

WEAR- AND EROSION-RESISTANT COATINGS FOR TITANIUM ALLOYS

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Diffusion-bonded electroless nickel plate was investigated as a wear-resistant coating for titanium alloys 6Al-6V-2Sn and 8Al-1Mo-1V. Plate adhesion and diffusion zone structure were assessed by metallographic and X-ray diffraction techniques. Effects of the diffusion heat treatments on mechanical properties and wear characteristics of the titanium alloys were determined. The diffusion bonding produced a surface of nickel-rich intermetallics which significantly improved the wear resistance of the titanium alloys without any appreciable degradation of their structural integrity.

Introduction

It was recognized early in the Army titanium program that the surface characteristics of titanium had many intrinsic shortcomings. For example, titanium alloys were very susceptible to galling, whereby transfer of metal occurs from one surface to another under sliding contact. Under severe conditions seizure occurs, whereby metal parts become welded together.

Recently, several problems associated with the use or potential use of titanium alloys in Army materiel have arisen. Dust ingestion has reduced time-between-overhaul (TBO) on helicopter jet engines operating from unimproved landing sites. This dust has been responsible for erosion which is particularly severe in the compressor section. Dust erosion within the compressor section destroys the aerodynamic profile of axial blades and vanes and wears radial blades of the impeller (Ti-6Al-4V) so thin as to promote fatigue failure. Titanium alloys are also candidate materials for advanced aircraft engines in the compressor section where light weight, high strength, and ductility up to 1200 F (high pressure end) are required. Similar dust erosion problems, aggravated by higher temperatures, are anticipated.

In addition, significant weight reductions in helicopters can be achieved by substituting titanium alloys for steel in transmission components. The poor bearing characteristics of titanium make the development of a composite coating/base alloy system necessary for the utilization of titanium gears.

Approaches to the solution of titanium surface problems involve improved or modified processes for carburizing, nitriding, siliciding, induction surface hardening, coatings and chemical treatments to produce conversion coatings. Our approach will be limited to coatings.

Electrodeposited coatings of chromium and nickel have been considered for obtaining a wear-resistant surface on titanium. Electroplating studies carried out at this Center (1,2) demonstrated that only a moderate degree of adhesion of nickel or chromium could be achieved on titanium alloy surfaces. The key to adherent plate probably lies in the removal or modification of the oxide film prior to plating. Recently, Piontelli (3) claimed to have plated adherent chromium to titanium alloys 6Al-4V and 5Al-2.5Sn. His process consisted of anodizing, cathodically reducing the anodic film to 0.1 micron thickness (this film prevents the titanium from oxidizing in air), and finally electrodepositing chromium. He has reportedly (4) experienced some defoliation of chromium at corners or edges in bend tests.

Electroless nickel has also been considered for applications where resistance to wear and abrasion was of prime importance. In

this technique a nickel salt is reduced with sodium hypophosphite on the surface being coated (5). The electroless plate is usually 93 to 95% nickel, the remainder being largely phosphorous, probably as nickel phosphide. One of the greatest advantages of the process is that the coating deposits uniformly over irregular shapes, crevices, blind holes, and recesses. It thus is ideal for applications requiring plating of roots and tips of threads, worms, gears, etc. The nickel deposit is hard, nonporous and has a low coefficient of friction. Adhesion of the plate is good on most metals. However, on titanium the adhesion is inadequate.

Levy and Romolo (6) improved the adhesion of electroless nickel plate to several older vintage titanium alloys (150A, 155A, 6Al-4V) by diffusion bonding treatments. The interdiffusion between the nickel and titanium produced a wear-resistant surface which, under specific test conditions, was comparable to steel (case-hardened to Rc 60). Our work reported here extends this study to include the more recently available titanium alloys 6Al-6V-2Sn and 8Al-1Mo-1V.

