ACTA TECHNICA CORVINIENSIS
– Bulletin of Engineering
Tome IX [2016], Fascicule 4 [October – December]
ISSN: 2067 – 3809

1.Aseel A. KAREEM, 2.Zaina RAHEEM

MICROHARDNESS AND ADHESION STRENGTH OF PMC'S COATINGS BY NiCr ALLOY

1-2.Department of Physics, College of Science, University of Baghdad, IRAQ

Abstract: The use of polymer matrix composites (PMC's) in the gas flow path of advanced turbine engines offers significant benefits for aircraft engine performance but their useful lifetime is limited by their poor environmental resistance. Flame sprayed NiCr graded coatings are being investigated as a method to address this technology gap by providing high temperature and environmental protection to polymer matrix composites. In this research coating was spread with two configuration, coating with bound coat and coating without bound coat. In general the coating with bound coat and coating without bound coat showed increase in micro hardness and adhesion temperature with increase curing temperature; this is due to the microstructural changes the physical splat structure of the coating also changes with heat treatment. All coating failed at the interface between the composites and the coating, failure occurs along the weakest plane within the system, some of the coating systems that have presented fracture at the bond coat/top coat interface. The surface topography of NiCr films was further examined by using AFM atomic force microscopy as a function of curing temperature at 100,200 and 300°C for 1 hour each, it can be clearly seen that the island structure was observed and the R_{max} increase, the surface became rougher with increasing curing temperature. The surface morphology and microstructure of the coating were examined using SEM.
Keywords: protective polymer fiber composites; polymer matrix composites in aerospace applications; high temperature flame spray coating; hard coating

INTRODUCTION

Coating and surface modification technologies allow the engineer to improve the performance, extend component. Surface engineering is defined as the design of a composites system of a surface and a substrate together to give a performance which cannot be achieved breather the surface or the substrate alone [1]. The primary benefit in replacing metals with lower density, higher specific strength PMC's is the weight savings. Additional advantages are the lower processing and fabrication costs [2]. Polymer matrix composites can be successfully deposited by with Thermal spray coating. Successful deposition of a wide array of materials shows that thermal spray coating is available technology for the polymer composites surface protection [3]. A graded coating composition or structure improves the load coatings is astright forwoard process and not as defecult as metallographic preparetion. The system can consist of a coating with or without an interface [4]. Since polyimides are thermally stable at high temperature they are a popular choice for structural parts in aerospace applications, where metal replacement is required with lightweight materials. Polyimide adhesives are used for joining metals and high temperature composites because their coefficient of thermal expansion is comparable to that of metals [5].

Applications of these coatings are widespread and can be found in aerospace, petrochemical [6]. The material selection for turbine engines is a balance between the cost and efficiency, high-strength NiCr alloys are often used in the aero-engine applications for weight reduction [7].

The microindentation indentation technique has been used to characterize the material properties and of coating materials because it is simple and can be performed on small specimens [8].

EXPERIMENTAL

A woven Carbon fiber epoxy composite was selected as substrate; the hand lay-up technique was used to prepare these composites with volume fraction 30%. The composites specimen was cleaned with acetone to remove moisture, dirt oil and other foreign particle. The coating that improves the adherence of the subsequent deposited is called bond coat. Polyimide are used as bond coat, In this work, pyromelliticdianhydride (PMDA) and p-phenylenediamine (PDA), which are commercially available from Sigma-Aldrich are used to prepare polyimide by thermal evaporation technique. These two monomers, 2 gm each, were evaporated from two separated boats to form a poly(amic acid) (PAA) thin film on substrate. The deposition process began at vacuum of 2 X 10^{-5} mbar. The resultant polyamic acid PAA film was then soft baked to remove nH_2O from the substrate followed by a thermal treatment at 250°C for 1 hour each in an air circulating oven, and deposited polyimide film into the composites substrate .The final thickness of films is 5±0.1μm

On the other hand NiCr is used as atop coat .The elemental composition of NiCr alloy samples used in this work was made by using X-ray fluorescence (XRF) analysis technique as shown in Table 1.

Table 1. Elemental composition of the powder used for deposition of coatings.

