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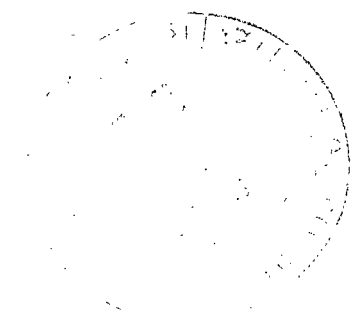
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Improved Adherence of Sputtered Titanium Carbide Coatings on Nickel- and Titanium-Base Alloys

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SUMMARY

Various techniques of applying radiofrequency-sputtered titanium carbide coatings to René 41 and Ti-6Al-4V alloys were evaluated. Coatings were tested with a pin-on-disk apparatus to relate adherence and wear protection to application technique. The coating-alloy interfacial region was analyzed by X-ray photoelectron spectroscopy in order to clarify the mechanisms of improved adherence that resulted with some techniques. For the René 41 alloy, the best adherence was obtained when a small partial pressure (6.7×10^{-3} N/m^2 (5×10^{-5} torr)) of acetylene was added to the plasma before, and during the initial minites of, sputter deposition. This technique produced a carburized alloy surface that bonded better to the carbide coating. For the titanium alloy, the insertion of a titanium metal interlayer approximately 30 nanometers (~300 Å) thick between the alloy and the titanium carbide coating markedly improved wear properties over those obtained with all other methods. The titanium interlayer appears to prevent the formation of the brittle, passive, aluminum oxide layer that normally forms on the surface and is the source of spalling failure.

INTRODUCTION

In the field of wear prevention, the use of hard coatings is often desirable. One of the most attractive methods of applying these coatings is by radiofrequency (rf) sputtering. Many hard coatings applied by rf sputtering have been examined for wear resistance (refs. 1 and 2). These studies have shown that coatings of refractory compounds, such as titanium carbide, can provide good wear protection and, in many cases, low friction. The durability of most sputtered coatings, however, is limited not by the coating properties, but rather by adherence of the coating to the substrate. Most failures of sputtered coatings are due to spalling from the surface to be protected.

Previous work has shown that, for many refractory compounds, adherence to steel surfaces could be significantly improved either by oxidizing the steel surfaces (ref. 3) or by sputtering them in a reactive nitrogen-argon plasma in the initial few minutes to form a nitride-mixed interface (ref. 4). However, many materials that require wear-resistant coatings are not iron alloys. Other alloys are often used as the substrate because of weight, strength, or high-temperature properties. In particular, titanium- or nickel-base alloys are often the substrates requiring wear protection. Therefore, this investigation was conducted to determine which coating techniques would improve the adherence of hard, refractory compounds to alloys of either titanium or nickel.

Titanium carbide (TiC) was chosen as the coating material because it is one of the more effective coatings on steel substrates (ref. 3). The alloys were titanium - 6-percent aluminum - 4-percent vanadium (Ti-6Al-4V) and nickel - 19-percent chromium - 11-percent cobalt - 10-percent molybdenum - 3-percent titanium (René 41). Sputtered TiC coatings were deposited under a variety of conditions onto both Ti-6Al-4V and René 41 disks. The coatings were analyzed by X-ray photoelectron spectroscopy (XPS) as well as evaluated in pin-on-disk sliding friction and wear experiments.

APPARATUS AND PROCEDURE

Radiofrequency Sputtering

The deposition of the coating materials used in this investigation was done in a commercial, multitarget radiofrequency diode sputtering apparatus operating at 13.56 megahertz. Targets of the coating material (either hot-pressed compacts or metals) were cemented with a silver conductive epoxy onto a copper backing plate (0.60 cm thick). The copper backing plates were mounted on water-cooled rf electrodes 15.2 centimeters in diameter. The specimen to be coated was placed on an electrically insulated block that was 2.5 centimeters below the target surface. This block in turn was placed on a rotary substrate table so that the specimen to be coated could be positioned under any of the three targets. In this manner, a specimen could be moved from under one target and placed under a second one so that different layers could be deposited without opening the vacuum system. Also, in this manner, the targets could be cleaned without contaminating the specimen or the targets. An additional voltage of 0 to -1500 volts dc could be applied directly to the specimen either for sputter etching (cleaning) or for biasing the specimens during film deposition and growth.

