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**SCANNING-ELECTRON-MICROSCOPY
OBSERVATIONS AND MECHANICAL
CHARACTERISTICS OF ION-BEAM-
SPUTTERED SURGICAL IMPLANT ALLOYS**

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1. Report No. NASA TM X-3553	2. Government Accession No.	3. Recipient's Catalog No.	
4. Title and Subtitle SCANNING-ELECTRON-MICROSCOPY OBSERVATIONS AND MECHANICAL CHARACTERISTICS OF ION-BEAM-SPUTTERED SURGICAL IMPLANT ALLOYS		5. Report Date August 1977	
		6. Performing Organization Code	
7. Author(s) Albert J. Weigand, M. Lise Meyer, and Jerri S. Ling		8. Performing Organization Report No. E-9056	
		10. Work Unit No. 506-22	
9. Performing Organization Name and Address Lewis Research Center National Aeronautics and Space Administration Cleveland, Ohio 44135		11. Contract or Grant No.	
		13. Type of Report and Period Covered Technical Memorandum	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D. C. 20546		14. Sponsoring Agency Code	
		15. Supplementary Notes	
16. Abstract <p>An electron-bombardment ion thruster was used as an ion source to sputter the surfaces of four orthopedic prosthetic metals (17% Cr - 14% Ni stainless steel, a Co - 20% Cr - 15% W alloy, Ti, and a Ti - 6% Al - 4% V alloy). Scanning-electron-microscopy (SEM) photomicrographs are shown of each ion-beam-textured surface. The effect of ion texturing an implant surface (Ti - 6% Al - 4% V) on its bond to a bone cement, polymethyl methacrylate, was investigated. The Co-Cr-W alloy and surgical stainless steel were used as representative hard tissue implant materials to determine effects of ion texturing on bulk mechanical properties (tensile ultimate strength and yield strength). Tensile specimens with untextured and textured surfaces were tested to failure. The ultimate strength of both bulk materials after texturing was essentially unchanged from the ultimate strength of untextured samples. Detailed results are shown in tables and stress-strain curves. An investigation was conducted to determine the effect of substrate temperature on the development of an ion-textured surface microstructure. Ti was used for this study. The results indicate that the microstructure will develop more rapidly if the substrate is heated prior to ion texturing. SEM photomicrographs were taken of the microstructure to show the development of the microstructure and to measure the microstructure height as a function of ion-texturing duration.</p>			
17. Key Words (Suggested by Author(s)) Ion sputtering Biological implants Tensile tests Polymethyl methacrylate		18. Distribution Statement Unclassified - unlimited STAR category 39	
19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 32	22. Price* A03

SCANNING-ELECTRON-MICROSCOPY OBSERVATIONS AND MECHANICAL CHARACTERISTICS OF ION-BEAM-SPUTTERED

SURGICAL IMPLANT ALLOYS

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SUMMARY

This report describes three separate ion-texturing investigations. Two were studies of biomedical applications of ion texturing, while the third investigated the effect of substrate temperature on the development of a surface microstructure that results from ion texturing. The effect of the duration of ion texturing on the surface microstructure was also investigated in the third experiment.

A cobalt-chromium-tungsten alloy and surgical stainless steel were used as representative hard tissue implant materials to determine effects of ion texturing on bulk mechanical properties (tensile ultimate strength and yield strength). Tensile specimens with untextured and textured surfaces were tested to failure. The ultimate strength of both materials after texturing was essentially unchanged from the ultimate strength of untextured samples. Detailed tensile test results are given in tables and stress-strain curves. Scanning-electron-microscopy (SEM) photomicrographs are shown of each ion-textured surface.

The objective of the second biomedical investigation was to produce an ion-textured surface to which a bone cement, polymethyl methacrylate, may bond more securely. Titanium - 6-percent-aluminum - 4-percent-vanadium alloy was used as a representative orthopedic implant material. Tensile data were obtained for textured and untextured titanium alloy samples which were bonded to polymethyl methacrylate. The results indicated that there was no significant improvement in bond strength due to the ion texturing. SEM photomicrographs are shown of the textured and untextured titanium alloy samples. An SEM photomicrograph of the polymethyl methacrylate bonding surface after tensile testing is also shown.

The rate at which the ion-textured surface microstructure of titanium developed depended on substrate temperature. Preheating the substrate to the steady-state operating temperature reduced the time required to develop a given microstructure. The maximum microstructure height was limited by the operating parameters and not by the total duration of ion texturing. There was no change in microstructure height after 32 minutes of ion texturing. SEM microphotographs were used to analyze the microstructure development and to determine microstructure height.

INTRODUCTION

A desirable feature for hard tissue implants (those adjacent to or in bone tissue) is mechanical fixation through ingrowth of the surrounding tissue (refs. 1 and 2). Increasing the surface area of an implant by roughening the surface increases tissue adherence. Numerous studies have shown the beneficial effect of surface roughness on tissue ingrowth (refs. 3 to 5). One means of roughening the surface of implant materials is by ion-beam sputtering (ref. 6). An electron-bombardment ion thruster that was originally developed for space application was modified and used as an ion source to texture implant material surfaces. Ion-beam texturing can produce a variety of controlled, precise surface microstructures which can be used to optimize tissue adhesion properties of surgical implants. Soft tissue implants made of ion-textured segmented polyurethane have been implanted into canine arteries (ref. 7). The initial thrombus formation (within 1 hr of implantation) on ion-textured surfaces was accelerated compared with the thrombus formation on unspattered surfaces. The final thrombus thickness and composition were nearly identical for textured and untextured samples. These results indicate that ion-textured surfaces may enhance thrombus attachment to implant surfaces.

The metallic implant materials that were ion-beam textured included a cobalt - 20-percent-chromium - 15-percent-tungsten alloy (Co-20Cr-15W), a 17-percent-chromium - 14-percent-nickel surgical stainless steel, unalloyed titanium (Ti), and a titanium - 6-percent-aluminum - 4-percent-vanadium alloy (Ti-6Al-4V). One of the objectives of this investigation was to determine the effects of the ion-produced microstructure on the mechanical properties (tensile ultimate strength and yield strength) of the Co-20Cr-15W alloy and stainless steel.

Another area of interest was the strength of the bond between bone cement, polymethyl methacrylate, and the stems of orthopedic prostheses. Polymethyl methacrylate is used to secure the implant to the bone tissue. Once cured, the polymethyl methacrylate has a tensile strength of approximately 2.1×10^7 newtons per square meter (3000 psi). However, the bonding strength between the implant and polymethyl methacrylate is not as good. Ion-beam texturing was used to try to improve the bonding. The material investigated for this part of the study was Ti-6Al-4V.

The biocompatibility of any ion textured surface must be established because of the alterations in composition, chemistry, and morphology on the surface of the original material that may result from the ion-texturing process. However, to date no data concerning ion-textured surface biocompatibility are known to exist.

The actual mechanism of the production of an ion-beam-textured surface is not completely understood. Investigations have proposed several theories to explain the phenomenon. These theories include nucleation of impurities (ref. 8) and differential component sputtering (ref. 9). One way to enhance the formation of the ion-textured surface

microstructure is to deposit a low-sputtering-yield material, called the seed material, onto the surface while ion etching the surface (ref. 10). With tantalum (Ta) as the seed material, this method was adapted to investigate the development of an ion-textured Ti surface. The Ta seed material provides a controlled amount of impurity to be present on the substrate surface while being ion textured. A scanning electron microscope equipped for X-ray energy dispersion spectrometry (EDS) was employed to document the microstructure formation. Spark-source light-emission spectroscopy was used to determine the amount of Ta on the surface as a function of texturing time. The time dependence of the height of the microstructure was also documented. The information obtained from this microstructure formation study can generally be applied to all ion-texturing results.

APPARATUS AND PROCEDURE

Ion Source

A schematic of the 5-centimeter-diameter ion-beam source which was used to ion texture Ti-6Al-4V, Co-20Cr-15W, and surgical stainless steel is shown in figure 1. This particular source generally uses mercury (Hg) as the propellant. The use of a low-work-function, emissive mix coated insert (refs. 11 and 12) inside the cathode ensures a copious flow of electrons into the discharge chamber. Mercury also flows through the cathode into the discharge chamber. The electrons are attracted toward the anode and ionize Hg through electron bombardment before reaching the anode. The ionized Hg is accelerated from the discharge chamber through the accelerator grid. This grid has a hexagonal array of 1.9-millimeter-diameter holes with a 2.8-millimeter centerline-to-centerline spacing. The neutralizer is a double strand loop of Ta wire coated with an emissive mix. It supplies the electrons needed to obtain a neutralized beam.

This source is capable of operating at net accelerating voltages between 400 and 2000 volts. The beam current can be controlled between 8 and 30 milliamperes. For this study a 30-milliampere Hg ion beam at a net accelerating potential of 2000 volts was used to texture all surfaces. This maximum beam power heated the samples to ensure atomic surface mobility needed for texturing. All samples were located on the source axis at a distance of 10 centimeters from the accelerator grid. At this location the beam current density was 190 microamperes per square centimeter. The source operated at a steady-state condition during each of the tests, which lasted up to 7 hours.

An 8-centimeter-diameter ion source was used for the Ti surface microstructure formation study. This source used Xe as the propellant. A range of ion-beam currents between 10 and 200 milliamperes could be obtained at net accelerating voltages between 500 and 2000 volts. For this study an ion-beam current of 100 milliamperes and a net

accelerating voltage of 2000 volts were used. The sample was located 10 centimeters from the grids for all tests, which resulted in an ion-beam current density of 2 milliamperes per square centimeter.

Both sources were operated in vacuum facilities at nominal pressures of 1.3×10^{-3} to 4.0×10^{-3} pascal (1×10^{-5} to 3×10^{-5} torr). A more detailed description of the 5-centimeter-diameter source can be obtained from reference 13, and reference 14 describes the 8-centimeter-diameter source.

Effect of Ion-Textured Surfaces on Bulk Tensile Properties

Figure 2 shows the type of cylindrical tensile test specimen used in testing Co-20Cr-15W alloy and stainless steel. The Co-20Cr-15W alloy had a composition designated by ASTM F90-68, while the stainless steel was made according to the ASTM designation F55-71. Each 0.29-centimeter-diameter sample was polished to a 0.1-micrometer- (4- μ in. -) rms-roughness lapped finish and placed in the beam so that its axis was perpendicular to the thruster axis. The sample was attached to a gear arrangement so that it could be rotated without turning off the ion beam. The sample was rotated four times (90° per rotation) to cover the entire surface with an ion-beam-produced microstructure. Table I gives the duration of ion-beam texturing for each specimen tested. The surface temperature resulting from ion bombardment for all samples was approximately 300° C during texturing.

