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TRIBO-CORROSION INVESTIGATION ON MATERIAL 316LX MANUFACTURING RESULT OF 3D PRINTING MATERIAL ADDITIVES IN 5% $H2SO_4$ SOLUTION

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Abstract

Tribocorrosion is a type of material degradation caused by simultaneous wear and corrosion of metal surfaces caused by laminar or turbulent flow. Additive manufacturing technology plays an important role in its application to precision components and complex assemblies. This study developed a 316LX material with Fe, Ni, Cr, and other powder alloys that was processed into an ultra-protective wire as a 3D printing filler. This simulation of tribocorrosion conditions was performed on a triboester machine. This simulation is expected to provide important insights and understanding into the behavior and properties of the 316LX 3D printing material, especially when exposed to abrasion and corrosion conditions in a sulfuric acid solution environment. Corrosion Rate Testing of 316LX Material Additives Using Potentiodynamic Methods in a Modified Rotating 5% Sulfuric Acid Fluid. In addition to corrosion rate, the Vickers hardness, metallography, and shrinkage of the 316LX green part material were also tested at 1000°C after sintering.

Keywords: *Tribo-Corrosion, 316LX, Additive Manufacture, 3D Printing, Surface Acid.*

1. INTRODUCTION

Corrosion is the surface of metals or alloys in a certain environment. Basically, some metals have a higher resistance to corrosion than others, and this can be due to a number of factors such as their chemical constituents, the nature of their electrochemical reactions, and others. The corrosion resistance of metals can be defined by their ability to withstand aggressive conditions. This largely determines the service life of the components used. However, corrosion has several definitions, and according to the International Union of Pure and Applied Chemistry (IUPAC), corrosion is the irreversible interface reaction of a material (metal, ceramic, and polymer) with the environment, resulting in wear or consumption of the material as an environmental component dissolve in the material [1].

Additive manufacturing is the official term for what used to be called rapid prototyping and is often called 3D printing. The term "Rapid Prototyping" (RP) is used in many industries to describe the process of quickly creating a system or partial representation before final release or commercialization. In other words, the focus is on rapid creation and that the output is a prototype or basic model, from which further models are obtained and finally the

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final product. Both management consultants and software engineers also use the term rapid prototyping to describe the piecemeal process of developing business and software solutions that allow customers and other stakeholders to test ideas and provide feedback during the development process [2].

Almost all metals react chemically with their environment and cause one form of corrosion that weakens the metal. Most systems operate in conditions of continuous mechanical interaction in addition to a corrosive environment. Mechanical interactions such as load, stress and friction can also weaken the mechanical properties. Abrasion caused by friction between two surfaces or the impact of solid particles or liquids can seriously damage the material. Material loss due to wear is often affected by a corrosive environment [3].

Users of RP technology have come to understand that the term is inadequate and, in particular, does not effectively describe newer applications of the technology. Improvements in the production quality of these machines have meant that the final product is often much more closely related. Many parts are now made directly on these machines, so we cannot call them "prototypes". The term "rapid prototyping" also does not capture the basic principle of these technologies, as they all produce parts using an additive approach. The newly formed ASTM International Technical Committee decided that new terminology should be adopted. Although there is still some debate in the recently adopted ASTM consensus standards, the term additive manufacturing is now used [4].

The key to how AM works is that parts are made by adding material layer by layer; each layer is a thin section of the part derived from the original CAD data. Obviously, in the physical world, the thickness of each layer must be limited, so the resulting part is an approximation of the original data, as shown in Fig. 1. The thinner each layer, the closer the final part is to the original. All AM machines on the market to date use a layer-based approach, and the main ways in which they differ are the materials used, layer formation and layer bonding. Such differences are determined by factors such as the precision of the final part and its material and mechanical properties. They also determine factors such as how quickly the part can be produced, how much post-processing is required, the size of the AM machine used, and the total cost of the machine and process. This chapter introduces the basic concepts of additive manufacturing and describes general AM process from design to implementation. It continues the discussion of the impact of AM on design and manufacturing and tries to help understand how it has changed the entire product development process. Because AM is an increasingly important tool in product development, some tools related to the product development process are discussed at the end of the chapter [5].

