The Effects of Cold Working on Corrosion Resistance of Implant Materials

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Abstract
This experimental work regarding the effect of cold working on corrosion resistance of stainless steel 316L as application metal materials in biomedical. The research aim to investigate the influence of deformation that occurs through a process of cold working (rolling) on the corrosion resistance as implant material stainless steel 316L. At the beginning, this material will perform cold working (rolling) to make several products with different thickness. Next, the products will perform different method to find the mechanical properties testing (hardness) and corrosion testing (dipping into Hydrochloric acid HCl). Lastly, the characteristics of the product will be achieved by micro structure observation. Optical microscope will be used. The result it showed the effect of thickness reduction during rolling process for mechanical properties (hardness) from specimens test is increasing relevant to hardness of specimens. The value of hardness on the degree of deformation is expressed in the strain that shows the value of hardness of the material will continue to increase along with the amount of strain that occurs. Pore located in the grain becomes larger, so it's getting easier corrosion occurs when acid liquid (HCl) is applied.
Keywords: Cold working, corrosion, stainless steel 316L

1.0 Introduction

Any time a foreign material is placed inside the human body, the manner in which that material will affect the body must be considered. There are many causes that contribute to the corrosion of metals when placed inside the human body. After surgery the pH surrounding the implant in reduced to a pH between 5.3-5.6 typically due to trauma of surgery. Infectious microorganisms and crevices formed between components can reduce oxygen concentration, both of which contribute to the corrosion of the implant. One serious problem with all implants is the common and serious condition called nickel allergy dermatitis (Efendy, Hady, 2010).

The research aims to investigate the influence of deformation that occurs through a process of cold working (rolling) on the corrosion resistance of 316L stainless steel as an implant material. When 2 articulated implants in contact with one another some wear cause by the friction can happen. Corrosion cause by friction is a big problem since it releases metallic ions that could cause a tissuelar reaction. Contact area was measured and contact stress was analyzed for the metal-on- metal components. Stainless steel is the generic name for a number of different steels used primarily because of their corrosion resistance. All stainless steels share a minimum percentage of 10.5% chromium. Chromium is always the deciding factor, although other elements, particularly nickel and molybdenum, are added to improve corrosion resistance. The success of the material is based on the fact that it has one unique advantage. The chromium in the stainless steel has a great affinity for oxygen, and will form a film of chromium oxide on the surface of the steel at a molecular level. The film itself is extremely thin, about 130 Angstroms and one Angstrom is one millionth of a centimeter. This layer is described as passive (does not react or influence other materials), tenacious (clings to the layer of steel and is not transferred elsewhere) and self-renewing (if damaged, more chromium from the steel will be exposed to the air and form more chromium oxide). Figure 1 presents the total hip joint prosthesis components.

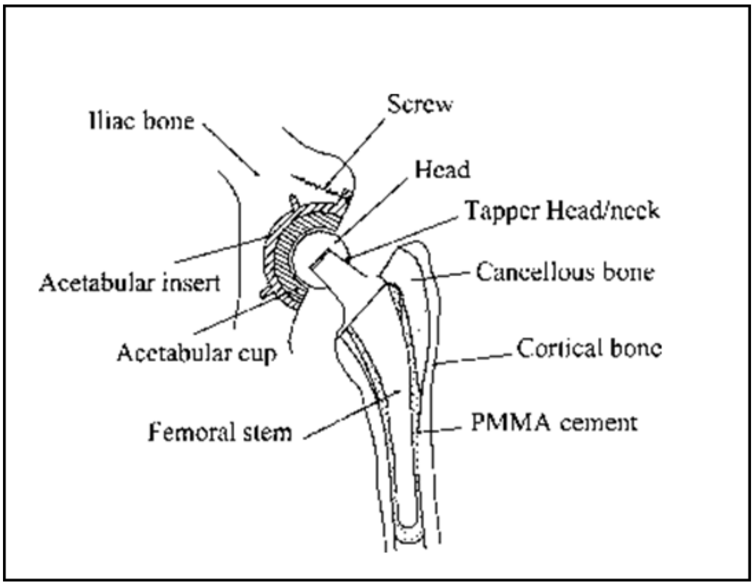

Figure 1: Total hip joint prosthesis components (L. Duisabeau, 2004)