Materials

Titanium Alloys

Test specimens for adhesion, wear, and mechanical property evaluation were machined from titanium alloys 8Al-1Mo-1V and 6Al-6V-2Sn. The alloys were purchased in the annealed condition.

Ti-8Al-1Mo-1V is a near-alpha alloy containing 10 volume percent beta which is distributed at the grain boundaries. The alloy has been mainly used where weldability and moderate strength at or below 1000 F are required. It has potential for jet engine compressor components where superior resistance to creep and good tensile properties at temperatures between 750 and 1000 F are required. The alloy was used in the as-received annealed condition and had the following room temperature mechanical properties: yield strength 120,000 psi; ultimate tensile strength 133,000 psi; elongation 20%; and reduction of area 49%. The chemical analysis was: C 0.22%, Fe 0.06%, N 0.008%, Al 7.6%, V 1.1%, Mo 1.1%, H 0.005% and O 0.09%.

Ti-6Al-6V-2Sn is an advanced alpha-beta composition which can be heat treated to very high strength levels. The alloy provides effective weight savings when extruded shapes, thick plate sections, or forged parts are required for air frame assemblies operating for long times up to temperatures of 700 F. It contains C 0.014%, Fe 0.79%, N 0.014%, Al 5.6%, V 5.7%, H 0.007%, Sn 2.0%, Cu 0.74%, O 0.14% and has the following room temperature mechanical properties in the as-received annealed condition: yield strength 137,000 psi; ultimate tensile strength 146,000 psi; elongation 20.5%, reduction of area 48.2%. For our study the Ti-6Al-6V-2Sn was heat treated to

160, 170, and 180 ksi yield strength levels. However, because of the similarities in results, only the data for the 160 ksi strength level alloy will be presented. Both alloys were processed for electroless nickel deposition and diffusion bonding heat treatments.

Plating Bath

The bath selected was a modified Brenner - Riddell (7,8), composition. The bath composition, preparation, and plating conditions have been described elsewhere. (6)

Experimental Procedure

The preparation of the test specimens involved the following sequence of operations: vapor degreasing, vapor blasting, activation, plating, aging, and heat treatment.

Prior to plating the specimens were trichloroethylene vapor degreased and vapor blasted with 100-grit glass beads at 70 to 80 psi. The specimens were immediately immersed in a slurry of glass beads for not more than 10 minutes, removed and placed in a stream of cold running water, and brushed until all grit was removed. The cleaned specimens were immediately immersed in an activating solution of hot (150 F), slightly acidic, 10% nickel chloride solution for 2 minutes and transferred into the plating bath. If plating did not start immediately, an aluminum rod was used to touch the samples, thus creating an internal galvanic cell, which initiated the reaction.

After plating 0.5 to 0.6 mils of nickel, the specimens were rinsed in a stream of hot running tap water, air dried, and stored in a desiccator for at least 24 hours prior to heat treatment. The plated specimens were vacuum diffusion bonded at 3×10^{-5} Torr at temperatures between 750 and 1550 F for 4 hours and furnace cooled in vacuum.

Plate adhesion and diffusion zone structure were assessed by metallographic and X-ray diffraction techniques. The effects of diffusion bonding on the mechanical properties of the alloys were determined. Wear resistance of the electroless nickel deposits, both diffused and undiffused, were obtained with a modified Mac Millan apparatus.

Results and Discussion

Metallographic Examination of Bond and Diffusion Zone Structure

Figure 1 shows the microstructure of a cross section of electroless nickel plate on Ti-8Al-1Mo-1V in the as-plated condition, heat treated at 750, 950, 1150 and 1350 F for diffusion bonding. The

Table I. X-Ray Diffraction Analysis of Diffusion Bonding of Electroless Nickel on Titanium Alloys