Three different sample treatments were used to determine the effect of ion texturing on the mechanical properties. Unspattered samples of each material were tensile tested to obtain control data for comparison with handbook data. Samples in the second set were heat treated. These samples were heated to 300° C in a furnace for 16 hours to determine the effects of high temperature on the mechanical properties. The ion-textured samples were the third set tested.

The cross-sectional area of each sample was determined by using a shadow graph. Then the ultimate strength and yield strength were determined by using a tensile testing machine. After a sample was fractured, the diameter at the fracture site and the total elongation were measured. The hardness of each type of sample was also measured.

Scanning electron photomicrographs were taken of the surface of each sample before and after testing. A Japan Electron Optics Laboratory, model JSM-2, scanning electron microscope was used to examine all the materials tested.

Bond Strength of Polymethyl Methacrylate Adhesive Joined to Ion-Beam-Textured

Titanium - 6-Percent-Aluminum - 4-Percent-Vanadium Alloy

Pieces of Ti-6Al-4V (ASTM designation F136-70) approximately 1.2 by 1.2 by 0.2

centimeter were ion-beam textured with the aid of a Ta seed (fig. 3). The Ti-6Al-4V sample was perpendicular to the thruster axis, and the Ta seed was positioned approximately 30° from the thruster axis. Each sample was ion textured for 4 hours.

The determination of the bond strength of polymethyl methacrylate to Ti-6Al-4V began with applying the bone cement between two parallel plates (fig. 4). Test samples of sputtered and unsputtered Ti-6Al-4V (polished to a $0.8\text{-}\mu\text{m}$ ($32\text{-}\mu\text{in.}$) rms roughness) and sand-blasted metal pieces were furnace brazed to bolt heads. The surgical grade polymethyl methacrylate was mixed according to the manufacturer's directions. A solution of 2 grams of polymethyl methacrylate powder and 1 milliliter of liquid monomer (methyl methacrylate) was used for each test. The polymethyl methacrylate mixture was pressed between a Ti-6Al-4V sample and a metal piece so that no polymer overlapped the edges of either metal component. Axial alignment was maintained by using pegs which centered each bolt relative to the other. After the polymethyl methacrylate was allowed to cure for 48 hours, the sample was tensile tested to determine the bond strength.

Microstructure Development Study

Samples 1.0 by 2.5 by 0.1 centimeter were used to investigate the formation of the textured surface on titanium. A Ta seed was positioned next to the Ti sample and oriented 10° from the thruster axis (an arrangement similar to that in fig. 3). A constantan-Alumel thermocouple was attached to the back of the Ti sample to provide temperature data during texturing.

The investigation included two series of tests. In the first sequence, samples initially at room temperature were ion textured for 1, 2, 4, 8, 16, 32, and 120 minutes. In the second sequence, each Ti sample was preheated by being placed in the ion beam and rotated so that the back face was being ion sputtered. The sample was allowed to reach the thermal equilibrium temperature (determined from the temperature data of the first test sequence) and was then rotated to expose the front surface to the ion beam for 1, 2, and 4 minutes.

Scanning electron photomicrographs were taken of each surface to examine how the microstructure developed. Spark spectroscopic analysis was used to document the amount of Ta on each surface. The samples were then "freeze fractured" in order to view and measure the depth of the microstructure. The method employed included putting each sample in clamping pliers, submerging the sample in a Dewar filled with liquid nitrogen, lowering the sample temperature to approximately -200°C , removing the sample from the Dewar, and immediately hitting the edge of the sample with a hammer until the sample cracked into two pieces. The fractured edges were examined with the scanning electron microscope to determine microstructure height.

RESULTS AND DISCUSSION

Ion-Textured Surfaces of Cobalt - 20-Percent-Chromium - 15-Percent-Tungsten Alloy and Stainless Steel and Their Effects on Tensile Properties

Figure 5 shows scanning electron photomicrographs of the Co-20Cr-15W tensile test sample surface before and after ion-beam texturing. The striations on the control sample followed the circumference of the rod. However, the cones produced by ion texturing formed rows that were parallel to the rod axis. This result was contrary to the theory of A. N. Broers of the University of Cambridge, Cambridge, England. His unpublished results showed cone formation following surface scratches. One difference between Broers' theory and the Co-20Cr-15W results was that he used a seed material to develop the microstructure while no seed was used with Co-20Cr-15W. Energy dispersion spectrometry results (fig. 6) indicated very little change in the surface composition of Co-20Cr-15W as a result of ion-beam texturing.

The ion-textured surface of surgical stainless steel is shown in figure 7. The microstructure (made without any seed material) was less pronounced than that of Co-20Cr-15W. However, if the microstructure affected the structural properties of the material, these effects should have been observed whether the topographical features were dense or sparse. Figure 8 shows that traces of Hg (present in the vacuum facility) and/or Mo (ion source grid material) may have been present on the surface, but the minor amounts indicated for either should not have affected the tensile results.

Figures 9 and 10 show typical stress-strain diagrams for Co-20Cr-15W and surgical stainless steel, respectively, for a control sample, a sample that was only heated, and an ion-textured sample. Table II lists all the parameters that were measured or calculated from the tensile tests. ASTM mechanical property values are noted in table II for comparison with control sample results.

For both materials the ultimate strength was essentially unaltered after heating and ion texturing. The ultimate strength of treated (ion-textured and heated) surgical stainless steel was within 4 percent of the control sample value, and the ultimate strength of treated Co-20Cr-15W was within 5 percent of the control sample values.

The measured yield strengths (at 0.2 percent offset) of the heat-treated surgical stainless steel samples were not significantly different from the control sample values. The average yield strength of the heat-treated samples was 11 percent higher than the average yield strength of the control samples. However, the average yield strength of the ion-textured samples was 20 percent greater than the average control sample value. This difference might be caused by the effects of heating the samples while ion texturing them and/or might be the result of the insignificant number of tensile samples tested. The latter may be the reason because metallographic examination of the cross sections

of ion-textured and control samples by an electron microprobe indicated no significant compositional difference between the ion-textured and control samples. There was no change in surface hardness (Rockwell hardness of approximately C-40) associated with the increase in yield strength.

The average yield strength of the ion-textured Co-20Cr-15W samples was nearly identical to the average yield strength of the control and heat-treated samples. And, like the stainless steel, Co-20Cr-15W showed no change in surface hardness (Rockwell hardness of approximately C-25) as a result of ion texturing.

The total elongations of the treated samples of Co-20Cr-15W and stainless steel did not change significantly from the control sample values, which indicates that ion texturing does not change the ductility of these materials.

These results indicate that ion texturing does not alter the mechanical properties of materials which are insensitive to heating effects.

Strength of Bond Between Titanium - 6-Percent-Aluminum - 4-Percent-Vanadium

Alloy and Polymethyl Methacrylate

Figure 11 shows scanning electron photomicrographs of Ti-6Al-4V surfaces before and after ion texturing with a Ta seed. The tensile test results of the bonded assembly of Ti-6Al-4V and polymethyl methacrylate are shown in table III. The reproducibility of the untextured samples was within 10 percent of the average bond strength. The bond strength between polymethyl methacrylate and Ti-6Al-4V increased by 10 percent when the surface was ion textured. This increase was within the margin of error of the untextured sample tensile tests.

Figure 12 shows the polymethyl methacrylate surface that was attached to the ion-textured Ti-6Al-4V surface. This photomicrograph indicates that the polymethyl methacrylate did not really conform to the microstructure of the ion-textured Ti-6Al-4V surface. A rougher surface structure such as that resulting from sputtering through a screen may allow a more macroscopic level conformation of the polymethyl methacrylate surface.

Since a Ta seed was used to accelerate the development of the ion texturing, a residual amount of Ta remained on the Ti-6Al-4V surface, as shown by the EDS spectrum of an ion-textured Ti-6Al-4V sample shown in figure 13. The iron peak was probably due to sputtering of the sample holder. One way to remove most of the Ta residue would be to remove the seed material and etch the sample for a short period (1 to 5 min, ref. 15). For this investigation it was not necessary to remove the Ta residue to determine the bond strength.

Surface Texture Development

Titanium samples were ion textured by a xenon ion beam for 1, 2, 4, 8, 16, 32, and 120 minutes with Ta as the seed material. The resulting ion-textured surfaces and two of the corresponding EDS spectra are shown in figures 14 to 20. The appearance of a microstructure could be seen after 1 minute of ion texturing (fig. 14). However, it was not until the surface had been exposed for 2 minutes that the microstructure covered the entire surface (fig. 15). By 8 minutes the microstructure had begun to take on the characteristic ridge structure (fig. 17). The surface macrostructure corresponding to grain boundaries was never completely eliminated, as verified in figure 20(a). The ion-textured surface became more complex and dense with time. A second series of samples was ion textured under the same operating conditions, and the results were almost identical to those for the first series.

Scanning electron photomicrographs of the freeze-fractured edges of the samples are shown in figures 21 to 26. Figure 27 is a plot of the average ridge height for ion-beam exposure time from 2 to 120 minutes. The microstructure height increased until it reached a maximum of 1.2 micrometers after 32 minutes of ion-beam sputtering. The microstructure height remained at 1.2 micrometers after 32 minutes. There may be several reasons for this result. Figures 23 to 26 show that most of the ridges had sides that were perpendicular to the substrate surface. Because the ridges were so thin (approximately $0.1 \mu\text{m}$), they were probably not structurally capable of becoming higher than a critical length. Spectroscopic analysis of the samples indicated that the amount of Ta on the surface increased with a decreasing rate for the first 32 minutes of texturing (fig. 28). The apparent asymptote of Ta deposition at approximately 180 micrograms per square centimeter may have been an equilibrium between Ta flux arrival rate and Ta sputter rate from the substrate.

The temperature dependence of the microstructure formation was demonstrated by taking temperature readings as a function of ion-beam exposure duration. Figure 29 shows the temperature profile of Ti exposed to a Xe ion beam; 4 minutes were required to reach thermal equilibrium (specimen temperature of 640°C). Figure 30 shows a scanning electron photomicrograph of a Ti surface heated to 640°C before being ion textured for 1 minute. An EDS spectrum is also shown. After a 1-minute ion-beam exposure, a thin texture covered the entire surface. The rate of formation of the texture was clearly enhanced by preheating the substrate, as can be seen by comparing figures 14 and 30.

