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# WEAR AND CAVITATION EFFECT IN AN EPOXY FILLED WITH BORON AND SILICON NANOCARBIDES

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**Abstract:** Cavitation is a complex process that includes the steps of nucleation, growth, coalescence, collapse and successive rebound of bubbles and/or clusters of vapour and/or gas in a liquid when varying its thermodynamic and hydrodynamic conditions during short periods of time. When cavitation occurs close to the surface of a solid, it causes localized damage due to the high impact pressures that exceed the yield strength of the material and/or as a consequence of the fluctuating stresses that promote surface fatigue. In many systems like hydraulic equipment, fluid pump impellers, ship propellers and steam turbines, etc. mechanical erosion can be a great problem. For this reason, a growing interest in polymer coatings for tribological applications has emerged in the last years, being epoxy resins are good candidates. This work focused on the analysis of surface damage of nanocomposites as epoxy-based coating. The epoxy resin used was a commercial resin, the fillers were boron and silicon carbides with particle size of 60 and 100 nm respectively, and they were used in two percentage (6 and 12 wt.%). Pin on disk test were carried out to evaluate wear. Cavitation test were performed on aluminium specimens coated with the nanoparticles/epoxy composites, to evaluate their effectiveness as wear-resistance coating. Wear tracks and cavitation samples were studied by Scanning Electron Microscopy (SEM). The results showed higher wear resistance of nanocomposites than clear epoxy resin, and also better cavitation resistance.

Keywords: Wear; Cavitation, Nanocomposites, Coatings.

#### 1. INTRODUCTION

In many systems like hydraulic equipment, fluid pump impellers, ship propellers, hydrodynamic bearings, fluid seals, inlets to heat-exchanger tubes, diesel engine wet cylinder liners, hydrofoils, liquid metal power plants, steam turbines, etc., mechanical erosion could be a great problem, induced by the pulse pressure exerted by the collapse of vapour bubbles, causing surface damage [1,2]. Such process of the formation of vapour bubbles in low pressure regions within a fluid (when the hydrostatic pressure acquires a value lower than the saturation vapour pressure) is called cavitation. The mechanical erosion brought about by the cavitation is called cavitation erosion [3]. A great variety of materials and many different surface engineering processes have been applied to increase the cavitation resistance [1,3,4]. Many metallic and intermetallic compounds have been used as coatings due to their good cavitation erosion resistance by their high work hardening rate, high hardness, good oxidation and corrosion resistance [1]; also, many ceramics compounds have been used by their excellent wear resistance and high hardness and they are considered to be potencial candidates for applications needing high resistance to cavitation erosion [4]. Some authors [5,6] have provided useful data related to erosion rates for many materials, including polymers, but there is still a lack of specific information related to polymer coatings for potential use in tribological applications. Epoxy resins have shown been good results when used as coating on stainless steels by Garcia et al. [7].

The aim of this work is the use of nanocomposites as coatings with high resistance to wear and cavitation erosion. An epoxy resin filled with nanocarbides has been used by the high hardness of carbides. Silicon and boron carbides are widely used for abrasion applications. In other works composites with microparticles of silicon and boron carbides have been studied under wear conditions [8,9], showing problems due to lack of anchorage lack between particles and resin. However the composite with nano SiC showed better wear resistance that unreinforced resin [8].

#### 2 EXPERIMENTAL PROCEDURE

# 2.1 Materials and fabrication of nano-composites

The used epoxy was a bi-component, with Epofer Ex 401 resin and Epofer E432 hardener (Feroca Composites, Madrid, Spain). It has low viscosity (1300 cps) at 25 °C, and it cures at room temperature for 24 h. The resin-hardener mass ratio is 100:32.



Fig. 1 Carbides used micrographs: (Left) B<sub>4</sub>C; (Right) SiC.

