostheses, vascular prostheses, and artificial heart components. Masking techniques and resulting surface textures are described along with progress concerning evaluation of the biological response to the ion beam sputtered surfaces.

INTRODUCTION

A new area of potential application for the electron bombardment ion thruster is in the field of biomedicine and, in particular, implantology. Surface morphology and chemistry are important factors when considering materials for biological implants. The ion thruster when used as a sputtering source is capable of modifying the surface morphology and chemistry of biocompatible metals, alloys, and polymers, such as polyurethanes and polyolefins. These modifications may influence the usefulness and/or biocompatibility of the materials.

The materials investigated during this study may be biologically categorized into two groups. The first group, soft tissue implants, is normally placed adjacent to or in connective tissue or in the blood. Examples of this kind of implant include vascular prostheses (artificial blood vessels), artificial heart pump diaphragms, pacemakers, and percutaneous (protruding through skin) connectors. These implants must meet stringent requirements before they can be considered biologically acceptable.

Vascular prostheses and artificial heart pump diaphragms must permit adequate attachment of thrombus (the layers of deposited blood components which form on surfaces of implanted materials exposed to blood). The blood response to vascular implants must be such that only a thin thrombus layer develops. This requirement is essential to prevent occlusion of prosthetic vessels. The thrombus must also firmly attach to the prosthetic vessel to prevent thrombus material

from detaching and possibly blocking vessels of vital organs. Soft tissue implants should also allow connective tissue in-growth to assure mechanical attachment to the implant. This requirement is particularly true for pacemakers and percutaneous connectors.

The second group of implants, the hard tissue implants, is normally implanted adjacent to or in bone tissue. Orthopedic prostheses and dental implants are two examples of hard tissue implants. Biocompatibility and tissue in-growth are also important factors when considering materials for hard tissue implants. Materials used for hard tissue implants include titanium, titanium 6-4 (6% aluminum, 4% vanadium), surgical stainless steel, and cobalt-chromium alloy (46% cobalt, 20% chromium).

The intent of this paper is to discuss the technique and results of ion beam sputter modification of various materials presently used or under consideration for implant devices.

IMPLANT MATERIALS

Both polymers and metals were ion beam sputtered. Each material will be briefly discussed in terms of its composition and implant application.

Segmented Polyurethane

Segmented polyurethane¹⁻³ is an elastic polymer used for vascular prostheses and implantable roller-type heart assist pumps. Segmented polyurethane is currently thought to consist of alternating hard segments of pipazine and toluenediisocyanate separated by soft segments of polytetramethylglycol.⁴ The polymer can be manufactured

into almost any shape by solution polymerization.³ The segmented polyurethane used for this study was cast into a shape suitable for subsequent evaluation as vascular implants.

Polyolefins

A 32% carbon-impregnated polyolefin that had been deproteinized and doubly centrifuged⁵ was obtained from its manufacturer, the Good-year Rubber and Tire Co., which developed this material specifically for biomedical application. This polymer demonstrated high flexure strength⁶ and is under evaluation as an artificial heart pump diaphragm material.⁷

Titanium

Pure titanium is used for endostial blade-vent dental implants.^{8,9} An alloy of titanium is used for orthopedic prostheses. Its composition consists of 6% aluminum and 4% vanadium and is manufactured according to ASTM standard specification designation, F136-70.

Cobalt-Chromium

Another orthopedic prosthesis material is a cobalt chromium alloy (ASTM designation: F90-68). The major components are 46% cobalt and 20% chromium. This material is also used for heart pacemaker enclosures.

Stainless Steel

The stainless steel used for surgical implants is made according to ASTM specification designation F55-71. Orthopedic implants such as bone pins, screws, and prostheses are made from this material.