SUMMARY OF RESULTS

The material properties of ion-beam sputtered surgical implant alloys were investigated. An ion-textured surface microstructure on samples of cobalt - 20-percent-chromium - 15-percent-tungsten alloy and stainless steel did not reduce the ultimate strength or hardness. The strength of the bond between polymethyl methacrylate and titanium - 6-percent-aluminum - 4-percent-vanadium alloy was not significantly increased as a result of ion texturing.

The initiation of a microstructure enhanced by a seed material (tantalum) was accelerated when the substrate was preheated to normal (ion-beam induced) operating temperature before the ion beam was applied to the test surface. The particular ridge structure developed on titanium for the experimental conditions investigated was limited to 1.2 micrometers in height after 32 minutes of ion texturing. The microstructure became more complex and dense as the ion-texturing duration increased.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, March 8, 1977,
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TABLE I. - ION-BEAM EXPOSURE DURATIONS

FOR Co-20Cr-15W ALLOY AND

STAINLESS STEEL SAMPLES

Sample material	Sample numbers	Ion-beam texturing time per rotation, hr	Number of rotations
Co-20Cr-15W alloy	9	7	4
	10	4 $2\frac{1}{2}$	2 2
	11	4	4
Stainless steel	4, 5	4	4

TABLE II. - RESULTS OF TENSILE TESTS OF Co-20Cr-15W ALLOY AND SURGICAL STAINLESS STEEL

Sample material	Sample number	Ultimate strength		0.2% offset yield strength		Elongation, percent	Average initial diameter		Reduction in area at fracture site, percent
		N/m ²	psi	N/m ²	psi		cm	in.	
Stainless steel Control ^a Heated (300° C) Ion-textured	1	1.23×10 ⁹	177 400	1.02×10 ⁹	147 000	9	0.29	0.114	65
	2	1.25	180 300	1.11	160 600	8	.23	.092	62
	3	1.24	179 200	1.14	164 000	4	.29	.114	31
	4	1.26	181 000	1.17	165 800	-	.29	.114	--
	5	1.37	197 400	1.25	180 400	7	.28	.110	60
	6	1.30	187 700	1.23	176 900	8	.27	.110	64
	7	1.24	178 900	1.28	176 900	7	.27	.107	72
	8	1.30	186 400	1.28	184 000	5	.19	.075	55
	9	1.28	185 000	1.31	188 000	7	.22	.086	45
Co-20Cr-15W alloy Control ^b Heated (300° C) Ion-textured	10	9.40×10 ⁸	135 600	4.62×10 ⁸	66 700	----	0.29	0.114	35
	11	9.35	134 800	4.56	65 800	57	.29	.114	32
	12	8.82	127 100	4.30	62 000	49	.29	.113	35
	13	8.93	128 600	4.53	65 400	53	.29	.114	45
	14	9.35	134 400	4.29	61 700	79	.29	.114	48
	15	9.30	134 000	4.74	68 300	53	.24	.096	38
	16	8.75	126 100	4.42	63 800	57	.27	.108	48
	17	9.05	130 400	4.70	67 800	49	.25	.100	38
	18	9.50	136 900	4.64	66 600	52	.25	.100	62
	19	9.64	138 700	4.72	68 000	53	.25	.097	53

^aASTM designation F55-71, surgical stainless steel; ultimate strength, 7.65×10^8 to 9.73×10^8 N/m² (110 000 to 140 000 psi).

^bASTM designation F90-68, Co-20Cr-15W alloy; ultimate strength, 8.69×10^8 N/m² (125 000 psi); yield strength, 3.12×10^8 N/m² (45 000 psi).

TABLE III. - STRENGTH OF BOND
 BETWEEN POLYMETHYL
 METHACRYLATE AND ION-
 TEXTURED AND
 UNTEXTURED Ti-6Al-4V

Surface condition	Bond strength	
	N/m ²	psi
Textured	3.4 × 10 ⁶	490
Untextured	3.2	455

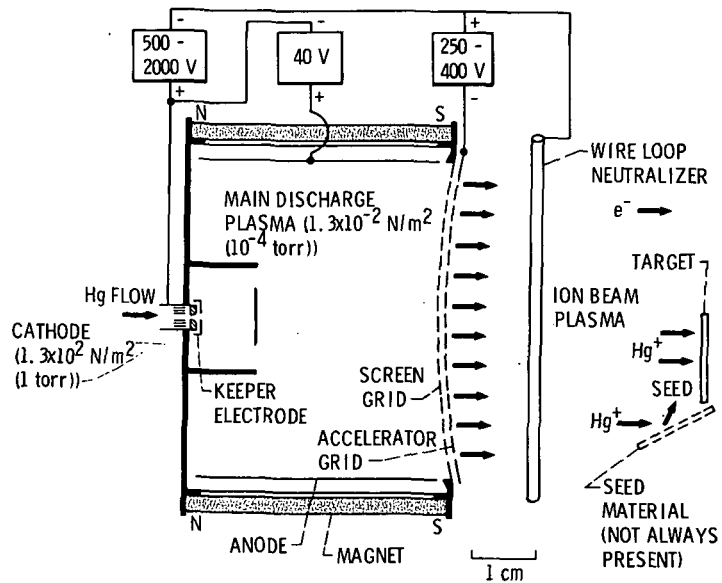


Figure 1. - Ion source.

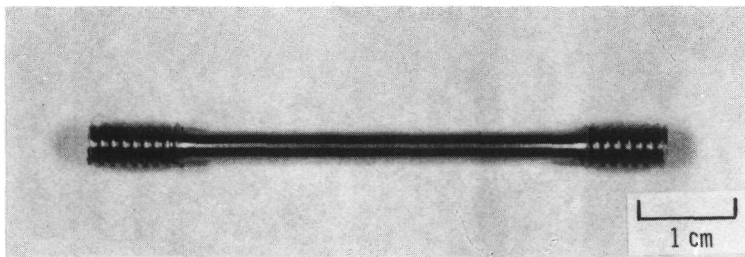
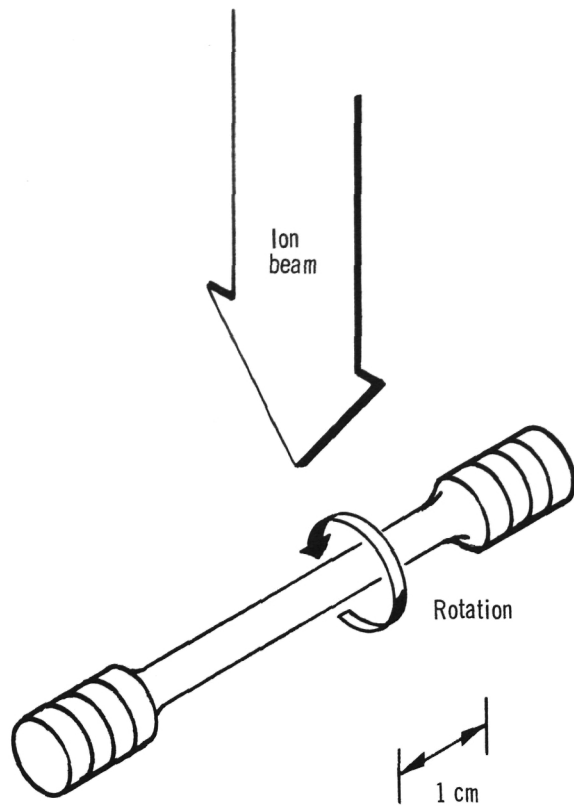


Figure 2. - Tensile test specimen and its relation to ion beam.

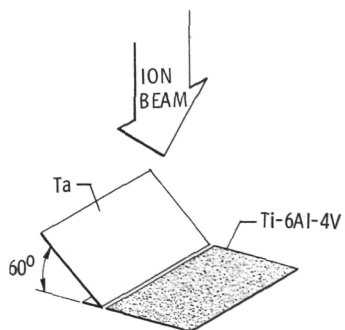


Figure 3. - Ion-beam texturing setup for Ti-6Al-4V and Ta seed.

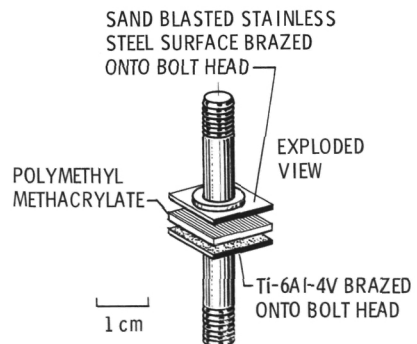
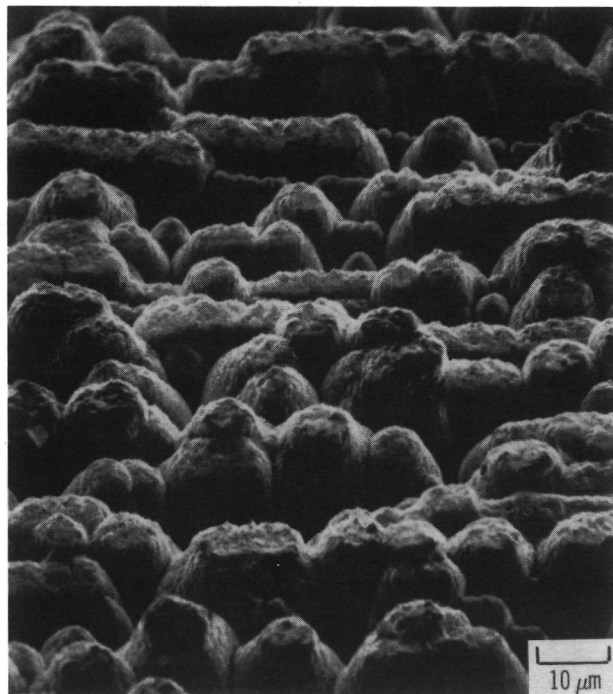


Figure 4. - Sketch of polymethyl methacrylate bond strength test sample.

