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Microstructural Analysis of Corrosion of Welded Joints in Stainless Steel Used in Manufacture of Bioreactors

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Abstract: *Stainless steel is widely used in the manufacture of biogas power generation plants, it is a corrosion resistant material with good toughness, ductility and low cost if compared to titanium and nickel alloys. These materials are used in the form of welded tanks, pipelines and compression equipment. Analysis of those materials and the type of welding in the manufacturing plants affects thereof durability. There for it is necessary to check the structure of the welding, the occurrence of post-sensitization of the material information and the welding structural properties of the material before and after the process. The objective of this study is to assess the effect of welding on the microstructure and structural steel property of AISI 304 and AISI 316. These stainless steels of austenitic class were welded by autogenous TIG and MIG with additional material. The welded materials were also exposed to a solution of ferric chloride 3 ($FeCl_3$). This solution was responsible for the attack of the material welded, being made as specified in ASTM 48G. Finally inspections of pits were made by weighing before and after immersion and tested in liquid penetrant.*

Keywords:*Biogas, Bioreactors, Corrosion, Stainless Steel, Welded joints.*

I. INTRODUCTION

All research related to energy efficiency technologies and the uses of renewable energy are important factors for sustainable energy [1]. The production of biogas occurs in sealed tanks, called bioreactors, with non-oxygen atmosphere, which is why they are calledanaerobic. Those tanks promote their generation by means of anaerobic bacterial digestion of the waste. They also have the role of reducing the rate of environmental contamination by confinement of the resulting gas, which no longer vented to atmosphere and treating the organic substrate material allowing further use of the natural cycle as biofertilizer [2]. Figure 1 shows a bioreactor built in stainless steel AISI 316L for this research.

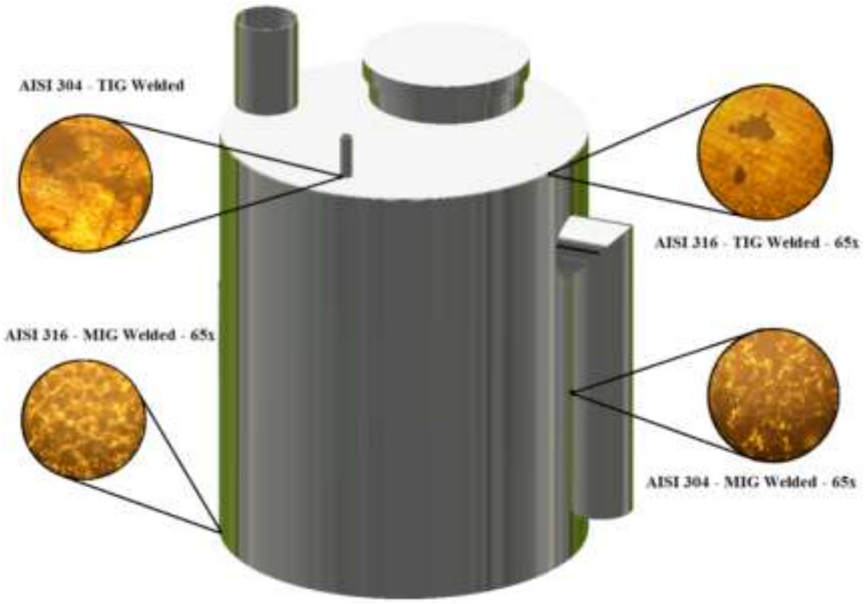

Figure1. Biorreactor –Stainless steelausteniticAISI316L.

The manufacturing material of the bioreactor and accessories such as loading and unloading ducts is usually composed of alloy steels, among them steel AISI 304 and 316 [3]. The bioreactors are manufactured by welding the plates and making large cylindrical vessel.

Four factors contribute to use austenitic stainless steels: a good weldability, corrosion resistance, oxidation resistance and mechanical properties. Also, the good appearance of stainless steel, which remains over time with a simple cleaning associated to mechanical strength, makes these materials suitable for use in tanks and bioreactors. The corrosion resistance of stainless steels in many chemical means allow its use in containers, piping, equipment components and processing of biogas. The oxidation resistance at higher temperatures makes possible its use in firing systems and biogas combustion, heat exchangers and motors. Finally, the relatively high strength at room temperature and low temperatures as well makes those material good enough to be used in components of machines and equipment which requires high reliability and performance like biogas turbine for energy production.

The corrosive attack caused by the biodigestion process on welddings and tank’s wall decrease their useful life. Wear of the bioreactor can compromise the entire system, requiring constant maintenance or even its complete replacement. The stopping of the biogas generation process determines high maintenance costs, due to the time required to reach full production again. The investigation of the erosion of materials is very important to prevent leaks and shutdowns in production [1].