1.1. Problem Statement

Effect of Cold Working deformation of austenitic alloys at room or slightly elevated temperature produces an increase in strength accompanied by a decrease in elongation value. Alloys 316L flat rolled products are generally available in the annealed condition as implant material as health and safety issues. High risk to human body effect the corrosion on implant material. This project presents a study influence of deformation that occurs through a process of cold working (rolling) on the corrosion resistance of 316L stainless steel as an implant material.

1.2. Objectives

The research aims to investigate the influence of deformation that occurs through a process of cold working (rolling) on the corrosion resistance of 316L stainless steel as an implant material. The objectives of the research are:

i. Development of properties of stainless steel material for implant material in biomedical system
ii. To investigate the influence of deformation that occurs through a process of cold working (rolling) on the corrosion resistance of 316L stainless steel as an implant material.
iii. Study the characteristics implant materials products.

1.3 Scope

The goal of the project to be achieved are can knowing the influence of cold working (rolling) of deformation to change in structure of the microstructure, hardness and corrosion resistance expressed by the rate of corrosion.

1.3.1.1 The scope of this project is limited only to the problem of fabrication and characterization of implant material (SS 316L).

1.3.1.2 Testing mechanical properties include hardness.

1.3.1.3 Micro and macro structure observation using Optical Microscope.

2.0 Material and Methods

At present, three metallic groups of materials are widely used in the prostheses Fabrication: 316L SS austenitic stainless steels, cobalt–chromium alloys and Ti–6Al–4V titanium alloys, much used since the 1970s because of their good mechanical properties combined with high biocompatibility (L. Duisabeau 2004). Type 316 is an austenitic chromium nickel stainless steel containing molybdenum. This addition increases general corrosion resistance, improves resistance to pitting from chloride ion solutions, and provides increased strength at elevated temperatures. Properties are similar to those of type 304 except that this alloy is somewhat stronger at elevated temperatures. Corrosion resistance is improved, particularly against sulfuric, hydrochloric, acetic, formic and tartaric acids; acid sulfates and alkaline chlorides. Type 316L is an extra-low carbon version of type 316 that minimizes harmful carbide precipitation due to welding. Typical uses include exhaust manifolds, furnace parts, heat exchangers, jet engine parts, pharmaceutical and photographic equipment, valve and pump trim, chemical equipment, digesters, tanks, evaporators, pulp, paper and textile processing equipment, parts exposed to marine atmospheres and tubing. Type 316L is used extensively for weldments where its immunity to carbide precipitation due to welding assures optimum corrosion resistance (AK Steel Data Sheet, 2007).

2.1. General Properties

Alloys 316 (UNS S31600), 316L (S31603) and 317L (S31703) are molybdenum-bearing austenitic stainless steels which are more resistant to general corrosion and pitting/crevice corrosion than the conventional chromium- nickel austenitic stainless steels such as Alloy 304. These alloys also offer higher creep, stress-to-rupture, and tensile strength at elevated temperature. In addition to excellent corrosion resistance and strength properties, the alloys 316, 316L, and 317L Cr-Ni-Mo alloys also provide the excellent fabricability and formability which are typical of the austenitic stainless steels (Sandmeyer Steel Company, 1952).

Austenitic or nonmagnetic stainless steels, are classified in the 200 and 300 series, with 16% to 30% chromium and 2% to 20% nickel for enhanced surface quality, formability and increased corrosion and wear resistance, and are nonhardenable by heat treating. These steels are the most popular grades of stainless produced due to their excellent formability and corrosion resistance. All austenitic steels are nonmagnetic in the annealed condition (depending on the composition, mainly the nickel content, austenitics do become slightly magnetic when cold worked). Austenitic stainless steel grades include: Type 201, Nitronic 30, 301, 304, 305, 309S, 316, 316L, and 321. Austenitics are used for automotive trim, cookware, food and beverage equipment, processing equipment and a variety of industrial applications (AK Steel Data Sheet, 2011).