Figure 1. Solidworks image CMAS Prototype

Fig. 1 shows he thinner each layer, the closer the finished part is to the original. All AM machines on the market so far use a layer-based approach, and the main ways in which they differ are the materials used, how the layer are created and how the layer are bonded to each other. Such differences are determined by factors such as the precision of the final part and its material and mechanical properties. They also determine factors such as how quickly the part can be produced, how much post-processing is required, the size of the AM machine used, and the total cost of the machine and process. 3D printing is one of the additive manufacturing processes [6].

The 3D printing process inside the machine consists of two steps: (1) direct transfer of software data to the printed structures, (2) repeated positioning of the print head in space in all three directions, layer by layer. More specifically mentioned how the printing process takes place, first a model is made using a CAD system and then the areas are printed using two assemblies. A 3D object is represented by dimensional parts that are printed layer by layer until the object is completed. The second stage of production can also be divided into two main stages "coating and melting", during which the material is applied to the surface and the layers are formed by the energy source. The energy source and raw material vary depending on the technology used [7].

Some technologies are commercially available prototype methods, others are rapidly becoming viable production forms, and new technologies are constantly being developed. These different methods of additive manufacturing can be classified according to the type of material used. One class of 3D printing equipment builds an object by gluing together successive layers of very fine powder. Such powder adhesion or bonding of granular materials can be achieved by spraying each powder layer with an adhesive or by melting the powder granules with a laser or other heat source. Other techniques melt and then melt granules of pulverized building material as it is deposited on the built surface. Various forms of powder bonding are already used in many materials in 3D printing. These include nylon, bioplastic, ceramic, wax, bronze, stainless steel, cobalt chrome, and titanium[8].

1.1. Tribocorrosion Process

Tribocorrosion as a result of the combined effect of two dissipative phenomena. These tribocorrosion systems are complex in nature and depend on several mechanical, material, chemical and electrochemical factors (Fig. 2).

The study of tribocorrosion is therefore a complex scientific task due to its industrial importance. In fact, tribocorrosion affects various technical systems, such as biomedical implants, mining equipment, food processing equipment, metal forming tools, nuclear power plants, transportation and energy systems. All these systems are characterized by the use of metals that are prone to wear by fluid flow (cavitation), particle movement (erosion) or contact with solid bodies (sliding and scratching), and in corrosive environments. The technical challenge is to propose prevention plans and material (and/or environmental) selection criteria to overcome or control tribocorrosion in various situations. Wear and corrosion are usually misleading. The example of TiN coatings illustrates this problem. These coatings combine excellent dry wear and corrosion resistance in most aquatic environments. However, their tribocorrosion performance was found to be very poor [9].

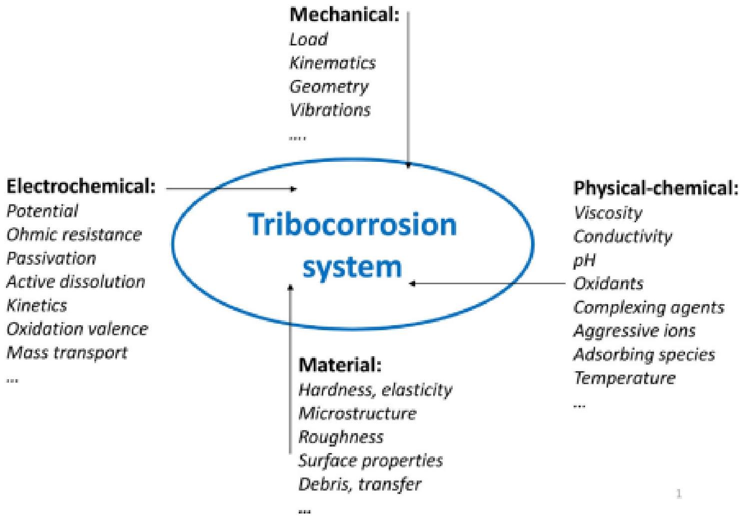


Figure 2. Factors affecting tribocorrosion systems

1.2. Mechanisms of Tribocorrosion

A purely tribocorrosion mechanism is already very complex, focusing on the degradation process and its time-dependent nature and non-linear behavior, but the inclusion of a corrosion component in the system increases its complexity even more. Often a tribological event is characterized by movement or articulation between two bodies. Corrosion is usually distinguished from an electrochemical process, which usually occurs in a stable environment with an exposed surface [10].

