



Resistance behavior to erosive wearing away of alumina coatings

Resistencia al desgaste erosivo de recubrimientos de alúmina

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ABSTRACT

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This article presents the results of the research related to the experimental behavior of alumina coatings obtained from micrometric size particles and deposited through a thermal spraying by flame process on an AISI 304 stainless steel substrate when it is subjected to erosive wear caused by cavitation through a vibratory apparatus. The methodology used to reach the proposed objective consisted of five phases in the first a morphological and chemical characterization of the materials used, was carried out; the second was the adaptation of UIP1000hd ultrasound equipment to the requirements demanded by the ASTM G32-16 standard (standard test method for erosion by cavitation using vibrating apparatus); afterwards, test pieces of AISI 304 stainless steel were tested to verify the performance of the equipment used, the validation of the wear phenomenon present in the specimens was carried out through scanning electron microscopy tests in order to observe the evolution of the footprint left over the specimen; as a fourth phase, the deposition of the alumina coatings was carried out through a conventional oxyacetylene combustion equipment and an Eutalloy 85 BX gun; finally micro-hardness and erosive wear resistance tests were carried out on AISI 304 stainless steel specimens without and with alumina coatings. The results obtained allowed to validate the operation of the adapted equipment for the performance of the tests since the percentage of average error between the experimental and theoretical data was of 4,5% for AISI 304 stainless steel; regarding the behavior of alumina coatings a 26,23% reduction of material loss was obtained with respect to the AISI 304 stainless steel which represents a significant improvement and encourages its use when mechanical elements are subjected to erosive wear caused by cavitation.

RESUMEN

Palabras Clave:

Alúmina
Desgaste
Erosión
Recubrimientos

Este artículo presenta los resultados de la investigación relacionada con el comportamiento experimental de recubrimientos de alúmina, obtenidos a partir de partículas de tamaño micrométrico y depositadas a través de un proceso de rociado térmico por llama, sobre un sustrato de acero inoxidable AISI 304, cuando están sometidos a un desgaste erosivo originado por cavitación a través de un aparato vibratorio. La metodología empleada estuvo conformada por cinco fases; en la primera se realizó una caracterización morfológica y química de los materiales utilizados; la segunda consistió en la adaptación del equipo de ultrasonido UIP1000hd a los requerimientos exigidos por la norma ASTM G32-16 (Método de pruebas estándar para erosión por cavitación usando aparatos vibratorios); en la tercera fase se ensayaron probetas de acero inoxidable AISI 304 para comprobar el funcionamiento del equipo utilizado y la validación del fenómeno de desgaste de las probetas se realizó a través de ensayos de microscopía electrónica de barrido con el fin de observar la evolución de la huella dejada sobre el espécimen; en la cuarta fase se realizó la deposición de los recubrimientos de alúmina a través de un equipo de combustión oxiacetilénica convencional y una pistola Eutalloy 85 BX; por último se realizaron ensayos de microdureza y resistencia al desgaste erosivo a probetas de acero inoxidable AISI 304 sin y con recubrimientos de alúmina. Los resultados obtenidos permitieron validar el funcionamiento del equipo adaptado para la realización de los ensayos ya que el porcentaje de error promedio entre los datos experimentales y teóricos fue del 4,5% para el acero inoxidable AISI 304; respecto al comportamiento de los recubrimientos de alúmina se obtuvo una reducción del 26,23% de pérdida de material con respecto al acero inoxidable AISI 304, lo que representa una mejora significativa e incentiva su utilización cuando se tienen elementos mecánicos sometidos a un desgaste erosivo originado por cavitación.

Introduction

The term wear refers to loss of material occurring at the interface of two surfaces, when there is relative movement between them, under a force in extreme conditions of mechanical or even environmental stress.

Erosion can lead to fatigue failure of the work piece, the principal factor causing loss of the material's mechanical properties. The manner in which wear, surface deterioration and loss of material occur, can be explained by two alternative or simultaneous mechanisms: wear caused by plastic deformation, mainly occurring in ductile materials and

secondly, wear through crack generation that causes peeling and originates mostly from brittle materials like ceramics.