	750 F				950 F				1150 F				1350 F				1450 F				1550 F				
Depth (mils)	Ni	Ni ₃ Ti	NiTi ₂	Ti	Ni	Ni ₃ Ti	NiTi ₂	Ti	Ni	Ni ₃ Ti	NiTi ₂	Ti	Ni	Ni ₃ Ti	NiTi ₂	Ti	Ni	Ni ₃ Ti	NiTi ₂	Ti	Ni	Ni ₃ Ti	NiTi ₂	Ti	
8Al-1Mo-1V																									
0.0	A	B	C	-	A	B	C	-	A	B	C	-	-	A	C	-	-	A	C	-	-	A	D	-	
0.2	A	B	C	D	A	B	C	-	A	B	C	D	-	A	C	-	-	A	C	-	-	A	D	-	
0.4	A	B	C	C	A	B	C	C	A	B	C	D	-	A	C	-	-	A	C	-	-	A	B	-	
0.5													-	A	C	-									
0.6	B	C	C	B					B	B	B	D	-	A	C	-	-	A	C	D	-	A	B	-	
0.7					D	-	-	B					-	A	C	C	-	A	B	C	-	B	B	C	
0.8	-	-	-	A	-	-	-	A	-	B	D	C	-	A	B	C	-	B	B	B	-	C	A	C	
0.9													-	B	B	C					-	-	A	B	
1.0					-	-	-	A	-	-	D	A	-	C	B	B	-	C	B	B	-	-	B	B	
1.1													-	-	-	A									
1.2					-	-	-	A	-	-	-	A					-	-	D	A	-	-	C	A	
1.4																					-	-	C	A	
6Al-6V-2Sn - 160 KSI Y.S.																									
0.0	A	B	C	-	A	B	C	-	A	B	C	-	A	B	B	-	D	A	B	-	-	A	D	-	
0.2	A	B	C	-	A	B	C	C	A	B	C	D	A	B	B	-	-	A	B	-	-	A	C	-	
0.4	A	B	C	C	A	B	C	C	A	B	C	C	A	B	B	-	-	A	C	-	-	A	C	-	
0.6	A	B	C	B	B	B	C	B	A	B	C	C	A	A	C	D	-	A	B	D					
0.8	-	-	-	A	D	-	-	A	A	B	C	B	A	B	C	C	-	A	C	C	-	B	B	C	
1.0									-	-	-	A	C	C	C	B	-	B	C	B	-	D	B	B	
1.2													-	-	-	A	-	C	D	A	-	-	C	A	
1.4																	-	-	-	A	-	-	-	A	
170 KSI Y.S.																									
0.0	A	B	C	-	A	B	B	-	A	B	C	-	A	B	B	-	D	A	B	-	-	A	D	-	
0.2	A	B	C	-	A	B	C	-	A	B	B	D	A	B	B	-	-	A	B	-	-	A	D	-	
0.4	A	B	C	C	A	B	C	D	A	B	B	D	A	B	B	-	-	A	C	-	-	A	D	-	
0.6	A	B	C	B	A	B	C	C	B	B	B	C	A	B	B	D	-	A	C	-	-	A	D	-	
0.8	-	-	-	A	-	-	-	A	C	C	C	B	C	B	B	D	-	A	D	-	-	A	C	D	
1.0									-	-	-	A	D	B	B	D	-	B	D	C	-	A	B	C	
1.2													-	C	C	B	-	D	-	A	-	B	B	B	
1.4													-	-	D	A	-	-	-	A	-	-	C	A	
180 KSI Y.S.																									
0.0	A	B	C	-	A	B	C	-	A	B	B	-	A	B	B	-	D	A	B	-	-	A	D	-	
0.2	A	B	C	C	A	B	B	C	A	B	B	-	A	B	B	-	-	A	B	-	-	A	C	-	
0.4	A	C	C	B	A	B	B	B	A	B	B	D	A	B	B	-	-	A	C	-	-	A	C	-	
0.6	C	D	D	A	-	-	-	A	A	B	B	D	B	B	B	D	-	A	C	D	-	A	C	-	
0.8	-	-	-	A	-	-	-	A	-	-	-	A	C	B	B	C	-	A	C	C	-	A	B	D	
1.0													-	C	B	B	-	B	D	C	-	A	B	D	
1.2													-	D	C	A	-	D	D	A	-	B	B	C	
1.4													-	-	-	A	-	-	-	A	-	-	C	C	A
A Predominant					B Moderate					C Slight					D Trace										