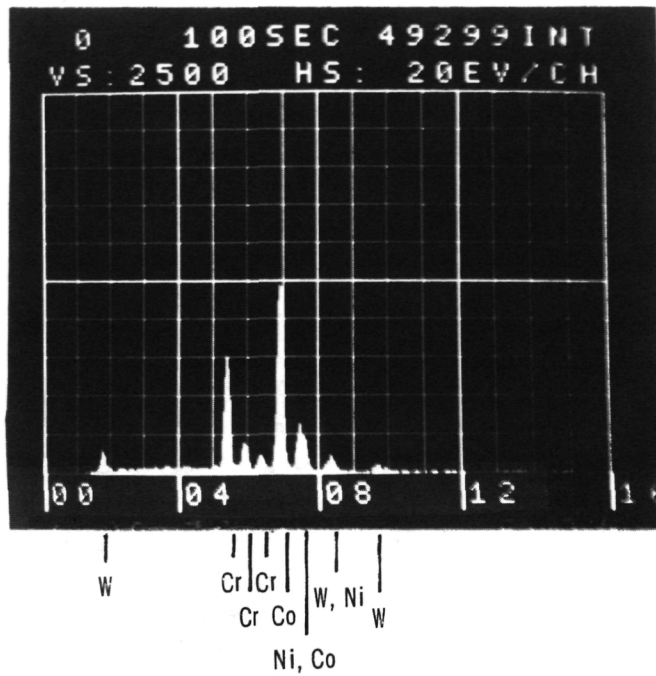


(a) Before.

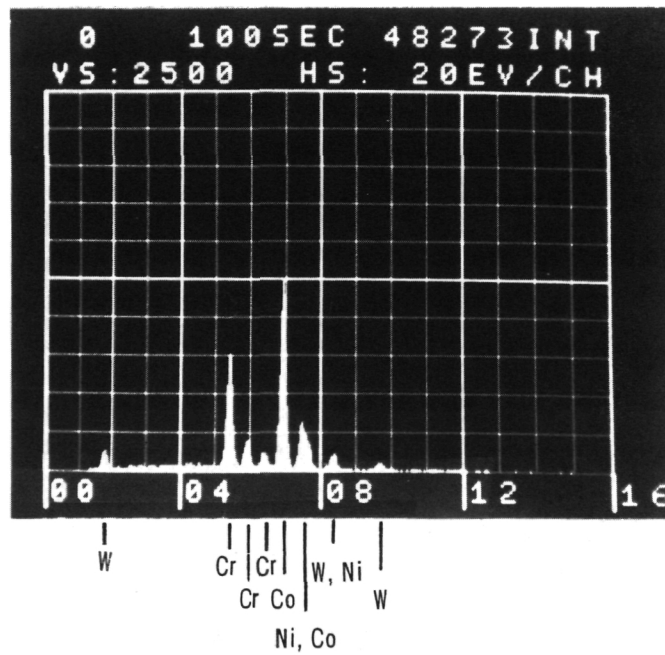


(b) After; 30° tilt.

Figure 5. - Scanning electron photomicrographs of Co-20Cr-15W before and after ion-beam texturing.

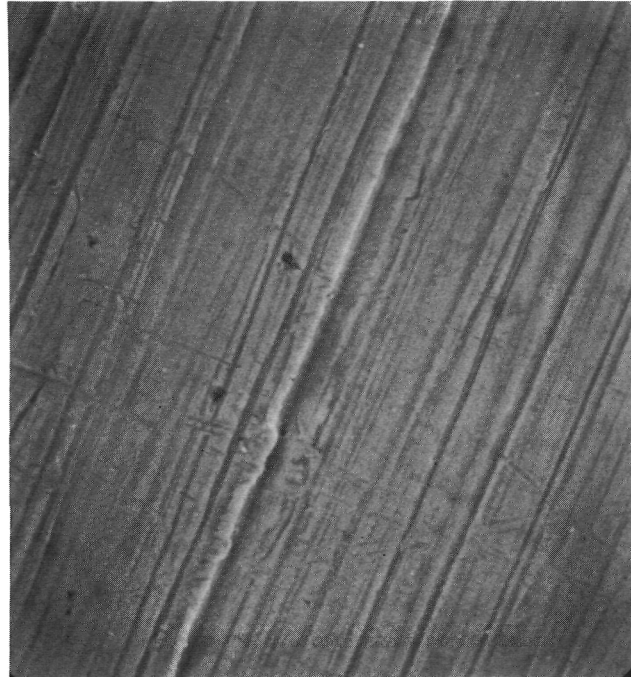


(a) Before.

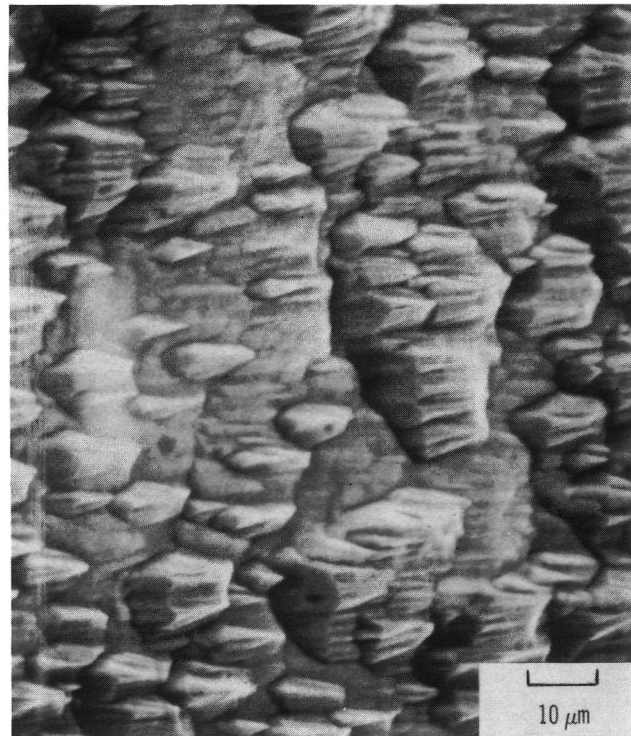


(b) After.

Figure 6. - Energy dispersion analysis of Co-20Cr-15W before and after ion texturing.

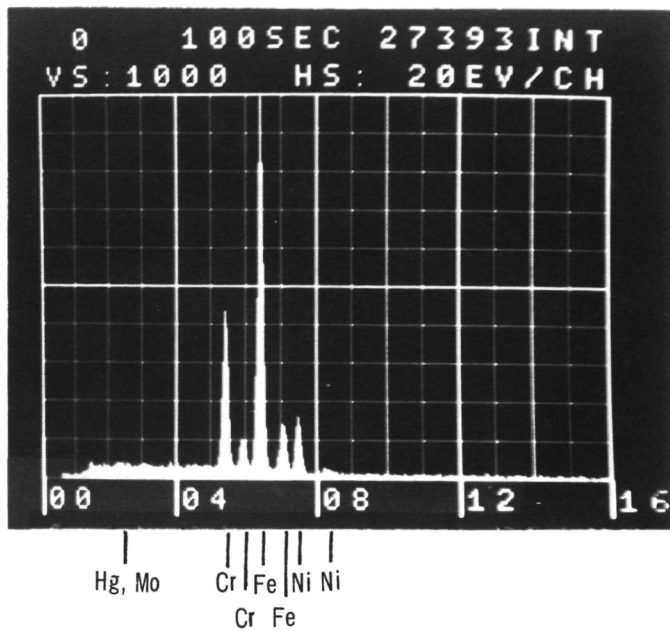


(a) Before.

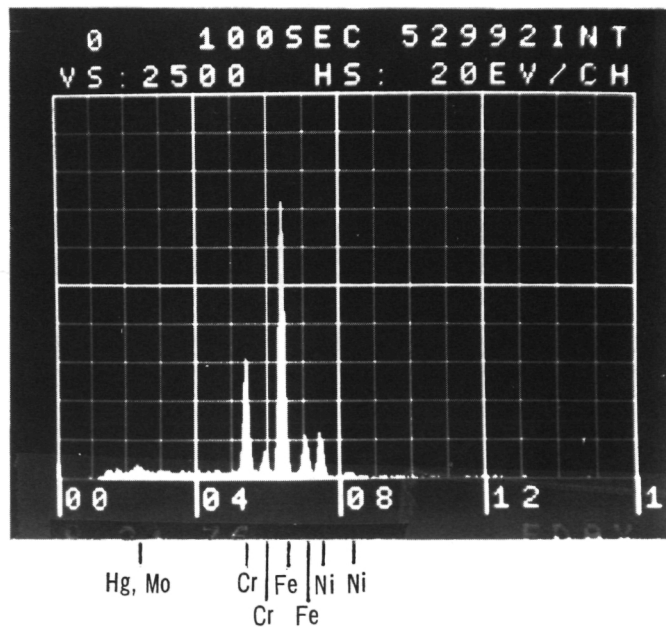


(b) After.

Figure 7. - Scanning electron photomicrographs of stainless steel before and after ion texturing.



(a) Before.



(b) After.

Figure 8. - Energy dispersion analysis of stainless steel before and after ion texturing.

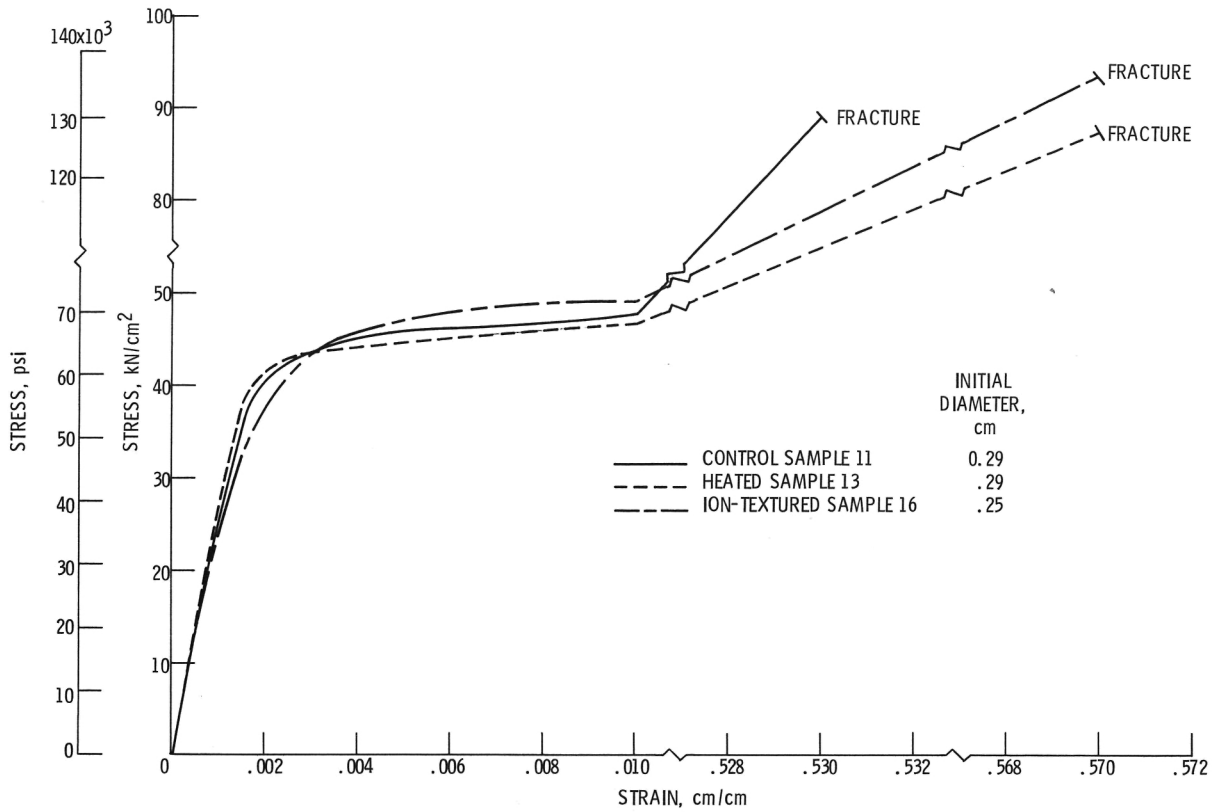


Figure 9. - Ion-texturing effects on stress-strain diagram for Co-20Cr-15W.