Simultaneous mechanisms: wear caused by plastic deformation, mainly occurring in ductile materials and secondly, wear through crack generation that causes peeling and originates mostly from brittle materials like ceramics.

Erosion by wear arises due to a surface's exposure to repeated impacts of solid particles or external agents such as gases or liquids; a phenomenon that causes this type of wear is cavitation.

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It is defined as the formation and subsequent collapse of cavities or bubbles containing vapor or gas-steam mixture, within a fluid [1].

Cavitation generally arises as a result of high pressure variations caused by the movement of a liquid. As this pressure suddenly falls below vapor pressure, the stress imposed on the liquid produces cavities due to the small nuclei of cavitation present in a real liquid [2]-[4]. Subsequently these cavities or bubbles violently collapse when subjected to a higher pressure.

They in turn cause shock waves at speeds between 500-600 m/s and high pressure that can be up to several GPa, causing fatigue, fracture and loss of solid surface material [5]-[7]. This phenomenon is one of the most damaging in nature. It affects the efficiency and proper functioning of almost all hydraulic systems such as: pipes, valves, water pumps, boat propellers, hydraulic turbine blades, diesel engines, fuel injectors and many other hydraulic devices used in industrial applications. In order to reduce the impact of the cavitation phenomenon in different mechanical elements, two alternatives can be adopted: the first is to make an optimum design that maximizes the exposed components' resistance to erosive wear through the implementation of materials with improved mechanical properties. Among the aforementioned materials, AISI 304 stainless steel is remarkable for its corrosion resistance and good performance facing said phenomenon. The second alternative encompasses the development or implementation of coatings that possess a better resistance to cavitation and erosion [8], [9]. The most commonly used alternative is the use of coatings as they are customarily cheaper and lead to good results.

The protection of metal substrates against wear can be obtained by using abrasive and abradable coatings (tribological), mainly composed of ceramics [10], [11]; coatings with alumina (Al_2O_3) are increasingly used in many industrial applications due to excellent properties such as high wearing resistance, elevated hardness, good tribological properties, excellent corrosion resistance, high thermal resistance and relatively low manufacturing cost [12]-[15].

Competently executing the ceramic coating is extremely important due to its high adhesion to the substrate. It is critical to obtain good wear resistance. Ceramic coatings can be attained through several methods such as thermal spraying [16], polymerizable complex [17], chemical vapor [18] deposition and plasma spraying [19]. These processes introduce some foreign materials on the surface of the matrix. One of the most popular processes for applying coatings on metal surfaces is thermal spraying, as a result of its versatility and the wide variety of materials that can be applied [20], [21]. These methods use a spray nozzle that controls an oxy-fuel flame, electrical or plasma arc [22]-[23] that allow the deposition of both metallic and nonmetallic materials. Progress in thermal spraying is widely accepted in the industry, both in the manufacturing as well as in the maintenance of parts, where the field of application is increasingly expanding and broadening due to the development of new alloys and processes [24].

There are several thermal spraying processes such as: thermal spray wire, metal powder, plasma, gun trigger for thermal spray, high-speed oxy-fuel and archwire spray; the coatings obtained have a layered structure from deposited material and can have porosities between 10% and 20%, caused by air and rust particles trapped by the high temperatures used [22].

In conventional processing, ceramic coatings are produced from micrometric sized powders and their properties have been studied in depth, showing that wear resistance is related to the coating's hardness and toughness [25]-[26].

The ultrasound is a physical principle widely used to determine erosive wear resistance caused by cavitation [27]-[30]. It is the use of mechanical waves whose frequency is above the audibility of the human ear, approximately 20,000 Hz.

Said waves spread by causing alternating cycles of low and high pressure, with intervals that depend on the frequency of application and that cause cavitation in the fluid where they are being applied.

