

Research Paper

Wear and Friction of Polymer Fiber Composites Coated by NiCr Alloy

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The use of polymer matrix composites (PMCs) 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. So the goal of this research was to develop new coatings to enhance the surface properties of PMCs. In this paper, flame sprayed NiCr graded coatings are investigated as a method to address a PMC poor resistance by providing high temperature and environmental protection to PMCs.

In this study, carbon fiber/epoxy composites are used as a substrate. A polyimide bond coating was prepared and deposited by a thermal evaporation technique onto the polymer composite substrate. The NiCr alloy was used as a top coating deposited by a flame spray technique. The coatings were spread with two configurations, a coating with a bond coat (with polyimide) and a coating without a bond coat (without polyimide).

Wear rate measurements for both coated and uncoated samples were performed by a pin-on-disc technique. The results show that PMCs with NiCr coatings had enhanced wear resistance as compared with the uncoated PMCs, and this was due to high hardness and wear resistance of the NiCr alloy. The wear rate increases much more with increasing temperature than it did for the coated PMCs at room temperature. The coated PMCs with bond coats (with polyimide in the bond coat) had lower friction coefficients than the coated PMCs without bond coats (without polyimide in the bond coat), and this was due to a significant increase in interface adhesion. The wear and friction resistance of all the coatings improved significantly with heat treatment. The worn surfaces and oxidation of the NiCr layer were observed by the scanning electron microscope (SEM) with X-ray diffraction (XRD).

Key words: protective polymer composites, protection by flame spray technique, high temperature coating, coating against wear.

1. INTRODUCTION

As is known, historically speaking, thermal (flame) spray technology has been limited to the application of metal alloys, cermets and some low-performance

polymers to form a protective coating on the substrate [1, 2–4]. One of the major advantages of flame spraying with coatings is that it creates a high-performance coating on parts that would be difficult or costly to coat by other means [5, 6]. In the aerospace industry, the applications of surface coatings on engine parts have been practiced for over 50 years [7–9].

Polymer matrix composites (PMCs) are widely used in the aerospace industry and in the military applications because of their low density, high specific strength and stiffness, and other unique properties such as ease of formation and machining [10], and good size stability. Surface coatings may make it possible to produce PMCs that are resistant to erosion wear, sliding wear and oxidation [11, 12]. Hard coatings have been especially useful in applications involving erosive and abrasive wear [13, 14].

However, by simply applying a hard metallic or ceramic coating on softer and durable, PMCs to improve erosion and abrasion resistance is not effective since coating durability may be short-lived [5]. Surface modification processes can be classified as hardening by flame, induction, laser or electron beam [16].

Major advances have been made in nickel alloy design over the past thirty years but many of the improvements in high temperature strength have been made at the expense of oxidation and hot corrosion resistance [17, 18].

Friction, corrosion and wear are the most common factors that cause engineering failures. Wear is the progressive loss of material from a surface. Among the different types of wear, abrasive wear and adhesive wear normally occur more often than other types. Abrasive wear is due to hard particles or hard protuberances forced against and moving along a solid surface [19]. One technique commonly used to improve coating adhesion and durability is the use of bond coats that are interleaved between a coating (topcoat) and a substrate with vastly different coefficients of thermal expansion. Bond coats sandwiched between high-quality flame-sprayed, wear-resistant coatings (top coats) and PMCs substrates have substantially improved the wear resistance of PMCs [20].

In this article, the thermal evaporation technique is used to deposit a polyimide bond coat onto a polymer composites substrate. The NiCr alloy as a top coat was deposited by the flame-spray technique.

2. EXPERIMENTAL ANALYSIS

A woven carbon fiber epoxy composite was selected as a substrate; the hand lay-up technique was used to prepare the composites with a volume fraction of 30%. The composite specimen was cleaned with acetone to remove moisture, dirt oil and other foreign particles.

The coating that improves the adherence of the subsequent deposited coating is called bond coat, and polyimides are used as bond coat. In this work,

pyromellitic dianhydride (PMDA) and p-phenylene diamine (PDA), which are both commercially available from Sigma-Aldrich Corporation, are used to prepare polyimide by the thermal evaporation technique. These two monomers, 2 g of each monomer, were evaporated from two separated boats to form a poly(amic acid) (PAA) thin film on the substrate. The deposition process began under a vacuum of $2 \cdot 10^{-5}$ mbar. The resultant PAA films were then soft baked to remove $n\text{H}_2\text{O}$ from the substrate followed by a thermal treatment at 250°C for 1 hour, each film, in an air circulating oven, and then deposited into the composite substrate. The final thickness of these films was $5 \pm 0.1 \mu\text{m}$.

