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The role of droplets on the cavitation erosion damage of TiN coatings produced with cathodic arc physical vapor deposition

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ABSTRACT

In this study, the role of droplet-related defects on the initiation and propagation of cavitation erosion damage of TiN coatings produced by Cathodic Arc Physical Vapor Deposition (CA-PVD) was investigated. Ultrasonic-aided cavitation erosion tests were conducted in distilled water, using a specially designed specimen holder. By using this holder, following of the defect-related damages on the same region of the sample became possible by scanning electron microscopy (SEM) during a total test duration of 12 h. Focused ion beam (FIB) investigations were also carried out on cross-sections of some selected defects to understand the damage mechanisms. Results of the study revealed the important role of droplet-related defects on the cavitation erosion damage. Deep cavities formed by detaching of conical droplets were determined as the most detrimental type of defects. At these sites, large impact craters were formed with sizes extending to 100 μm with a substrate-reached area at their center after 3.5 h of cavitation erosion test. However, the damage created by buried droplets was very limited when compared to the cavities. Their role on the initiation and propagation of the cavitation damage depends on their shape and position in the coating. Conical droplets embedded in the coating resulted in the capping of a coating layer above them. These damages were not deepened or extended during the test duration. On the other hand, wide droplets with a flat top that sat on the substrate did not result in the formation of neither capping of the coating nor impact craters after 12 h of testing.

1. Introduction

Cavitation is a dynamic phenomenon that generally appears in fluid systems as a result of fast formation and collapse of vapor bubbles in liquids due to the strong pressure fluctuations. Cavitation bubbles collapse violently, emitting micro-jets and pressure waves featuring high velocities, pressures and temperatures. The amplitude of pressure pulses may exceed 1 GPa within an impact time duration of 1 ns-μs. The size of the impact area created by the collapsing bubbles is in the range of few μm^2. Accordingly, if the collapse of bubbles happens close to a solid surface, the material surface will go through a local dissipation of a large density of energy. The gradual degradation of materials under cyclic impact of imploding cavitation bubbles is called cavitation erosion, which exhibits fatigue characteristics [1,2]. This phenomenon is one of the most drastic types of damage that affects the efficiency and causes undesirable problems in almost all fluid systems and hydraulic machinery, such as feed water pumps, propellers of ships, hydraulic turbine blades, diesel engines, fuel injectors as well as valves of piping in the industrial applications.

Bulk materials or coatings with high hardness and Young's modulus, fatigue strength, homogeneous and fine-grained structures are highly resistant to cavitation [3–7]. Since cavitation erosion takes place at the liquid/solid interface, surface properties of the materials are more relevant than the bulk properties. Therefore, coating of the surface with an appropriate protective film is an efficient way to optimize the cavitation erosion resistance. Additionally, as very well known, attainment and tuning of the above mentioned properties for coatings are easier and cheaper than changing bulk material properties. Different types of coatings have already been applied [8–12] for increasing the useful life of the machine elements such as fuel injector parts, steam turbine blades and aircraft parts exposed to rain during flight.

As stated above, from mechanical and structural point of view, expectations for a good cavitation erosion resistance are not different for bulk materials and coatings. However, for coatings, very good adhesion of the coating to the substrate is a prerequisite since even a high end coating will be useless without good adhesion under cavitation erosion effects. In addition to the above-mentioned parameters, cavitation erosion behavior of the coated materials is also influenced by the surface characteristics of the coatings and hardness of the substrate [4]. Surface roughness, scratches and pits on the coated substrate surface as well as any irregularities and defects in the coating structure such as inclusions

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and droplets are determined as preferred initiation sites for cavitation [3].

CA-PVD currently represents one of the most widespread methods for deposition of wear-resistant hard coatings of industrial interest owing to their inherent benefits of increased deposition and ionization rates and producing coatings with very good adhesion to the substrates [13,14]. Thus, these coatings performed very well for cavitation erosion applications as already shown in previous studies [15–18]. However, one of the major drawbacks of CA-PVD coatings is droplets, which may have a negative effect on the surface roughness, corrosion and oxidation stability, and subsequent tribological properties [14,19]. Droplets are formed as a result of ejection of liquid metal particles from the target material. The amount and distribution of droplets are strongly dependent on the process parameters (e.g. deposition temperature and pressure, cathode current, applied bias voltage) and could be substantially reduced by the adoption of several filtering systems [13,20]. The role of droplets on the cavitation erosion properties of CA-PVD coatings has been the subject of several studies. According to the study conducted by Krella [21], the number of droplets that have been generated during the coating deposition by the CA-PVD is one of the key parameters that determine weight losses in the initial stages of cavitation erosion tests. During the tests, some droplets were removed from the surface of the coating leaving behind pits, while others remained at their original sites. In other studies [2,5], the cavities left behind after the detachment of the droplets and the boundary between the droplets and the TiN coating were determined as micro-cracks initiation sites. Similar results were obtained in the study conducted on cavitation erosion of CrN coatings [6]. Although the droplet- related defects on these coatings acted as crack initiation sites, substantial improvement in cavitation erosion performance is achieved owing to their very well adhesion to the substrate.