This investigation is aimed at determining the behavior of alumina coatings obtained from micrometric sized particles deposited through a process of thermal flame spraying, when subjected to erosive wear caused by cavitation.

Materials and methods

Morphological and chemical characterization of materials

The morphological and chemical characterization of alumina (Al_2O_3) to be deposited was performed using the Electron Microscope (SEM), a scanning technique; in which the FEI Quanta FEG 650 model (microscopy unit) was used. The microscopic images obtained helped to identify the shape and calculate the size of particles constituting the material.

Additionally, the microscope has an integrated microanalysis system in concert with Energy Dispersive Spectroscopy EDS (Energy Dispersive X-ray Spectroscopy). It delivers a report containing the identified spectrum and elements, with their percentages by atomic weight. Similarly, the chemical composition of AISI 304 stainless steel used as substrate is related in Table I.

Tabla I. Chemical composition of AISI 304 Stainless steel

Elementos	N	Mn	S	P	Si	Cr	Ni	N	Fe
Porcentajes	0,06	1,8	0,02	0,04	0,6	19,5	9,5	0,1	68,38

Adaptation of Ultrasound unit to execute erosive wear testing

The UIP 1000 hd unit generates a 1000 W ultrasonic power at a frequency of 20 kHz, and it has an automatic frequency adjustment, that is, an adjustable 25 micron range, between 50% and 100% of this value. It is also fitted with a titanium sonotrode; its main industrial applications are the dispersion, homogenization and deagglomeration of particles, grinding materials, emulsification and cell disintegration.

In order to use this equipment in the testing of erosive wear, it was necessary to tailor it to the requirements demanded by the ASTM G32-16 standard, "Standard Test Method for Cavitation Erosion Using Vibratory Apparatus" in which the sonotrode was modified through the realization of a threaded hole in the lower area to facilitate the assembly of the specimen to be tested. Additionally, the flow recalculation system fitted to the unit was disconnected since it requires a vessel that permits temperature control. Furthermore, it must avoid the effects of the sound wave reflections as they clash with the limits of the vessel and the interaction with waste caused by cavitation.

Execution of erosive wear tests by cavitation on AISI 304 steel samples Erosive wear tests by cavitation were performed in distilled water with the ultrasound unit UIP 1000 hd adapted to the requirements of ASTM G32-16 standard, whereby pressure fluctuations are induced by vibrations as a means of generating the erosion by cavitation phenomenon [1]. In figure 1 a schematic overview of the assembly used for conducting the tests is shown.

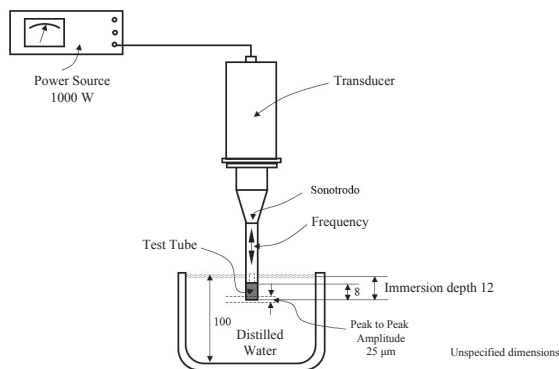


Figure 1. Mounting used for testing of erosive wear cavitation

The depth of distilled water used in the container was 100 ± 10 mm, the test surface of the specimen was submerged to an immersion depth of 12 mm with a temperature of 25°C within the liquid. The testing was conducted at an atmospheric pressure of 95 hPa and the displacement amplitude, peak to peak of the test surface of the sample was $25\ \mu\text{m}$; the diameter

of the samples used was 20 mm, with a thickness of 8 mm. A total of 35 tests were performed, five for each of the following times: 30, 60, 120, 240, 480, 960 and 1140 minutes. The variable measured was the specimen's loss of mass following each period of exposure to cavitation and it was executed through a digital scale model, BP 211 D with a resolution of 0.1 mg.