On the other hand NiCr is used as a top coat. The elemental composition of NiCr alloy samples used in this work was studied by using the 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

Rototec 80 spray gun made in Germany by Castolin Eutectic Company, was used for thermal spraying by flame. In this process, the oxygen-acetylene mixture is passed through a nozzle and ignited to form a combustion flame.

The NiCr coating powder with particle sizes ranging from 50 to 90 μm was fed into the flame, accelerated and projected onto the substrate to form a top coating with a thickness of about $70 \pm 2 \mu\text{m}$. The flame temperature was limited to around 1400°C , and particle velocities were relatively slow.

The operating parameters during the 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 the coating, the samples were cured at (100, 200, 300) $^\circ\text{C}$.

Wear and Friction Testing. Wear tests are carried out in a pin-on-disc type wear monitoring test rig (supplied by DUCOM) as per the ASTM G 99 test method. The counter body is a disc made of hardened ground steel with a hardness of 385 HV. The specimen is held stationary and the disc is rotated, while a normal load is applied through a lever mechanism. A series of test

were conducted with sliding velocities of 12 000 cm/min under a normal loading of 20 N.

The wear rate was measured by using the weighing method, and each specimen was weighed before and after the wear testing by using sensitive digital balance with an accuracy of 1 mg. The wear rate values were calculated by using the following equations:

$$(2.1) \quad \text{Wear rate (WR)} = \frac{W}{SD} \quad [\text{g/cm}].$$

ΔW is a weight difference [g]:

$$(2.2) \quad \Delta W = W_1 - W_2,$$

where W_1 and W_2 are the weights of a specimen before and after the wear test, respectively [g].

The sliding distance SD (cm) is measured by the following equation:

$$(2.3) \quad SD = 2\pi \cdot r \cdot n \cdot t,$$

where r is radius from the center of the specimen to the center of the disc (7 cm), n is number of cycles (500 cycles/min), and t is time of sliding (10 min).

The coefficient of friction (μ) was found by dividing the friction force (F) by the applied normal load (N) according to the following equation:

$$(2.4) \quad \mu = \frac{F}{N}.$$

Frictional force is found by a specially designed steel ring taken from a calibration apparatus of TQ strain meter mounted on U-angle block fixed on the bench and aligned with the left side of the lever arm. The force was transmitted from the left side of the fulcrum lever to the ring using a 0.5 cm diameter and 3 cm long sliding rod. Two half-bridges were formed on inside and outside the ring surfaces by means of four strain gauges with gauge factor of 2.065. These strain gauges were then connected to a TQ-S6072 digital bridge strain meter. Before running the experiment, the strain micrometer digital display (cathode ray) screen was calibrated to record the microstrain reading before and after the running. The reading from the strain meter was taken and the calibration curve of load (force) versus strain was plotted at constant sliding time (20 min), sliding speed (2.7 m/s) and steel disc hardness of 35 HRc.

3. RESULTS AND DISCUSSIONS

It can be clearly seen in Table 3 that PMCs with NiCr coatings had enhanced wear resistance, and this is due to the hardness of NiCr. For the uncoated PMCs,

Table 3. Wear rate of coated and uncoated PMCs at room temperature.

Type of composites	Wear rate [g/cm]
Uncoated	$5.00 \cdot 10^{-6}$
Coated without bond coat	$8.80 \cdot 10^{-7}$
Coated with bond coat	$6.10 \cdot 10^{-7}$

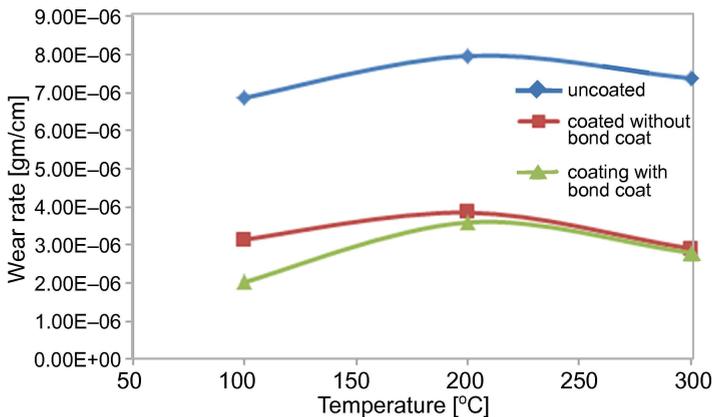
the wear rate increased much more that it did for the coated PMCs at room temperature.