The nanoparticles used in this work were: nano SiC (Bioker Research S.L., Oviedo, Spain) with average particle size 80-120 nm (Fig. 1 right), and nano  $B_4C$  (PlasmaChem GmbH Berlin, Germany) with average particle size 30-60 nm and its morphology shown in Fig. 1 left. The nanoparticles were added in two different percentages 6 and 12 wt.%. Among the methods used to disperse nanoparticles in a polymer, an extrusion method was selected in this work, with a previous deaeration of the resin. The mixture was poured into silicon moulds with shapes adequate for the different tests.

In the text and figures, the nomenclature will be: E (for the clear resin, Epofer), ESC (for nanocomposite with nano silicon carbide), EBC (for nanocomposite with nano boron carbide), and including 6 or 12 according to filler amount.

# 2.2 Wear test

Dry wear tests were carried out at room temperature using a pin-on-disk tribometer (Microtest, Madrid, Spain). A 6 mm diameter alumina ball was used for the pin. Test conditions were a speed of 120 rpm, with an applied load of 15 N, relative humidity below 30% and friction radius was 8 mm. The sliding distance was 1000 m.

For the wear test the bulk composites were used. The wear tracks were studied by scanning electron microscopy (SEM) Philips X-30, Philips Electronic Instruments, Mahwah, NJ, USA), in order to determine the mechanism of wear.

The wear resistance was calculated by weight loss according to ASTM G99 standard. Also coefficient of friction was measured following the same standard.

# 2.3 Cavitation erosion test

For cavitation tests, clear resin and nano-composites were used in form of coatings. Aluminium tips were cleaned with MEK (methylethylketone), and silanized with  $\gamma$ -GPS (3-Glicidoxypropyl-trimetoxysilane, Sigma Aldrich, Saint Louis, USA) to improve coating adhesion, and finally coatings were casted on them. The 1% solution of silane was hydrolysed for one hour in MilliQ water at pH 5 and dried in oven for 1 h at 100 °C.

The cavitation tests were done according the ASTM G32 standard, with a Digital sonifier model S-450 (Branson Ultrasonic, Dambury, USA). The vibratory apparatus used for these tests produce axial oscillations of a test specimen inserted to a specified depth in the test liquid (water in the present experiments).

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The conditions of cavitation test were a frequency of 20 kHz and amplitude of 25 microns. The tests were carried out in intervals of 5 min. Every 5 min the tips were dried and weighed, to determine the cumulative mass loss and the erosion rate. The tests were carried out until the stabilization the mass loss. As-manufactured samples were used for cavitation tests, as flat surfaces were obtained from casting. It is well known that roughness influences erosion cavitation [10], and processed surfaces have less roughness than the one obtained if samples were sanded.

Surfaces after cavitation test were evaluated by SEM. Before the analysis, the samples were gold coated using a sputtering system.

#### 3 RESULTS

### 3.1 Wear

In the wear test, nanocomposites have higher wear resistance than clear resin, except for E12SC (Fig. 2). E6SC has the highest wear resistance. The EBCs present similar wear resistance than clear resin for both amounts of nanoparticles. Friction coefficients are also observed in Fig. 2. Only 6% nanocomposites have friction coefficients lower than clear resin.



Fig. 2 Wear resistance and friction coefficient for clear resin and its nanocomposites.



(E)

This behaviour can be explained in terms of wear mechanisms, as anchoring between nanoparticles and resin plays a very important role. In general wear tracks present abrasive wear, as it is observed in Fig. 3, where abrasion lines are found. These abrasion lines are defined more clearly in clear resin and nanocomposites with 12% of particles, which derives in high friction coefficients. Small cracks (produced by fatigue) are also found in clear resin and 12%-addition nanocomposites (Fig. 3 A, D and E). However in nanocomposites with 6% of filler, adhesive wear also takes place. Therefore they have low wear and friction coefficient (Fig. 2). The difference found between E12SC and E12BC is due to anchoring of nanoparticles (Fig. 3 D and E). The B<sub>4</sub>C nanoparticles have some hydroxyls groups in their surface; these groups come from the manufacturing process. The OH groups can join better to the clear resin and they can not get loose so easily, as occurs in E12SC. In E12SC the nanoparticles can exert as third body and increase abrasive wear (Fig. 3 D).