The entire diode system was contained in a glass bell jar, 45 centimeters in diameter. The system was mechanically forepumped and oil-diffusion pumped through a liquid-nitrogen-cooled baffle. Two variable-leak valves were also attached to the system. One valve regulated the flow of high-purity argon (99.9995 percent); the other valve controlled the flow of either acetylene or nitrogen, depending on which gas cylinder was attached to the bleed-in line.

Before deposition, the targets were cleaned by rf sputtering until there was little pressure rise due to outgassing as a result of target heating. The disks could be cleaned, if desired, by sputter etching with argon at -1200 volts for 15 minutes at a pressure of 2.7 N/m^2. At this time, if acetylene or nitrogen was to be added to the plasma, the argon leak valve was closed and the system was pumped to the base pressure (1.3×10^{-4} N/m^2 ($\sim 10^{-6}$ torr)). Then acetylene or nitrogen was bled into the system to the desired partial pressure. Once the partial pressure was set (in the range 6.7×10^{-3}

to 6.7×10^{-2} N/m^2 (5×10^{-5} to 5×10^{-4} torr)), the argon valve was again opened and the total pressure was raised to the rf sputtering pressure of approximately 2.7 N/m^2. The voltage on the disk was then reduced to either -500 or -1000 volts, and the disk was exposed to the reactive gas-containing plasma for a few seconds to 30 minutes. After this exposure the bias voltage of -500 volts was set, and the rf power was turned on to begin deposition. The specimen was then rotated under the target and deposition began.

After the first 2 minutes of deposition, during which time the critical interface was formed, the valve controlling the acetylene or nitrogen flow was closed and the sputtering environment was again reduced to pure argon without interrupting the sputtering process. The balance of the deposition was then conducted. For samples prepared without acetylene or nitrogen, deposition was begun immediately after the sputter etching of the disk. In all cases the power density was 1.64 W/cm^2 and deposition times were from 5 to 40 minutes; the shorter deposition time was used to make XPS samples a few tens of nanometers thick. For friction and wear testing, the coatings were made approximately 300 nanometers thick. Thicknesses were measured by surface profilometry of a deposition step.

Friction and Wear Testing

The rf-sputtered films were tested in a pin-on-disk apparatus. The pin-on-disk configuration is often used for solid-film lubrication testing. The apparatus is shown in figure 1. The disk (6.4 cm in diam) was René 41 or Ti-6Al-4V rf sputter coated with TiC. A 0.476-centimeter-radius AISI 304-stainless-steel pin was loaded against the surface of the disk. The pin was mounted in a holder on the end of, and perpendicular to, a gimbal-supported arm. The pin was loaded against the surface of the disk by weights hung on the arm halfway between the pin and the gimbal. Normal loads from 0.1 to 20 newtons (10 to 200 grams force) were used. The end of the arm opposite the pin holder was attached to a strain-gage bridge, which measured the friction force. A new pin was used for each test. The entire apparatus was enclosed in a plastic box. A constant flow of dry nitrogen (<20-ppm H$_2$O) was maintained in the box before and during friction tests in order to minimize environmental effects.

The experiments were conducted for 30 minutes at a constant speed of 25 centimeters per second. After friction testing, the disks were examined by optical microscopy and surface profilometry to determine the extent, if any, of film wear or spalling from the substrate. Film spalling is evident on a surface profile by sharp, vertical-sided, flat-bottomed depressions, which are characteristic of a brittle film spall. Under the conditions and for the materials used here, film failure increased rapidly once spalling

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began. The average friction coefficient and rider wear increased because of the greater metal-to-metal contact.

The pin-on-disk configuration provides high contact stresses, in contrast to a conforming type of contact. Because of the uncertainties with regard to modulus values for coated substrates, no attempts to calculate Hertzian stresses were made for the coated sample. Any coating that performs well under such extremes would probably perform as well or better under less severe conditions. Thus, in this respect, the pin-on-disk test can be regarded as an upper-limit screening test for these coatings.

Specimen Preparation

The René 41 and Ti-6Al-4V disks and the AISI 304-stainless-steel pins were mechanically polished in the same manner before they were coated with TiC. The pins and disks were abraded on silicon carbide paper down to 600 grit. Then they were polished with 3-micrometer diamond paste and lapped with 1-micrometer alumina. After lapping, the disks were rinsed in alcohol and dried. For some tests, the disks were placed in a furnace to oxidize their surfaces before coating. The René 41 specimens were heated at 550° C for either 24 or 120 hours since surface oxides differ with oxidation time (ref. 5). The Ti-6Al-4V alloy was oxidized for approximately 20 hours at 400° C.