Powder	Elemental composition (%)					
	Ni	Cr	Si	C	Fe	other
NiCr	43.4	52.6	0.13	0.62	0.17	0.08

Spray Gun (rototec 80), it's used for thermal spraying by flame which was made in Germany by (Castolin+Eutectic) Company. In this process oxygen-acetylene mixture is passed through a nozzle and ignited to form a combustion flame. Ni-Cr Coating powder with particle sizes ranging from 50 to 90 μm were used is fed into the flame, accelerated and projected onto the substrate to form a top coating with thickness about 70±2 μm calculated by magnetic induction measurement methods. The flame temperature is limited to around 1400°C, particle velocities are relatively slow.

Operating parameters during coating deposition process are listed in Table 2.

Table 2. Operating parameters during coating deposition process

Operating Parameters	Values
Oxygen pressure	4 bar
Acetylene pressure	0.7 bar
Standoff distance	200 mm

Before coating the samples are cured at (100,200 and 300)°C.

Hardness type Vickers was conducted for all samples by using (Hensddt-Wetzlar). Vickers hardness values were calculated according to the following equation:

$$HV = 1.8544\frac{F}{d^2}\ (kgf/mm^2) \qquad (1)$$

where F is applied load (kgf) and d is the main diagonal of indentation (mm).

The controlled electronic universal testing machine used for pull off adhesion tests, and it is type is (WDW-200E). The bond strength is found from the simple relation:

$$UTS = L / A \qquad (2)$$

where: UTS = cohesive or adhesive strength - force per unit of surface area, L = load to failure (force), A = cross sectional area of specimen.

RESULTS AND DISCUSSIONS

Hardness is described as resistance to surface indentation of the material.

It can clearly see in the Figure1. At room temperature PMCs with NiCr coatings had enhanced high hardness this is due to the hardness of NiCr. The increase in the hardness in the composites coating with polyimide bound coat is the indication of good polyimide bonding between the composites and the NiCr top coating [9,10].

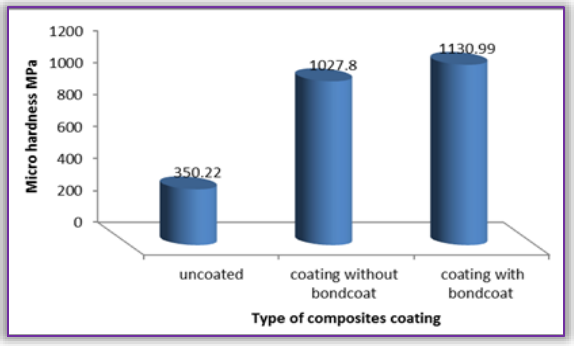

Figure1. Microhardness of coated and uncoated PMCs at room temperature

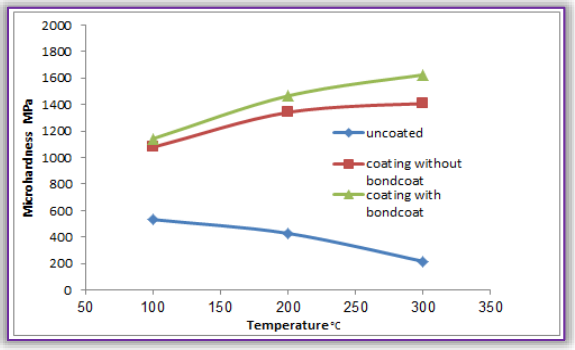

Figure 2. Microhardness of coated and uncoated PMCs as a function of temperature

In Figure 2, in general, the response of the uncoated composites to heat treatment induced softening of the microstructure and account for the reduction in hardness. Heat treatment in air generated higher average hardness values in coating systems, the coating with bound coat and coating without bound coat showed increase in micro hardness with increase temperature; this is due to the microstructural changes the physical splat structure of the coating also changes with heat treatment [11]. It is found that the degree of fusion of the particles and the presence of an oxide phase have effect on the microhardness of the coatings [7,12].

The results of pull off tests are shown in Figure3 adhesion strength for the PMCs coating with polyimide bound coat is higher than PMCs coating without bound coat. The adhesive strength between the polyimide and metal was affected by the chemical state of bonding on the surface in polyimide films; the hydrophilic bonding such as C~O bonding is believed to be suitable for enhanced adhesion between polyimide thin films and NiCr [13]. During the spray process, there is some partial formation of intermetallic phases. Subsequent fusing of the coating causes a complete transformation of the materials [14].

