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A STUDY OF HIGH TEMPERATURE OXIDATION RESISTANCE OF PLASMA TREATED Nb-Ti-Al ALLOYS

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ABSTRACT

The effect of plasma treatment on the oxidation behavior of Ti-22Al-27Nb (atomic%) has been studied. The alloy, cast as an ingot by triple VAR and worked to a billet, was cut into smaller samples and oxidation plasma pre-treated. Oxidation plasma pre-treatments were conducted at four ambient temperatures: 25°C, 200°C, 400°C, and 500°C. Both the untreated and plasma pre-treated samples were oxidized at 700°C and 1000°C for one hour, in a muffle furnace. The oxidation behavior of the samples led us to do an extended oxidation of 100 hours at 700°C. The 200°C plasma pre-treated samples showed a definite leveling out of oxidation rate, while the non-treated samples had a continually increasing oxidation rate at times at or past 100 hours.

INTRODUCTION

Much interest is being shown in the development of alloys based on the ternary Nb-Ti-Al system for use as advanced aerospace materials. For years, titanium aluminides have been of great interest to aircraft-engine builders due to their inherent low density and high temperature strength compared to conventional titanium alloys. Recently, efforts have been made to create alloys with higher amounts of niobium, with higher potential service temperatures than titanium aluminides, using various surface treatments such as cladding, electroplating, chemical deposition, and plasma treatment. The primary objective of this research is to determine the influence of an oxygen plasma pre-treatment on high temperature oxidation resistance of a particular Nb-Ti-Al alloy, Ti-22Al-27Nb (atomic %).

EXPERIMENTAL TECHNIQUE

The Nb-Ti-Al alloy used in this research project was cast as an ingot by triple VAR and worked into a billet. The original sample was cut down to a size of approximately 7.62 x 3.81 x 1mm in size. The samples were then ground on 600 grit sand paper to remove any extreme surface roughness. Four pieces of the alloy were subjected to an oxygen plasma treatment, at

temperatures of ambient, 25°C, 200°C, and 400°C. The plasma pre-treatments were conducted for a total of 12 hours, 6 hours per side. Two of the treated samples from each treatment temperature were then oxidized at 700°C for one hour. The other two treated samples were oxidized at 1000°C for one hour. Finally, four pieces of the alloy were subjected to an oxygen plasma treatment, at temperatures of ambient 25°C, 200°C, 400°C, and 500°C. These samples were then subjected to 100 hours at 700°C with weight measurements and observations being taken every 24 hours. A standard resistance coil muffle furnace with a normal atmosphere was used. Weight change was recorded for each sample tested, using a Mettler AT261 Delta Range balance. Experiments were conducted to determine whether or not the surface deformation that takes place during sawing and cutting of the samples preferentially oxidized, the results showed no effect. Samples were washed with methanol after cutting to remove any lubricating residue that might affect surface oxidation.

Results and Discussion

Visual Observations

After Plasma Treat

The room temperature (25°C) plasma treated sample looked like a non-treated sample.

The 200°C plasma treated sample appeared to have yellow-brown patches on it.

The 400°C plasma treated sample appeared to be a dark blue color.

The 500°C plasma treated sample appeared to be an even darker blue in color.

After 1 Hour at 700°C

The 700°C samples had no apparent change, other than a darkening in color.

After 1 Hour at 1000°C

At 1000°C the non-treated samples had a very small whitish speck dispersed throughout the dark metal surface, with no scales or flakes apparent.

The 200°C plasma treated samples were about 35% - 45% white/yellow surface scale.

The 400°C plasma treated samples were about 15%- 20% yellow scale and 15% white scale on the dark metal surface.

After 24 Hours at 700°C

The non-treated samples showed only a slight color change.

The room temperature samples were almost 100% bluish/white surface scale.

The 200°C samples were 5% - 10% bluish/white surface scale.

The 400°C samples were 20% - 25% bluish/white surface scale.

The 500°C samples varied from 20% - 90% bluish/white surface scale.

After 48 Hours at 700°C

The non-treated samples were dark gray in color.

The plasma treated samples showed no change at this time interval.

After 72 Hours at 700°C

The non-treated samples showed no apparent change.

The 400°C samples were approximately 25% - 40% bluish white surface scale.

The other plasma treated samples showed no apparent change.

After 100 Hours at 700°C

The non-treated samples turned a darker gray.

The room temperature and 200°C plasma treated samples showed no apparent change.

The 400°C samples had trace amounts of yellow in the surface scale.

The 500°C samples showed more surface scale present.

Discussion

The plasma pre-treatment seemed to affect the oxidation resistance best at the 700°C range, for the 200°C sample and especially for the 400°C sample. Due to these initial results at the one hour oxidation and consideration of the aircraft engine application of this material, it was decided to extend oxidation times to 100 hours with varying plasma pre-treatments, and periodic weight measurements. Table I shows the weight gains of each of the plasma pre-treatments at each temperature.