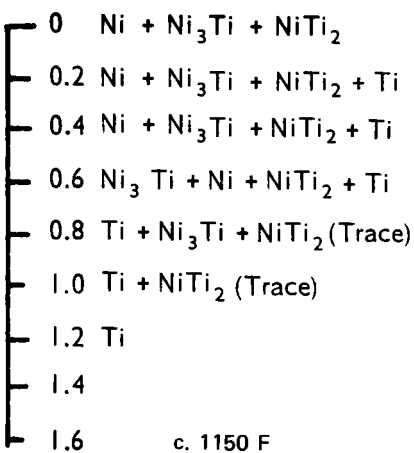
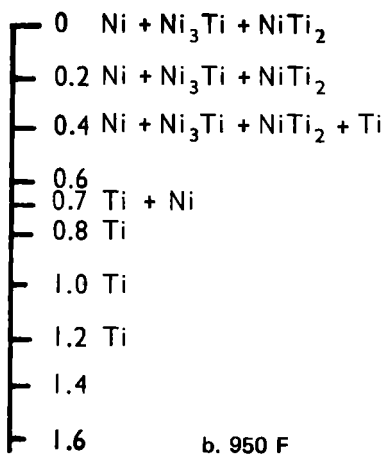
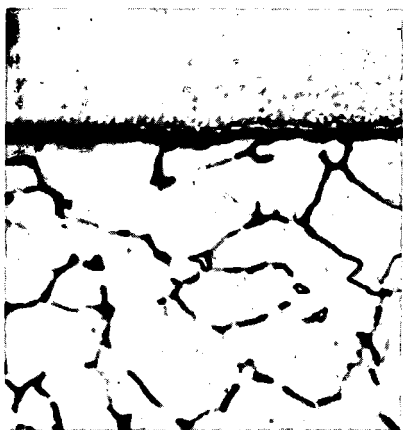
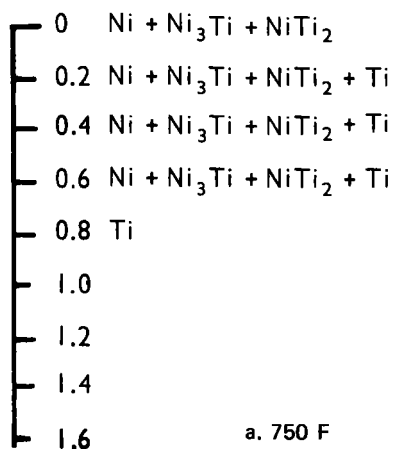


Fig. 1. a-750°F, b-950°F, c-1150°F, electroless nickel diffusion bonded to Ti-8Al-1Mo-1V at various temperatures - diffusion zone structure. X1500

