Trans. Indian Inst. Met.
Vol. 61, Nos. 2-3, April-June 2008, pp. 99-101

TP 2188

Microhardness Of Laser Ablated Alumina Coating On Ti-6Al-4V

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(Received 26 November 2007 ; in revised form 5 January 2008)

ABSTRACT

Alumina coated on Titanium alloys find wide tribological applications due to the improvement in hardness of substrate. This paper presents the effect of deposition of alumina by pulsed laser ablation on Vickers hardness of Ti-6Al-4V substrate. Nd: YAG laser of wavelength 1064nm is used with sintered alumina disc as target for ablation. The variation of Vickers microhardness with load in Ti-6Al-4V shows indentation size effect. Proportional Specimen Resistance (P.S.R) model is applied to separate load –independent hardness from the load – dependent hardness. Composite hardness of Alumina coated Ti-6Al-4V is measured for different laser processing conditions. The film hardness has been separated from the composite hardness of the film-substrate system by the use of an approach based on the law of area of mixtures model taking into an account of ISE, due to proportional specimen resistance of the material to indentation. Film hardness of different films produced by varying the target – substrate distance is presented. As the film thickness increases its hardness decreases as compared to bulk hardness. These studies will be useful in the selection of appropriate coating thickness and substrate hardness to achieve a required composite hardness in the design and production of wear-resistant parts of engineering devices.

1. INTRODUCTION

Titanium and its alloys are the most attractive materials to be used in aerospace applications due to their high strength, low density, relatively high melting point, excellent corrosion resistance etc. Titanium alloys show low hardness, very low load bearing capacity and poor sliding wear resistance, which restricts their usage in many applications[1]. To overcome this drawback, material surfaces are modified by coating with materials of higher hardness. Ceramics are found to be promising materials for these surface coatings, which can provide enhanced surface properties like wear and hardness[2]. Al_2O_3 coatings are of interest because of their excellent hardness, high wear resistance and high melting point. Alumina coatings can be produced by chemical vapor deposition and various physical vapor deposition processes such as thermal evaporation, arc ion plating, magnetron sputtering, thermal spraying etc[3]. Among these methods, PLD (Pulsed laser deposition) is found to be powerful technique to produce adherent thin films of exact stoichiometry as that of target material. The microstructure and thickness of PLD films can be easily altered by varying processing parameters such as pressure, substrate temperature, laser fluence & target substrate distance[4]. Much work has not been done on ceramic coatings of Titanium alloys using PLD. In addition to this, hardness is one of the important properties of the film that determines the service effectively and reliability of the composite film substrate system in tribological applications. Thickness variation of the film results in different film hardness due to microstructure variation. Our study aims at studying the variation of microhardness of the alumina-coated films by PLD technique on Ti-6Al-4V at different target substrate distances and as a function of variation of duration of ablation.

2. EXPERIMENTAL DETAILS

2.1 Deposition of the film

Titanium alloy Ti-6Al-4V slabs of size 2x2 cm size was used for the Al_2O_3 coating. The surface of the alloy was sequentially polished with SiC water proof abrasive papers form 320 grit to 1500 grit size. The surfaces are then polished to mirror finish of average roughness (R_a) of 20nm using alumina suspension of 1, 0.5, 0.3micron size. Samples are then cleaned using acetone in an ultrasonic cleaner. Cleaned samples are used for alumina deposition. Nd:YAG laser of wavelength 1064 nm, frequency 10Hz is used for ablation. The laser beam has been directed at an incident angle of 45^o on to the rotating target. The substrate is placed at distances of 3,5, 10cm from the target during deposition and the pressure in the chamber is maintained at 10^{-2} Pa at room temperature. After the deposition, sample is heat treated at 600^{0}C for an hour. Alumina discs sintered at 1600^{o}C are used as target.

2.2 Hardness measurements

Hardness measurements are performed using CLEMEX microhardness tester equipped with standard Vickers pyramidal indenter. On each specimen, indentations were made between 25 gf to 1 kgf. At each load, average of 10 indentations is taken as the representative microhardness value. The thickness of deposited films varies from 0.3 to 0.8 μm. The depth of indentation is more than the thickness of the film. Hence the measured hardness is the composite hardness of the film–substrate system. At lower loads coating dominates more on the composite hardness but at higher loads substrate dominates more than the coating on the composite hardness. Relative contributions of the film and the substrate to the hardness have to be separated. These studies will give information about the selection of appropriate coating thickness and substrate hardness to give a required composite hardness[5]. Numerous mathematical models were proposed on the basis of different assumptions. Most common model is based on the "area of mixtures" approach[6] in which composite hardness of the film substrate system is given by

$$H_c = (A_f/A)\ H_f + (A_s/A)\ H_s$$

$$H_f = (A/A_f)[Hc - (A_s/A)H_s] \qquad (1)$$

where A is the total contact area; H is hardness; subscripts f and s denote film and substrate respectively. $A = A_f + A_s$ is the total contact area.

