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The effects of solid particle erosion of ZrN coating material on GF/EP and CF/EP composites by using PVD method

CE/EP ve KE/EP kompozitler üzerine FBB metodu kullanılarak ZrN kaplama malzemesinin katı partikül erozyonuna etkileri

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CE/EP ve KE/EP Kompozitler Üzerine FBB Metodu Kullanarak ZrN Kaplama Malzemesinin Katı Partikül Erozyonuna Etkileri

Araştırma Makalesi / Research Article

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ÖZ

Fiziksel Buhar Biriktirme (FBB) metodu kullanılarak üretilen koruyucu kaplamalar sayesinde makine parçalarının çalışma ömrü arttırılabilir. Bu çalışmada da bu faydanın sağlanabilmesi için ≈ 0.25 μm kalınlığındaki ZrN kaplamalar Cam Elyaf Takviyeli Epoksi (CE/EP) ve Karbon Elyaf Takviyeli Epoksi (KE/EP) kompozitlerin üzerine Magnetron Sıçratma Yöntemi ile kaplanarak erozyon direncinin arttırılması amaçlanmıştır. Deneyler, 53 m/s çarpma hızı ile 30°, 60° ve 90° çarpma açılarında gerçekleştirilmiştir. Deneylerde 200 μm ortalama çapındaki Al_2O_3 aşındırıcı partiküller kullanılmıştır. Farklı parametrelere bağlı olarak deneyler yapılmasına rağmen tüm numuneler için literatürde sünek malzemelerde karşılaşılan durum gözlemlenmiş olup 30° çarpma açısında maksimum erozyon oranı belirlenmiştir. Erozyon mekanizmasını karakterize etmek için yüzeylerin optik mikroskop görüntüleri elde edilmiştir. Aşınma sürecinin ilerlemesinde matris ve kaplama formunun her ikisinin etkisinin de baskın bir rol oynadığı bulunmuştur. Altlık malzeme ile kaplama tabakasına ait ara yüzeydeki değişimi hassas bir şekilde açıklamak için malzeme kaldırma işlemi ile erozyon oranının değişimindeki uyuma dikkat edilmiştir.

Anahtar Kelimeler: Katı partikül erozyonu, ZrN kaplama, CE/EP, KE/EP, FBB.

The Effects of Solid Particle Erosion of ZrN Coating Material on Gf/Ep and Cf/Ep Composites by Using PVD Method

ABSTRACT

Protective coatings produced by using Physical Vapour Deposition (PVD) method can increase the life time of the components. Therefore; in this study, ≈ 0.25 μm thickness ZrN coatings are applied on Glass Fiber Reinforced Epoxy (GF/EP) and Carbon Fiber Reinforced Epoxy (CF/EP) composites by magnetron sputtering to gain an improved understanding of the erosion resistance. The impingement angles used in the tests were 30°, 60° and 90°, while the impact velocity was 53 m/s. Al_2O_3 abrasive particles with an average diameter of 200 μm was used. All test specimens regardless of their various properties exhibit maximum erosion rates at 30° impingement angle and thus exhibiting similar behavior as that observed for ductile materials. Optic microscopic views were performed on the surfaces in order to characterize the erosion mechanism. The erodent particles of the both coating layer and composite matrix were found of main role in governing the wear progression. The measured erosion rates were sensitively correlated with the material removal process in order to explain the changes within the coated interfaces.

Keywords: Solid particle erosion, ZrN coating, GF/EP, CF/EP, PVD.

1. INTRODUCTION

Solid particle erosion impresses many industries such as aerospace, power generation, coal mining, as well as in manufactured goods like pipe line in petroleum refining, helicopter rotor blades, pump impelling blades, rocket nozzles in jet engines, which caused great economic losses. The erosion wear system is characterized as material loss at surface caused by continual impact of particles having various shape, dimension, structure and

a certain velocity carried by liquid or gas ambient for composite materials.

At the studies in literature the parameters affecting erosion and erosion process are examined under two headlines as additive and coated composites. As in other tribological systems, erosion is related with operation parameters (particle velocity and flow rate, impact angle, temperature) erodent properties (shape, dimension, hardness, density), target material properties and environmental test conditions. As it is not possible to express a lot of parameters and their effects to process on a single variable, alternative additives and coating

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materials were derived in researches aiming at a modeling erosion.