# 3.2 Cavitation

Fig. 4 shows mass loss rate produced in cavitation tests for clear resin and the nanocomposites. Clear resin (E) shows a higher mass loss rate than the nanocomposites. Fig. 5, plotting cumulative mass loss vs time for materials, clearly shows that mass loss is higher for clear resin than for nanocomposites. Maximum values of mass loss are delayed for the clear resin.



Fig. 4 Mass loss rate obtained by cavitation test for clear resin and nanocomposites.



Fig. 5 Cumulative mass loss obtained by cavitation test for clear resin and nanocomposites.

The solid material absorbs the impact energy as elastic deformation, plastic deformation or fracture; the last two processes lead to the erosion of material. The more elastic or plastic deformation energy a material can absorb, the higher its resistance to cavitation erosion. Erosion is generally associated with mass loss of the surface, and it takes place after an incubation time. During this time, the materials are deformed elastically or plastically [11]. According to ASTM 32 standard, maximum and terminal erosion rate and incubation time (Table 1) can be calculated from Fig. 5.

Table 1 shows that maximum and terminal erosion rate in nanocomposites are smaller than in the clear resin. Among nanocomposites, slight differences are also found. In ESC, maximum and terminal erosion rates are similar (their values are inside measurement error,  $\pm 0.02$ ); while in EBC maximum erosion is lower than in E12BC. This can be explained because of the lower density of E12BC than that of E6BC (Table 1), as porosity has a considerable influence on incubation time and on rate of erosion [12]. The incubation time is similar for all materials (clear resin and nanocomposites). The slight differences found can be due to measurement error ( $\pm 1$  min). Anyway, these times are very low when they are compared to metals, but the easy replacement of these coatings against metal repair allows its industrial use.

Mean depth of erosion (MDE) is obtained from mass loss, density and sample area by Eq. 1. The sample area was 2.778 cm<sup>2</sup> and density was calculated measuring volume and mass (Table 1). MDE is according to the data of mass loss, so depth of erosion is lower for nanocomposites than for clear resin. In EBC, MDE values lower than in ESC are found, being measurement error ±5  $\mu$ m. Moreover the higher depth always corresponds to nanocomposites with 12% load.

$$MDE = \frac{\text{Mass loss}}{\text{Area * density}} \tag{1}$$

Materials	Maximum erosion rate (mg/min)	Terminal erosion rate (mg/min)	Incubation time (min)	Density (g/cm³)	Relative density (%)	Mean depth of erosion at 60 min (μm)
E	2.11	0.26	7.3	1.07	99.07	222
E6SC	0.59	0.16	6.1	1.10	97.35	65.5
E6BC	0.39	0.08	5.8	1.10	98.21	54.7
E12SC	0.56	0.18	4.7	1.11	94.87	69.5
E12BC	0.48	0.06	6.4	1.12	96.55	59.2

**Table 1** Maximum and finish erosion rate, incubation time, density and mean depth of erosion at 60 min for<br/>clear resin and nanocomposites.

The micrographs of cavitation samples show generalized cavitation in clear resin (Fig. 6 A); however in nanocomposites the cavitation is more localized in areas with defects, as porosity. This porosity is according with density lower than clear resin. In all nanocomposites, it is observed an outer ring without cavitation (Fig. 6 B, C, D and E) and also interior areas without cavitation (Fig. 6 B).



#### 4 CONCLUSIONS

In this study it has been proved possible to use epoxy matrix nanocomposites as coatings against cavitation erosion. The percentage of load (filler) and its anchoring with matrix and a good mixture are key aspect to increase the resistance to cavitation and erosion wear.

Nanocomposites with 6% fillers present better behaviour than nanocomposites with 12%. Between E6BC and E6SC, the results have demonstrated better properties in the E6BC, for its best anchoring with the matrix.

The manufacturing of these nanocomposites as paints could allow fast repairs industrially.

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