For the coated PMCs, the wear occurred predominantly by ductile fracture. For the uncoated PMCs, the wear occurred predominantly by the cutting and ploughing deformation of an abrasive disk [21].

The coated PMCs without polymer content in their bond coats (without polyimide) had higher wear rates than the coated PMCs with polymer content in their bond coats (with polyimide). The use of polyimides in bond coat have become important due to their excellent thermal stability and mechanical strength, leading to functionalized interfacial bonding increases and thus wear resistance, which all accounts for the lower wear rate than in PMCs without polymer content in their bond coats at room temperature [7, 22].

When the NiCr coated samples come in contact with the environment, a formation of oxides layer takes place, and during the sliding wear this oxide film breaks and provides the lubricating effect, which in turn reduces the wear of the composites. This could be attributed to the strong mechanical bonding between the substrate surface and the coating semi-molten particles derived on a surface formerly prepared for sprayed coatings [23].

In Fig. 1, it can be seen that the wear rates for the uncoated PMCs increase with temperature increase from 100°C to 200°C. This is due to the formation of

**FIG. 1.** Wear rate of coated and uncoated PMCs as a function of temperature.

microcracks within the fiber and the matrix, leading to the weak bond strength between the fiber and the matrix. The decrease in wear rates for the uncoated PMCs at the temperature increase above 200°C may be due to a significant increase in the ductility of the epoxy matrix [5, 24].

When the temperature increases above 200°C, the wear rates for both coated PMCs decrease. This is possibly due to a slight oxidation when the temperature was around 300°C and some oxides (NiO or Cr₂O₃ and NiCr₂O₄) were formed on the surface. The presence of a lubricating oxide film reduces the tendency for adhesive wear occurrence [19, 23].

Table 4 shows that the uncoated PMCs had the highest friction coefficient in comparison to the coated PMCs at room temperature, In addition, it can be seen the coated PMCs with polymer content in their bond coats (a polyimide bond coat) had lower friction coefficients than the coated PMCs without polymer content in their bond coats (without polyimide), and this is because polyimide shows a well adhered interface between PMCs and NiCr top coat [7].

Table 4. Friction coefficient of coated and uncoated PMCs at room temperature.

Type of composites	Friction coefficient
Uncoated	1.2587
Coated without bond coat	0.6195
Coated with bond coat	0.32

Figure 2 shows a slight decrease in friction coefficient for the uncoated PMCs as the temperature increases, and this is due to the increased ductility of epoxy matrix that allows for a less plastic deformation and is prone to fracture [25].

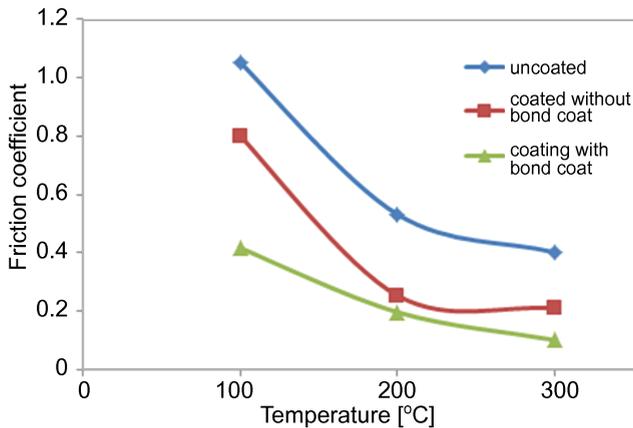


FIG. 2. Friction coefficient of coated and uncoated PMCs as a function of temperature.

For the NiCr alloy coated with and without bond coat, the friction reduction within the temperature increase may also be affected by the presence of some oxide films (NiO , Cr_2O_3 and NiCr_2O_4), which leads to solid lubricant [19, 26].