The world of advanced science and technology has always been in need of materials with excellent properties for better operational performances. Parallel to such needs, the importance and industrial utilization of polymer composites have tremendously increased. The composites are extensively used as structural materials in various components and engineering parts in automobile, space craft industry, marine and energy conversion systems due to their unique specific properties. The underlying reason for this is the fact that these composite materials exhibit high strengths, possess better coating properties and are economic as well. Meanwhile, advanced scientific researches on these materials are progressively carried out.

Many researchers have been interested in the solid particle erosion behavior of metal, polymer and their composite materials worn by erodent. While fiber reinforced polymers take place in most of the studies conducted on erosion of composites, studies involving erosion on composites with coating materials can hardly be encountered. The poor erosion wear resistance of thermoplastic composites causes severe problems if the material is subjected to solid particle erosion. To overcome this problem, erosion wear resistant magnetron sputtered PVD coatings were deposited on Glass Fiber Reinforced Epoxy (GF/EP) and Carbon Fiber Reinforced Epoxy CF/EP substrates. This is due to the fact that it is not easy to clearly understand wear mechanisms of these types of composites, properties of their components and their interface interactions.

In studies made in this field, additive materials were generally developed for ductile and brittle materials and in case single abrasive particle or multiple abrasive particles track the surface.

Srivastava [1] scrutinized the effects of particle, impingement angle and particle velocity on the erosion wear behavior of E-glass fiber reinforced epoxy resin composites. In that study, the erosive wear of wheat flour powder filled composites is evaluated at different impingement angles from 30° to 90° and at three different velocities of 24, 35 and 52 m/s. Pure glass epoxy without any filler shows the highest erosion rate due to weak bonding strength. Mohan et al. [2] reviewed the effect of incorporation of tungsten carbide (WC) powders on erosive wear behavior in glass fabric-epoxy (G-E) composites. The effect of different impact velocities (40 and 80 m/s) and impact angles (30° to 90°) on the performance of the wear resistance of the composites were measured. The results of erosive wear losses, impact angle, and impact velocity and erosion rate of filled and unfilled glass-epoxy composites were analyzed and discussed. The WC filled G-E composite exhibited a lower erosion rate as compared to that of unfilled G-E composites. Yang et al. [3] investigated the erosion behavior of EB-PVD thermal barrier coatings (TBCs) with real morphology based on FE simulation. An FE

modeling method was developed to establish a geometric numerical model using a scanning electron microscope. An empirical correlation between the depth of penetration and some characteristic parameters of the erodent particle was obtained by means of simulation. The crack driving force to produce a surface crack was analyzed. The results indicate that the real morphology would be more susceptible to erosion failure compared to the results of previous theoretical or numerical estimations. Rout and Satapathy [4] described the development of multiphase hybrid composites consisting of epoxy reinforced with glass fabric and filled with rice husk particulates. This study revealed that hardness, tensile modulus, impact energy and erosion resistance of these new class hybrid composites improve with filler addition, while a steady decline in tensile and flexural properties were observed. Mahapatra and Patnaik [5] investigated the erosion wear response of multiphase hybrid composites consisting of polyester reinforced with E-glass fiber and ceramic particulates and presented a comparison of the influence of three different particulate fillers [cement by-pass dust (CBPD), alumina (Al₂O₃), and silicon carbide (SiC)] on the wear characteristics of glass-polyester composites.

Studies continued with Bağcı's [6] development of a multi-component composite system consisting of thermoplastic epoxy resin reinforced with E-glass fiber and (Al₂O₃+SiO₂) particles, and studies its erosion behavior under different operating conditions. With this target in mind, this empirical study investigates the solid particle erosion wear behavior of a new composite material made of glass fiber and epoxy as the main materials and (Al₂O₃+SiO₂) particles added into the structure at the amount of 30% [15% (Al₂O₃) + 15% (SiO₂)] of the resin used for the composite.

Shin and Hamed [7] presented the results of an experimental investigation of the effect of topcoat micro-structure on the erosion resistance of air plasma sprayed 7wt% YSZ thermal barrier coating (TBC) at high temperature. Two sets of air plasma sprayed YSZ TBCs of differing micro-structures were tested in a high temperature erosion tunnel to simulate modern gas turbine engine's operating conditions. The results demonstrate reduced erosion resistance associated with increased porosity in the topcoat layer. Maurer and Schulz [8] were analysed different 30 µm thick metallic (Ti, Al, Cr) and multilayer (Ti/TiN) coatings in order to investigate the influence of the coating hardness and micro structure regarding the erosion performance. The results indicate that the erosion process of PVD coatings on CFRP can be divided into coating and substrate driven erosion mechanisms.