SPUTTERING APPARATUS AND TECHNIQUE

Ion Thruster

A 5-cm-diam electron bombardment mercury ion thruster was used as the ion beam source for most of the results reported herein. Figure 1 shows the schematic of the thruster which is similar to the thruster reported in Ref. 10 except a double strand tantalum (Ta) wire loop coated with an emissive mix is used for a neutralizer instead of a plasma bridge hollow cathode.¹¹ When a segmented polyurethane sample was to be implanted into an animal, the source used was an 8-cm-diam electron bombardment argon-ion thruster to eliminate sample contamination from mercury. The design of the 8-cm thruster is basically the same as the smaller 5-cm thruster.¹²

Polymers

Segmented polyurethane vascular implant samples were molded on a 5-mm-diam glass rod mandrel to a thickness of 0.25 mm. Pieces 3-mm wide and 5-mm long were cut from the mold and used as samples. Two sutures were attached to the convex surface of implant. A special sample holder shown in Fig. 2 was used to minimize backspattered facility material from depositing on the implant. An additional piece of segmented polyurethane was used as a back shield to further protect the sample from back sputtered facility material. The sutures were threaded through a protective tubular support and enclosed in a triangular chamber that prevented ion beam sputtering or deposition on the sutures. The holder was placed on a retractable rod which was part of a gate valve port. The sample was positioned and etched for

30 minutes at a 20 cm ion source-to-sample distance, 500-eV ion beam energy, and 10-mA ion beam current. The current density was $20 \mu\text{A}/\text{cm}^2$. When the beam voltage was turned off, the sample was extracted through the gate valve. An air filter was attached to the bleed valve to provide a dust free bleed up. After bleed up, a dust free bottle was immediately placed over the sample as it was taken from the port. The sample was transported to a clean room where it was removed from the holder and placed in another dust free bottle in which it would be transferred to a hospital for final autoclave sterilization prior to implantation.

Polyolefin samples 2 cm x 2 cm x 1.8 mm were positioned downstream of the ion thruster such that the plane of the sample was closer to the ion source than any part of the sample holder. This arrangement prevented any accumulation of backspattered material. As with the polyurethanes, the samples were 20 cm from the ion source. An ion beam current of 25 mA and an accelerating potential of 700 V (current density of $70 \mu\text{A}/\text{cm}^2$) was used without causing thermal damage to the sample. In some tests an electroformed nickel mesh was used as a mask over the surface of the sample during sputtering. The mesh wire was 2 μm wide, the spacing between wires was 50 μm , and the mesh was 5 μm thick. This technique resulted in an array of small uniform depressions sputtered into the polyolefin. This technique represents a controlled approach to studying the effects of surface texture on the mechanical attachment of thrombus to implants.

Metals

The surfaces of the metals discussed in this paper were textured by simultaneous ion beam sputtering of the implant material and Ta. The tantalum, called the seed material,¹³ was located in proximity to the implant material (Fig. 1) so that sputtered Ta atoms would be deposited on the implant target. Sputter resistant sites of Ta were formed and fostered the generation of a rough, sputtered surface.¹⁴ The seed material atoms can be removed from the implant surface by removal of the Ta from the ion beam and subsequent sputtering of the implant for 1 to 5 minutes. Analysis of the surface using a scanning electron microscope accessory which measures the X-ray from the sample indicated that almost all of the Ta was removed after 5 minutes. All the metal samples were sputtered at a 10-cm ion-source-to-sample distance, a 2000-eV ion beam energy, and a 30-mA ion beam current. The current density was $190 \mu\text{A}/\text{cm}^2$.

RESULTS AND DISCUSSION

Polymers

Figure 3 shows scanning electron photomicrographs of segmented polyurethane before and after ion beam sputtering. After sterilization the samples were implanted into canine carotid and femoral arteries. (See Fig. 4.) Blood response results to date indicate an accelerated rate of thrombus formation on ion beam sputtered samples implanted for 1 hour. Longer in vivo implant tests (24 and 96 hr) seem to indicate less difference in blood response between the ion sputtered and unsputtered segmented polyurethane.

The difference in initial blood response may be caused by changes in the surface texture and/or changes in the chemical composition of the surface of the segmented polyurethane. Further testing is needed to evaluate if an improvement in the attachment of the thrombus has been achieved.

Figure 5 shows scanning electron photomicrographs of carbon-impregnated polyolefin before and after ion beam sputtering. A polyolefin sample with a 5- μ m thick nickel mesh superimposed was also sputtered (Fig. 5(c)). The depth of the sputter roughened microstructure is approximately 8 μ m. The microstructure of the ion beam sputtered polyolefin is probably due to the difference between the sputtering yield of the carbon and polyolefin. Because carbon has a relatively low sputtering yield, it is likely that surface abundance of carbon exceeds that of the initial carbon-polyolefin mixture. This result may enhance the overall performance of the polyolefin since carbon is quite biocompatible.