The microstructure of the stainless steel provides characteristics that are really relevant when corrosion is observed in stainless steels and also in welding, since alloying elements have a strong influence on the corrosion resistance and weld ability of those steels [4].

Austenitic stainless steels are known for their excellent corrosion resistance in many aggressive environments. Other elements such as molybdenum, titanium and niobium may be added to improve resistance to general corrosion, pitting, crevice and to minimize the intergranular corrosion, stabilizing carbides present. Of the three groups, these steels are those with higher corrosion resistance. They combine low yield strength with high tensile strength and good elongation, offering the best properties for cold working [5].

For the micro structural characterization of stainless steel AISI 304 and 316 welded samples were used. Analysis of the structure of the steel allows the observation of structural changes caused by welding process. For those analyses, we used a light microscope to evaluate structural spectrometry to obtain the components of the matrix material mentioned above.

II. MATERIALS AND METHODS

The pitting corrosion that affects the austenitic stainless steel is a type of localized corrosion, with diameters ranging from micrometers to a few millimeters and can penetrate the material in a short time. These cavities may be filled by corrosion products [6]. Metals and alloys containing passive thin skin are usually susceptible to formation of pits. Austenitic stainless steels immersed in an aqueous solution containing high concentrations of halogen ions, especially chloride (Cl-) and bromine (Br-), are susceptible to localized attack and punctiform. From the point of view of oxide film’s theory, chlorine penetrates the oxide layer through pores and imperfection more easily than other ions. The pitting corrosion potential increases with ion concentration, as lower pH and increased temperature also. The addition of molybdenum (Mo) over than 2% and nickel (Ni) in AISI 316 steel, reduce the number of inclusions and precipitated, minimizing the incidence of pitting corrosion [7].

Among the most common factors in cases of localized attack is the relationship between cathodic and anodic areas. In addition, we have the differential aeration, pH variation and foreign elements in the layer of passivating material, for example oxides, present on the metal surface or formed during the corrosion process. And surface defects in passivation layer must be taken into account, since the pitting corrosion is a localized corrosion and perforating, not requiring a large surface area for its occur [8].

According with the same author, the chemical composition of corrosive means and the metallic material can greatly influence the pitting attack. Thus, the presence of chloride in the corrosive means accelerates the formation of pits in stainless steel.

Experimental studies have shown that the occurrence of pitting corrosion potential decreases with decreasing temperature. This behavior explains the origin of the critical pitting temperature in the immersion tests in the presence of an oxidant, fixing the potential for corrosion potential. The temperature that corresponds to pitting temperature where Eb potential (transpassivation potential) becomes equal to or lower than the corrosion potential. The steels are classified according to their resistance to pitting corrosion, according to the critical temperature of formation [9].

The corrosion resistance of stainless steels depends primarily on the chemical composition and microstructure. Therefore, we ought to analyze the phenomenon of passivation and the influence of alloying elements on corrosion resistance.

The passivation in stainless steel is obtained by presence of a thin layer of hydrated metal oxide on the surface. The presence of the layer depends on the nature of the means and determines the behavior of the steel. When this layer is present, the stainless steel approaches the behavior of noble metals, otherwise resembles, the activity of regular steel. The destruction of the thin layer at a specific point can lead a quickly corrosion, following types of corrosion: pitting, by gaps, intergranular and voltage [10].

Depending stainless steel type and conditions of the environment, corrosion is avoided or manifests itself quickly and destructive. Austenitic steels are considered the most resistant (having increased resistance) to corrosion in industrial environments air or acid mains, keeping the surface bright and substantially free of corrosion products.

The formation of pitting is slow, but when formed, there is an autocatalytic reaction, which produces conditions for growth of pitting, particularly when exposed to a solution rich in chloride. In the anode, within the pit, oxidation of the steel occurs with formation of ions according to Equation 1 and 2.

$$Fe^{2+}Cr^{2+}Ni^{2+} \tag{1}$$

$$Fe - Fe^{2+} + 2e \tag{2}$$

With production of positive charges in this area occurs a migration of chloride ions into the pit, which causes an increase in salt concentration, FeCl2, shown on Equations 3 and 4.

$$FeCl_2 + 2H_2O - Fe(OH)_2 + 2H^+ + 2Cl^+ \tag{3}$$

$$Fe^2 + 2H_2O - Fe(OH)_2 + 2H^+ \tag{4}$$

With increasing concentration of H+ ions, there is a decrease in pH, which causes the formation of HCl, which accelerates the corrosion process, as can see in reaction 5 and 6.