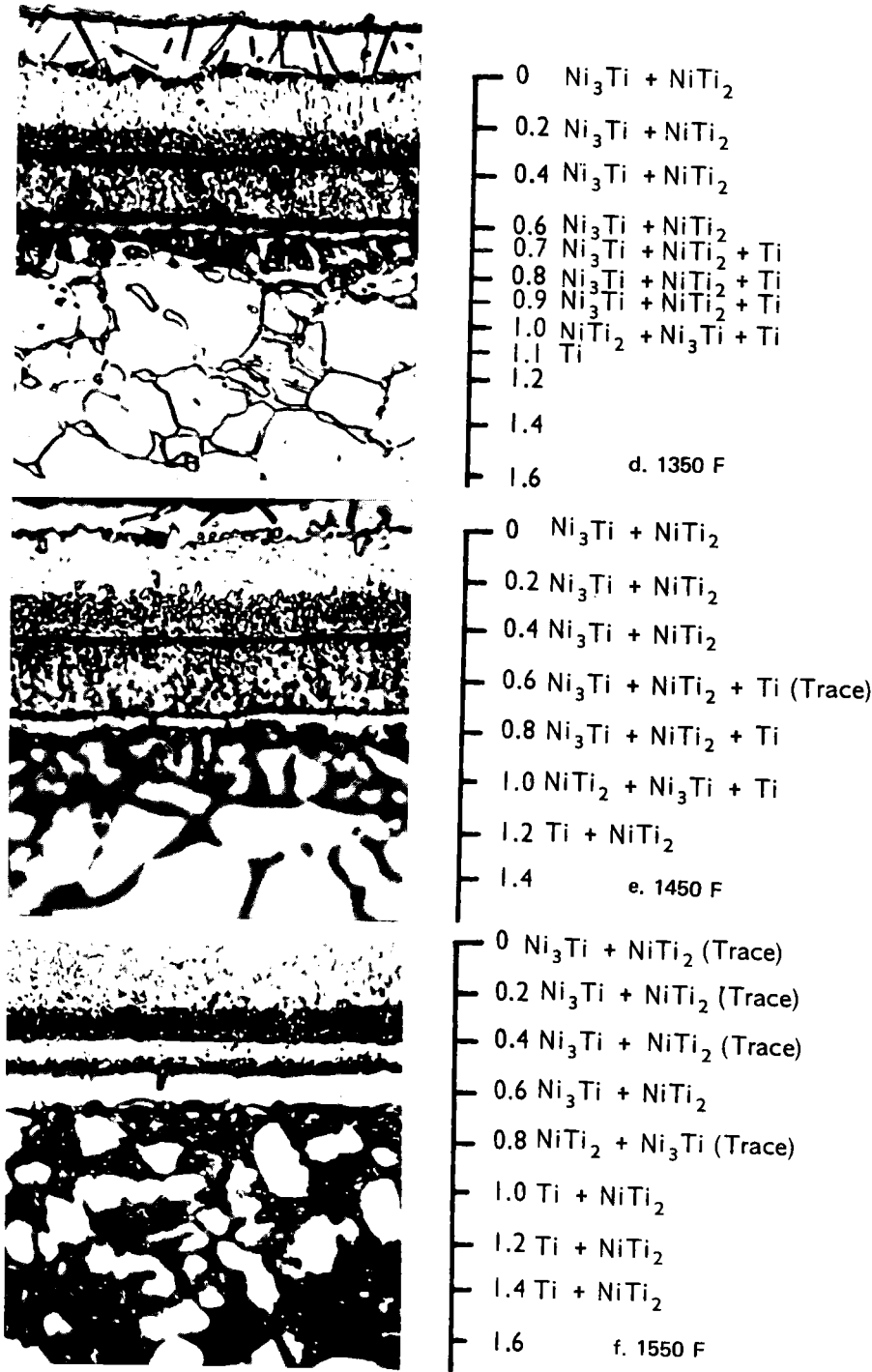


Fig. 1. (cont'd) d-1350°F, e-1450°F, f-1550°F

phases present at the various levels of penetration are identified by X-ray diffraction analysis after selective removal of material in 0.2 mil increments. In the as-plated condition adhesion is relatively poor. Adhesion is improved with increasing temperature and diffusion bonding is achieved at temperatures as low as 750 F. At 1350 F and above, several interdiffusion zones have been formed. Below 1350 F the main constituents of the outermost layer are Ni, Ni_3Ti , and NiTi_2 . Above this temperature the nickel disappears and only the intermetallics are present. Similar results were obtained for Ti-6Al-6V-2Sn with one exception. Nickel remains a constituent of the outer layer at 1450 F and disappears at 1550 F. For both titanium alloys the predominant phases present in the outermost layer and up to 0.5 mil into the diffusion zone are Ni and Ni_3Ti (see Table I). The diffusion zone depth increases with increasing temperature and is approximately the same for both alloys. A maximum penetration of 1.4 mils is noted.