To test the correct functioning of adjustments made to the ultrasound unit, AISI 304 stainless steel samples were analyzed in order to compare the results with data reported in the literature and determine the percentage of error in the tests. As the value obtained was less than 5% the result was considered invalid and then proceeded to perform tests on alumina-coated samples.

Attaining Coatings The alumina coatings were achieved through the process of thermal flame spraying, using the Eutalloy BX 85 gun, adapted to conventional oxy-acetylene combustion equipment.

The distance used between the torch and the base material was approximately 30 cm, with an inclination in relation to a 60° horizontal. The pressure of oxygen used was 25 psi and the acetylene was 5 psi, defining a pressure ratio of 1:5.

The morphological and chemical characterization of the deposits is completed with the same equipment and techniques that are used in the characterization of the alumina used for coatings.

Testing specimen with and without alumina coatings

Micro-hardness and erosive wear resistance on AISI 304 steel samples with and without alumina coatings were tested. Micro-hardness tests were performed on a Wilson Tukon Wolpert-2100B micro durometer from INSTRON.

The type of micro-hardness measured was Knoop, because the depth of penetration is less than those obtained through the Vickers micro-hardness testing. Trials were conducted adhering to the ASTM E384 - 11 Standard Test Method for Knoop and Vickers Hardness of Materials.

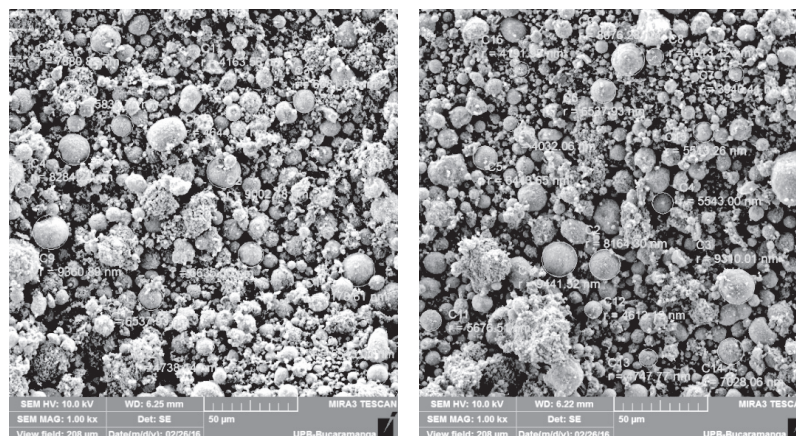


Figure 2. Images Scanning electron microscopy of the alumina particles used for the realization of coatings

A manufactured indenter with a diamond tip was employed; its geometrical shape is a pyramidal square base, which is pressed on the surface of the sample with a controlled force through high precision testing machinery.

This procedure leaves a trace on the surface of the specimen. A total of 30 trials for each type of sample were executed: 10 for each load used (50, 100 and 200 grams force).

The application time was 12 seconds and its maintenance, 15 seconds. The testing of erosive wear resistance for AISI 304 stainless steel samples, with and without alumina coatings were carried out according to the procedure described in section 2.3 of this document.

Results and Discussion

By scanning electron microscopy, the morphological characterization of the alumina used for making coatings was executed; the images obtained from the procedure permit the quantification of the size of particles present in the sample, as shown in Figure 2. From the measurement of 50 particles, an average value of 11.27 μm was obtained and a size distribution as shown in Figure 3.