In this experimental study, ≈0.25 µm thickness ZrN coatings are applied on GF/EP and CF/EP by magnetron sputtering to gain an improved understanding of the erosion resistance. All test specimens regardless of their various properties exhibit maximum erosion rates at 30° impingement angle and thus exhibiting similar behavior as that observed for ductile materials. Firstly, pure

chrome coating is applied on the GF/EP and CF/EP composites to increase the adhesion of the zirconium nitride to the surface. Moreover, the surface topography of the eroded composites was investigated by an optical microscope and a non-contact 3D digital mapping method.

2. MATERIALS AND METHODS

In this experimental study, the unidirectional CF/EP and multidirectional GF/EP composites were used as substrate materials. Fibers with diameter of 17 μm, thickness of 0.20 mm and mass per unit area of 200 g/m² arranged in a uni/multi directional location provide homogeneously distributed fibers in the matrix form. All test specimens were produced as plates by “hand lay-up” technique (110 Bar pressure, 120 °C temperature and a time of 3 top of the hour) with a thickness of 3 mm and dimensions of (1x1 m²). Then, to enable the attachment of specimens to the holder, a diamond-impregnated slitting saw was used to cut notches on the samples with size of 30×30×3 mm³ from the manufactured composite plate for the erosion tests. Substrate materials (GF/EP and CF/EP test specimen) are given before and after the coating in Figure 1.

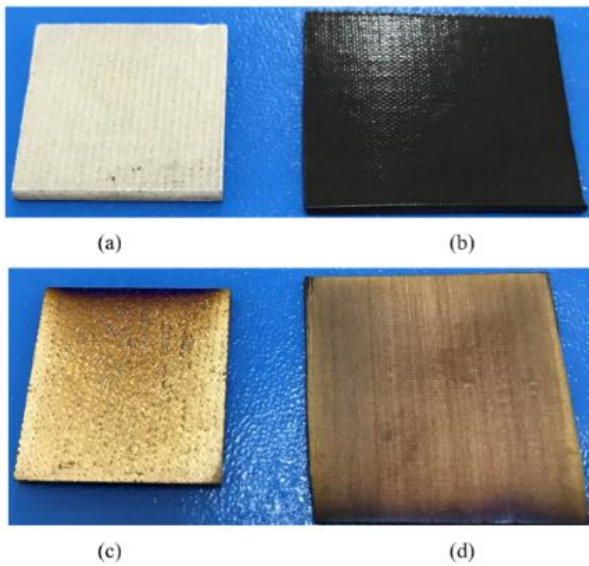


Figure 1. Substrate materials (x2); a) GF/EP (before the coating), b) CF/EP (before the coating), c) GF/EP (after the coating), d) CF/EP (after the coating).

In Table 1, mechanical and physical properties of the test specimens as per the ASTM standards {ASTM D3039/ D3039M-17 [9] and ASTM D2583-13a [10]} are given and abbreviations are used, whereas in Figure 2, SEM

view of angular Al₂O₃ erodent {density (3.94 g/cm³), hardness (9 Mohs) and melting point (1950 °C)} are shown. As the test specimens are deformed due to particle bombardments, the abrasive particles also undergo some deformations and fractures. In order to prevent this condition from affecting the tests conducted, fresh particles were used in each test.

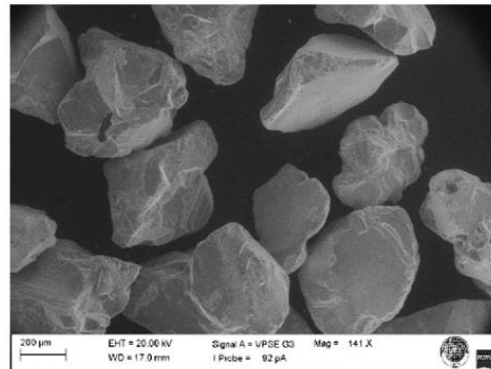


Figure 2. SEM view of angular Al₂O₃ erodent particles.