Coating Analysis

The rf-sputtered coatings and the interfacial regions were examined by X-ray photoelectron spectroscopy. The principles of XPS are presented in reference 6. The apparatus used here consisted of a commercial electron energy analyzer and X-ray source in a vacuum system designed to accommodate a number of samples. An ion gun in the system was used to gradually sputter away the sample surface and thus to determine the composition as a function of depth. The gun used a beam of 5-kiloelectron-volt argon ions that were rastered by deflection plates over the specimens in order to obtain a uniform sputtering rate over the area being analyzed. The sputtering rate was measured on a sputtered TiC coating deposited on a glass slide and on a 55-nanometer-thick gold film evaporated onto a silicon substrate. In both cases the sputtering rate was 0.4 to 0.5 nanometer per minute.

The position of peaks on the XPS spectrum identifies the elements present in the sample surface and gives some information about their oxidation states. The area under a peak is related to the amount of the corresponding element present in the film.

RESULTS AND DISCUSSION

Friction Results

Rene 41. - Several Rene 41 disks that had been sputter coated with TiC under several conditions were prepared and evaluated in pin-on-disk tests. Before sputter coating, the disk surfaces were either sputter-etch cleaned, oxidized, or exposed to a reactive gas-containing plasma. The oxidized surfaces were primarily chromium oxide (Cr_2O_3) or nickel oxide (NiO), depending on oxidation time. Figure 2 shows XPS depth profiles of oxidized Rene 41 substrates, one oxidized for 24 hours and the other for 120 hours. Clearly the shorter oxidation time produced a thinner oxide that was primarily Cr_2O_3 and the longer time produced a thicker NiO layer.

The TiC coating was also applied in the presence of a small partial pressure of acetylene. Acetylene was added to the plasma after the Rene 41 disk was sputter-etch cleaned. The disk was exposed to this plasma for various periods before the rf power was turned on and sputter deposition begun. After the first 2 minutes of deposition, during which the interface was formed, the acetylene was turned off. The friction and wear performance of these coatings is summarized in figures 3 and 4. The bar charts of figure 3 show the average wear and friction for three similarly prepared samples; the surface profile tracing of figure 4 was chosen to be representative.

Oxidation of Rene 41 to either NiO or Cr_2O_3 before sputter coating did not improve the friction and wear performance of the coating. The coatings spalled from these alloy surfaces shortly after testing began (figs. 3, 4(b), and 4(c)), resulting in high pin and disk wear from metal-to-metal adhesive contact. In contrast, the samples prepared with an acetylene partial pressure of 6.7×10^{-3} N/m^2 (5×10^{-5} torr) demonstrated good adherence and thus greater protection against friction and wear at loads of 1.0 newton (figs. 3 and 4(d)). Samples either prepared with higher partial pressures of acetylene (e.g., 1.3×10^{-2} N/m^2 (1×10^{-4} torr)) or exposed to the acetylene-containing plasma before deposition for much longer times (e.g., 30 min) peeled off spontaneously and were not run in friction tests. XPS results (disussed later) indicate that a graphite layer formed on the disk surface in these cases, and this layer could account for the poor adhesion.

Ti-6Al-4V. - As with Rene 41, several techniques were used in sputter coating Ti-6Al-4V samples with TiC. The alloy was sputter etched, as well as preoxidized. The alloy was also exposed to a nitrogen-containing plasma under conditions that improved the adherence of TiC on steel surfaces (ref. 4). These results are summarized in figure 5.

Clearly, neither sputter etching, oxidation, nor exposure to a reactive nitrogen-containing plasma yielded coatings with good friction and wear properties. The coatings

spalled quickly, resulting in high alloy wear even at very light loads (0.10 N). On the other hand, a sample prepared with an interlayer of pure titanium demonstrated very good wear protection. The titanium interlayer was deposited at -500 volts bias to a thickness of approximately 30 nanometers onto a sputter-etched Ti-6Al-4V alloy disk.

After the pure-titanium interlayer was applied, the disk was rotated from under the titanium target to a position under the TiC target and sputter coated with TiC in a continuous process. Samples prepared in this manner were tested successfully at loads to 1.0 newton, even though at no time was any test successfully performed at even 0.10 newton on a sample sputter coated by any other method. The choice of a titanium interlayer was motivated by knowledge that Al_2O_3 was likely to be present at the interface. It was thought that titanium might combine with the oxide and also present a metallic surface to the coating.