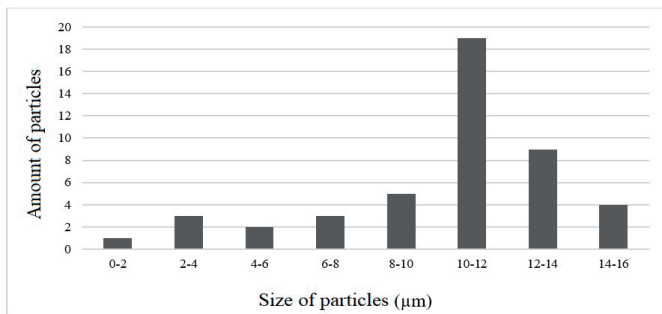


Figure 3. Distribution of particle size in the sample alumina analyzed for conducting coatings

The chemical characterization of alumina used for the coatings was attained through Energy Dispersive X-ray Spectroscopy analysis (EDS); the results obtained are listed in Figure 4 and they generate some atomic percentages of oxygen and aluminum, 51.2% and 48.8% respectively, which ensure that the material examined is 99.5% pure alumina.

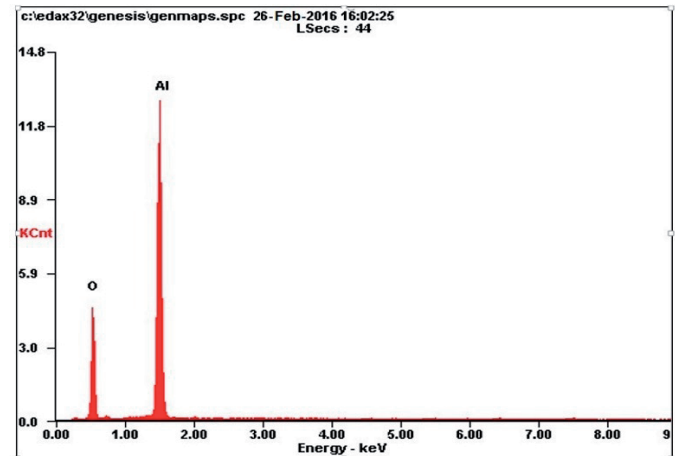


Figure 4. Results of spectrometry energy dispersive X-ray on the material used to make the coatings

The morphological analysis of the coating carried out through the process of thermal flame spraying (see Figure 5), was also accomplished through SEM analysis.



Figure 5. Deposits of alumina coatings through thermal flame spraying process

In Figure 6 depicts the discontinuity of the coating, an increase in particle size reaching maximum values of approximately 50 microns and a vast quantity of pores occurring due to the technique. This concurs with the information reported by several researchers [21]-[24].

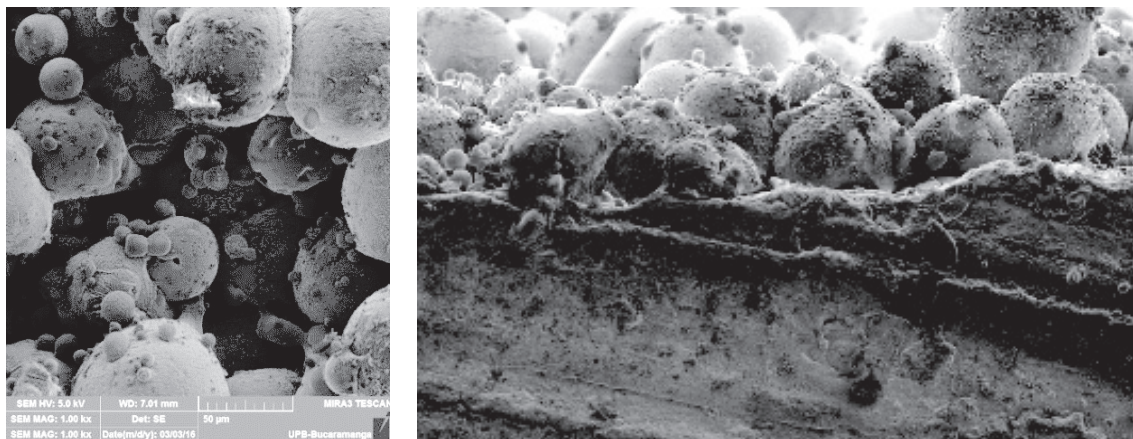


Figure 6. Upper and lateral view of alumina coatings made by the thermal flame spraying process

The Knoop micro-hardness testing of alumina coatings demonstrated a 220.3% increase in its magnitude when compared to the values obtained from AISI 304 stainless steel (see Figure 7), which was the material used as matrix for deposition of the coating.