In addition to all these details which define the test specimens, as shown in Figure 3, the X-ray diffraction patterns were obtained in order to prove the presence of the glass/carbon fiber and epoxy. A sharp peak at 2θ=17.6° for glass fiber and at 2θ=26.9° for carbon fiber can be distinguished. However; a different diffraction pattern is observed for epoxy resin in which a broad peak appears around 2θ=21.5°. The observed peaks for GF, CF and EP indicate that all the layers have been sufficiently disordered.

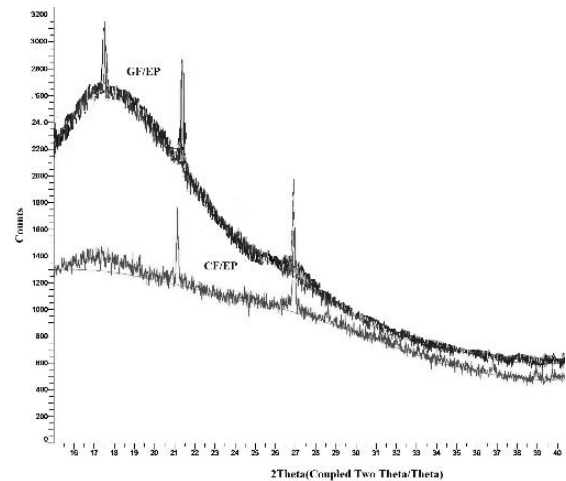


Figure 3. XRD patterns of test specimens.

The standard test process was performed in accordance with ASTM G76-95 [11] for each erosion test. Erosion

Table 1. Mechanical and physical properties of test specimens.

Material	ρ (g/cm ³)	σ _t (MPa)	H (HB)	E (MPa)
GF/EP (uncoated, A)	1.683	533	87	144
GF/EP (ZrN coated, B)	1.726	597	106	167
CF/EP (uncoated, C)	1.497	1713	133	869
CF/EP (ZrN coated, D)	1.553	1824	117	921

wear losses in the test specimen with an electronic balance on accuracy of 0.1 mg was measured. Then 40 kg of erodent particles (approximately 6 minutes of experiment time) were sputtered on the specimen and then, the latter was weighed again to determine its weight loss.

At the same time, these measurements as well as volumetric losses occurring in the test sample for detecting the digital map method of the surfaces is simulated using the point cloud and the mesh modeling. In addition, volumetric losses occurring in the test sample was determined. Surface mapping of the numerical method for three-dimensional (3D) scanning has been used on the Figure 4. Weight loss and volumetric loss changes of a similar trend has emerged in terms of the comparison of results.

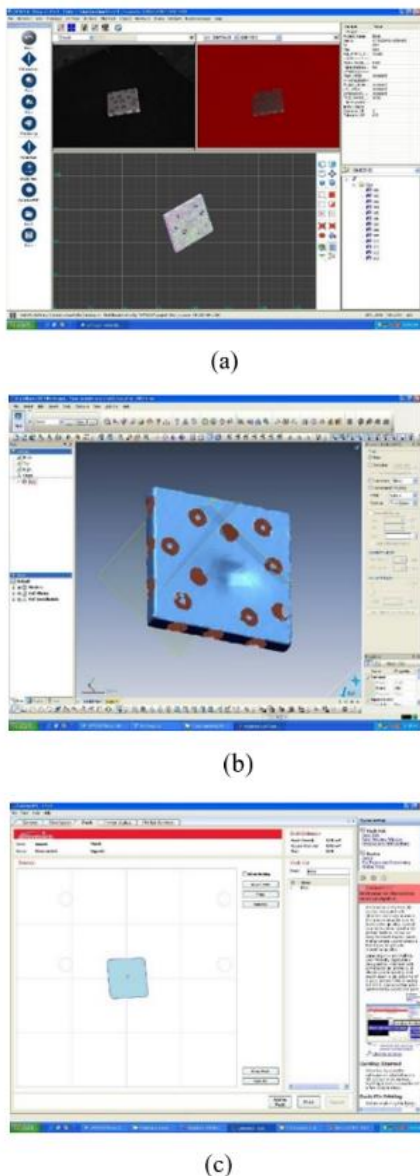


Figure 4. The digital map method of the surfaces; (a) Point cloud with OPTOCAT, (b) Mesh modeling with Rapidform XOR/Redesign, (c) Volumetric loss with CatalystEX.