XPS Results

René 41. - To understand and better characterize the effect of the acetylene plasma on the René 41 coating interface, samples of René 41 were prepared by sputtering in high (1.3×10^{-2} N/m^2 (1×10^{-4} torr)) and low (6.7×10^{-3} N/m^2 (5×10^{-5} torr)) partial pressures of acetylene at -500- and -1000-volt bias for each pressure. These samples were then analyzed by XPS after they were argon sputter-etched for 1 minute to remove the surface contamination produced by exposure to air. The amount of carbon on the surface increased with a higher partial pressure of acetylene and with greater negative bias voltage. Table I shows this dependence.

Not only did the carbon (1s) peak height vary, but its binding energy was different for the two pressures used. This is illustrated in figure 6 for the -1000-volt samples. The binding energy of 284.5 electron volts of the carbon (1s) peak for the high-pressure sample is approximately the value for graphite (ref. 7). The binding energy of this peak for the low-pressure sample is about 1.3 electron volts lower, at 283.2 electron volts. This would be consistent with the value for a metal carbide (ref. 7), although standards for the particular carbides of interest here were not available to confirm this conclusion.

If the 283.2-electron-volt carbon (1s) peak is due to the presence of a carbide, it would be interesting to know whether it is nickel or chromium carbide. Previous experience (ref. 8) has shown that the XPS peaks due to metal carbides are almost indistinguishable from those due to virgin metal. Nevertheless, the nickel and chromium (2P 3/2) peaks were examined closely for changes that might be correlated with the presence of the 283.2-electron-volt carbon (1s) peak. The signal-to-noise ratio for chromium was so low that subtle changes could not be observed and no conclusion could

be reached about the possible presence of chromium carbide. However, a weak satellite of the nickel (2P 3/2) peak occurred at 860 electron volts. Figure 7 shows that this satellite peak increased in intensity on its high-binding-energy side when the 283.2-electron-volt carbon (1s) peak was present. Again the lack of standards prevented absolute identification of this feature with nickel carbide. However, we tentatively concluded that the 283.2-electron-volt peak was due to the presence of nickel carbide (and possibly chromium carbide) and that the 284.5-electron-volt peak was due to graphite or amorphous carbon. Any one carbon (1s) peak could be a composite of these two peaks (fig. 6(a)), e.g.). An analog curve resolver was then used to separate the two components.

Depth profiles of the major elements present in the high- and low-pressure samples prepared at -1000 volts are shown in figure 8. Sputtering time in these profiles may not be proportional to depth below the surface since there is significant carbon present and carbon has a notoriously low sputtering rate. There are two major features of the profiles to notice. First, the low-pressure sample has a surface with very little graphite but relatively abundant carbide, in contrast to the totally graphite-covered surface of the high-pressure sample. Second, the high-pressure sample is somewhat depleted of chromium in the surface region. This depletion seems to be associated with the presence of graphite and could be due to preferential sputtering in the depth profiling or sample preparation.

Ti-6Al-4V. - The adhesion of TiC to clean Ti-6Al-4V was very poor. Adding oxygen or nitrogen in the sputtering environment did not improve the adhesion. It was thought that, since many aluminum-containing alloys are known to have a passive Al_2O_3 surface layer, such a layer could be contributing to the poor adhesion in this case. It is known that poor adhesion of electroplated films on aluminum is due to the presence of aluminum oxide (ref. 9). Sputter cleaning the surface could remove this layer, but the initial burst of oxygen-containing gas from the TiC target could well reform it, even if a bias voltage is employed, because Al_2O_3 is not reduced to aluminum by bias sputtering (ref. 2). In an attempt to provide a metallic surface to which the TiC might adhere and which, itself might adhere to the surface of the Ti-6Al-4V, a pure titanium interlayer was tried, as previously described. It was markedly successful.

To determine the reason for this success, XPS depth profiles were made of samples prepared with and without the titanium interlayer. These profiles are shown in figure 9. The aluminum metal and aluminum oxide were easily distinguished in the XPS spectra. No distinction between titanium in TiC, pure Ti, or Ti-6Al-4V could be seen, but the presence of carbon and vanadium serves to roughly identify the coating and substrate regions, respectively. In figure 9 these regions are set off by vertical lines.