During the manufacture of the composite specimens, a matrix layer is formed on the surface that has a square type structure, see Fig. 3.

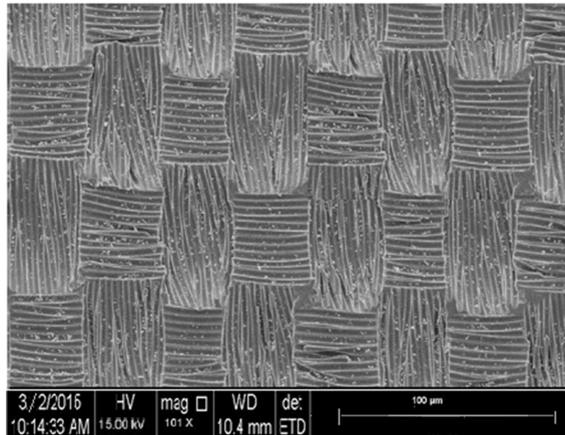


FIG. 3. Original PMCs surface.

In Figs. 4 and 5, the boundary between a worn surface and an “unworn” surface can be seen. In the images, the relative sliding direction is vertical. In Fig. 6, the mating surfaces are not perfectly flat, especially the composite surface, and the worn area will have an irregular shape. In this figure, the surface is slightly worn. But, it is far from being worn away down to the fibers. Therefore,

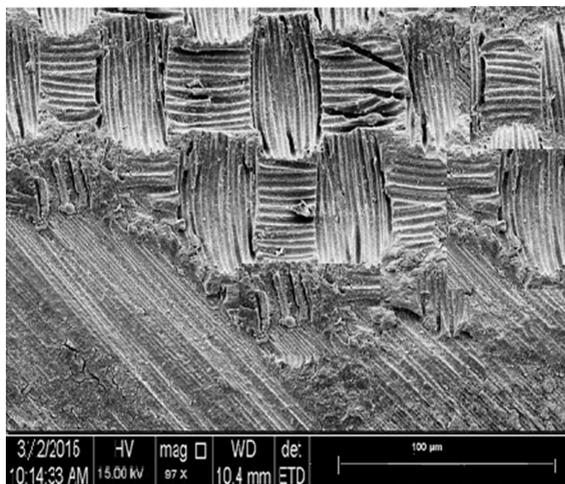


FIG. 4. Boundary between a worn original surface and fibers for PMCs.

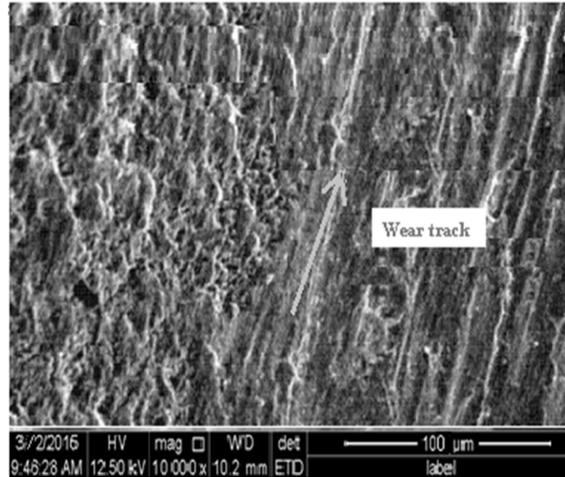


FIG. 5. SEM micrograph of the worn surface of NiCr coating.

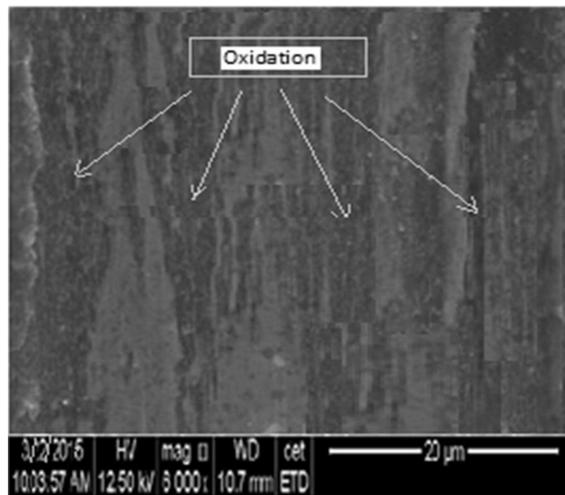


FIG. 6. Formation of oxide films on the worn surface of NiCr at high temperature (300°C).

the main wear mechanism cannot be slow wearing of the matrix. Instead, the wear of the matrix layer is probably mainly due to fragments breaking off from the boundary between the area where the entire matrix layer has been removed and where the matrix layer is fairly unworn [27].