From the geometrical conditions Equation 1 can be written as

$$H_f = H_s + (H_c - H_s) / [2c\ (c/d) - c^2\ (t/d)^2] \qquad (2)$$

where c≈2sin^2 11≈0.07 for brittle film on soft substrate; D is the indentation depth which is about (1/7)th of diagonal length d and t is the film thickness. This model does not consider the load dependence hardness known as Indentation Size Effect (ISE). ISE can be analyzed using P.S.R model[7]. It is suitable for analyzing both the load dependence and the saturation hardness. According to PSR model, the indentation test load P is related to indentation diagonal d as follows.

$$P = a_1 d + a_2 d^2 \qquad (3)$$

$$P/d = a_1 + a_2 d \qquad (4)$$

The value of a_1 and a_2 can be evaluated through linear regression of P/d vs. d. In this model the physical meaning of the two PSR parameters i.e. a_1 and a_2 have been very effectively addressed[8]. It is proposed that the a_1 value consist of two complimentary effects i) the elastic resistance of the test specimen ii) the indenter facet/test specimen interfacial friction. The a_2 value is related to the load independent hardness of the substrate.

By combining the equation 4 with standard Vickers formula, $HV = 1854.4\ P/d^2$

$$HV = 1854.4\ (a_2 + a_1/d) \qquad (5)$$

$$HV = H_o + B/d \qquad (6)$$

Hardness variation with applied load was introduced by Vingsbo et al[9] through Equation (6). is

$$H_f = H_{fo} + B_f / d \qquad (7)$$

$$H_s = H_{os} + B_s / d \qquad (8)$$

Where the H_{fo} and H_{os} are intrinsic hardness of the film and substrate respectively, B_f and B_s are constants. Introducing this variation in Equation (2), and neglecting the second – order 1/d term, the composite hardness is given by

$$H_c = H_{so} + [B_s + 2c_1 t(H_{fo} + H_{so})]/d \qquad (9)$$

$$H_c = H_{so} + [B_s + t(H_{fo} + H_{so})]/d \qquad (10)$$

Where c_1 = c(d/D)≈0.5 for hard film on softer substrate. According to Equation (10) the experimental data on the composite hardness is plotted against 1/d, and the intrinsic hardness of the film is calculated from the slope of regression line. Intrinsic hardness of the substrate is calculated separately from the measured hardness value of the substrate material.

3. RESULTS AND DISCUSSION

The average thickness of films obtained at different target –substrate distances and different deposition duration at the same beam fluence is given in Table 1. The measurement of the intrinsic hardness of the substrate (Ti-6Al-4V) is performed before the deposition of the film. Hardness variation is found for different indentation loads as shown in Fig. 1. Due to the ISE higher hardness value is obtained for lower loads and it is reduced as the load increases. ISE is a result of P.S.R of the test specimen described by the a_1 term of the Equation 3. The value of a_1 depends on the elastic resistance of the test specimen and the friction developed at the indenter facet/specimen interface. Using Eq.4 P/d vs d is plotted for substrate material before and after deposition (Fig. 2). Linear relation confirms that PSR model is applicable for explaining ISE behavior. In our study a_1 value increases with film thickness obtained by varying target – substrate distance. So thicker films show higher contribution to a_1. The second term of the Equation 3, a_2 is related to the load independent hardness by multiplying it with Vickers conversion factor 1854.4. In the present study, load independent hardness is found to be 358HV, which is

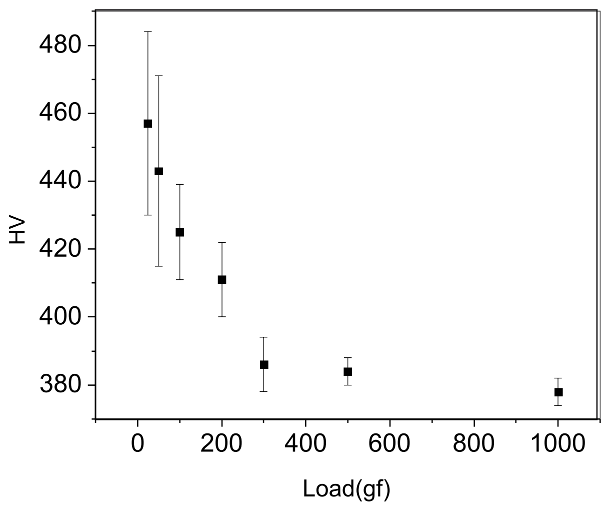

Fig. 1 : Hardness versus load for Ti-6Al-4V substrate.