Metals

Scanning electron photomicrographs of ion beam sputtered Ti, Ti 6-4, cobalt-chromium alloy, and stainless steel are shown in Fig. 6(a) to (d). Each microstructure is characteristic of the material using Ta as the seed material.

Figure 7 shows a Ti surface after sputtering first with a Ta seed followed by 5 minutes of sputter etching with no seed. Samples of ion beam sputtered Ti in the form of endostial blade-vent implants are being evaluated in canines under NASA grant NSG-3110, "Endostial

Blade-Vent Implants Modified by Ion Beam Sputtering Techniques," at Mt. Sinai Hospital, Cleveland, Ohio. The rough microstructures may provide improved mechanical fixation of the bone tissue. The biocompatibility may also be modified by changes in the surface composition (residual Ta atoms). As with the polymers, the roughness is only surface roughness.

CONCLUDING REMARKS

The electron bombardment ion thruster when used as a sputtering source can microscopically roughen the surfaces of implant polymers, metals, and alloys. The morphology of the sputtered surfaces indicates that ion beam sputtering can produce a controlled roughness in terms of surface feature size, spacing, and depth for each implant material investigated. The resulting surface morphology may potentially be useful to improve the biological response to surgical implants.

ACKNOWLEDGEMENT

The authors would like to thank Craig Van Kampen for implanting ion beam sputtered segmented polyurethane samples into canines. With the aid of his co-advisers, Dr. R. Jones, Surgical Research at St. Luke's Hospital, Cleveland, Ohio, and Dr. D. Gibbons, Department of Biomedical Engineering at Case Western Reserve University, Cleveland, Ohio, Mr. Van Kampen provided the evaluation of blood responses to the samples.

The authors would also like to thank Dr. Albert Burstein of Case Western Reserve University, and the Goodyear Rubber and Tire

Company for contributing helpful suggestions and providing implant material samples for this ion beam texturing investigation.

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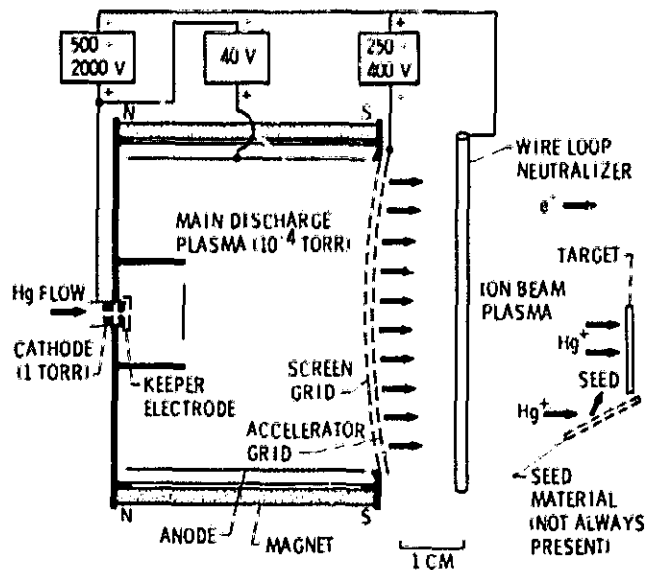


Figure 1. 5-cm ion thruster with hot wire neutralizer and relative relation of target and seed material.