$$Fe + 2HCl - FeCl_2 + H_2 \tag{5}$$

Or:

$$Fe + 2H^+ - Fe2^2 + H_2 \tag{6}$$

The corrosive process remains with the formation of reaction $FeCl_2$ already presented in Equation 5.Sulfide inclusions reduces pit's pH, which produce the solubilization of Manganese sulfide (MnS) as can be seen in Equation 7.

$$MnS + 2H^+ - Mn^2 + H_2S \tag{7}$$

For the corrosion test in the laboratory of the welded joint was used to ASTM G48, which deals the pitting corrosion of stainless steels, a very aggressive corrosion and very common in stainless steels, may cause perforations in the metal, which can cause leaks and contamination in bioreactors [11]. The welding was performed by MIG and TIG (autogenous).

The experimental procedure was divided into:

- Metallographic analysis carried out with material welded;
- Spectrometry for the analysis of structural components;
- Corrosion tests performed according to ASTM G48, and analyzed the pitting corrosion of the joints, subjected to the welding process;
- Inspection of pitting formation, for the materials tested according to ASTM G46 [12].

Table1. Weldingof specimens.

Weldingtype used	Specimens	Thickness (mm)	Current (Amperes)	Voltage (Volts)
TIG	316	3.2	90	-
MIG	316	3.2	110	38
TIG	304	2.0	60	-
MIG	304	2.0	63	14

In the experiments we used the TIG welding process, autogenously, without adding material or MIG, where the welding are made with wire ER 308 L AS, for the ASTM 304 and ER 316 AS, to the 316. The shielding gas used was argon, and has been taken into consideration welding parameters and material thickness. The specimens were welded and a sample was obtained of each material. A top view of the welding can be seen in Figure 2 and 3.

Figure 2. Aspects of the weld in TIG and MIG steels AISI 316.

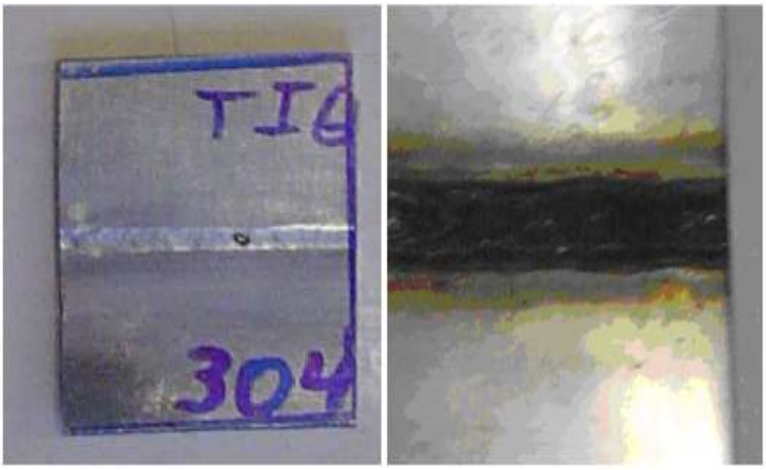

Figure 3. Aspects of the weld in TIG and MIG steels AISI 304.

For metallographic analysis a sample was removed after the welding of each material. This material has been built and subjected to metallographic, sanded, polished with alumina and diamond paste and wholesale reagent according to the standard (ASTM E407) [13]. The reagent used to reveal the structure of the material was 10 ml HNO_3 (nitric acid), 10 ml CH_3COOH (acetic acid), 15 ml HCl (hydrochloric acid) and 5 mL glycerin. After the attack we can visualize the material's microstructure.

To perform the metallographic analysis the material was cut on his welded transverse plane to the line welded, after the material was embedded in Bakelite sanded, with the following particle sizes of sandpaper 220, 400, 600, 800, 1000, 1200 and 1500. Following the polishing was performed with samples of alumina (Al_2O_3) and with diamond paste of 1 micrometers (µm). It was attacked with chemical reagents described above, according to ASTM E407 and analyzed under a microscope.

For the corrosion test was taken a sample of AISI 304 and AISI 316 steel, which were prepared according to ASTM G1, which deals with the preparation and evaluation of clean specimens for corrosion testing [14]. The cleaning was done by immersing the sample in 100 ml of nitric acid at 60°C for 20 minutes.

For further analysis of the board subjected to the ferric chloride solution III was performed using the ASTM G46, which indicates that the visual inspection is important for the identification of pits, but not sufficient, then it will use a magnifying glass for viewing under increased the formation of pits, and liquid penetrant test, for better visualization of the disposition of pits on the surface of bulk material, a procedure performed according to ASTM G46. The test piece after cleaning was performed with running water and brush nylonto remove the deposit of material in the valleys of corrosion pits arranged on the surface in accordance with ASTM G48.