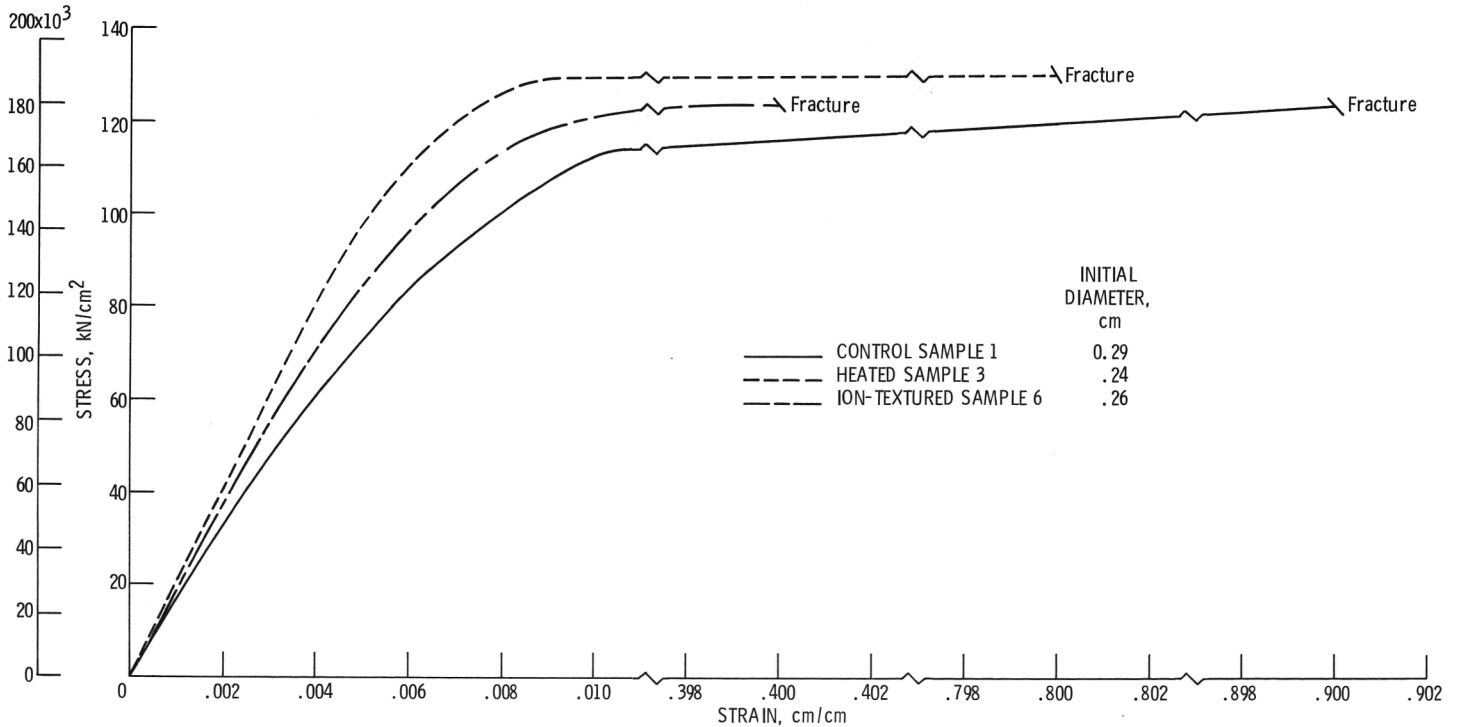
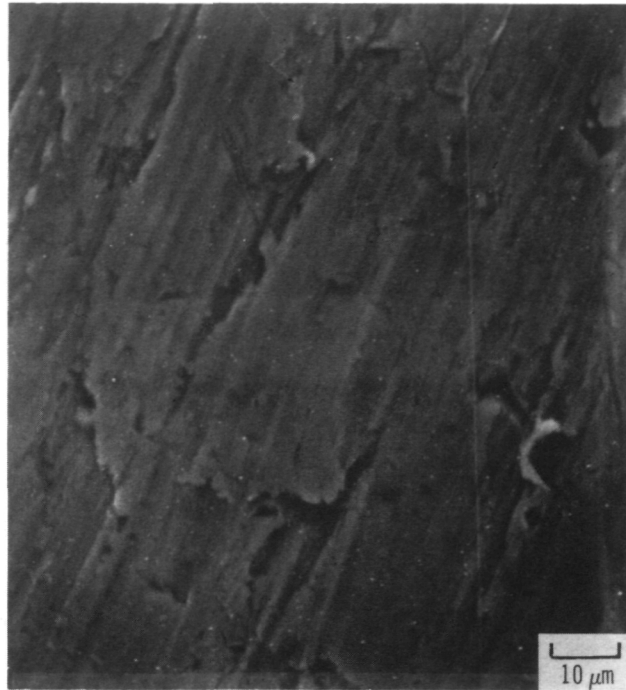
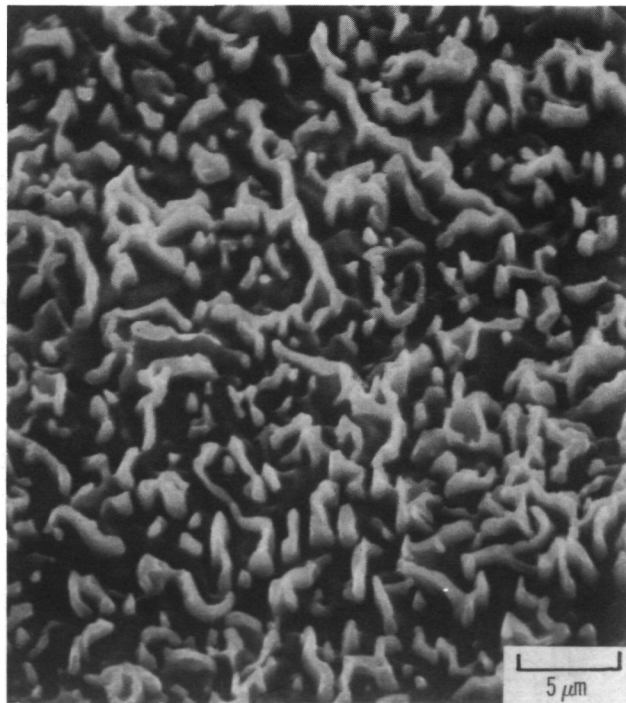


Figure 10. - Ion-texturing effects on stress-strain diagram for surgical stainless steel.



(a) Before.



(b) After; 30° tilt.

Figure 11. - Scanning electron photomicrographs of Ti-6Al-4V before and after ion texturing.

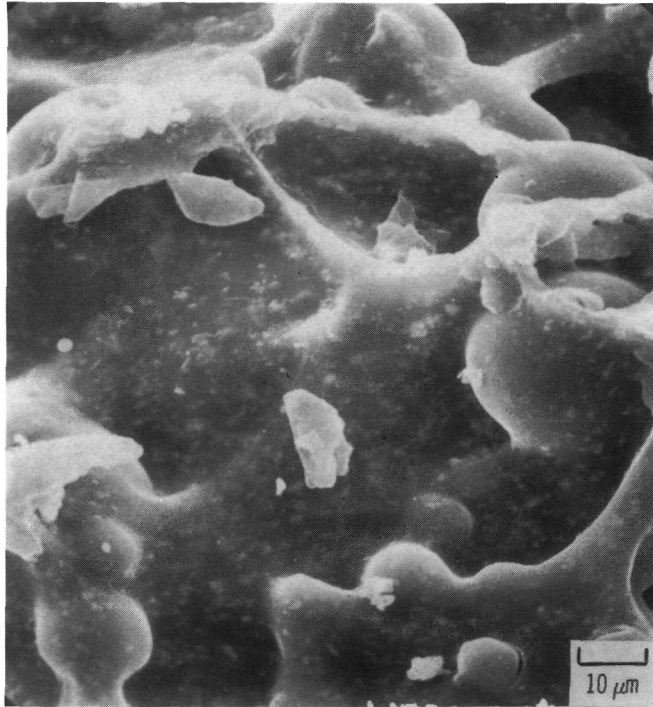


Figure 12. - Scanning electron photomicrograph of polymethyl methacrylate surface at interface with textured Ti-6Al-4V.

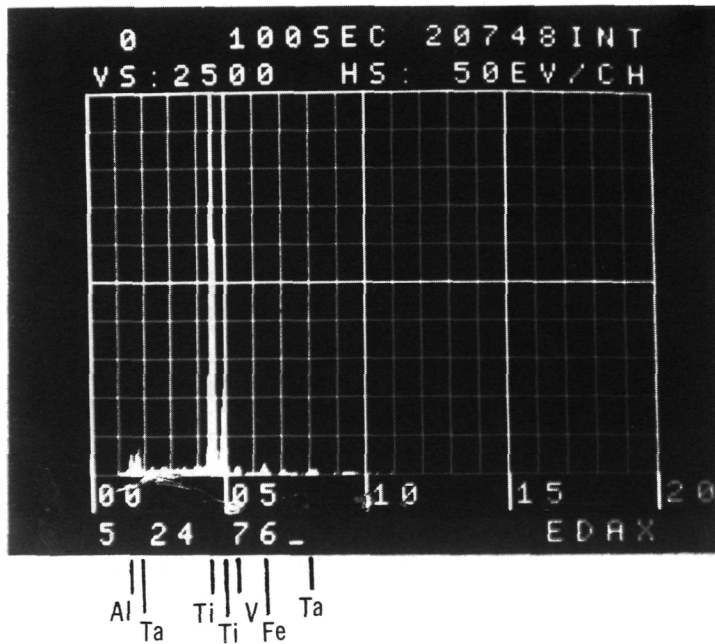


Figure 13. - Energy dispersion analysis of Ti-6Al-4V after ion texturing.