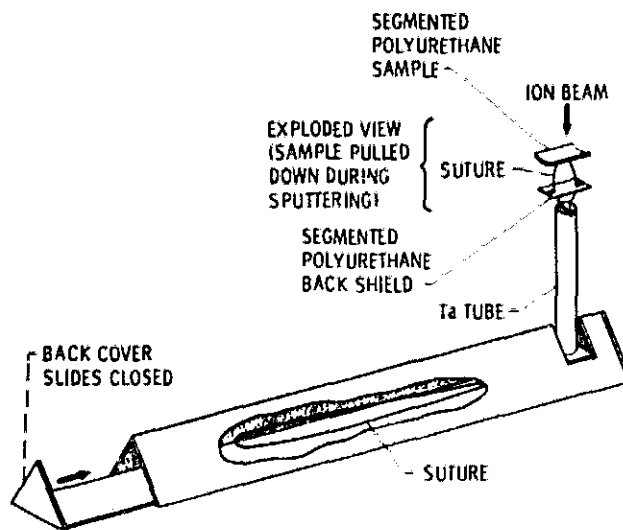
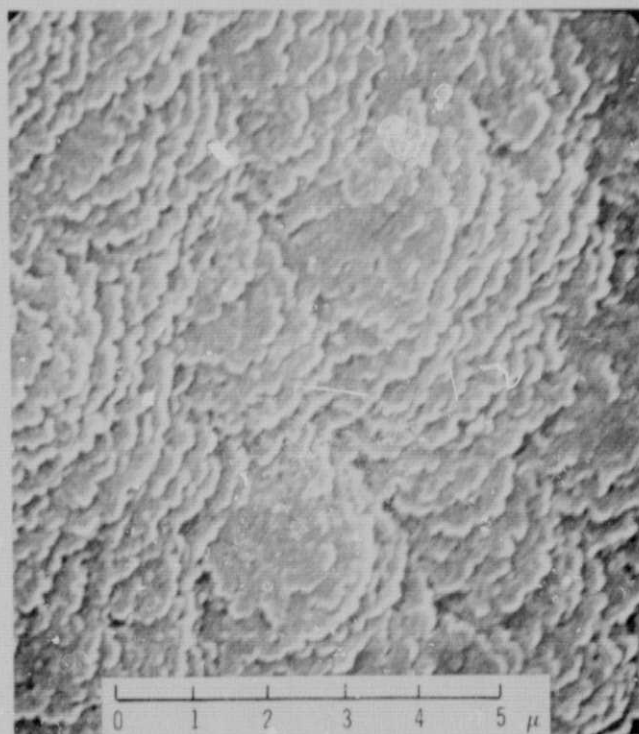


Figure 2. Segmented polyurethane sample holder



(a) Unspattered.



(b) Sputtered.

Figure 3. - Segmented polyurethane before and after ion beam sputtering.

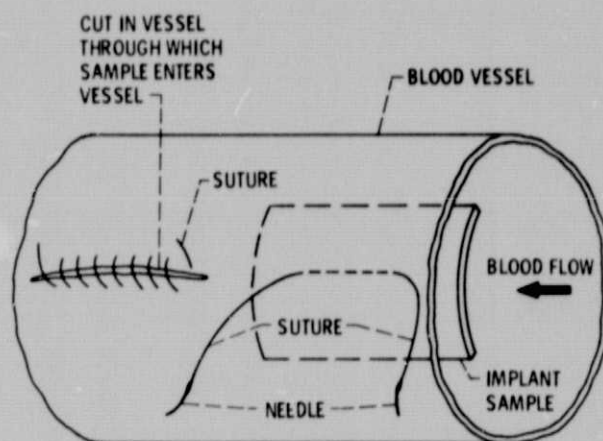
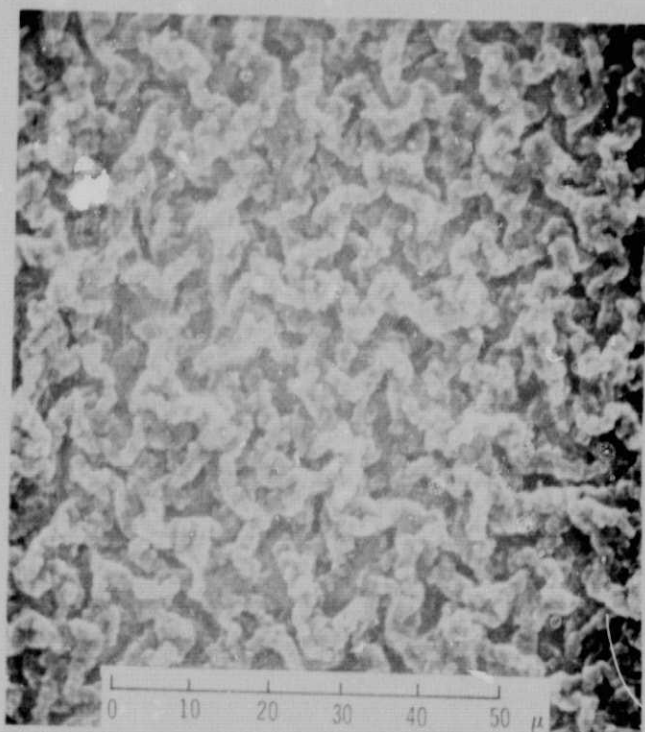


Figure 4. - Position of implanted segmented polyurethane sample in blood vessel.