Table 1
Processing parameters during PLD

Target-substrate distance (cm)	3	5	10	10
Deposition duration (min)	30	30	30	70
Film thickness (μm)	0.6	0.4	0.3	0.8

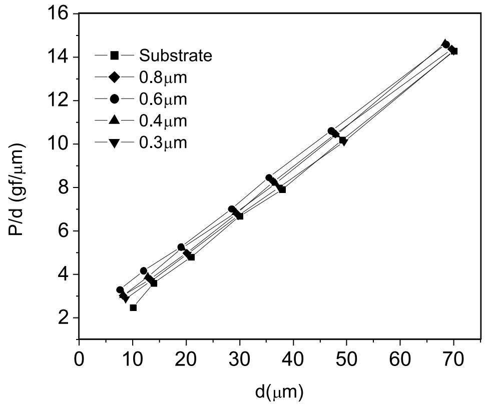

Fig. 2 : P/d plotted against d for Ti-6Al-4V substrate and alumina film-substrate system of different thickness.

close to the value obtained using variation of H_s with $1/d$ [4]. The intercept gives the load independent hardness, which is found to be 369HV. The experimental plots of the H_c vs 1/d are approximated by the linear regression. A least square fit of the plots to the Equation (9) results in the slope $[B_s + 2c_1 t (H_{fo} + H_{so})]/d$ from which film hardness H_{fo} is calculated. At higher loads composite hardness will approach asymptotically the value of the intrinsic substrate hardness, which is found to be 342, 347, 336, 339HV for films having thickness 0.6, 0.4, 0.3, 0.8μm respectively. From the Fig. 3, it is clear that film hardness decreases as its thickness increases. This behavior is similar to that observed on TiC films on silicon substrate by D.Ferro et al[4], which is attributed to the microstructure obtained under different ablation conditions. Variation of target–substrate distance results in different microstructure as reflected by the different a_1 value, which in turn results in different composite hardness. At same target – substrate distance, longer duration deposited film – substrate system shows higher value of a_1. If the film thickness is increased to few hundred nm, resulting film hardness will be same as that of bulk hardness. Thus, the thickness affects the intrinsic hardness of the thin films of alumina coatings. This data can be used in design and production of wear–resistant parts of the engineering devices.

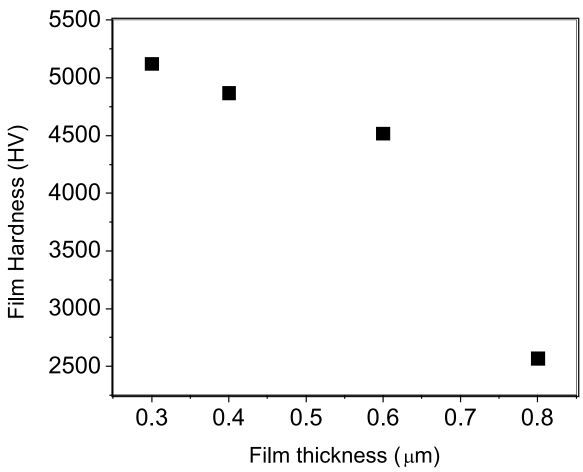

Fig. 3 : Variation of film hardness with film thickness.

Table 2

Values of a_1 & a_2 for different coating thickness

Film thickness (μm)	a_1(gf/μm)	a_2(gf/μm^2)
0.0	0.74	0.19
0.3*	1.18	0.18
0.4	1.39	0.19
0.6	1.84	0.19
0.8*	1.35	0.19

* Deposition at same target-substrate distance (10cm) for different duration (30 & 70 min)

4. CONCLUSIONS

Thin films of alumina can be deposited on Ti-6Al-4V substrate by pulsed laser ablation using Nd: YAG laser of wavelength 1064nm. A proportional specimen resistance model was successfully applied to explain the ISE and to obtain the intrinsic hardness of the Ti-6Al-4V. The film hardness has been separated from the composite hardness of the film-substrate system by the use of an approach based on the law of area of mixtures model taking into an account of ISE. Film hardness is higher than the bulk hardness. Variation in microstructure obtained under different ablation conditions results in different hardness. As the film thickness increases its hardness decreases to that of bulk hardness.

ACKNOWLEDGEMENTS

The authors thank ISRO for Financial assistance in carrying out this research work and Vikram Sarabhai Space Centre (VSSC), Trivandrum for supplying substrate materials.

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