The data of figure 9 do not show, unambiguously, the reason for the improved adhesion with a titanium interlayer. There are, however, three factors that could con-

tribute to the improvement. First, it is clear that in figure 9(b) there is a much more gradual change from coating to substrate. None of the constituents change as rapidly as they do in figure 9(a). Second, there is less aluminum and Al_2O_3 adjacent to the TiC coating in figure 9(b), although some of these constituents seem to be incorporated into the titanium interlayer. However, it is the aluminum rather than the Al_2O_3 that dominates at the TiC boundary, but in figure 9(a) the Al_2O_3 is the major constituent at the boundary. Finally, in figure 9(b) there is an excess of titanium in the interlayer region, as would be expected. It does not seem possible to decide whether the improved adhession of the TiC on the titanium interlayer is due to the graded interface produced or to the reduced amount of Al_2O_3 and increased amount of titanium at the interface. Indeed, it seems likely that both factors are important.

SUMMARY OF RESULTS

The application and testing of titanium carbide coatings on René 41 and Ti-6Al-4V alloys yielded the following results:

1. For René 41 alloys, the best adherence and wear performance was obtained when the sputtered coatings were applied with a small partial pressure of acetylene added to the plasma. This is related to the formation on the René 41 surface of metal carbides, which bond better to the titanium carbide.

2. For Ti-6Al-4V alloys, the best adherence and wear performance was obtained when an interlayer of pure titanium metal was deposited onto the Ti-6Al-4V alloy surface before deposition of the carbide coating. The interlayer reduced the amount of brittle aluminum oxide that could form at the interface and promoted a more graded interface.

Lewis Research Center,
    National Aeronautics and Space Administration,
   Cleveland, Ohio, January 16, 1979,
            506-16.

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TABLE I. – CARBON (1s) PEAK HEIGHTS ON

RENÉ 41 EXPOSED TO ACETYLENE

[Measured after 1-min argon ion etch.]

Bias voltage, V	Partial pressure of acetylene, N/m^2 (torr)	
	1.3×10^{-2} (1×10^{-4})	6.7×10^{-3} (5×10^{-5})
	Carbon (1s) peak height, arbitrary units	
–500	2250	600
–1000	4600	1170
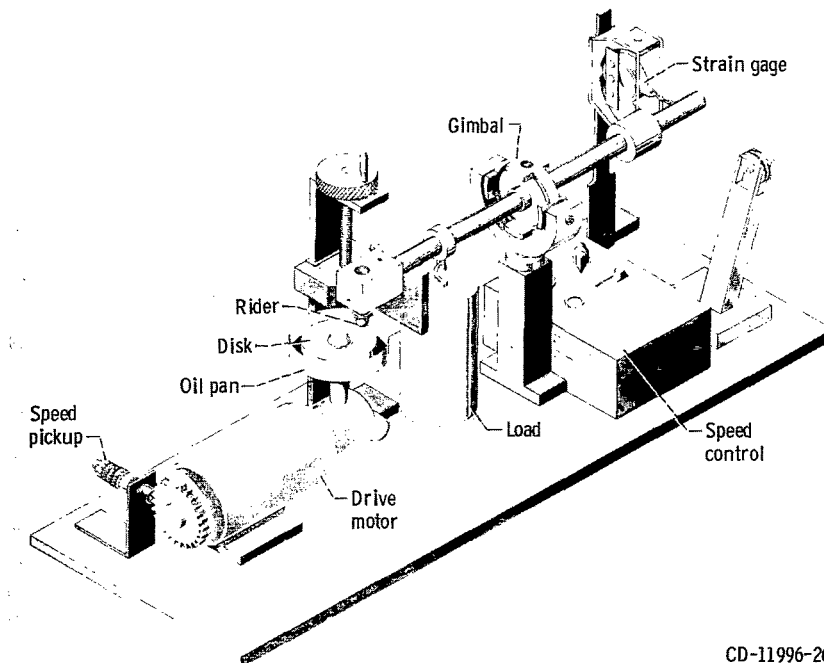
Figure 1. – Pin-on-disk friction and wear apparatus.