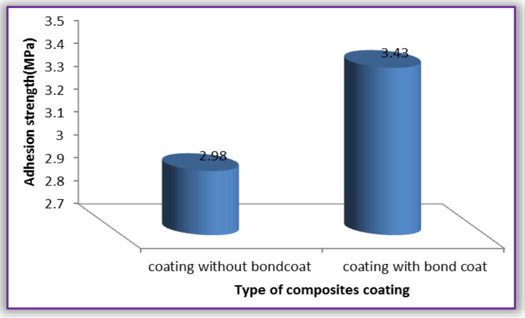

Figure 3. Adhesion strength of coated and uncoated PMCs at room temperature

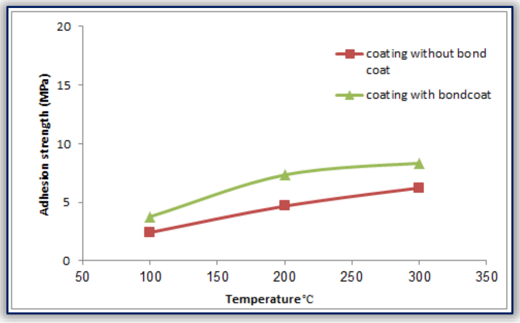

Figure 4. Adhesion strength of coated and uncoated PMCs as a function of the temperature

We can see from Figure 4. When curing temperature increase the interlocking (and then adhesion) increase because of diffusion into the substrate also occurs, improving bonding. Porosity is nearly eliminated, with no interconnecting porosity and the formation of hard oxide phases leads to increases the roughness of substrate surface [10,15].

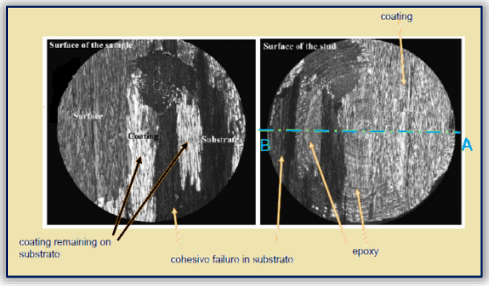

Figure 5. Microscope pictures of failed specimens showing types of failure

Figure 5 shows that all coating failed at the interface between the composites and the coating failure occurs along the weakest plane within the system, some of the coating systems that have presented fracture at the bond coat/top coat interface. In most cases there is a cohesive failure occur of the substrate [15].

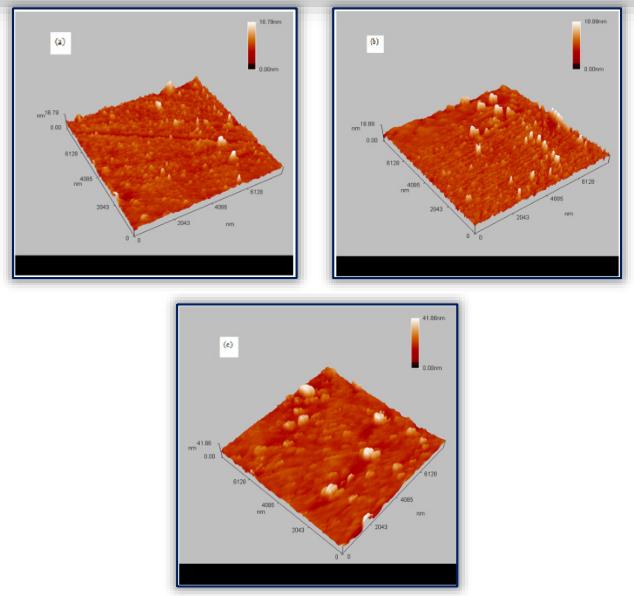

Figure 6. 3D AFM images of NiCr films with different curing temperature from (a) to (c) are 100,200 and 300°C.