2.2. Specifications

Types 316 and 316L Stainless Steel sheet and strip are covered by the following specifications: Type 316 Type 316L AMS 5524 AMS 5507 ASTM A 240 ASTM A 240 ASTM A 666 ASTM A 666. The composition ranges, mechanical properties and grade specifications are tabulated in Table 1, Table 2 and Table 3 respectively.

Table 1: Composition ranges for 316L stainless steels (University of Puerto Rico Mayaguez, 2003)

Grade	Tensile Str (MPa) min	Yield Str 0.2% Proof (MPa) min	Elong (% in 50mm) min	Hardness	
				Rockwell B (HR B) max	Brinell (HB) max
316L	485	170	40	95	217

Table 2: Mechanical properties of 316L stainless steels (University of Puerto Rico Mayaguez, 2003)

Grade	Tensile Str (MPa) min	Yield Str 0.2% Proof (MPa) min	Elong (% in 50mm) min	Hardness	
				Rockwell B (HR B) max	Brinell (HB) max
316L	485	170	40	95	21 7

Table 3: Grade specifications for 316L stainless steels (University of Puerto Rico Mayaguez, 2003)

Grade	UNS No	Old British		Euronorm		Swedish SS	Japanese JIS
		BS	En	No	Name		
316L	S31603	316 S11	-	1.4404	X2CrNi Mo1 7-12-2	2348	SUS 316L

2.3 Cold Working Process

Cold rolling is a process by which the sheet metal or strip stock is introduced between rollers and then compressed and squeezed as shown in Figure 2. The amount of strain introduced determines the hardness and other material properties of the finished product. The advantages of cold rolling are good dimensional accuracy and surface finish. Cold rolled sheet can be produced in various conditions such as skin-rolled, quarter hard, half hard, full hard depending on how much cold work has been performed. This cold working (hardness) is often called temper, although this has nothing to do with heat treatment temper.

Figure 2: Rolling Process

In skin rolling, the metal is reduced by 0.5 to 1% and results in a surface that is smooth and the yield point phenomenon excessive stretching and wrinkling in subsequent operations, is eliminated. This makes the metal more ductile for further forming and stretching operations. Figure 3 shows the effect of cold working on mechanical properties.

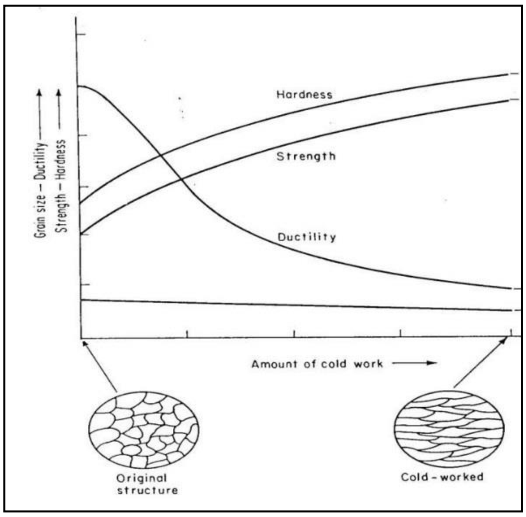

Figure 3: Effect of cold working on tensile strength, hardness, ductility and grain size (the curve below ductility represents the change

in grain size)

3.0 Methodology

In the experiment, the corrosion test were performed dipping in acid solution Hydrochloric acid (HCl). Measurement taken using conventional method and observation with optical microscope to find the microstructure. . Characterization and material performance obtain when the material after on cold working (rolling) operation. To understand the relative implant material characterization after cold working, it is necessary to completely understand the corrosion mechanism and mechanical properties. Table 4 represents the sample category and Figure 4 shows the stainless steel 316 L used in this study.