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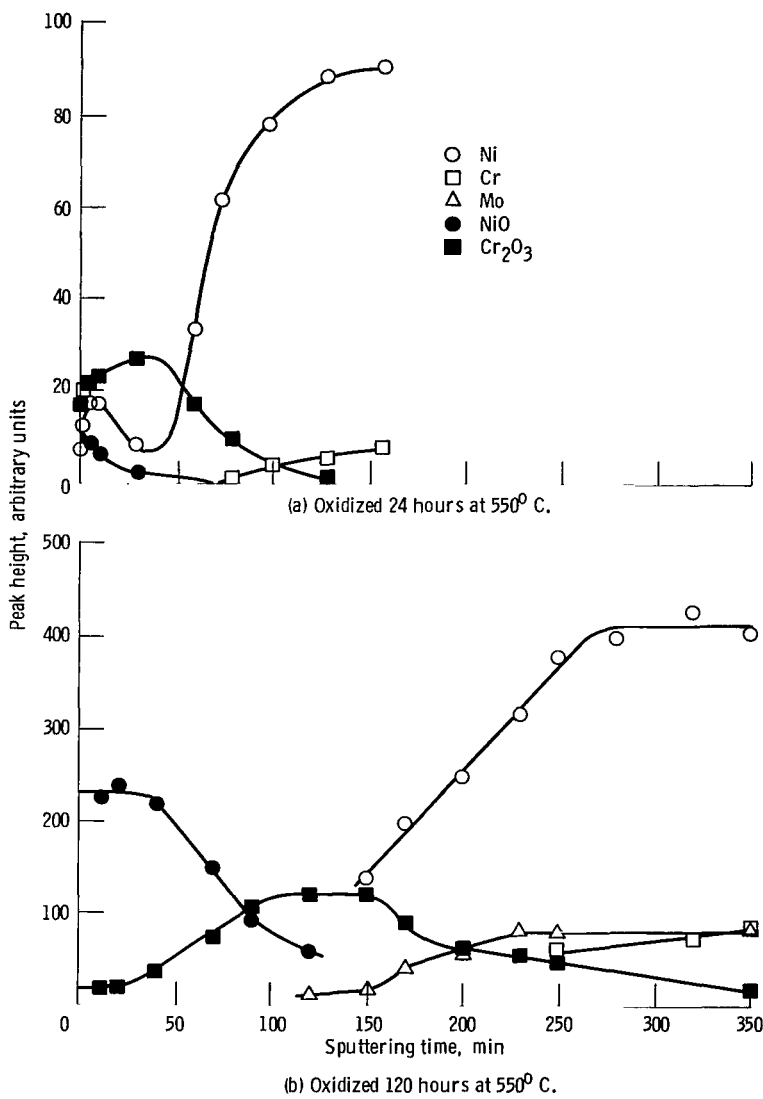
Figure 2. - X-ray photoelectron spectroscopy depth profiles of René 41 alloy.

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(a) Surface sputter etched; average friction coefficient, 0.8.

(b) Surface oxidized 24 hours at 550° C; average friction coefficient, 1.0.
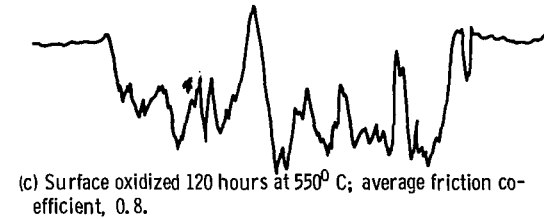
(c) Surface oxidized 120 hours at 550° C; average friction coefficient, 0.8.

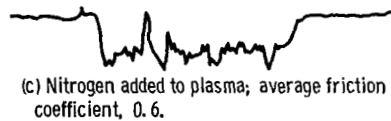(d) Acetylene added to plasma; average friction coefficient, 0.4.

Figure 4. - Surface profilometer traces of wear tracks on titanium-carbide-sputter-coated René 41 alloy. Sliding velocity, 25 cm/sec; run duration, 30 min; pin material, AISI 304 stainless steel; load, 1 N; atmosphere, gaseous nitrogen; average friction coefficients from figure 3.

Figure 3. - Pin wear volume and average friction coefficient for titanium-carbide-sputter-coated René 41 alloy. Sliding velocity, 25 cm/sec; run duration, 30 min; pin material, AISI 304 stainless steel; atmosphere, gaseous nitrogen.
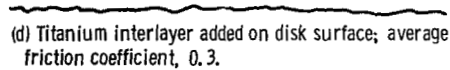
(a) Surface sputter etched; average friction coefficient, 0.5.