Tribocorrosion events can be divided into the following categories: erosion, abrasion, adhesion, scratching and fatigue. Therefore, tribocorrosion is used to cover them in a wider area and in all interactions related to wear and corrosion. The common types of tribocorrosion are sliding corrosion, sliding corrosion and microfriction corrosion [11].

1.3. 316 LX

Fig. 3 shows the 3D printing material for 316LX, which is made of 316L stainless steel powder, which is then printed in BCC (Body Centered Cubic) format, weighing 58 grams and 4.1mm thick on both sides. The side length of the first, second, third, and fourth box are 39.67, 29.98, 20.76 and 13.42 mm, respectively. The box has multiple circle diameters, where the first, second, third, and fourth circle diameter are 35.54, 25.40, 16.38, and 9.23 mm, respectively.

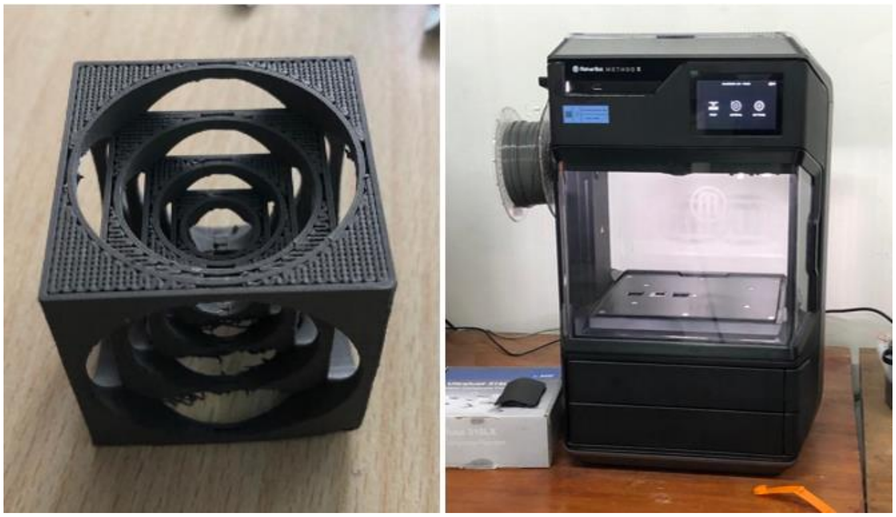


Figure 3. 316LX Cellular Form 3D Printing material with MakerBot Method X

2. EXPERIMENTAL METHOD

This study aims to investigate the tribo-corrosion phenomenon of 316LX material manufactured from 3D printing process in 5% H2SO4 solution. 316LX samples were prepared by cutting them to the desired dimensions and polishing them to a smooth surface. This test uses a magnetic stirrer to add fluid flow in its environment. Electrochemical testing was carried out by immersing the 316LX sample in 5% H2SO4 solution as a corrosive environment. Open circuit potential (OCP) measurements were taken to monitor the corrosion potential of the samples during the test. Furthermore, potentiodynamic polarization measurements were performed to determine the corrosion potential (Ecorr) and corrosion current density (icorr) of the samples. Polarization resistance (Rp) was measured using electrochemical impedance spectroscopy (EIS) to evaluate the corrosion resistance of the material. Tribo-corrosion testing was carried out by inserting 316LX samples as test pieces on a potentiodynamic. The tribocorrosion simulation is run for a specific time period or a specific number of cycles to simulate simultaneous wear and corrosion. Before the test, hardness ickers and metallographic tests were conducted. The data obtained from the tests were evaluated and analyzed to determine the corrosion rate and tribo-corrosion rate of the 316LX material. Fig. 4 shows the flowchart of the research process is explained.