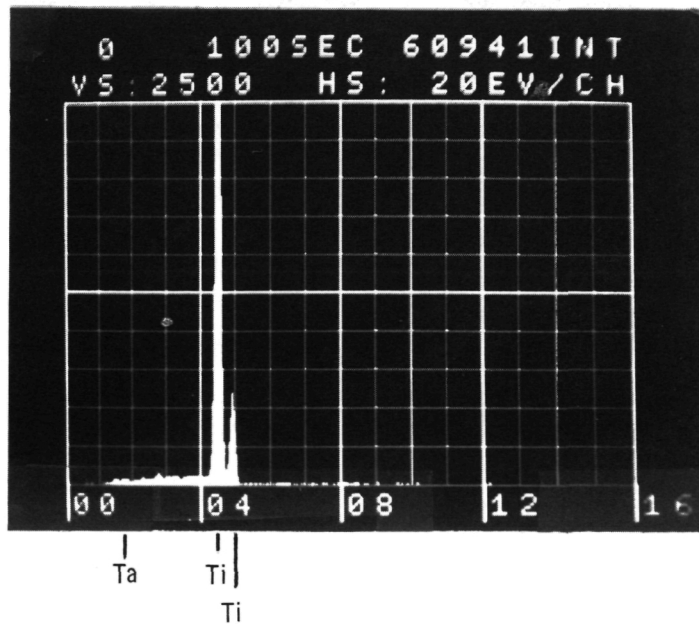
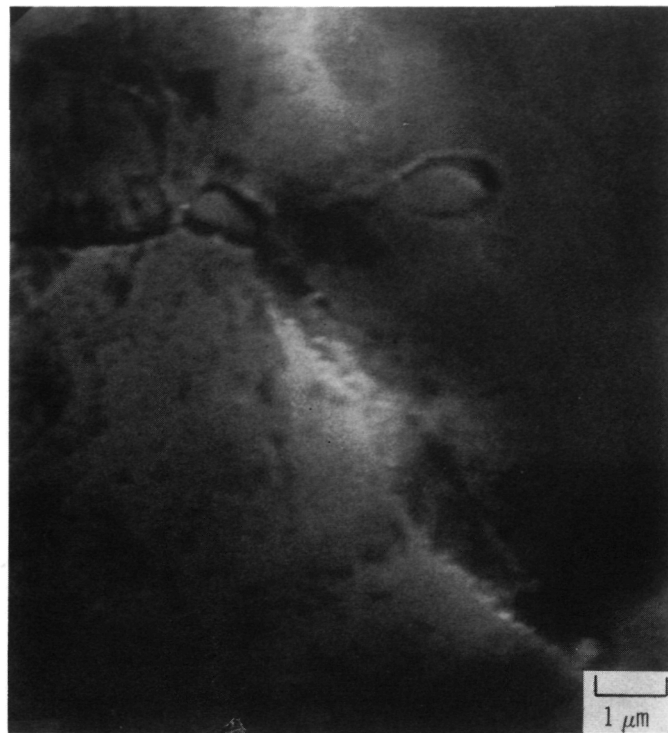


Figure 14. - Titanium ion textured for 1 minute.

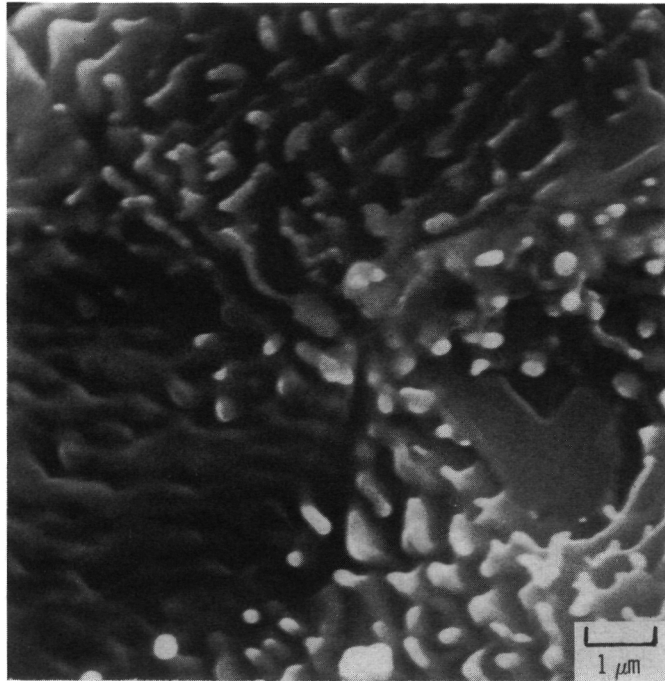


Figure 15. - Titanium ion textured for 2 minutes.

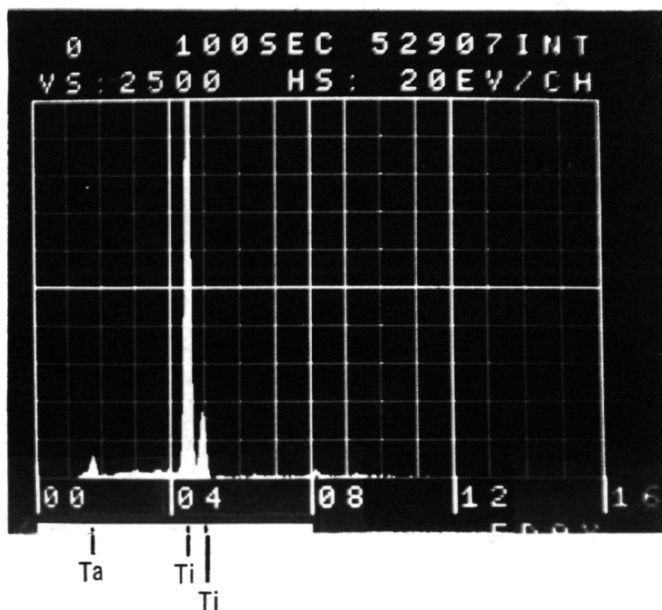
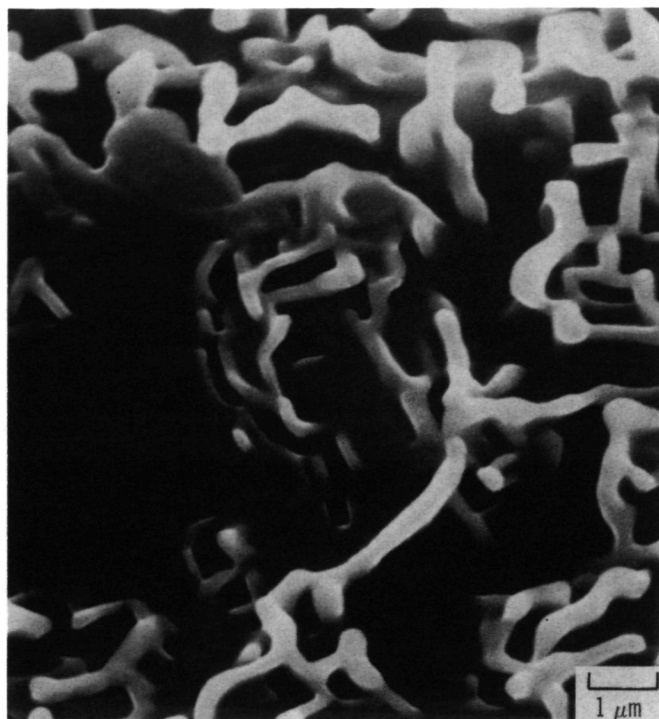


Figure 16. - Titanium ion textured for 4 minutes.

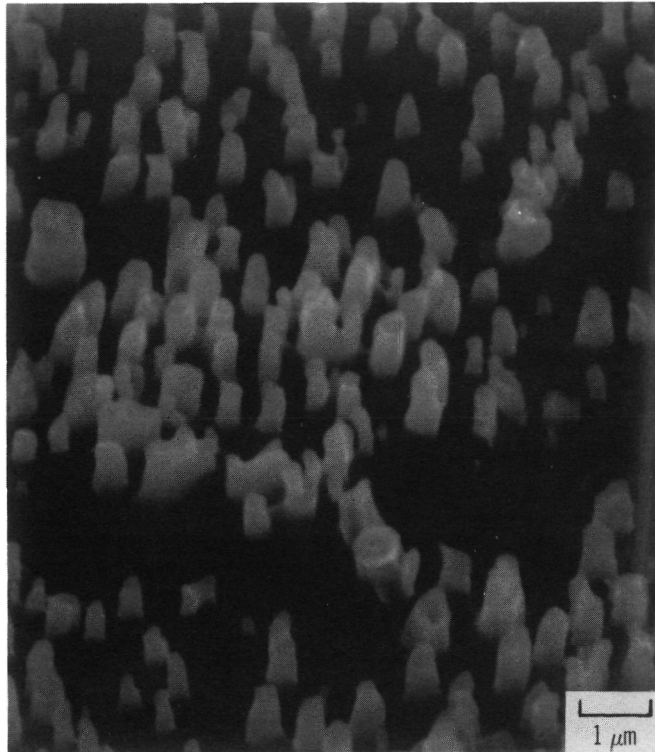


Figure 17. - Titanium ion textured for 8 minutes.