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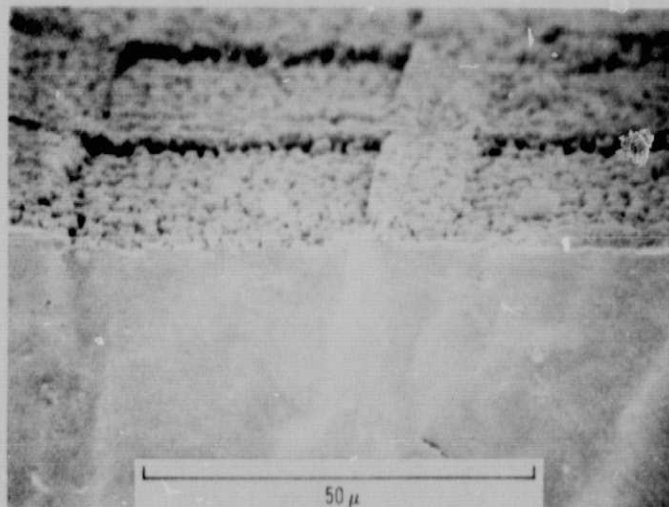


(a) Unspattered.



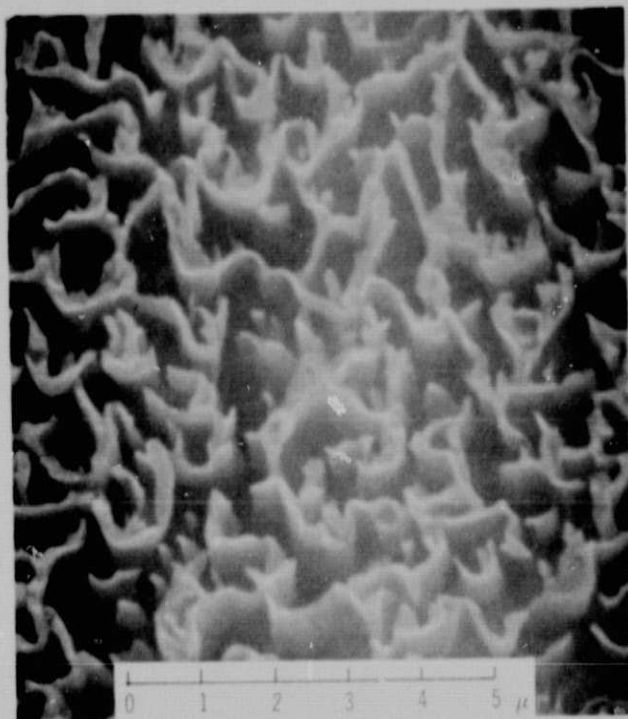
(b) Sputtered.

Figure 5. - Polyolefin (carbon impregnated) before ion beam sputtering, after 4 hours of ion beam sputtering and 4 hours of ion beam sputtering while covered with nickel mask.



(c) Sputtered with mask.

Figure 5. - Concluded.