X-ray diffraction analysis did not yield any traces of NiTi or NiP. Duwez and Taylor (10) reported that eutectoid decomposition of TiNi occurs on prolonged heating at 1202 and 1472 F, the phases Ti_2Ni and TiNi_3 being formed. Poole and Hume-Rothery (11) confirmed their conclusion. According to Purdy and Parr (12), TiNi does not undergo a low-temperature eutectoid decomposition. However, the possible decomposition of TiNi could explain its absence in our X-ray diffraction tracings. Chemical analysis of our electroless nickel plate showed the phosphorous content to be 6% on Ti-8Al-1Mo-1V and 9% on the Ti-6Al-6V-2Sn. According to Gutzeit (9) the phosphorous content of electroless nickel plate varies between 3 and 16% depending on the bath and plating parameters. Graham et al. (13) believe that the deposit is a supersaturated solution of phosphorous in crystalline nickel. Randin et al. (14) confirm this and report that the X-ray diffraction lines of nickel and nickel phosphide coincide wholly or in part and since the grain size of the as-plated electroless nickel is extremely small, broadening of the diffracted lines occurs. Thus the phosphide phase would be hardly discernible.

Effect of Diffusion Bonding Treatment on Mechanical Properties of the Alloys

Table II gives the microhardness and converted Rockwell C hardness values for the titanium alloys before and after the electroless nickel plating and subsequent diffusion bonding treatment. In the unplated condition, the higher strength Ti-6Al-6V-2Sn alloy was markedly harder than Ti-8Al-1Mo-1V, which was expected. Significant increases in hardness were achieved for both alloys by the plating and subsequent diffusion bonding treatments at the two extremes of temperature, 750 and 1550 F (Rc 57 for Ti-6Al-6V-2Sn and Rc 50 for Ti-8Al-1Mo-1V). The effects of the plating and diffusion bonding on the tensile strength, yield strength, impact energy, elongation,

and reduction of area of the alloys are shown in Table III. The nickel plating had little or no effect on the mechanical properties of both alloys. For Ti-8Al-1Mo-1V the diffusion bonding treatments (between 750 and 1350 F) had no degradative effects on room temperature tensile and yield strengths. There was an approximately 10% reduction in elongation and reduction of area while Charpy impact resistance was enhanced. Where diffusion temperatures exceed the aging temperature for the alloy, some degradation in mechanical properties can be expected. For the Ti-6Al-6V-2Sn tensile and yield strengths were unaffected by heat treatments between 750 and 1150 F. The heat treatment at 1350 F caused approximately a 5% reduction in these properties. Again Charpy impact resistance increased, but a significant increase rather than decrease occurred in elongation and reduction of area at 1350 F.

Evaluation of Wear Characteristics

The wear resistance of titanium alloys, the electroless nickel deposits both diffused and untreated, were determined with a modified MacMillan Wear Tester which has been described in detail elsewhere (6). Briefly, it uses the other race of a tapered bearing as a specimen which rotates against the full width of a stationary block under load. Failure occurs when either the preset torque of 11 to 13 ft-lb is

Table II. Microhardness of Diffusion Bonding
of Electroless Nickel on Titanium Alloys