Inspection pitting of metals was performed by observing the ASTM G46. After weighing the test was performed to validate the test, visual inspection and microscopically. Finally, the use of penetrating liquid to location of the points of attacked part and the density of pitting definition compared with the standard ASTM

G46. The pitting density is a factor of great importance, since with this data we can assess the depth of pits, according Figure 4 is part of ASTM G46.

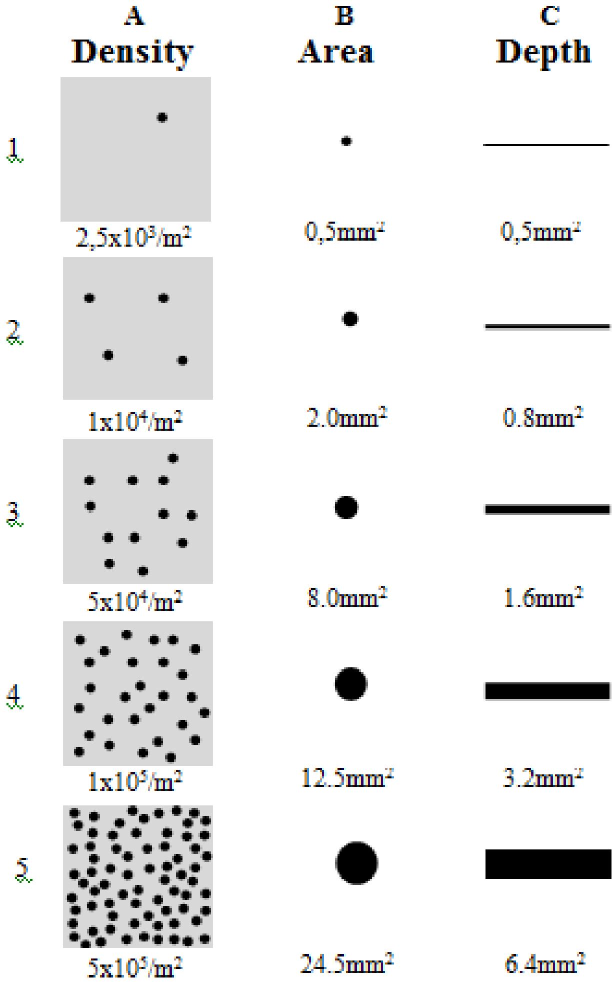

Figure 4. Density ratio, area and depth of pitting.

III. RESULTS

Metallographic Analysis results were obtained from the chemical composition of steel without welding, as shown in Table 2.

Table 2. Sample (316 and 304) Chemical Composition.

Sample	Element	Concentration (%)
AISI 316	Carbon (C)	0.04
	Silicon (Si)	0.57
	Manganese (Mn)	1.64
	Phosphorus (P)	0.067
	Sulfur (S)	0.007
	Chrome (Cr)	18.01
	Molybdenum (Mo)	2.12
	Nickel (Ni)	10.31
AISI 304	Carbon (C)	0.07
	Silicon (Si)	0.50
	Manganese (Mn)	1.35
	Phosphorus (P)	0.077
	Sulfur (S)	0.022
	Chrome (Cr)	18.00
	Molybdenum (Mo)	0.218
	Níckel (Ni)	7.32

Table 3 is given to the chemical composition of the consumable welding wire ER 308L AS, for the welding of steel AISI 304 and ER 316L AS, for the welding of steel AISI 316 in MIG welding process.

Table 3. Chemical composition of welding wire supplies.

Sample	Element	Concentration (%)
ER 308 L AS	Carbon (C)	<0,03
	Silicon (Si)	0.30 – 0.65
	Manganese (Mn)	1.00 – 2.50
	Phosphor (P)	-
	Sulfur (S)	-
	Chrome (Cr)	19.50 – 22.50
	Molybdenum (Mo)	-
	Nickel (Ni)	9.00 – 11.00
ER 316 L AS	Carbon (C)	<0.03
	Silicon (Si)	0.30 – 0.65
	Manganese (Mn)	-
	Phosphor(P)	-
	Sulfur (S)	-
	Chrome (Cr)	18.20 – 20.00
	Molybdenum (Mo)	2.00 – 3.00
	Níckel (Ni)	11.00 – 1.00

The materials AISI 304 and AISI 316 has a thickness of 2 mm and 3.2 mm respectively, so it is found a difference in mass due to variation in thickness. It was also observed a variation in the mass of heavy specimens after welding by TIG and MIG, and this is coming from the addition material applied to MIG and TIG process being autogenous does not use additional material explaining the mass variation. The welding consumables were selected to ensure the quality of the weld in MIG for steel AISI 304 wire used the ER AS 308 L and AISI 316 steel wire was used ER AS 316 L, the composition according to Table 3.