TABLE I. ONE HOUR WEIGHT GAINS

Temperature	Treatment	Gain g/cm^2
700°C	None	0.000125
	25°C	0.000133
	200°C	0.000131
	400°C	0
1000°C	None	0.000646
	25°C	0.00222
	200°C	0.0009125
	400°C	0.0007095

The plasma pre-treatment of 200°C seemed to affect the oxidation resistance of the alloy the most at the 100 hour oxidation times. A 500°C plasma pre-treatment was added, since the one hour oxidation of the 400°C sample was so successful. Table II shows the results of the cumulative weight gain. Figure 1 shows the cumulative weight gain. None of the plasma pre-treatments seemed to compare favorably to the non-treated sample in cumulative weight gain. However, when the data is looked at as incremental weight gain per period (figure 2.), it shows that the oxidation rate of the non-treated sample and that of the 200°C sample seem to be

converging. From this data we might expect that at longer periods, the oxidation rate of the 200°C sample would level off while that of the non-treated sample would continue increasing. It should also be noted that the slight upturn of the curve in figure 1 comes from the fact that the final time period between 72 and 100 hours was 4 hours longer than the other weight gain intervals. Consideration should also be given to the possibility of plasma pre-treatments at temperatures such as 100°C and 300°C to see if these intermediate temperatures have an effect or not. Research on this alloy is continuing, 100°C and 300°C samples will be oxidized. Also, longer oxidation times are also being considered to show whether or not the 200°C sample's oxidation rate will level off.

TABLE II. CUMULATIVE WEIGHT GAIN (100 HRS. @ 700°C)

Treatment	Gain 24 hrs	Gain 48 hrs	Gain 72 hrs	Gain 100 hrs
None	.000117g/cm^2	0.000169	0.000196	0.000287
25°C	0.00054	0.000755	0.000898	0.00112
200°C	0.000256	0.000383	0.000469	0.000577
400°C	0.000443	0.000623	0.000719	0.000848
500°C	0.000544	0.000723	0.000811	0.000994

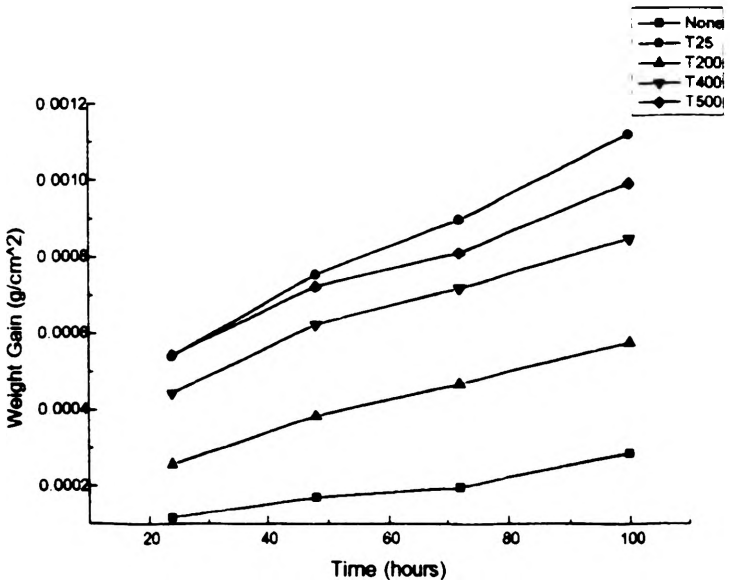

Figure 1 Cumulative Weight Gain (100hrs. @ 700°C)

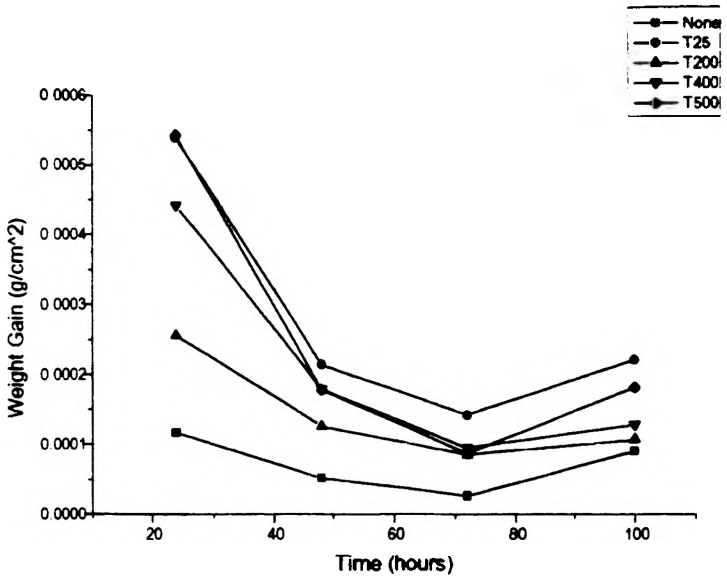

Figure 2. Incremental Weight Gain (100hrs @ 700°C)

SUMMARY

Oxygen plasma treatment in the temperature range 100°C - 300°C shows promise of a possible leveling out of the oxidation rate. Other temperatures did not show oxidation improvements that were expected. More research is being conducted to see if the oxidation rate does level out, and to see if the 100°C and 300°C plasma pre-treatments will have a beneficial effect.

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