Microscopic observations of the worn surfaces indicated that the wear was taking place mainly by adhesion mechanism. Polishing lines along the sliding direction, together with micro-ploughing traces are clearly seen in Figs. 4 and 5.

The examined metallic surface exhibited local oxidation areas verified via the XRD microanalysis as shown in Fig. 7. The formation of oxide layer on

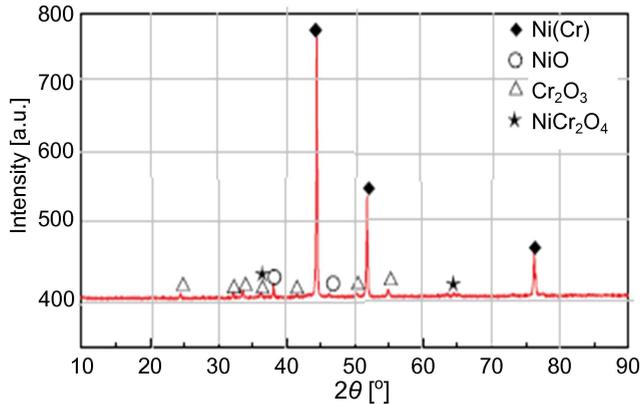


FIG. 7. XRD patterns of worn surface of NiCr at 300°C.

the surface of NiCr at 300°C is shown in Fig. 6, indicative of the activation of a tribo-oxidation mechanism [28].

Figure 7 shows the XRD patterns of the worn surface of NiCr after the wear test at 300°C. It can be seen that NiO, Cr₂O₃ and NiCr₂O₄ phases are identified on the worn surface of specimen. It is known that the oxidation of Ni and Cr results in a triboreaction film well distributed on the worn surface, and this film performs a lubricating effect [11, 29], because NiO and Cr₂O₃ have excellent plasticity and glutinosity and they are solid lubricant at high-temperature.

In Fig. 8, a cross-section of the coating system shows that the polyimide bond coating provided coating adhesion with good bonding between layer to layer and coating to substrate. Another indicator that the NiCr top coat has

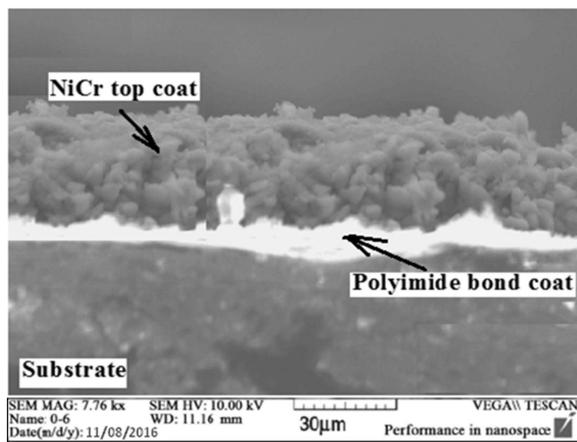


FIG. 8. SEM images of cross-section of PMCs with polyimide bond coating layer and NiCr top coating layer.

become fused with the polyimide bond coating leads to the penetrated NiCr top coating in the polyimide bond coating [13].

4. CONCLUSIONS

This paper presents an experimental process to protect PMCs by metallic flame spray coating.

The results of the presented investigations provide useful information for applying the NiCr coating to improve the wear and friction resistance of PMCs. According to the results of this study, the wear rate and friction coefficients for the uncoated PMCs were higher than for the coated PMCs at room temperature.

The coated PMCs without polymer content in their bond coats (without polyimide) had higher friction and wear rates than the coated PMCs with polymer content in their bond coats (with polyimide), because using polyimides in bond coat leads to functionalized interfacial bonding increases and thus wear resistance. The wear and friction resistance of all the coatings improved significantly with the heat treatment. Microscopic investigation of worn surfaces was done. The results of X-ray diffraction are presented and indicate the presence of oxides at the surface.

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