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Cavitation resistance of epoxy-based multilayer coatings: Surface damage and crack growth kinetics during the incubation stage

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ABSTRACT

Four epoxy-based multilayer coating systems, with thicknesses of 380 ± 20 μm, 650 ± 10 μm, 720 ± 30 μm and 920 ± 20 μm, were applied manually onto stainless steel samples and subjected to vibratory cavitation tests according to ASTM G32-09 standard. In order to correlate the cavitation resistance of the coating systems with some of their mechanical properties, instrumented micro indentation tests were performed to determine hardness, resilience, total plastic work, among others, as a function of the thickness of the coatings. Examination of the surfaces by Scanning Electron Microscopy (SEM) revealed that the surface damage in all the coatings was caused by incubation and growth of cracks. Statistical analysis of crack growth data allowed determining a behavior law characteristic for each coating system, which was adjusted with proper parameters related to the mechanical properties measured by micro indentation. In particular, a good correlation was obtained among cavitation resistance, coating thickness and hardness-to-Young modulus ratio *H*/*E*.

1. Introduction

Cavitation is a complex process that includes the steps of nucleation, growth, coalescence, collapse and successive rebound of bubbles and/or clusters of vapor and/or gas in a liquid when varying its thermodynamic and hydrodynamic conditions during short periods of time. Although this phenomenon has been studied for over a century it is not yet completely understood since it involves multiple variables and parameters related to the fluid, the surface and the testing technique [1–4]. 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 [4,5].

The surface damage mechanisms that take place during cavitation are divided into two main types, namely microjet impact and shock wave formation [6–8]. A complete agreement among researchers regarding the relative importance of each of these two mechanisms has not been achieved, although it is commonly accepted that when the bubble and/or cluster of bubbles collapse on or close to the solid surface both mechanisms are present, causing a synergistic effect [7–9]. Several authors have found that, when acting separately, the damaging effect of microjets and shock waves can be reasonably predicted, but due to surface's anisotropy and inevitable variations of pressure and velocity within the fluid the size and shape of the affected areas vary significantly, so comprehensive models for cavitation damage are still to be developed [8,10,11]. Similarly, it has not been possible to establish a general model to predict cavitation damage that can be applied to different families of materials, due to the fact that each material exhibits a characteristic behavior and the pressure distribution on the surface varies with position and time.

Besides the experimental setups available in flow systems and vibratory equipment for cavitation accelerated testing, other methods and techniques to create appropriate conditions for cavitation were also used in the laboratory as well as in the field, but unfortunately the results of the different tests are not fully comparable to each other [12–14].

Experimental correlations between mechanical properties and cavitation resistance measured in terms of mass and/or volume loss when the material has completed its incubation period have been previously proposed by several researchers [5,12,15]. A number of mechanical properties such as hardness, yield strength and ultimate tensile strength have been measured, in most cases, by techniques traditionally developed for bulk material testing [15–17] and eventually through surface characterization techniques such as instrumented indentation, being a number of metals [18,19] and some hard coatings [20,21] the most common objects of study. However, the correlations between mechanical properties

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and cavitation resistance are not straightforward and experimental data typically exhibit large dispersions.

During the incubation period, there are different stages related to strain hardening, nucleation and propagation of cracks near the surface, surface fracture processes with hardly noticeable mass loss, among others. According to Haosheng et al. [22], the damage of the material is due to both the plastic energy absorbed and a portion of the elastic energy that propagates through the solid and contributes to the nucleation and propagation of cracks and defects. This causes weakening of the material and facilitates its plastic deformation process. In other words, when the material completes its hardening stage it undergoes a period in which many defects require low energy to form and propagate. During this weakening stage the elastic energy plays an important role, before the development of fatigue mechanisms that occur in the final stage of the incubation period.

Because of the need for research on the phenomena involved during the incubation stage in cavitation processes, as well as the growing interest in polymer coatings for tribological applications, this work focused on the analysis of surface damage of four epoxy-based multilayer coating systems submitted to cavitation. The coatings were applied manually onto stainless steel samples and subjected to vibratory cavitation tests according to ASTMG32-09. The statistical analysis of crack growth data allowed determining a law of behavior characteristic for each coating system, which was adjusted with proper parameters related to the mechanical properties measured by instrumented micro indentation.