The metallographic wire welded and heat-affected zone in the steel AISI 316, are shown in Figure 5, 6, 7 and 8 respectively.

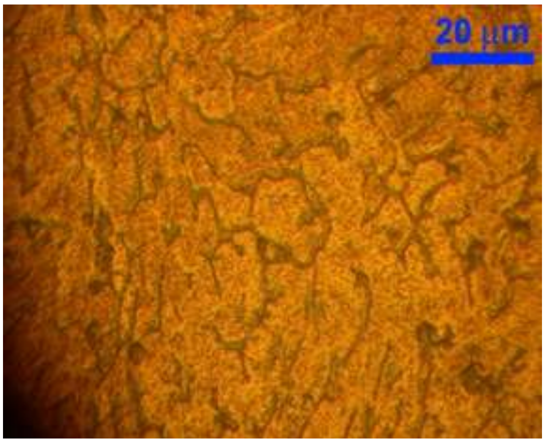

Figure 5. Sample(316)weldedprocess (TIG) autogenouscross sectionthroughthe bead (1000X).

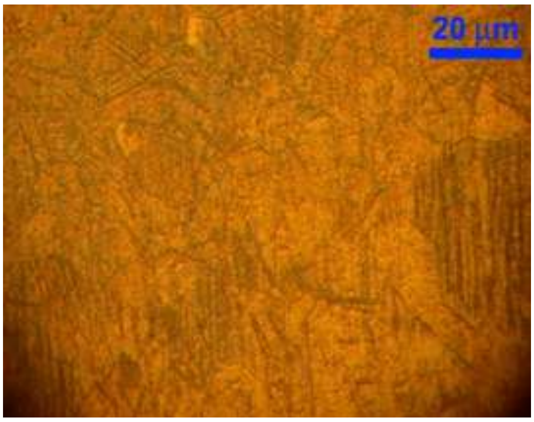

Figure 6. Sample(316)weldedprocess (TIG) ZACautogenouscross section(1000X).

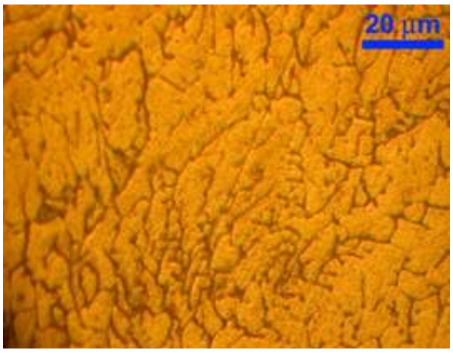

Figure 7. Sample(316)weldedprocess (MIG) cross sectionthrough thebead (1000X).

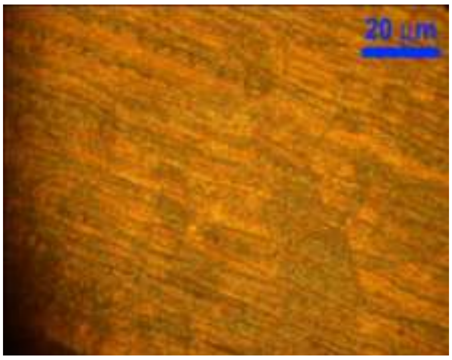

Figure 8. Sample(316)weldedprocess (MIG) cross sectionthrough thebead (1000X).

The middle of the bead, both for the MIG, TIG process as in, compositions are consist of delta ferrite structure from the melting of the material (Fig. 7). The ZAC weld for both processes continued austenitic, but with different structural, which reduced its resistance to chemical attack used to perform the metallography. The welded metalografhicbead and heat affected zone for the AISI 304 steel are presented in Figures 9, 10, 11 and 12.

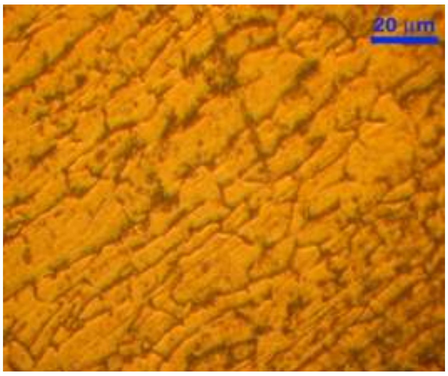

Figure 9. Sample (304) welded process (TIG) autogenous cross section through the bead (1000X).