(b) Surface oxidized; average friction coefficient, 0.5.

(c) Nitrogen added to plasma; average friction coefficient, 0.6.

(d) Titanium interlayer added on disk surface; average friction coefficient, 0.3.

Figure 5. - Surface profilometer traces of wear tracks on titanium-carbide-sputter-coated Ti-6Al-4V alloy. Sliding velocity, 25 cm/sec; run duration, 30 min; pin material, AISI 304 stainless steel; load, 1 N; atmosphere, gaseous nitrogen; average friction coefficients from figure 3.
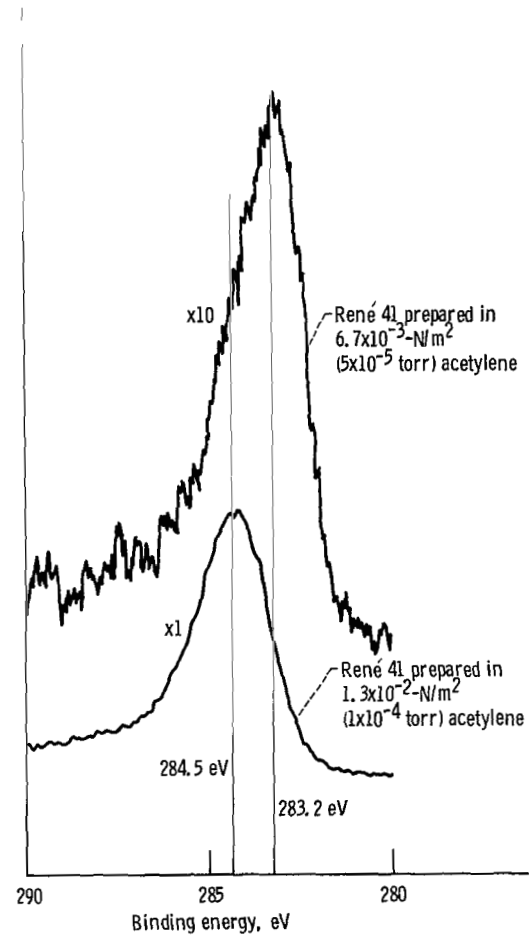

Figure 6. - Carbon (1s) peak after 3 minutes of sputter etching at 5 kilovolts. Emission, 25 mA; argon pressure, 6.7×10^{-3} N/m^2 (5×10^{-5} torr).
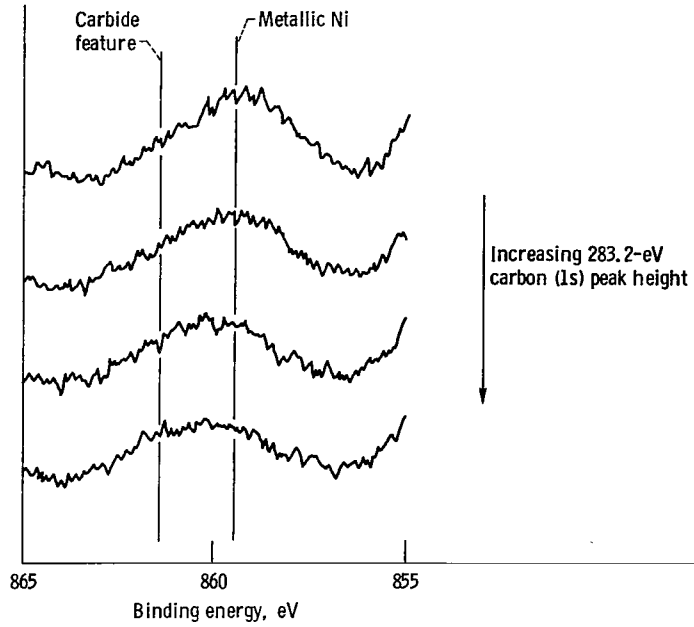
Figure 7. - Change in 860-electron-volt nickel (2P 3/2) satellite peak with increasing carbon content.

(a) Partial pressure of acetylene, 6.7×10^{-3} N/m^2 (5×10^{-5} torr).

(b) Partial pressure of acetylene, 1.3×10^{-2} N/m^2 (1×10^{-4} torr).