Diffu- sion Temp, (deg F)	Hardness									
	Ti-6Al-6V-2Sn									
	160 ksi	170 ksi	180 ksi	160 ksi	170 ksi	180 ksi	Average		Ti-8Al-1Mo-1V	
	Knoop			Rc		(conv)	Knoop	Rc	Knoop	Rc
Not plated	507	518	563	47.8	48.5	51.3	-	-	367	37.0
R.T.	535	539	526	49.6	49.8	49.0	533	49.5	538	49.8
750	668	651	718	56.9	56.0	59.4	679	57.4	534	50.0
950	550	548	572	50.5	50.4	52.0	557	51.0	415	41.0
1150	433	448	465	43.0	42.9	45.0	449	43.6	392	39.0
1350	378	398	380	37.7	39.6	38.0	385	38.4	452	46.0
1450	549	509	537	50.4	48.0	49.7	532	49.4	-	-
1550	684	688	684	57.7	58.0	57.1	685	57.6	-	-

Table III. Mechanical Properties of Titanium Alloys
Electroless Nickel Plated and Diffusion Bonded

Diffusion Temperature (deg F)	Ti-6Al-6V-2Sn							
	Ti-8Al-1Mo-1V		160 ksi		170 ksi		180 ksi	
	Not Plated	Not Plated	Not Plated	Not Plated	Not Plated	Not Plated	Not Plated	Not Plated
a. Tensile Strength (ksi)								
Room Temp.	-	137.0	166.8	169.6	172.1	171.8	182.3	182.7
750	137.4	136.9	171.5	170.0	173.0	170.8	187.9	187.3
950	137.1	137.1	169.4	170.0	171.4	170.9	175.8	183.4
1150	138.8	135.3	168.6	167.1	169.5	168.6	180.3	178.5
1350	135.3	136.0	159.0	159.4	158.0	153.4	158.0	156.4
b. 0.2% Yield Strength (ksi)								
Room Temp.	-	127.5	162.3	160.9	168.5	167.5	179.3	179.3
750	124.8	127.3	165.5	161.8	169.0	167.8	184.0	182.5
950	129.8	137.1	165.0	163.2	167.5	167.5	175.5	175.3
1150	131.3	128.3	164.5	161.0	163.9	165.8	176.8	175.0
1350	127.0	125.8	155.0	152.5	152.0	151.5	153.0	153.0
c. Impact Energy (ft-lb)								
Room Temp.	18.9	17.8	10.2	11.1	9.2	9.2	8.1	8.3
750	17.0	18.3	10.3	10.1	8.5	9.6	8.0	8.0
950	17.7	20.0	8.7	10.0	7.7	9.1	6.4	7.8
1150	19.1	20.0	9.3	12.9	8.3	12.3	7.6	8.8
1350	20.1	24.5	13.0	15.5	10.9	11.5	11.5	14.0
d. Elongation (%)								
Room Temp.	-	17.9	12.5	12.2	11.5	10.0	10.0	10.7
750	14.7	17.2	10.4	11.8	9.3	12.2	8.6	7.1
950	15.7	19.3	7.1	9.3	7.9	10.0	5.7	6.8
1150	17.1	17.5	14.3	14.3	10.7	12.2	7.2	8.2
1350	14.7	17.9	17.9	16.4	15.0	17.9	13.3	16.4
e. Reduction of Area (%)								
Room Temp.	-	40.5	29.5	27.6	17.1	23.3	31.5	34.0
750	33.8	34.7	18.9	30.5	22.5	34.2	21.6	20.1
950	34.3	37.4	10.3	16.9	18.2	24.1	10.9	21.3
1150	35.2	33.8	35.3	36.5	30.2	33.2	30.3	32.2
1350	28.5	38.9	38.9	42.8	37.6	52.1	29.8	48.2

exceeded or a rise in temperature of 100 F above ambient occurs. The wear data obtained are compared with 52100 steel (case hardened to Rc 60) in Table IV. Both titanium alloys in the unplated and as-plated conditions fail in 6 minutes or less. The as-plated material fails after defoliation of plate occurs, attesting to the poor bond in the as-plated condition. The diffusion bonding treatments markedly improve wear resistance which increases with increasing temperatures. Wear resistance is almost comparable to steel at the 750 F heat treatment and significantly better than steel under the same conditions at the higher temperature treatments.