This information coincides with particulars found in the literature [10]-[15], which state that the protection of metallic substrates against wear can be achieved by using coatings composed of mainly ceramic materials. That is, they exhibit a significant improvement in the material's hardness, therefore a greater resistance to wear.

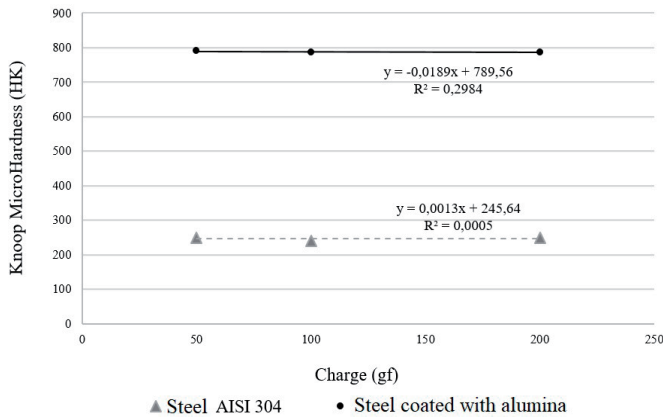


Figure 7. Results of Knoop micro-hardness on a AISI 304 stainless steel and alumina coatings deposited through a thermal flame spraying process

Validating the use of ultrasound unit UIP 1000 hd in the execution of erosive wear by cavitation testing was done by making use of images from electron microscopy scanning.

Through said scanning, the variation of the dimensions of the traces left on AISI 304 stainless steel samples was quantified within specific periods of exposure to the phenomenon. Figure 8 shows the images for testing periods of 240, 960 and 1440 minutes.

A directly proportional relationship between these two variables is noted. Essentially, there is a simultaneous increase in the sample time as well as in the dimensions of the traces.

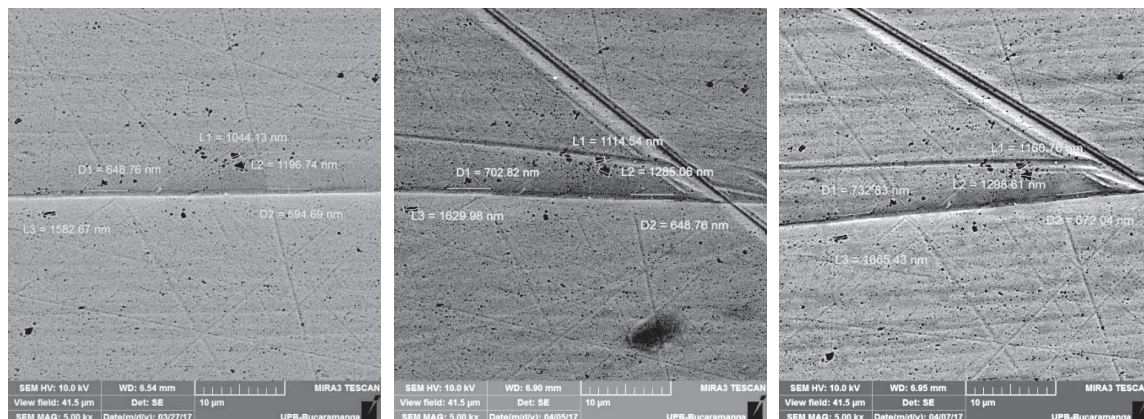


Figure 8. Dimensions of traces present in the AISI 304 steel specimen subjected to erosive wear by cavitation for periods of a) 240 min, b) 960 min c) 1440 min

Such behavior is expected since the sample is losing mass as a result of wear caused by cavitation. The appearance of new traces is also noted. It confirms, to an even greater extent, the wear phenomenon presented in the test specimen.