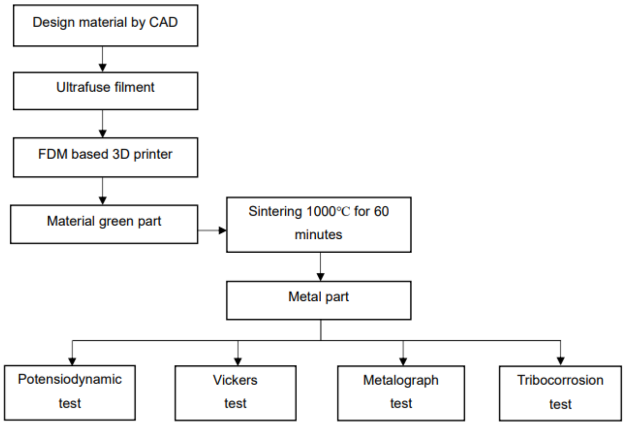


Figure 4. Flowchart Addictive Manufacturing Test

2.1. Chemical and Mechanical Properties Test

2.1.1. Potensiodynamic Test

Potentiodynamic polarization is a method for determining corrosion behavior metals based on potential relationships and anodic/catodic currents. If the metal is in contact with a corrosive solution, a reaction may occur on the metal surface reduction and oxidation reactions are simultaneously caused on the formed metal surface many micro-anode and micro-cathode microcells. Metal corrosion occurs when an anodic current is present which is the same magnitude as the cathodic current, even though no current is supplied from the outside system. This is because there is a potential difference between the metal and the solution environment. This potential difference is called the corrosion potential, E-corr. If into the cell system electrochemistry is given direct current from outside (DC Source) or added substances that can if it affects the cell potential, the metal potential will be more positive or more negative compared to its corrosion potential, produces anodic currents or cathodic currents. Changes the potential in the metal is called polarization or over potential. Metal polarization characteristics determined based on the potentiodynamic polarization curve which represents the current flow or log current a function of the generated potential. Polarization or over potential, η, is the change in the electrochemical potential of a half-cell from its equilibrium position with environment in an electrochemical process. Relationship over potential and current can used to express the corrosion rate. [12]

For potential differential testing, sample preparation was performed to obtain samples according to the standard used for potentiodynamic testing, namely ASTM G5 with a sample size of 1 cm x 1 cm. The samples used were 3D printed using Ultrafuse 316LX material to form a BCC (Body Centered Cube) and then sintered at 1000°C for 1 hour holding time to form a metal part. The material was cut into 1 cm x 1 cm size using a hand grinder according to the potentiodynamic test method. The cut material are grounded sizes of 240 mesh, 400 mesh, 800 mesh, 1000 mesh and 2000 mesh. The grinding process aims to remove the metal layer and any existing contaminants such as dust and rust that adhere to the surface of the metal layer being inspected so that they can be easily read with a potentiodynamic tool. After that, it is soldered to the copper wire so that it can be easily fixed for the purpose of installing it as a connection material with the WE terminal in the potentiodynamic test. After soldering, the soldered areas are coated with resin and covered with a layer of paint, so the test focuses on the readable area of the potential measurement. Corrtest was used as a potentiodynamic test meter connected to WE, CE and RE cables in the potentiodynamic test procedure. In this test CE for platinum and RE for Ag/AgCl were used. While, WE was used from his 316LX material using his 1 cm x 1 cm piece of material cut in the form of his BCC.

2.1.2. Metallographic Test

Metallographic examination combines science and art to examine the microstructure of metals and alloys using light, electron, or other types of microscopes. Metals’ performance and material properties, especially mechanical properties, are determined by their microstructure. The performance and reliability in the field were need to be understood through the microstructure of materials analyzing. The basic principles of metallographic examination fall into his two categories: macrography and micrography. A practical application of metallography is to study grain size, phase distribution, and the presence of impurities in metals. The metallographic results, which describe the processes that has been undergone on a material was used as reference to determine the required specifications for a material. Metallographic observation results are affected by the surface treatment of the observed specimen [13].