There are many different ways to deposit materials such as metals, ceramics, plastics and composites onto a surface (substrate) and to form a thin film. PVD (also known as thin film deposition at temperatures between 150 and 500 °C) is an environmentally friendly vacuum coating process with no hazardous by products resulting brilliant decorative finishes with excellent wear and corrosion resistance. Common industrial coatings applied by PVD are TiN, ZrN, CrN, CrCN, TiAlN etc. one common way to do this is to use what is known as a Magnetron Sputtering System. The system has the addition of a strong magnetic field near the target area. This field causes travelling electrons to spiral along magnetic flux lines near the target instead of being attracted toward the substrate. The end result is the deposition of a thin, compact coating with the desired structure and composition (Figure 5).

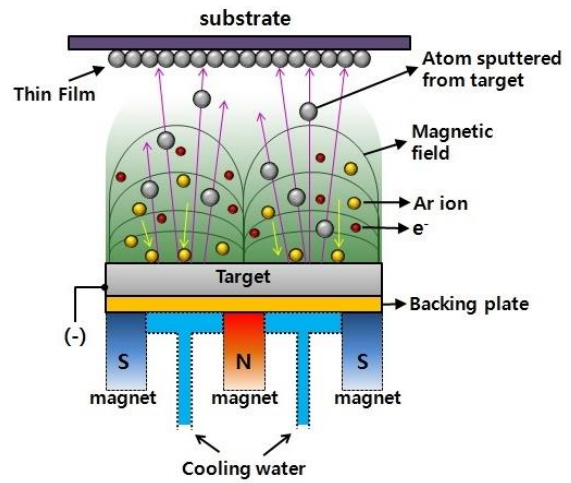


Figure 5. Schematic view of the magnetron sputtering system.

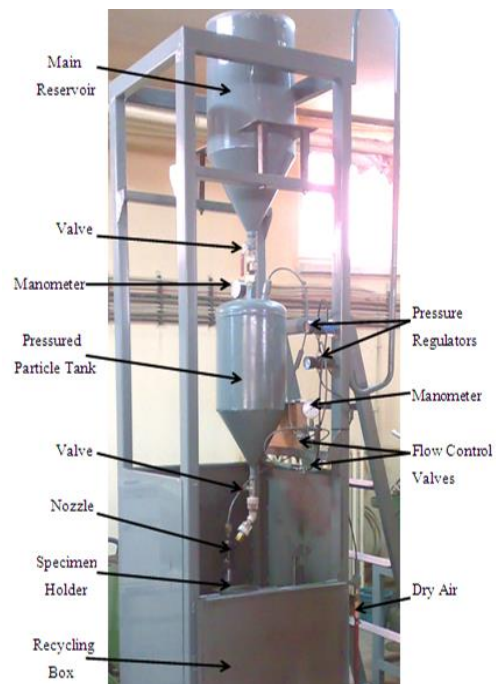


Figure 6. Detailed view of the test device.

Zirconium nitride (ZrN) is an inorganic compound used in a variety of ways due to its properties. ZrN grown by PVD is a light gold color similar to elemental gold. The hardness of single-crystal ZrN is 22.7 ± 1.7 GPa, elastic modulus is 450 GPa and density is 7.09 g/cm^3 . Zirconium nitride is a hard ceramic material similar to titanium nitride and is a cement-like refractory material. Thus it is used in refractories, cermets and laboratory crucibles. When applied using the physical vapor deposition coating process it is commonly used for coating medical devices, industrial parts, automotive and aerospace components and other parts subject to high wear and corrosive environments.

The testing device used in this scientific work are detailed in Figure 6. The impact velocity of the particles can be varied by varying the pressure of the compressed air. In order to determine the velocity of the eroding particles, the most common method [12] was used previously. The particles impact velocities used in the experimental tests (53 m/s) were adjusted by using the double disc method (Figure 7) in which two discs were connected to a common shaft from a driving prime mover.