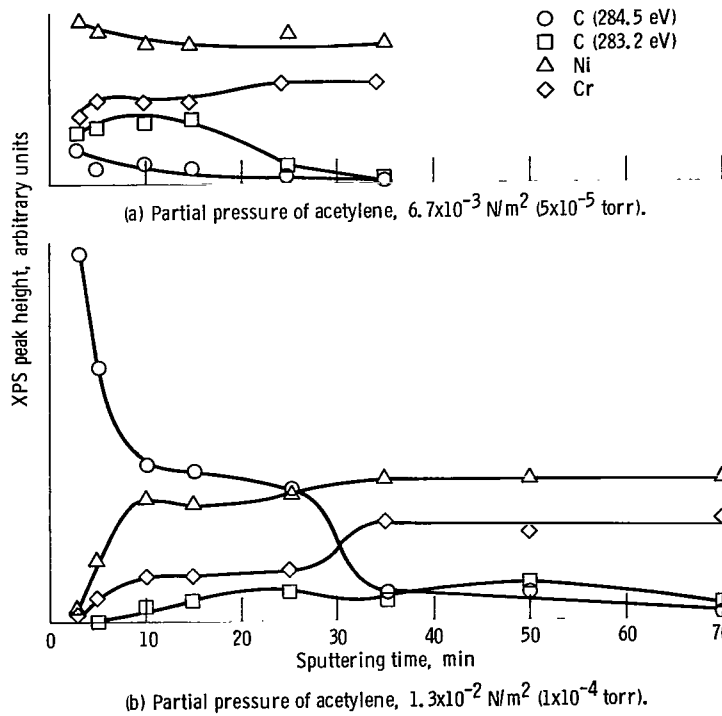Figure 8. - X-ray photoelectron spectroscopy depth profiles of René 41 alloy sputter coated with titanium carbide in acetylene-containing argon at -1000 volts for 2 minutes. Sputtering rate, ~0.5 nm/min.
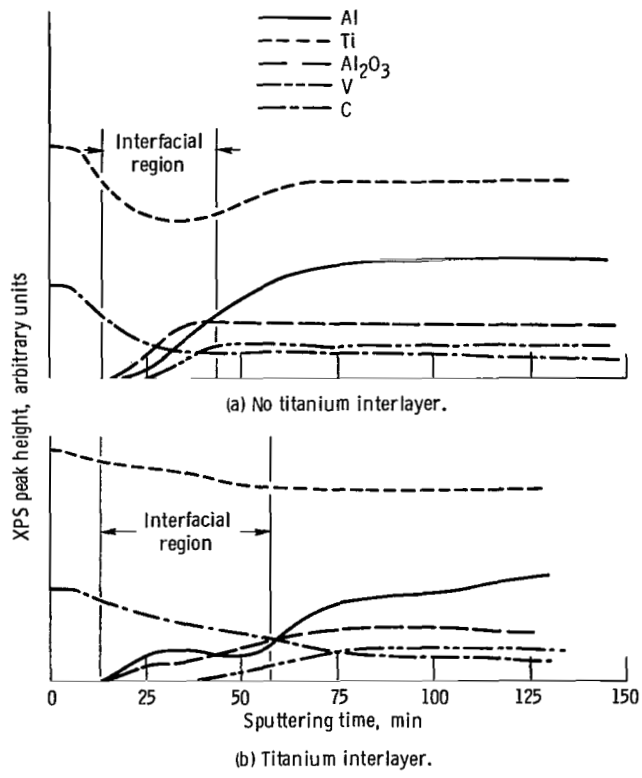
Figure 9. - X-ray photoelectron spectroscopy depth profiles of titanium-carbide-sputter-coated Ti-6Al-4V alloy with and without a titanium interlayer. Sputtering rate, ~0.5 nm/min.

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15. Supplementary Notes

16. Abstract

René 41 and Ti-6Al-4V alloys were rf sputter coated with titanium carbide by several techniques in order to determine the most effective. Coatings were evaluated in pin-on-disk tests. Surface analysis by X-ray photoelectron spectroscopy was used to relate adherence to interfacial chemistry. For René 41, good coating adherence was obtained when a small amount of acetylene was added to the sputtering plasma. The acetylene carburized the alloy surface and resulted in better bonding to the TiC coating. For Ti-6Al-4V, the best adherence and wear protection was obtained when a pure titanium interlayer was used between the coating and the alloy. The interlayer is thought to prevent the formation of a brittle, fracture-prone, aluminum oxide layer.

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