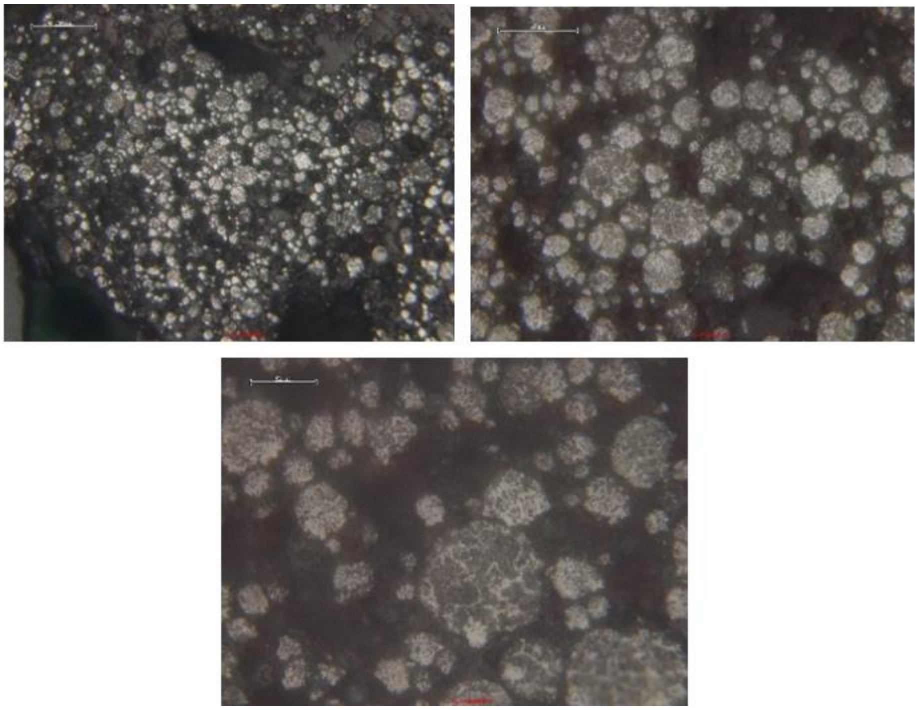


Figure 5. Metallography

The metallographic examination is performed according to the ASTM E3 standard as part of the metallographic examination. 316LX specimens cut into 1 cm x 1 cm pieces were used for metallographic examination. For convenience, cutting the materials with a hand grinder. The sample is composed of a cylindrical frame made of resin, and after the sample is put into a mold, it is put into the mold made of resin. This resin is made with a resin: catalyst ratio of 10:1, so the resin is neither too soft nor too hard. Pour the resin into the mold container and wait for the resin to dry. After the resin has dried, remove the resin and perform a polishing operation on the surface of the sample not covered by resin with 80-mesh sandpaper until the surface is flat and any contaminants such as dust and rust are removed. Then, smooth the surface of the sample with 120-mesh, 240-mesh, 400-mesh, 800-mesh, 1000-mesh, and 2000-mesh sandpaper. The sanding process is performed using a rotating sanding table with low speed for coarse sandpaper and high speed for fine sandpaper. The sanding process is performed using a small amount of water to facilitate the sanding process and remove any sandpaper remaining on the surface. After grinding, apply Cr2O3 paste to a soft cloth and polish the sample surface. Next, after etching the polished surface of the sample using aqua regia 3% as an etchant for a holding time of 5 seconds, the etchant on the surface of the sample was dried using a dryer, and the sample was dried with a dwell time of 10 minutes. Fig. 5 shows the metallographic examination process was then performed by zooming in until the grain boundaries of the 316LX sample were visible.

2.1.3. Micro Vickers Test

Micro Vickers test is a type of hardness test used to measure the hardness of materials, especially those with fine microstructures or thin coatings. It is a widely used method for measuring hardness on the microscopic scale and is particularly useful for evaluating small or delicate samples that cannot undergo conventional Vickers or Brinell tests.