Figure 6 gives 3D topography of films. For film surface, R_{max} is explained as maximum height of peak to valley for the depicted surface. Σ is the root mean square roughness. With curing temperature ranging from 100°C to 300°C, it can see the R_{max} is equal (0.8, 0.9 and 2.28) nm and σ is equal (1.16, 1.47 and 3.54) nm. When the film is cured at100°C, islands with small size are observed. However, when the film is cured at 300°C, the islands have agglomerated or coalesced to form bigger structure. The phenomenon can be explained by film growth process: during deposition process, particles are deposited and form nucleus first and then islands on substrate. This is mostly caused by atomic shadowing effects, which makes R_{max} reach 2.28 nm and σ 3.54 nm, and the film surface turns rough correspondingly as shown in Figure 6.(c).

However, when surface diffusion dominates the growing process, the coalescence of neighboring islands makes the valleys become higher and the peak become lower, consequently the surface becomes flat and R_{max} is decrease as known in Figure 6.(a). The film growth is finished by coalescence of neighboring islands. Surface morphology not only relates to film thickness but also to substrate type, works pressure, annealing and so on [14,16].

When the temperature reaches 100°C, the substrate was covered completely by spherical grains with similar radius. With an increase of temperature, the lateral grain size tends to increase. As seen from Figure 7, the lateral grain size changes from about 14.8 nm to 36.7nm when temperature ranges from 100°C to 300°C. The increase of lateral grain size with temperature is common for films [16].

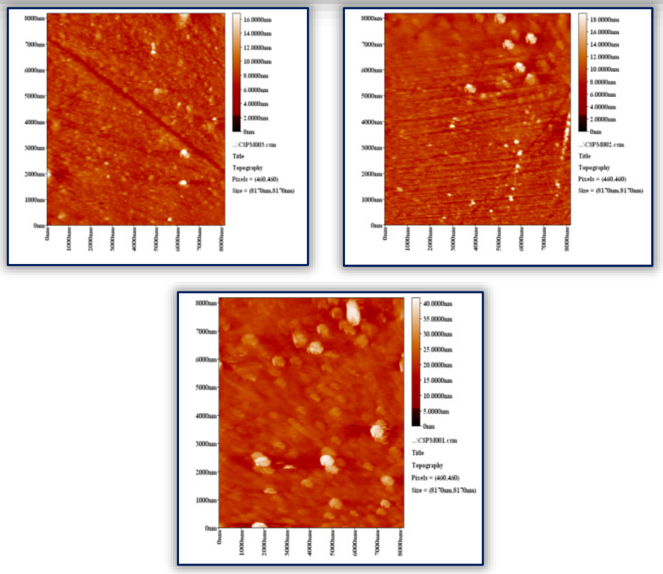

Figure 7. 2D AFM images of NiCr films with different curing temperature from (a) to (c) are 100, 200 and 300°C.

In Figure 8 an abrupt transition from the bond coating to the top coating that leads to their top coating in intimate contact with bond coating [2].

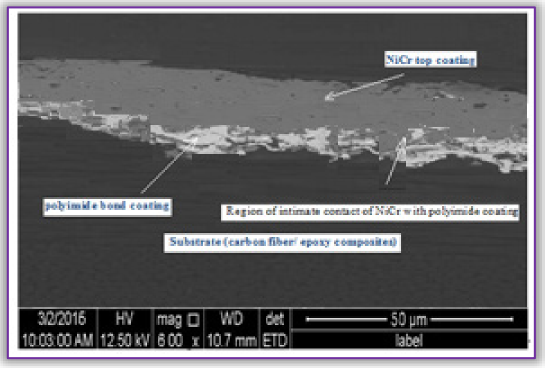

Figure 8. Microstructure of cross-section of carbon-epoxy CMPs with polyimide bond layer and NiCr top coating layer

CONCLUSIONS

This paper presents an experimental process to protect polymer matrix composite (PMCs) by metallic flame spray coating.

The results of the investigations provide useful information for applying the NiCr coating for the improvement of the hardness of PMCs. According to the results of this study, In general the coating with bound coat and coating without bound coat showed increase in micro hardness and adhesion strength with increase temperature. The adhesion strength for the PMCs coating with polyimide bound coat is higher than PMCs coating without bound coat.

The AFM analysis also provides information on the changes in the surface morphology and roughness introduced by the heat treatment. When a temperature change from 100°C to 300°C, the island structure was observed and the R_{max} increase from (0.8 to 2.28)nm, and σ increase (1.16 to 3.54)nm.

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