Table 4: Sample Category

Sample	Dimension (mm)
A	50x50x2.5
B	50x50x2.5
C	50x50x2.6
D	50x50x2.7
E	50x50x2.8
F	50x50x2.8

Figure 4: Stainless Steel 316L (sheet)

4.0 Results

4.1. 6 Sample Stainless Steel 316L (50mmx50mm)

Table 5 shows the measurement the thickness of specimen before and after the rolling process. In this process each sample was rolling to reduce the thickness and percent reduction of thickness of each sample are different.

Table 5: Measurement the thickness of specimen before and after the rolling process

Sample	Origin Thickness, t_0 (mm)	Final Thickness, t_f (mm)	Change of Thickness, Δt (mm)	Percentage of Thickness Reduce (%)
A	2.5	2.5	0	0
B	2.5	2.4	0.1	4
C	2.6	2.35	0.25	9.62
D	2.7	2.2	0.5	18.52
E	2.8	2	0.8	28.57
F	2.8	1.7	1.1	39.29

4.2 Strain of Each Sample

Table 6 shows the calculation value for strain from the equation

$$\in = \frac{\Delta t}{t}$$

below:

Table 6: Value strain of each sample

Sample	Strain, ϵ
A	0
B	0.04
C	0.09
D	0.18
E	0.28
F	0.39

The strain value increases of each sample. This is because; when the greater the percent reduction of thickness, the strain will increase. Figure 5 shows the strain versus sample results. From the graph, it shows that the sequence of sample based on their thickness which is sample F (0.39) is greater the percent reduction of thickness than sample B (0.04).

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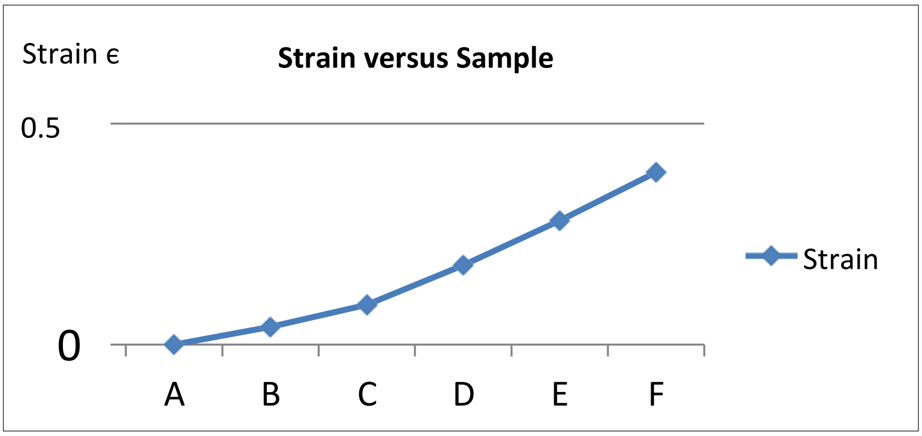

Figure 5: Graph of strain versus sample

4.3 Percentage Reduction of Each Sample

Figure 6 shows the percentage reduction versus sample results. From the graph, it shows that the sequence of sample based on their percentage reduction of thickness which is sample F (39.29%) is greater reduction of thickness than sample B (4%)