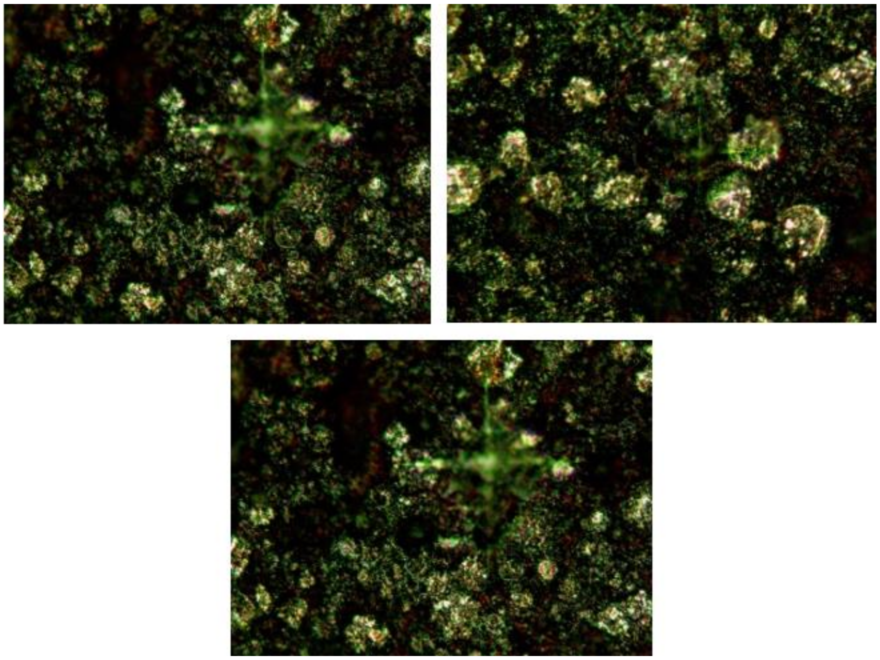


Figure 6. Micro-Hardness Vickers

Fig. 6 shows in the micro-Vickers test, a square pyramid-based diamond indenter is pressed into the surface of the material with a specified load. The indentations are usually very small, typically 10 to 100 microns. After forming the indentation, measure the diagonal length of the indentation with a microscope. A hardness value is calculated using the applied load and the average diagonal length of the indentation. Micro vickers testing has several advantages, such as high accuracy, good reproducibility, and the ability to test small and complex areas. Commonly used in research, quality control, and materials characterization applications.[14] On table 1 shows the result of the hardness materials 316LX with Diamond Indentor through three point of hardness test on the surface materials 316LX with the first point of surface hardness test is 167,15 HV, the second point test is 207,71 HV and the last point test is 203,45 HV. on fig. 10 is the result of the vickers hardness test which is micro-seen on the 316LX material and the vickers hardness test on the 316L material. For hardness value data from the vickers 316LX hardness test can be seen in table 3 and for the vickers hardness value at 316L can be seen in table 4.

Table 1. Vickers Result

Label	HV	D1	D2	Mean
1	167.15	42.30	51.90	47.10
2	207.71	44.05	40.45	42.22
3	203.45	47.02	38.86	42.69

2.1.4. Tribocorrosion Test

Tribocorrosion is corrosion that results from the combined phenomena of corrosion and wear occurring at sliding contacts and is defined as wear phenomena occurring at sliding contacts and can be defined as an irreversible an irreversible transformation caused by the simultaneous action of chemical, mechanical (wear) and electrochemical (corrosion) interactions occurring on surfaces subjected to sliding contact, mechanical (wear) and electrochemical (corrosion) interactions occurring on surfaces subjected to contact motion. subjected to contact motion. When tribocorrosion systems exist, the synergism that resulting from the simultaneous action of mechanical and environmental effects is observed over a period of time.[15]

As already explained, which roles as mechanical in this case is the rotation of the magnetic stirrer which creates a flow which can affect the corrosion of the test material, and the environmental here lies in the solution used, namely 5% H2S04. as described in Fig. 7. Fig. 8 and 9 show the results of the tafel diagram from the tribocorrosion and potentiodynamic testing of the 316LX. 316LX material in the tribocorrosion testing process has a corrosion rate of 17.067 mm/a with a relative corrosion resistance with an unacceptable level and for testing 316LX with the method without tribocorrosion has a corrosion rate of 0.02 mm/a with relative corrosion resistance with an outstanding level as shown is in table 2.