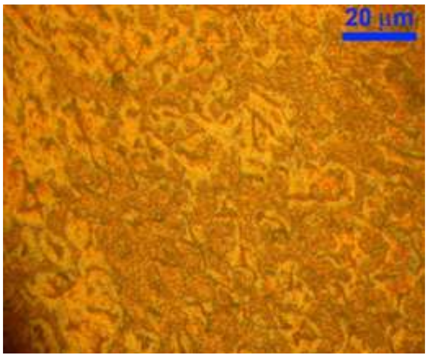

Figure 10. Sample (304) welded process (TIG) ZAC autogenous cross section (1000X).

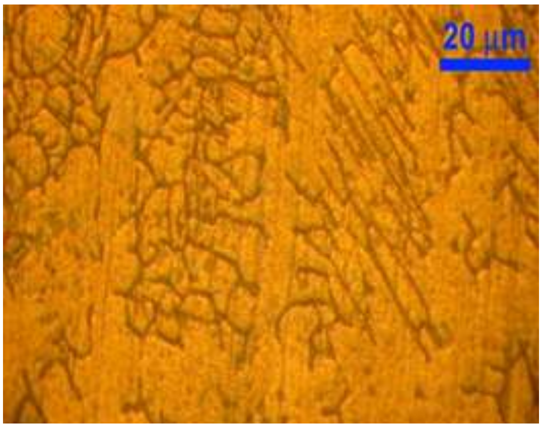

Figure 11. Sample (304) welded process (MIG) cross section through the bead (1000X).

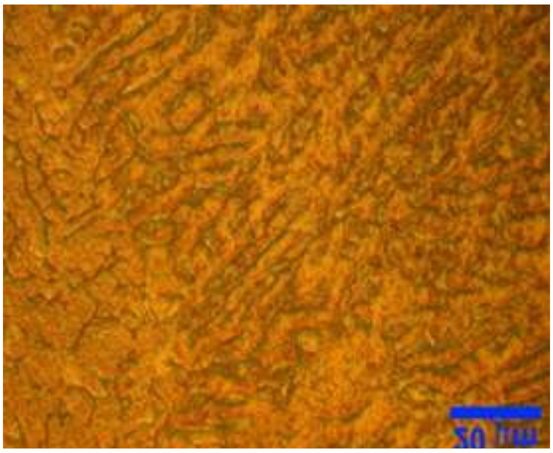

Figure 12. Sample (304) welded process (MIG) cross section ZAC (1000X).

The metallographic images in middle of the bead for the welding process TIG and MIG were presented by the Figures (9 and 11) revealing a structure comprised of delta ferrite in the ZAC welds is carried out by TIG welding can check a recrystallization from the heating during welding as shown in figure (10). In the ZAC process for the MIG welding is possible to verify the structural changes that this region weakened when exposed to metallographic etching reagent.

After preparation and cleaning provided in ASTM G1 norm, performed with nitric acid HNO_3, the material was weighed on an analytical balance accurate to four decimal places, weight in grams. The solution of $FeCl_3$, was prepared in the 900 ml of H_2O to 200 (g) of $FeCl_3$ then the samples were placed inside a beaker and immersed with the ferric chloride solution III. After were brought to a chapel that maintained the temperature of 22°C for 72 hours. The elapsed time required for the test specimens were cleaned in water and aid of a nylon brush in order to withdraw the product from corrosion.

The inspections were performed by weighing after testing, penetrating liquid and optical microscopy.We also observed the validation condition of the test, which allows a weight loss of up to 4 g/m^2.

All samples were immersed in a solution of $FeCl_3$ (ASTM G48A), the condition of maximum mass loss for validation of the test is 4g/m^2, the materials were weighed on an analytical balance to four decimal places, the mass loss is analyzed in Table 4 and in the graph of Figure 13.

Table 4. Samples of test of loss of mass for AISI 316 and AISI 304.

Typesteel 304	Initial weight (g)	Final weight(g)	The loss in weight(g/m^2)
316 TIG	49.1806	49.0999	0.5044
316 MIG	51.0417	50.4666	3.5944
304 TIG	37.3336	37.1210	1.3287
304 MIG	47.2829	46.6666	3.8519

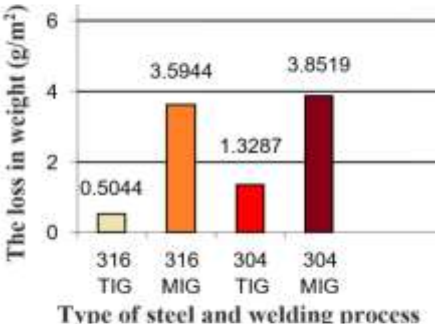

Figure 13. Mass loss for stainless steel.