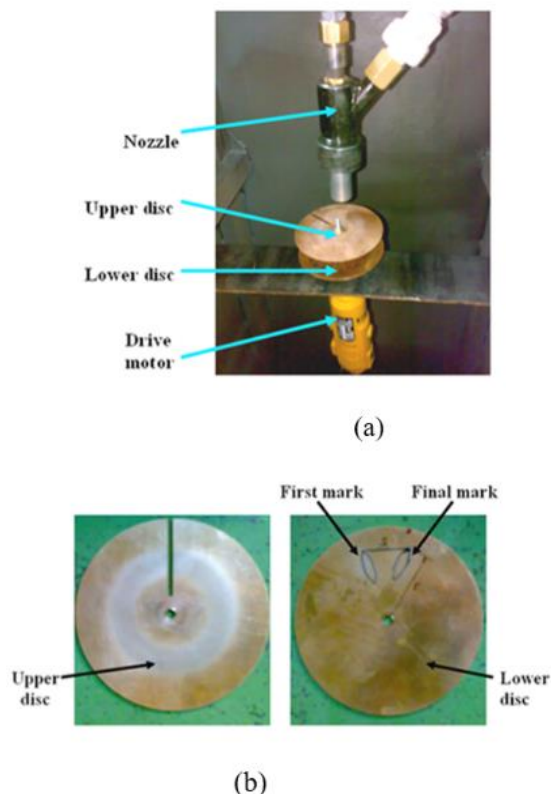


Figure 7. (a) Phosphorous bronze discs with drive motor used in the determination of particle velocity in the double disc method and (b) erosion marks formed on the discs.

3. RESULTS AND DISCUSSION

To ensure a sufficient lifetime of GF/CF epoxy components which are exposed to solid particle erosion, protective coatings are needed. Metallic coatings on

composites are mainly produced by electro plating, thermal spraying, or PVD. When deposited on GF/CF, electro plated coatings require extensive pre-treatment processes to create conductivity and to ensure adhesion. Coatings produced by thermal spraying can increase the surface roughness and an additional post-treatment may be needed to guarantee adequate flow conditions at the surface. In contrast, simple processing can be realized by PVD methods.

The graph in Figure 8 shows variations of erosion rates with test specimens (ZrN coated and uncoated composites) at impingement angles of 30° , 60° and 90° with an impact velocity of 53 m/s where the slightly rounded and irregular Al_2O_3 abrasive particles used had average diameters of $200 \mu\text{m}$. When the graph is examined it is seen that regardless of the fact the graph is at different materials, the specimens in both sets of graphs seem to undergo much erosion rates at impingement angle of 30° . Together with this, it has also been observed that, parallel to the increase of the impingement angles (60° – 90°), the erosion rates tend to reduce abruptly. This situation shows that, a similar erosion trend is observed as that seen in literature for ductile materials [13-17]. It was determined that beside the remarkable effects of ZrN coating material also have considerable effects on solid particle erosion wear. By studying the graph plotted from the experimental result data, it is seen that the effects of the GF/EP composites with ZrN coated on erosion wear are more evident than those of the CF/EP composites with ZrN coated.

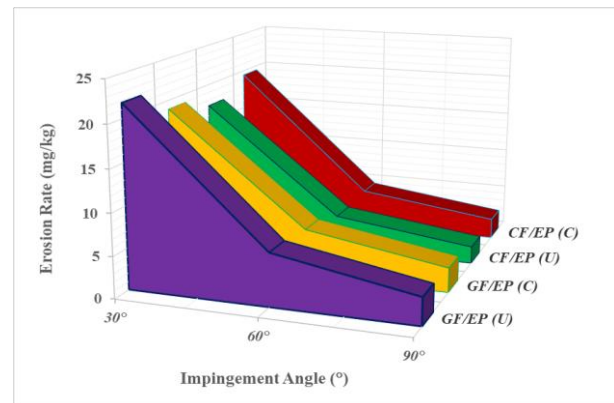


Figure 8. Variations of impingement angle & erosion rate of ZrN coated and uncoated test specimens.

When the results and optical microscope views based on the tests done are studied, it is seen that GF/EP and CF/EP of the ZrN coating material have had positive and negative effects respectively on erosion. This condition though can be perfectly recognized from the results, the microscope views also indicate extensive surface resistivity for the specimens with GF/EP. ZrN coating material adhered to the GF/EP substrate surface and reacted to the attack of abrasive particles. But CF/EP composites could not match with ZrN coating materials and the surface resistance is weakened (Figure 9).