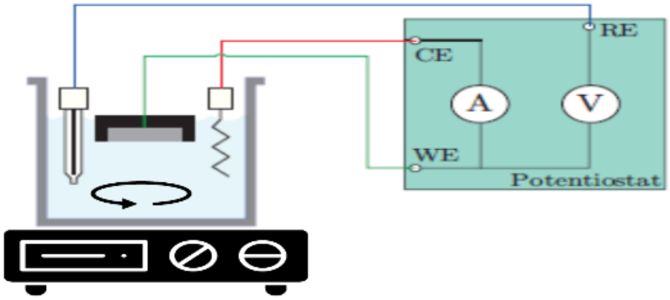


Figure 7. Tribocorrosion Test

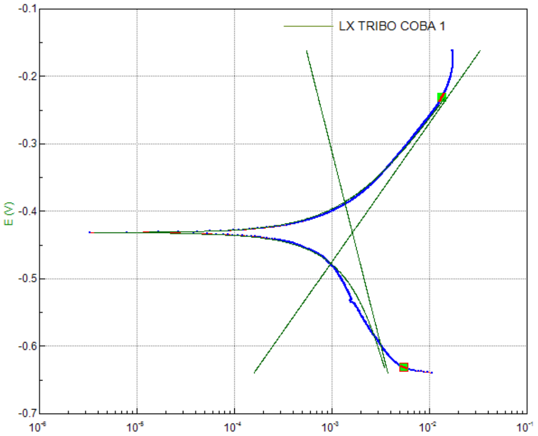


Figure 8. Diagram Tafel 316LX with Tribocorrosion Test

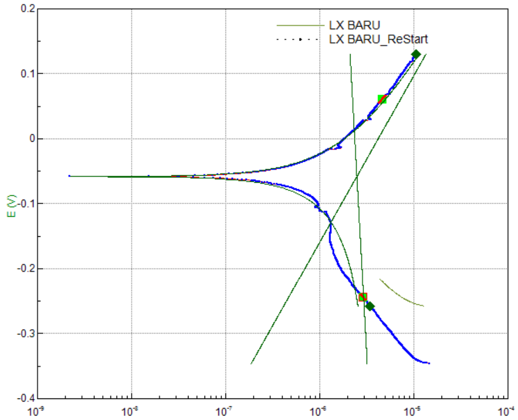


Figure 9. Diagram Tafel 316LX with Potentiodynamic Test

Table 2. Corrosion Rate 316LX

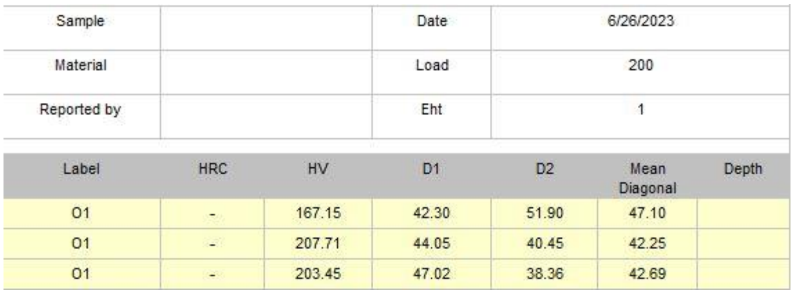
Specimen	Rate Corrosion	Relative Corrosion Resistance
316 LX Tribocorrosion	17.067 mm/a	Good
316 LX Without Tribocorrosion	0.02 mm/a	Outstanding

3. CONCLUSION

Based on the data obtained that in the microstructure of the metallographic results there are perite and ferlite phases but there are still many shafts that occur where the image is black. In the metallographic test there is a black color due to the presence of porosity on the surface of the material caused by the manufacturing process using a 3D printing machine which has a low density with a size of 0.25 µm so that in the sintering process the porosity cannot be removed and causes the display on the metallographic test black.

For tribocorrosion test using a simulation with a magnetic stirrer at rotation using 700 rpm with the solution used is 5% H2SO4. The results of the corrosion rate obtained through tribocorrosion simulation turned out that those given fluid flow had a greater corrosion value than those not. This means that fluid flow is very influential on the corrosion rate. In the next experiment, rotation with a larger rpm will be used, ranging from 1000-3000 rpm accompanied by pressure generated from oxygen with a minimum pressure of 1 bar. In the next process, a corrosion rate reading sensor based on CMAS (Coupled Multielectrode Array Sensor) made of 3D printing based on stainless steel is also used with several other developments. For the 316LX hardness tests using the Vickers test which was carried out using a diamond indenter and the load used was 200 kgf and for 316L using 300 kgf load. The results of the vicker hardness test obtained an average value of 192.77 HV for 316LX material and as a comparison the average value of 316L material was 172.52 HV. In this case, the 316LX material has a higher hardness value than the 316L material.