From the analysis of the chart (Fig.13) it can be concluded that for the AISI 316 and AISI 304, the weight loss was less than 4 g/m² which validates the test method. Austenitic stainless steel AISI 316, showed a weight loss of 0.5044 g for TIG (autogenous) and 3.5944 g which indicated a significant difference in weight loss, demonstrating the superiority of the process applied to autogenous TIG welding of AISI 316 steel in view of corrosion resistance.

Similarly it was observed a wide variation in weight loss for the AISI 304 compared the process of TIG and MIG welding, and the mass loss to the TIG welding process was 1.3287g, process for the MIG was of 3.8519g. It is clear superiority of the TIG process for the autogenous welding of the AISI 304 steel in view of corrosion resistance of the material.

Also can be verifying the superiority of AISI 316 stainless steel welded by the TIG process on AISI 304 welded by the same process. For the mass loss was 0.5044 g for the steel AISI 316 and 1.3287 g for the AISI 304 stainless steel welded by the same process. For the MIG process was observed superiority of the steel AISI 316 with respect to the steel AISI 304, because the mass losses were 3.5944 and 3.8519 g respectively.

The specimen in Figure 14 is the material AISI 316 TIG welded by autogenous material and the figure 15 is the steel AISI 316 welded by MIG process, after testing in liquid penetrant can estimate the density of pitting. The density of pitting is estimated in view of the standards ASTM G 46. In order to compare the TIG welding process and MIG welding process, is possible to demonstrate the superiority of the TIG process for the AISI 304 steel in terms of density test according to pitting can be seen in Figures 14 and 15.

Figure 14. Test of the steel penetrating liquid AISI 316 TIG welded.

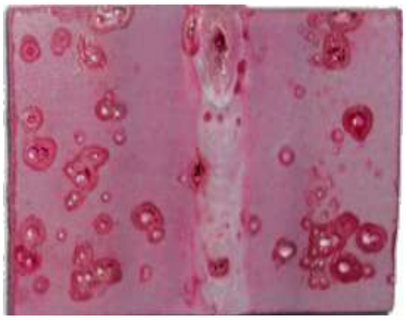

Figure 15. Test of the steel penetrating liquid AISI 316 MIG welded.

The samples of AISI 316 in Figure 16 and the Figure 17 were welded by autogenous TIG and MIG, respectively. After testing in penetrating liquid can be estimated the density of pitting in view of ASTM G46 standards. Comparing the processes of autogenous TIG welding and MIG in the same type of steel was possible to demonstrate the superiority of the TIG process for steel quoted in terms of density of pitting.

Figure 16. Test of the steel penetrating liquid AISI 304 (MIG).

Figure 17. Test of the steel penetrating liquid AISI 304 (TIG).

The density of pitting was evaluated based on the standard of ASTM G46 shown in Figure 3, this parameter is used to evaluate the depth of drilling. The results are shown in Table 7.

Table7. Resultsof density area and depth.

Steel and welding process	Density (m^2)	Area (mm^2)	Depth (mm)
AISI 316 TIG	1 x 104/m^2	2.0	0.8
AISI 316 MIG	5 x 104/m^2	8.0	1.6
AISI 304 TIG	1 x 104/m^2	2.0	0.8
AISI 304 MIG	5 x 104/m^2	8.0	1.6

The images shown below indicate the formation of pits on the surface of AISI welded by TIG welding as shown in Figure 18.

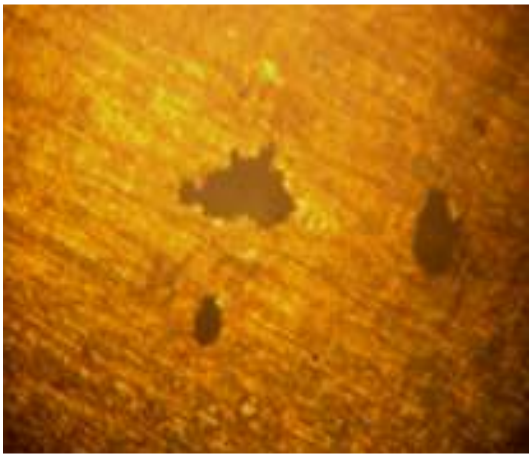

Figure 18. Picture of the formation of pitting of stainless steel AISI 316 (65x) welded by the process TIG.

Formation of pitting in AISI 316 welded by MIG process as shown in Figure 19.