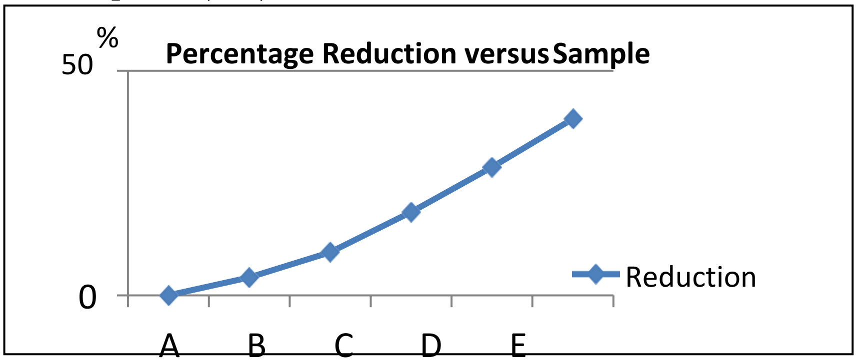

Figure 6: Percentage reduction versus sample

4.4 Hardness Test

Table 7 shows the result of value hardness test in VHN (Vickers Hardness Number). From the result, it shows five time hardness test act for each sample and the best data are taken from the average value. Figure 7 shows the VHN versus sample results. From the graph, it shows that the sequence of sample based on their average data from the hardness test value which is sample F (295.2 VHN) is greater than sample A (169.06 VHN).

Table 7: Vickers hardness test result (VHN)

Test	A	B	C	D	E	F
1	174.8	182.7	220	231	285	285
2	160.7	182.7	231	243	270	285
3	174.8	182.7	220	256	270	285
4	167.5	191.3	231	243	285	302
5	167.5	191.0	231	243	270	302
Average	169.06	186.08	226.6	243.2	276	295.2

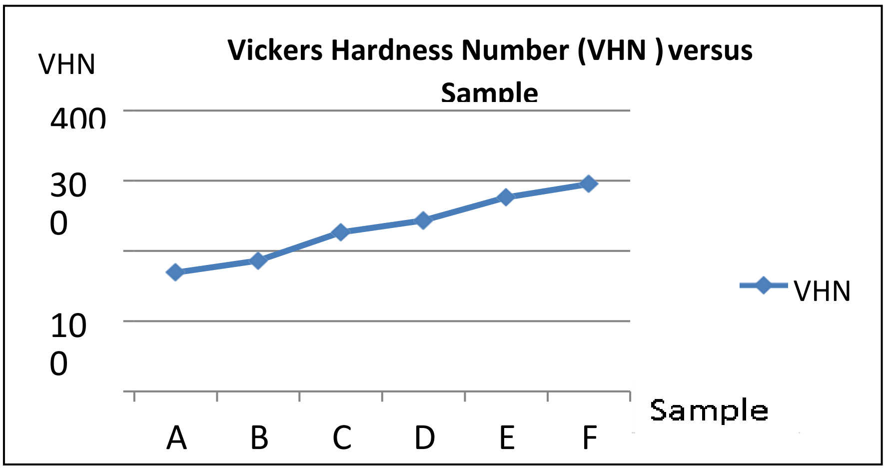

Figure 7: Vickers Hardness Number versus sample

4.5 Optical Microscope

Figure 8 shows the grain boundary for the samples is taken by optical microscope. This figure shows the origin grain boundary disposition without the rolling process. For sample B the figure shows the transformation grain boundary size dislocation after the rolling process for 4% thickness reduction. The transformation grain boundary size dislocation after the rolling process for 9.62% thickness reduction and sample D shows the transformation grain boundary size dislocation after the rolling process for 18.52% thickness reduction. While E and F shows the transformation grain boundary size dislocation after the rolling process for 28.57% thickness reduction and rolling process for 39.29% thickness reduction.