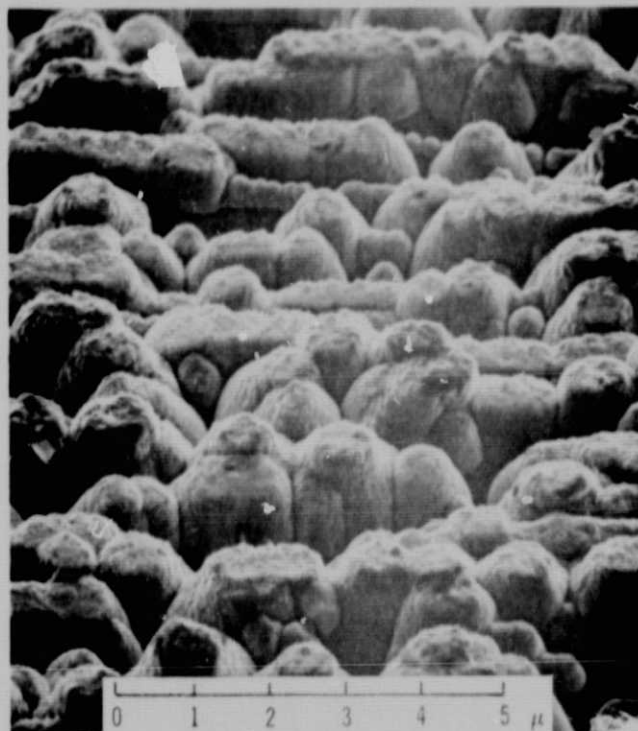
(a) Titanium



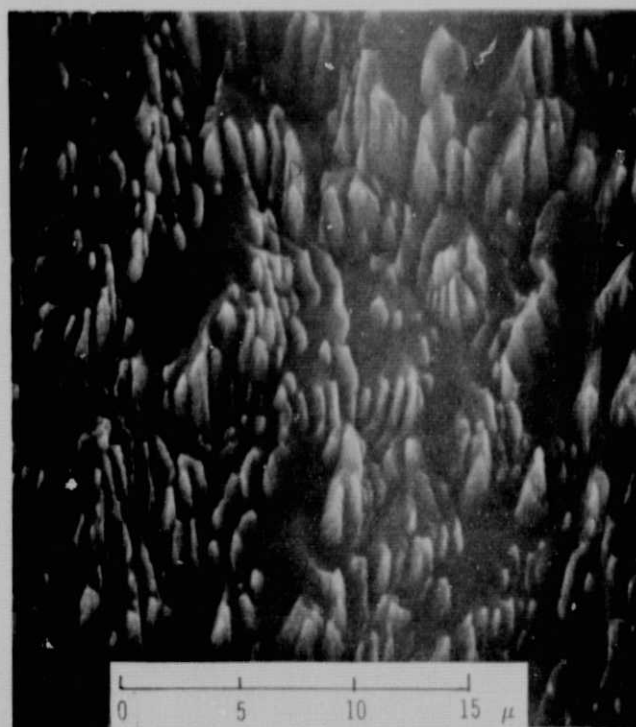
(b) Titanium 6%Al4%V

Figure 6 - Scanning electron photomicrographs of titanium, titanium (6%Al, 4%V), 46% chromium and 20% cobalt, and stainless steel 316 after ion beam sputtering with a tantalum seed. Sputtering durations were 4 hours except stainless steel which was 7 hours.

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(c) 46% chromium, 20% cobalt alloy.



(d) Stainless steel.

Figure 6. - Concluded.

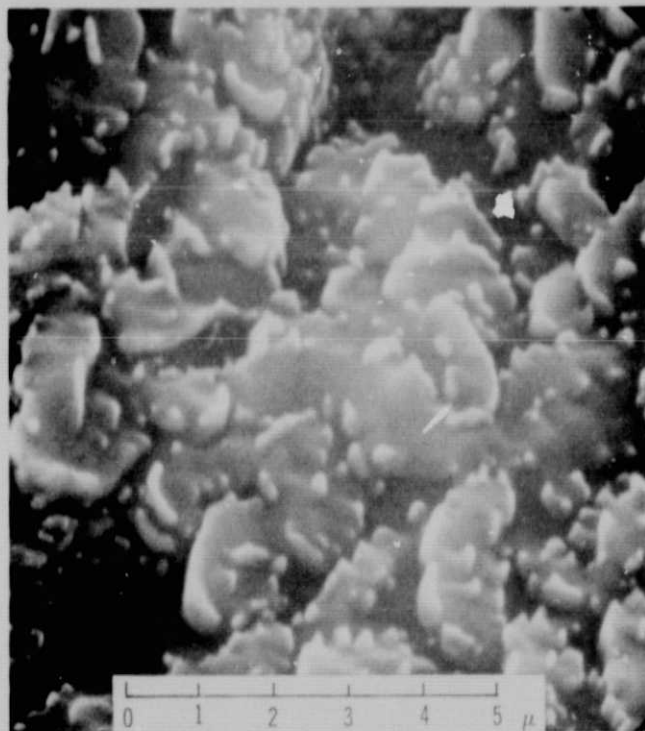


Figure 7. - Surface mophology of Ti sample after 4 hours of ion beam sputtering with Ta seed and then 5 minute etching with no seed.