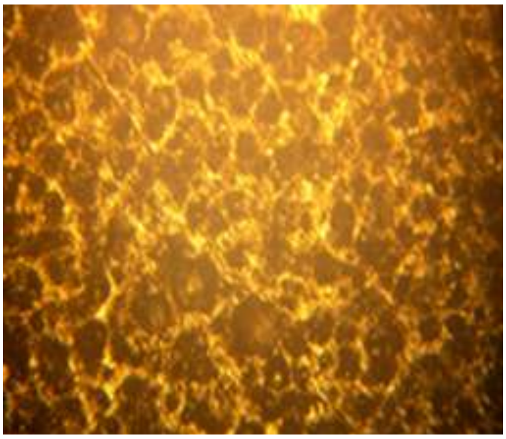

Figure 19. Image formation of pitting in stainless steel AISI 316 (65x) MIG welded.

In Figure 20 we can see the formation of pitting in AISI 304 welded by the TIG process.

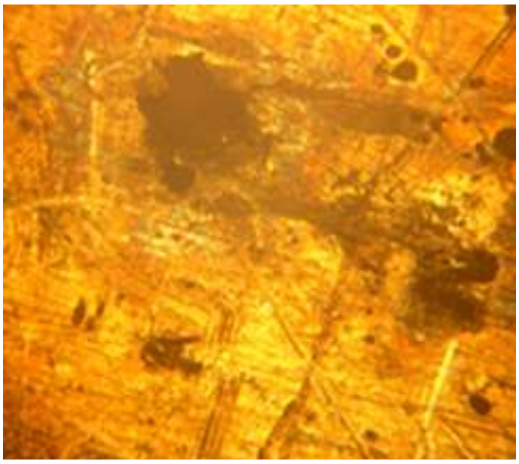

Figure 20. Formation of pitting in AISI 304 welded by the TIG process (65x).

In Figure 21 we can see the formation of pitting in AISI 304 welded by MIG process.

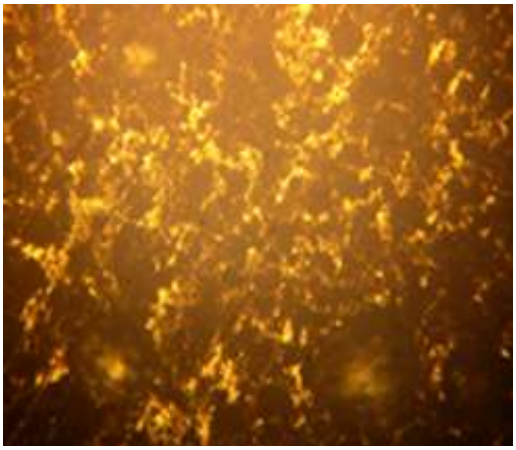

Figure 21. Formation of pitting in the weld bead of AISI 304 (65x).

IV. CONCLUSION

The austenitic stainless steel AISI 316 and AISI 304 have proven that it's indicated for use in construction of bioreactors when welded by TIG autogenic process and when the corrosion resistance are the objective. The AISI 316 stainless steel welded TIG autogenic process proved very resistant to solution, being almost free of pits after 72 hours immersion in $FeCl_3$ solution, has the same material when welded by MIG process were susceptible to the formation of pits mainly in ZAC is then given the TIG autogenic process for welding of plates used in the construction of bioreactors.

When compared AISI 304 and AISI 316, both welded by TIG autogenic process, it's possible to check a higher weight loss of AISI 304, with the weight loss of 1.3287 g/m² and AISI 316 with the weight loss of 0.5044 g/m² on weighing the material. It shows that the AISI 316 steel have a performance better, as can be observed a higher incidence of pitting in AISI 304.The structures presented in the welding of AISI 316 and AISI 304 are made in the area of delta ferrite and austenite fused bead with morphological changes in the heat affected zone.

The material AISI 316 welded by the TIG process were very resistant to corrosion. There was a weight loss of 0.5044 g/m² and a density of small pitting, as MIG welded when there was a considerable loss of mass, value of 3.5944 g/m^2 and is much higher compared to the TIG process for the same steel.

Similarly were the AISI 304 steel welded by TIG process proved more resistant to attack with ferric chloride III, in comparison with MIG and the weight loss of 1.3287 g/m² for the TIG process and 3.8519 g/m^2 this highlights the superiority of autogenous TIG welding corrosion resistance in the question. It's also possible to check a preferential attack in ZAC of MIG welding applied to the steel AISI 304, became evident the fragility generated by the welding process. Thus is indicated the TIG welding process (autogenous) for welding of stainless steel AISI 304 and AISI 316, for improved performance when exposed to chloride solutions, which have also a better finish bead avoiding the use of abrasion to remove the same, improving the resistance of the material and the finishing as well.

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