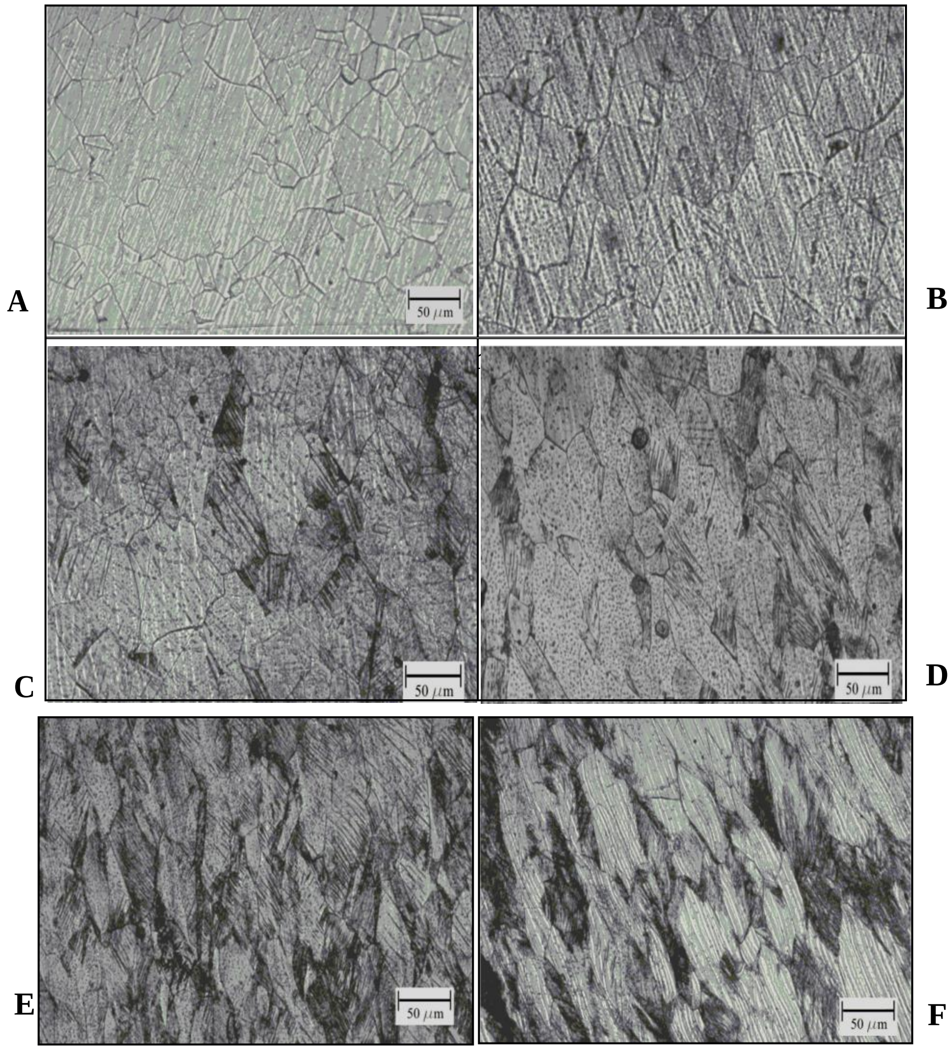

Figure 8: Microstructure of sample A, B, C, D, E and F

4.6 Corrosion Resistance Test

For this test discussed about the result gained from the corrosion resistance test and the analysis from testing. For process of corrosion resistance, it's just using two samples and dipping into the HCL solution to compare the reaction on the surface of the rolling sample between samples has not already rolling.

5.0 Discussion

5.1. Mechanical Properties (hardness)

Hardness tests was carried out on 5 points made at random. Hardness testing conducted to determine the hardness values before and after deformation. Besides hardness testing is also conducted to determine changes in the value of hardness on the degree of deformation is performed.

From the graph (Figure 7), the value of hardness on the degree of deformation is expressed in the strain shows the value of hardness of the material will continue to increase along with the amount of strain that occurs. This shows that a given strain increases the value of hardness and increase the strength of the material. The graph showed effect thickness reduction during rolling process for mechanical properties (Hardness) from specimens test. Reduction of thickness is increasing relevant to hardness of specimens. It is effect of increased amount of dislocation in materials during rolling process.

5.2. Cold Rolling Effects on Micro-structure

From the optical microscope observation in Figure 8, showed a different grain form and size after and before rolling process. Grain form can give different effect for properties of materials. Microstructure resulting from cold rolling microstructure is the result of plastic deformation that occurs in the lattice structure. Micro structural observations made by metallographic techniques with imaging using optical microscope.

The microstructure of the image obtained and shown in Figure 8 at the plates that have strain points 0:04 until 0:09 which are formed still point equated. Then with the increasing strain that occurs is the strain to strain 00:41 and 00:17 deformed grains tend to be flattened and elongated (elongated grains). Grain shape change indicates that plastic deformation occurred during the shift position of the atoms in the lattice structure which causes the formation of grain structure that is different from the initial condition.