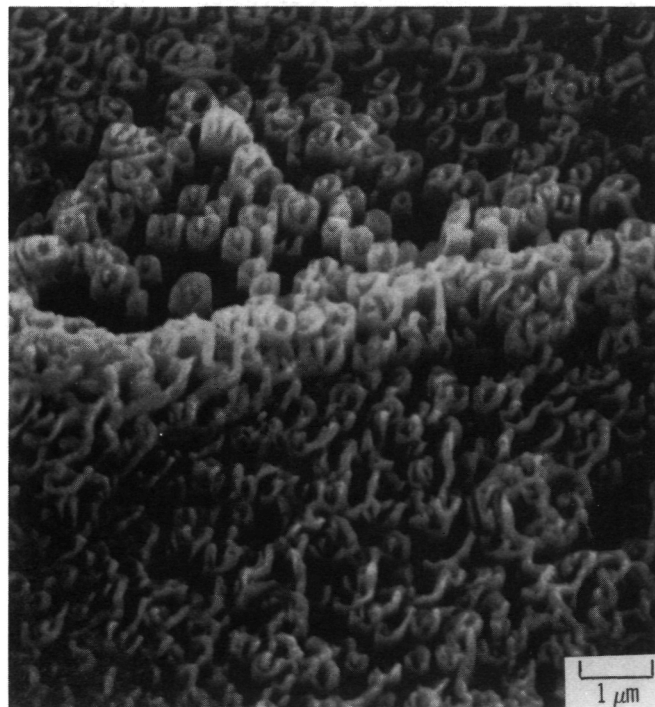


Figure 18. - Titanium ion textured for 16 minutes.

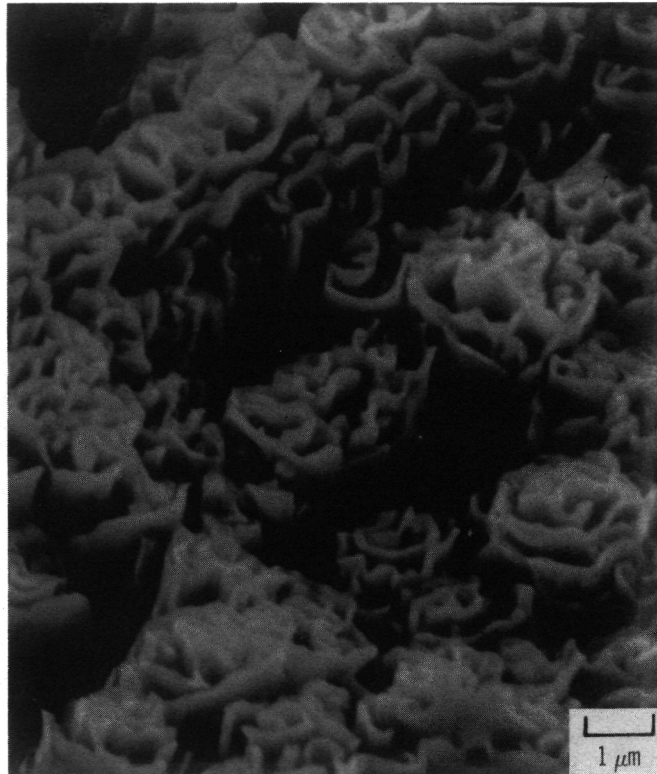
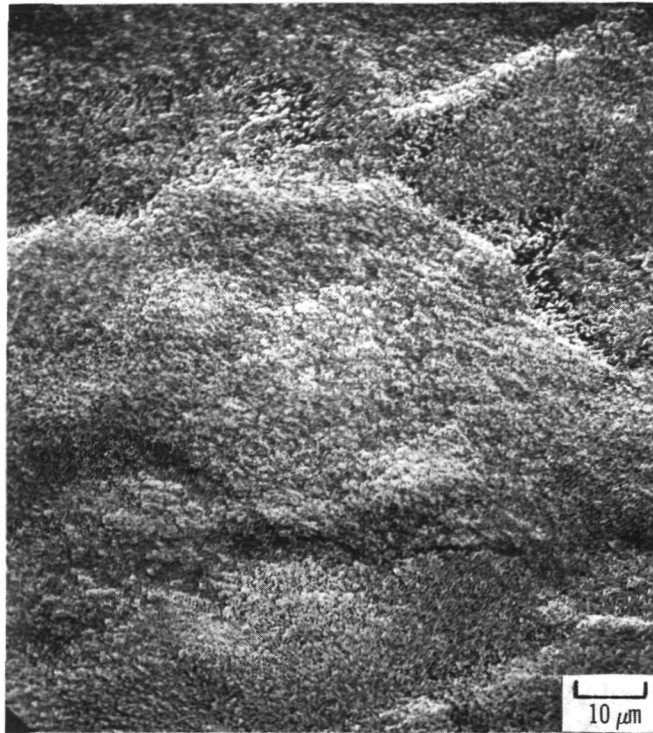
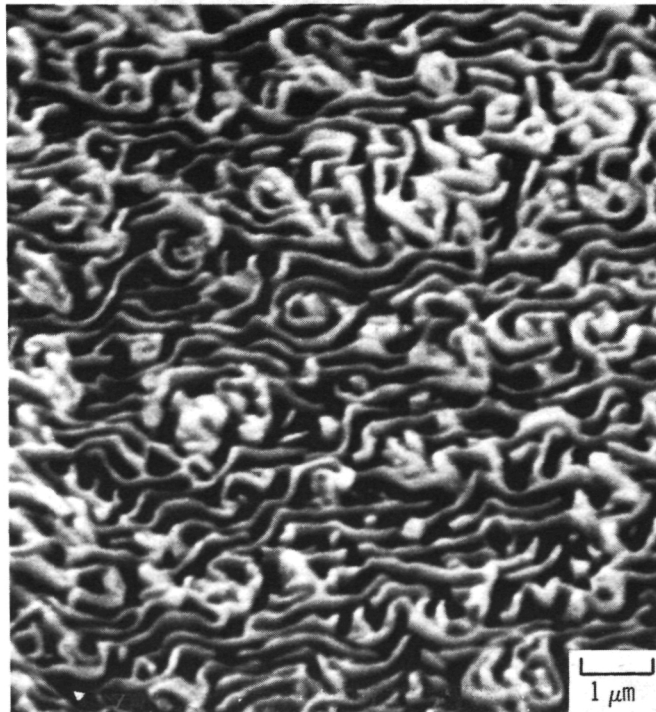


Figure 19. - Titanium ion textured for 32 minutes.



(a) ×1000.



(b) ×10 000.

Figure 20. - Titanium ion textured for 2 hours.

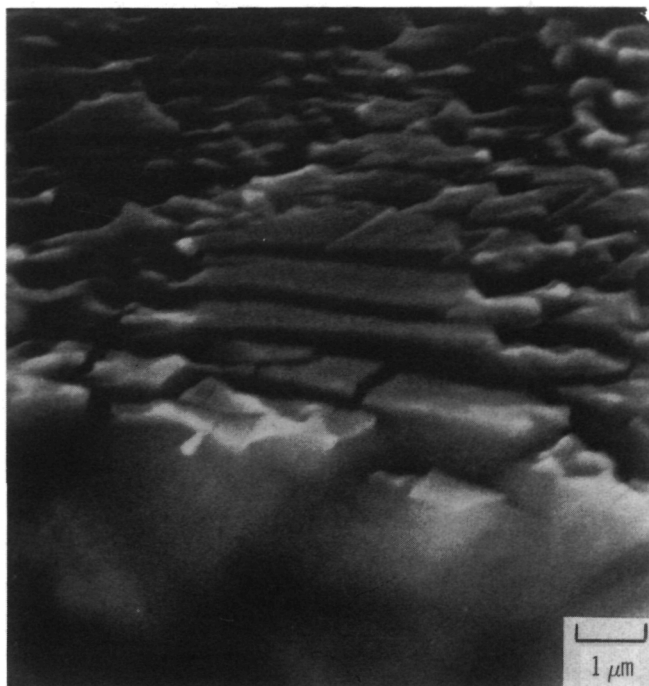


Figure 21. - Scanning electron photomicrograph of freeze-fractured Ti exposed to ion beam for 2 minutes.

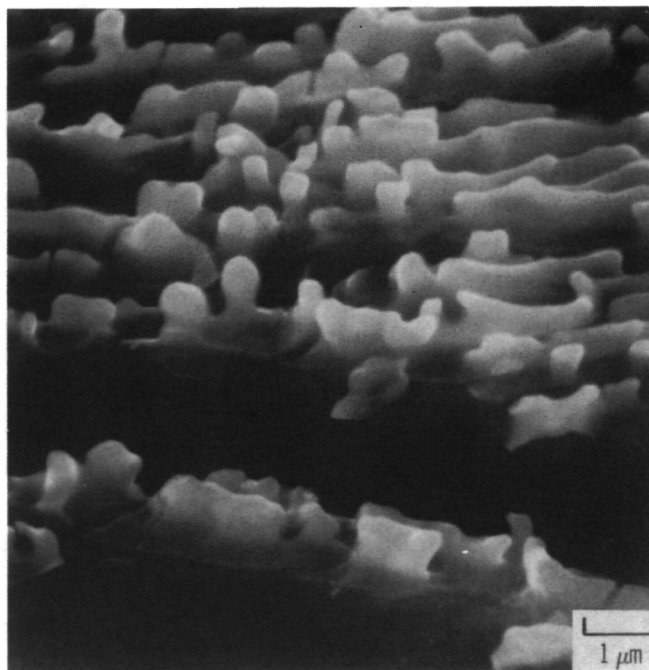


Figure 22. - Scanning electron photomicrograph of freeze-fractured Ti exposed to ion beam for 4 minutes.

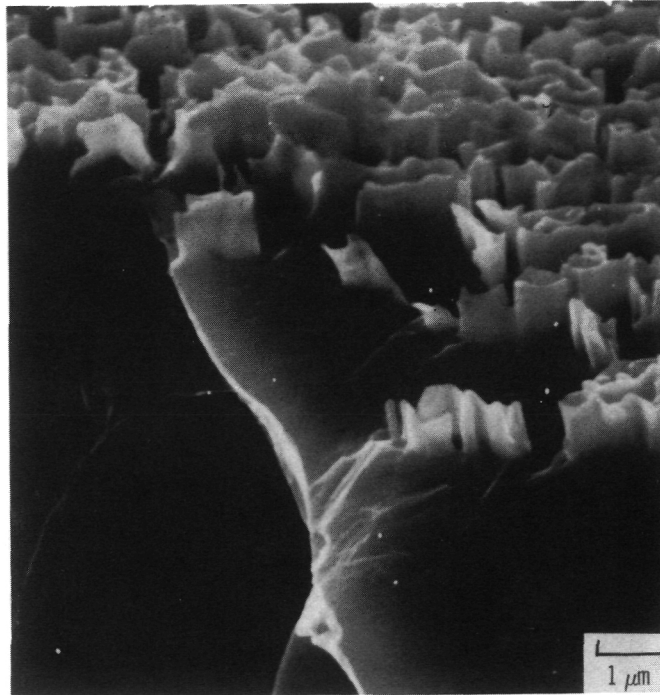


Figure 23. - Scanning electron photomicrograph of freeze-fractured Ti exposed to ion beam for 8 minutes.