Figure 9 illustrates the experimental and theoretical values for loss of mass obtained from AISI 304 steel samples for different periods of exposure to the erosive wear phenomenon by cavitation and its trend versus time.

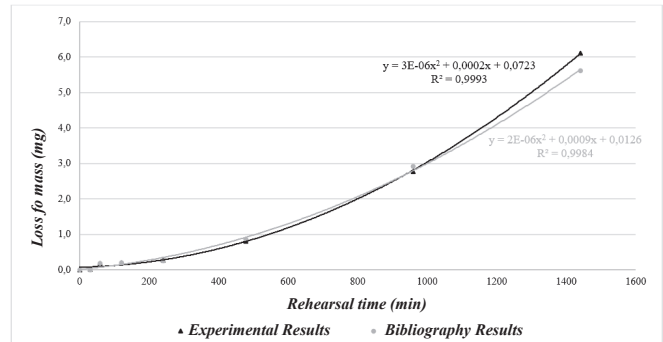


Figure 9. Loss of mass of AISI 304 steel sample exposed to erosive wear phenomenon by cavitation

It is observed that the trend of the values obtained from the test is similar to results found in the literature [31] and it is second order polynomial; furthermore, differences in the loss of mass within determined periods of testing are identified, the most significant having presented itself at 1440 minutes of exposure, wherein which an error rate of 4.5% between these values is attained.

Despite the aforementioned variation, experimental results are considered acceptable, therefore the modifications to the ultrasound equipment used for testing erosive wear by cavitation are validated. The machine is used for obtaining the performance of alumina coatings deposited through a thermal flame spraying process against this class of wear.

Figure 10 shows the behavior of the loss of mass of alumina coatings when exposed to cavitation. It is possible to observe the behavior of the third order polynomial, presenting a large emission during the first 240 minutes of testing.

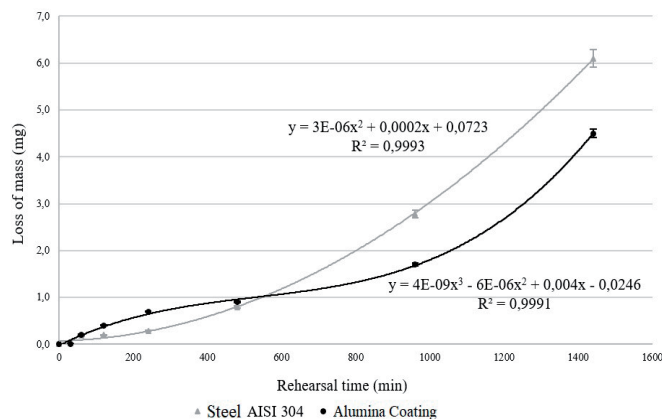


Figure 10. Behavior of the mass loss caused by the cavitation phenomenon in samples of AISI 304 stainless steel and coated alumina samples

It can be proffered that it is due to the numerous irregularities and porosities occurring in coatings made by thermal flame spraying [22].

Subsequently, an area of low material loss within periods ranging between 240 and 800 minutes is identified and eventually, the behavior is similar to that of AISI 304 stainless steel but with a 26% reduction in loss of mass; this permits the testing of the improvement in the resistance to erosive wear by cavitation that is achieved through the use of Al_2O_3 coatings, also agreeing with the information reported in the literature [12]-[14].

Conclusions

- It was possible to adapt a conventional ultrasound unit for the testing of resistance to erosive wear that complies with the ASTM G32-16 standard.
- The tests performed on AISI 304 stainless steel showed that the maximum error obtained during the tests of resistance to erosive wear, with respect to the data found in the literature, is approximately 4.5%.
- Coatings made from micrometric alumina by thermal flame spraying showed a 26.23% reduction in the material lost during the erosive wear resistance test pertaining to the samples of AISI 304 uncoated stainless steel.
- During low periods of testing, the alumina coating presented a higher loss of material due to the surface irregularity and the large number of pores obtained when a thermal flame spraying process is used.

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