Table 3. Vickers Result of Material 316LX



Sample		Date	6/26/2023
Material		Load	200
Reported by		Eht	1

Label	HRC	HV	D1	D2	Mean Diagonal	Depth
01	-	167.15	42.30	51.90	47.10	
01	-	207.71	44.05	40.45	42.25	
01	-	203.45	47.02	38.36	42.69	

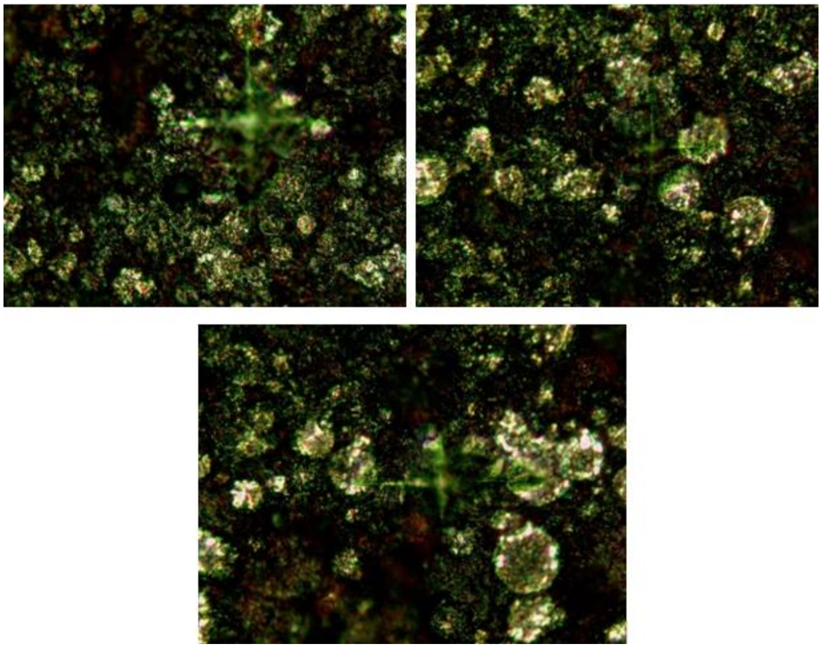
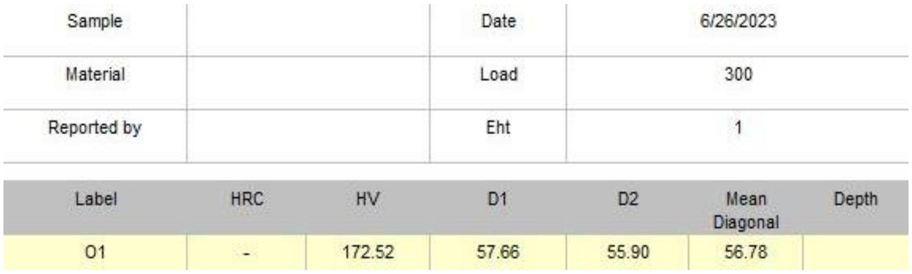


Figure 10. Result Micro-Hardeness Vickers 316LX

Table 4. Vickers Result of Material 316L



Sample		Date	6/26/2023
Material		Load	300
Reported by		Eht	1

Label	HRC	HV	D1	D2	Mean Diagonal	Depth
01	-	172.52	57.66	55.90	56.78	

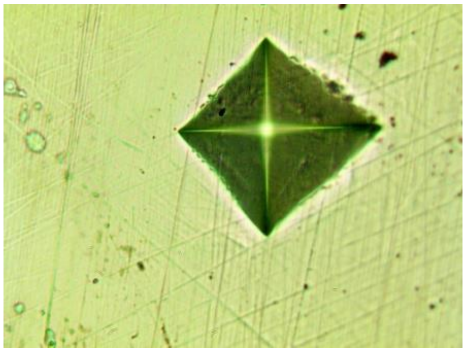


Figure 11. Result Micro-Hardness Vickers 316L

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