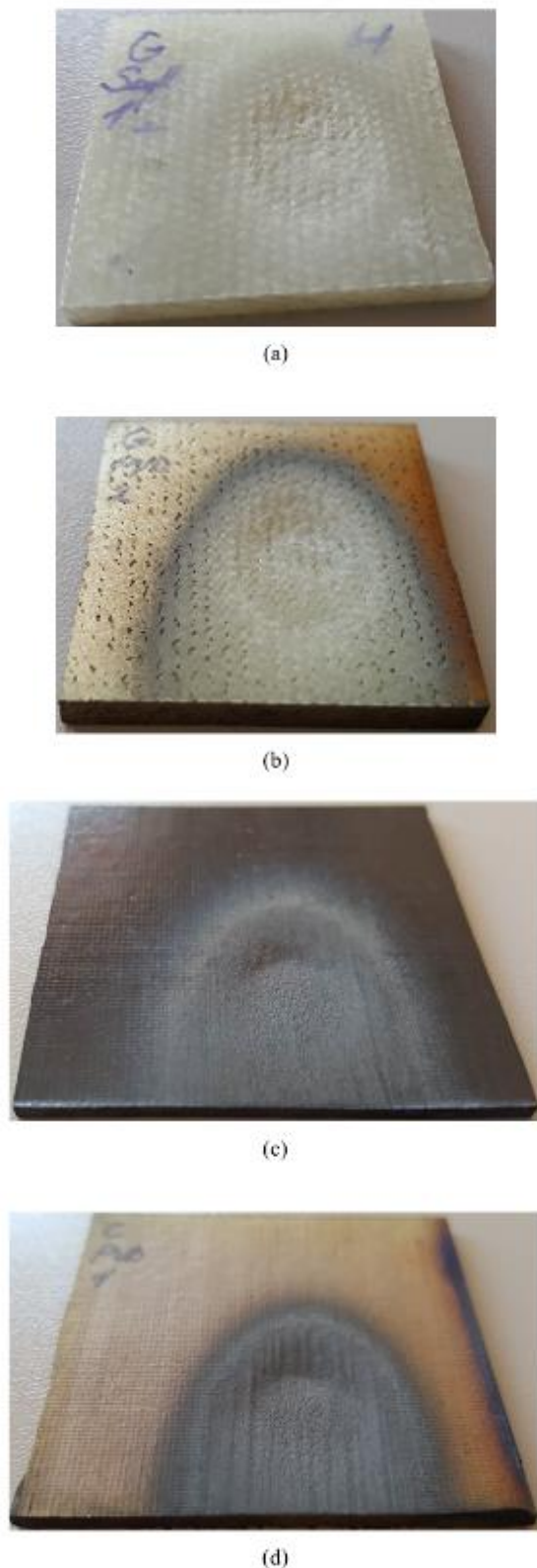


Figure 9. Optical microscope view of test specimens (x2); (a) A, (b) B, (c) C and (d) D.

4. CONCLUSIONS

It was found that material removal from the surfaces of the test specimens as a result of solid particle erosion

wear took place at several different stages. First of all, micro cracking appeared on the surfaces as the particles stroked the surface and as the particle bombardments continued, material delamination took place over the affected areas. The structural bonds got damaged as the particles kept striking the surfaces. Due to this damage, the rate of material delamination off the surfaces increased. Consequently; the cracks and grooves on the surfaces became more vivid. It has been observed that the application of coating to the materials caused the differentiation of these processes.

According to the experimental results,

ZrN coated and uncoated composites exhibit maximum erosion rates at 30° impingement angle and thus exhibiting similar behaviour as that observed for ductile materials. Inclusion of ZrN coating material in GF/EP composites considerably increases the value of hardness, tensile strength, modulus of elasticity and density. The erosion rate of ZrN coated GF/EP gives the lower value as it restricts surface delamination. GF/EP composites without any coated show the upper erosion rate due to weak bonding strength. In CF/EP composites, it decreased the erosion resistance due to the thermal and physical effects applied to the surface. The difference in erosion of GF/EP and CF/EP materials should be even higher. The difference in fiber orientations has been influential in this case. Experience shows that every tribological system can be optimised by selecting the right coating. As a future study, inclusion of new composites using different coating material and concentration combinations can be taken into consideration and the resulting experimental findings can be similarly analyzed.

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