The incorporation of droplets in the coatings during different stages of deposition and also the types of defects created by their presence may show distinct differences [19,20,22,23]. However, the effects of these different types of defects on the cavitation erosion are not investigated in detail. These differences are expected to exert strong influences on the initiation and propagation of the cavitation erosion damage. Additionally, cavitation erosion performance of the coated parts can be further improved by a better understanding of the role of different droplet-related defects formed during different stages of deposition; e.g. by tuning the deposition parameters to minimize the number of the most detrimental type of defects. This study aims to investigate the role of different droplet-related defects on the cavitation erosion resistance of CA-PVD TiN coated hardened and tempered high speed steels (HSS). A hard substrate is selected to minimize the early cavitation erosion damage of the coating resulting from the undulation of the soft substrate [4]. For the realization of this aim, the cavitation erosion tests were performed on TiN coatings using an ultrasonic apparatus in distilled water. To survey the evolution of the damage, a specific area of the coating was selected and continuously followed for the determination of the initiation and propagation of the droplet-related defects by investigating the same area with SEM after each cavitation test step. For a better explanation of the damage initiation and propagation mechanism, FIB cross- section investigations were also conducted on some selected defect areas.

2. Experimental

2.1. Coating deposition and characterization

Quenched and tempered HSS samples (HS 6–5-2) with dimensions of 20 × 20 × 5 mm and hardness of 60–62 Rc were used as substrates for deposition of TiN coating for cavitation erosion tests. Samples were metallographically grinded and polished (final polishing with 1 μm diamond paste). Before placing of the samples into the vacuum chamber, the samples were cleaned ultrasonically in an alkaline solution for 15 min, washed with distilled water and dried with ethanol. After the cleaning procedure, they were immediately placed into the vacuum chamber. The coating process was carried out in a cathodic arc PVD unit (Novatech-SIE, Model: NVT-12). Ti cathode operating at 60 A arc current was used both during the ion etching and coating steps. In order to perform heating and sputter cleaning of the substrates prior to the deposition of TiN, samples were Ti ion etched using −600, −800 and −1000 V DC bias voltages each for 1 min. During this procedure the substrates were heated to 500–550 °C. Before introducing of nitrogen into the system, a thin intermediate Ti layer was deposited using −150 V bias voltage for 1 min with a thickness of 250–300 nm to have a better adherence. TiN deposition process was conducted under the same bias voltage using N_2 gas pressure of 1 Pa. Deposition time and temperature of the films were 15 min and 550 ± 50 °C, respectively.

Surface morphology of both the coatings and Ti ion etched surfaces were investigated with SEM (Jeol JSM-5410).

FIB (JEOL JEM-9320) cross-section analysis was performed to investigate the selected surface defects. A thin protective carbon layer was deposited on the surface prior to FIB milling to obtain sharper top surface of the cross-sections. A preliminary high ion current milling (5 nA) followed by two lower ion current milling steps (0.5 nA, 0.1 nA) were used until the desired cross-sections were obtained.

Hardness of the samples was determined after slight polishing with 1200 grade SiC paper using ultra micro-hardness tester (Fischerscope HU1000) with a Vickers indenter. During the measurements, 50mN load was applied. Hardness measurements were conducted on randomly selected 30 points. The penetration depth of the indents varied between 0.3 and 0.4 μm, staying within 1/10th of the total coating thickness of 6 μm.

2.2. Cavitation erosion tests

To estimate the effect of droplet-related defects on the cavitation erosion damage, an ultrasonic system (SONICS Vibra Cell) with a horn tip diameter of 13 mm was used. During the experiments, horn oscillation rate and peak-to-peak amplitude were selected as 20 kHz and 40 μm giving a cavitation intensity of 45 kW/m^2. The sample was attached to a substrate holder, the z position of which can be controlled with 50 μm precision. Sample to horn tip distance was set to 0.5 mm. This distance is an optimum value for providing the maximum intensity of bubble collapse and the highest damage [9].

Prior to cavitation tests, sample surface had been subjected to a slight polishing using 1200 grade SiC paper, to remove loosely attached droplets from the surface. This is standard industrial practice that is conducted either by polishing or brushing of the CA-PVD coated surfaces.

To be able to follow time dependent evolution of the cavitation damage, a specific area of the sample was sequentially followed. For this purpose, the sample to be subjected to cavitation tests was mounted on the eucentric SEM sample holder and the area to be followed up was set by fixing the stage position of the SEM instrument.

Cavitation erosion tests were conducted in distilled water. A 2 L water jacketed glass cell was used to keep the temperature of the solution at 25 ± 2 °C. The time dependent cavitation damage propagation was determined in 7 steps (time intervals: 1, 2, 3.5, 4.5, 6, 8 and 12 h). Following each step, the degradation phenomena were followed using SEM (JEOL JSM-5410) and field emission SEM (FE-SEM) (JEOL 7100F).

Selected surface defects on the sample that were subjected to 12 h of cavitation test were investigated by FIB cross-section analysis.

3. Results and discussion

3.1. Investigation of surfaces prior to cavitation erosion experiments

The hardness and thickness of the CA-PVD TiN coatings used in this study were 32 ± 0.3 GPa and 6 ± 0.5 μm, respectively. The elasticity