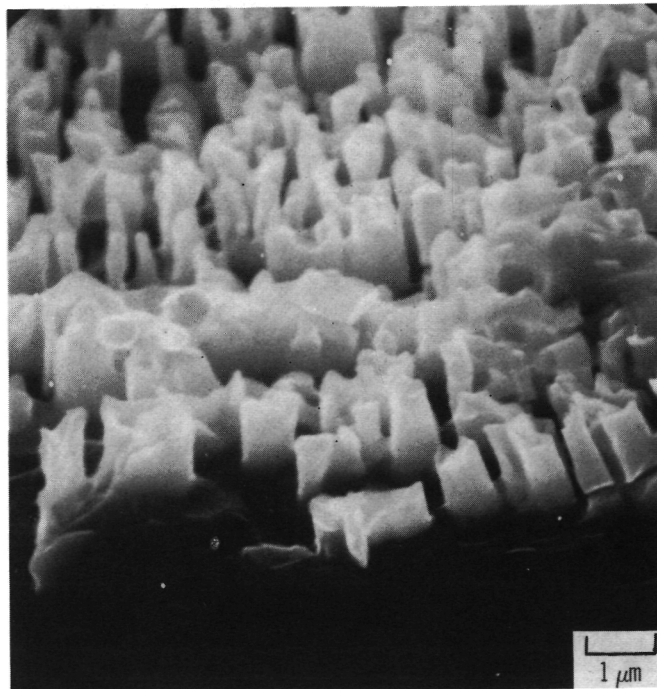


Figure 24. - Scanning electron photomicrograph of freeze-fractured Ti exposed to ion beam for 16 minutes.

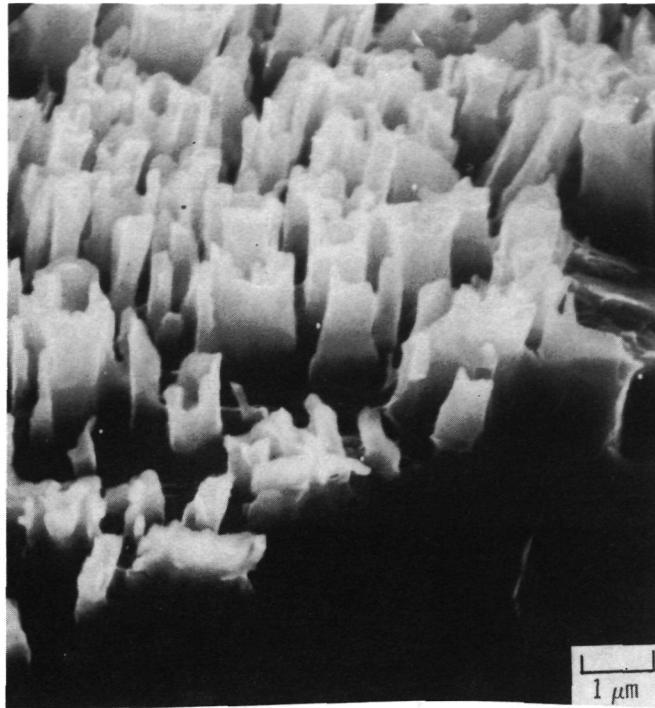


Figure 25. - Scanning electron photomicrograph of freeze-fractured Ti exposed to ion beam for 32 minutes.

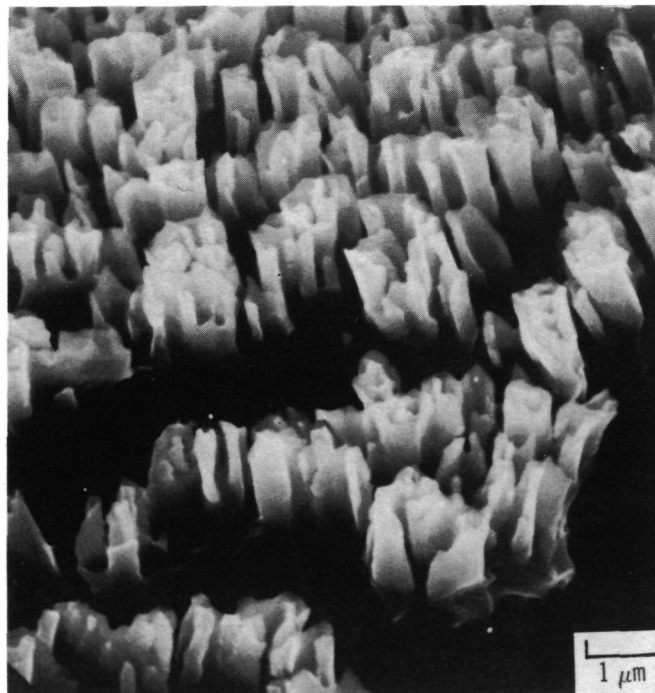


Figure 26. - Scanning electron photomicrograph of freeze-fractured Ti exposed to ion beam for 120 minutes.

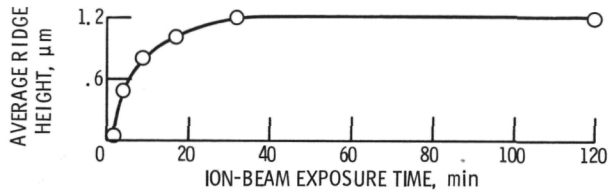


Figure 27. - Microstructure height on Ti samples as function of Xe ion-beam exposure time.

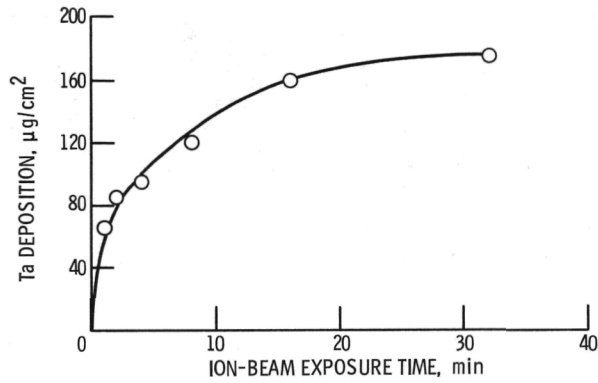


Figure 28. - Amount of Ta seed material on surface of Ti as function of duration of Xe ion texturing.

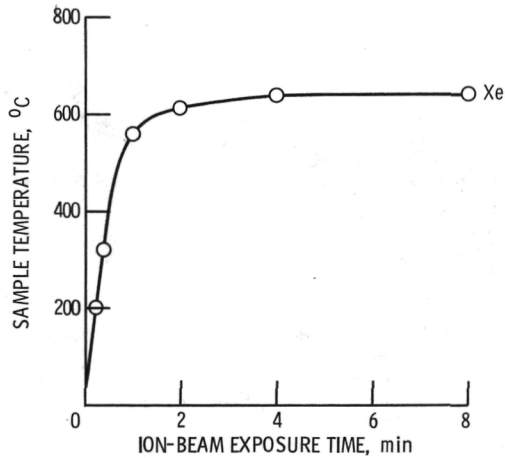


Figure 29. - Sample temperature as function of ion-beam exposure time with Xe as propellant. Ion-beam current, 100 milliamperes; net accelerating voltage, 2000 volts; ion current density, 2 milliamperes per square centimeter.

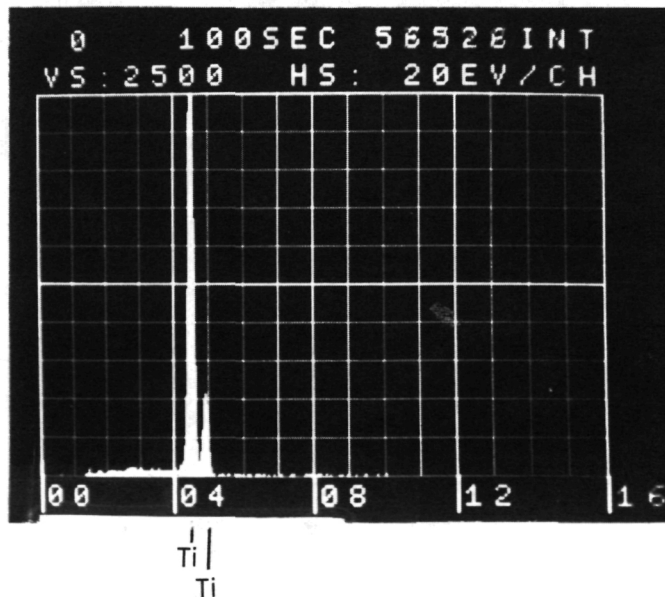
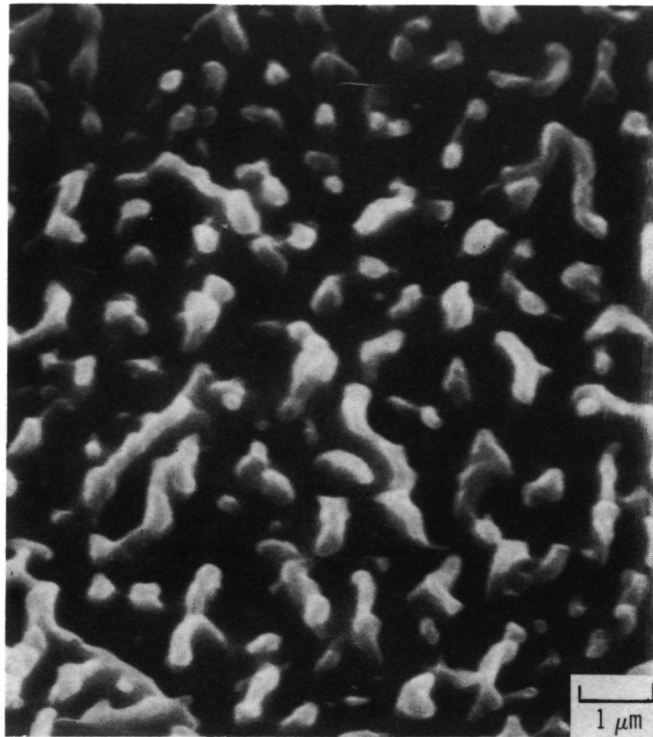


Figure 30. - Titanium ion textured for 1 minute at 640°C (equilibrium temperature).



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—NATIONAL AERONAUTICS AND SPACE ACT OF 1958

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