Summary

Electroless nickel in the as-plated condition is poorly bonded to titanium alloys. The diffusion treatments between 750 and 1550 F significantly improve adhesion. For the Ti-8Al-1Mo-1V, the diffusion treatments between 750 and 1150 F produce Ni, Ni Ti, and NiTi (in order of decreasing amounts) in the outer layer; above this temperature (1350 to 1550 F), Ni Ti and NiTi. For the Ti-6Al-6V-2Sn alloy, Ni, Ni Ti and NiTi are formed between 750 and 1350 F, while at 1450 to 1550 F Ni Ti and NiTi only are present. We see that a higher temperature is required to completely transform the nickel into the intermetallics for the 6Al-6V-2Sn alloy (1550 F versus 1350 F).

The diffusion treatments between 750 and 1350 F did not degrade the mechanical properties of the Ti-8Al-1Mo-1V alloy. There was a 5% reduction in ultimate tensile strength and yield strength of the Ti-6Al-6V-2Sn alloy at 1350 F. Wear data show a marked improvement

Table IV. Effect of Diffusion Bonding of Electroless Nickel on Titanium Alloys on Wear Resistance Using the Modified MacMillan Wear Tester

Diffusion Temperature (deg F)	Steel on Steel		Ti-6Al-6V-2Sn		
	52100 Rc 60	Ti-8Al-1Mo-1V	160 ksi	170 ksi	180 ksi
Not plated	136	6.0	5.3	-	-
Room temp.	-	6.0	5.0	5.1	5.7
as plated					
750	-	91	115	128	124
950	-	202	210	210	200
1150	-	334	252	302	296
1350	-	574	326	371	347

attributed to the electroless nickel plate and diffusion treatments. Best results were obtained at the highest diffusion temperatures, 1450 and 1550 F where the major constituents of the outer surface are the intermetallics Ni_3Ti and NiTi_2 . The best wear characteristics are therefore obtained at diffusion temperatures which degrade the mechanical properties of the alloys by about 5%. One therefore has to make a compromise in selecting the optimum diffusion treatment, that is, the temperature at which the best wear is obtained without any degradation of mechanical properties. On this basis 1350 F is recommended for Ti-8Al-1Mo-1V and 1150 F for Ti-6Al 6V-2Sn. At these diffusion temperatures the wear resistance of the coated alloys is still far superior to the wear resistance of case-hardened steel under the conditions of test. In general, the best compromise will most likely depend on the prior heat treatment of the alloys.

Conclusions

We have demonstrated that the diffusion bonding of electroless nickel plate is a promising technique for producing a wear-resistant surface for titanium alloys. However, at this point we cannot infer that this development will solve important Army problems associated with titanium compressor components of gas turbine engines where erosion resistance is of prime importance, or transmission gears in the drive system of helicopters where contact stresses and sliding velocities are all-important. For such applications further testing is necessary whereby the conditions of the test better simulate actual service conditions. Accordingly, in the near future we plan to carry out the following testing programs:

1. erosion testing utilizing a system which is capable of multi-angular abrasive particle impingement at temperatures ranging from ambient to 800 F, with controlled air and particle velocities - simulates dust ingestion from unimproved landing sites.
2. wear testing whereby the combined effects of contact stresses and sliding velocities will be exerted on the coated alloy system in a geared roller tester; and
3. for both applications, fatigue testing utilizing rotating beam specimens.

Since the diffusion bonding treatments have provided a metallurgically bonded nickel-rich surface on titanium, the feasibility of depositing adherent electroplated chromium over titanium alloys is greatly enhanced. Indeed, initial experiments have shown that adherent chromium plate can be deposited over titanium alloys which have been electroless nickel plated and diffusion bonded. Further evaluation for adhesion, wear, and erosion resistance is planned.

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