2. Materials and methods

The polymer coatings were applied manually with synthetic brush. This process was carried out by applying successive layers to obtain different thicknesses on a substrate of ASTM A743 grade CA6NM martensitic stainless steel and to avoid stress concentration due to shrinkage during polymer curing as well. The mechanical properties of the coatings were measured by instrumented micro indentation technique and the final thickness of the coating systems was measured by Scanning Electron Microscopy (SEM). The coatings were tested in vibratory cavitation for several periods of time, always within the incubation stage. The worn surfaces were observed and analyzed also with the aid of SEM and digital image processing was carried out to measure the length of cracks formed as a consequence of cavitation.

2.1. Preparation of coating systems

2.1.1. Stainless steel substrates

The surfaces of the 75 mm × 25 mm × 3 mm stainless steel substrate samples were subjected to the following procedure:

- A first finishing process was performed with abrasive wheel in order to ensure homogeneous and topographically flatter surfaces (free of defects, oxides, higher roughness and large ripples).
- A second mechanical finishing process was performed with motor-tool using sand paper abrasive discs (ASTM 120 grit) at 20,000 rpm.
- Then, the surfaces of the samples were degreased with acetone for 10 min in ultrasonic device.

2.1.2. Application of coatings

All samples were multilayer polymeric coating systems consisting of two kinds of resin. An epoxy-phenolic (EPF) resin without reinforcements was applied directly onto the surface of the stainless steel substrate to ensure adhesion. Then, an epoxy resin (EP) without reinforcements was applied in several layers onto the EPF resin. This kind of coating system was selected because it presented the best adhesion, porosity and wear resistance properties when compared to other systems consisting of polyurea, polyurethane and phenolic coatings with and without inorganic reinforcements, which were previously tested [23]. The polymeric resin system (EPF+EP) was prepared as follows:

Epoxyphenolic resin (EPF) without reinforcements: the (EPF) resin required a chemically compatible liquid catalyst, which was mixed in a proportion of 6:1 by weight and the induction time was 15 min to ensure the initiation of the chemical reaction. Then, this resin was applied manually in two layers with synthetic brush on the stainless steel substrate. The average film thickness was between 100 and 130 μm. The waiting time between applications of the layers was 90 min with the purpose of ensuring adhesion to the surface substrate as well as adhesion between layers of the (EP) resin.

Epoxy resin (EP) without reinforcements: Its preparation required the use of a liquid epoxy catalyst that was mixed at a ratio of (3.8:1) by weight. The induction time was also 15 min. Then, dissolution of the catalyzed product was performed with a solvent adjuster in a ratio of (4.8:1) by weight, which helped control the formation, growth and size of the bubbles in the liquid mixture and the surface porosity between layers of multilayer epoxy coating system. The waiting time between applications of the layers was 10 min. The average thickness obtained for each layer of the multilayer epoxy system was between 120 and 160 μm.

The samples analyzed in this study were classified into three groups according to the distribution of coating thickness obtained as follows: low thickness coatings with 380 ± 20 μm; medium thickness coatings with 650 ± 10 μm and 720 ± 30 μm and high thickness coatings with 920 ± 30 μm. The thickness of each system was measured with the aid of a portable ultrasonic Coating Thickness Gauge specially designed for measuring the thickness of non-metallic coatings applied onto metallic surfaces (reference Elcometer 456) and through SEM observation (JEOL 5910LV).

2.2. Instrumented micro indentation tests

A number of mechanical properties of the coating systems were measured by instrumented micro indentation tests. This technique involves the controlled application of a load using a Vickers micro indenter according with DIN 50359-1, during a full loading-unloading cycle, recording the indenter penetration depth during the cycle [24–26]. A Zwick/Roell instrumented micro indenter equipped with an optical microscope and a built-in CCD camera was used. The operation parameters used were as follows:

- Maximum load: 5 N
- Speed of application and removal of load: 1 N/s
- Creep time at maximum load: 10 s
- Type of micro indenter: Vickers standard, according with DIN 50359-1

The mechanical properties and some of the elastic–plastic characteristics reported by the micro indentation instrumented testing are summarized below:

- Microhardness: (H), in N/mm^2 (MPa)
- Young's modulus: (E), in kN/mm^2 (GPa)
- Indentation elastic work: (W_{Elast}), in N-mm (mJ)
- Indentation plastic work: (W_{plast}), in N-mm (mJ)
- Indentation total work: ($W_{Total}=W_{Elast}+W_{Plast}$), in N-mm (mJ)