When deformation occurs, the movement of dislocations in the shear field will be limited by the roll on both surfaces so that the shear field on each grain will rotate to the plane of rolling so as to produce metallographic texture. It also occurs at grain boundaries which tend to result in alignment with the field of rolling due to the shift of the dislocation. Greater degree of deformation of the micro structure of the rolling results will further show that the relative grain boundary parallel to the rolling fields and orientation in the direction of rolling.

5.3. Cold Rolling Effects on Corrosion

The effect of sample by HCl solution shows the corrosion can be seen on the side surface of the sample only (Figure 9). It is because not from the result of cold working process for this experiment, but due to the effects of cutting machines from the samples supplier. While in other sample, shows the effects of corrosion for the samples were rolling, causing the entire surface has been eroded. This is caused by the sample has been undergoing a process of cold working in this experiment.

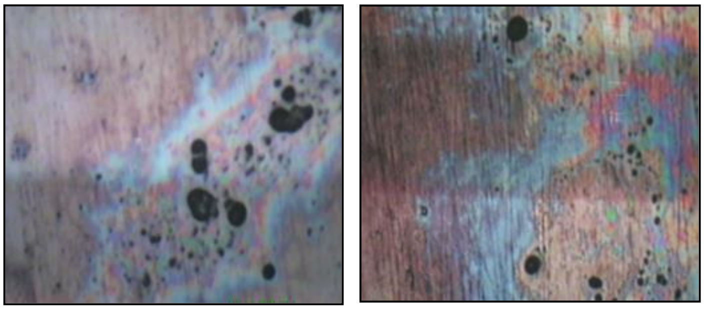

Figure 9: Effect HCl to corrosion in a surface of specimens

6.0 Conclusion

Form the results, it can be concluded that the achievements in the development of the material properties of stainless steel for biomedical implant systems have been successful in doing, which in this study, as an implant material in a process through which cold working (rolling). After going through this process of change in mechanical properties can be viewed in a hardness test. It also changes the grain of the material can be seen through the optical microscope. Through this process, the greater of percentage reduced changes in thickness is increased the hardness values, as well as changes in the grain boundary material which can be seen on the results of which the extension of the greater grain size after the rolling process. Indirectly this is one way to improve the content of hardness, strain and quality of the products. This can be evidenced in the results Table 4 and Table 5.

To determine the effect of deformation by cold working process (rolling) on the corrosion resistance of 316L stainless steel as an implant material in which the corrosion resistance tests carried out in which the material was dipping in HCl acid solution to determine the corrosion rates before and after the rolling process done. As a result was found in the levels of corrosion becomes more rust as compared with materials that do not do the rolling process. The whole of this experiment can be concluded that this method can only improve the hardness of the material, but for the rust resistance is weak. This is because the effect from the cold working (rolling) process that is done.

7.0 Recommendations

Recommendation and suggestion for future work not only used for the extension of the project but also share the relevant information to others. This recommendation made to complete the scope that does not be covered in the research. The future work may be contributing more concrete result for research and development soon.

From what has been discussed in the conclusions above, cold working (rolling) only can increase the hardness of material but for the corrosion resistant it is very weak. Then as a reserve in this project, the study may proceed with due the heat treatment. Heat treatment is a thermal cycle caused by welding, have little influence on mechanical properties of 316L stainless steel for this experiment. Although the strength and hardness can be increased by cold working (rolling), which will also rates reduce ductility. Usually of stainless steel full solution annealing will restore the material to its original condition, removing alloy segregation, sensitisation, sigma phase and restoring ductility after cold working. It controlled heating and cooling of metals to alter the physical and mechanical properties without changing the product shape and reduce of stress concentration. However the temperature and treatment time depends on the size of which is usually for the stainless steel temperature between 980°C to 1050°C with is 2/3˚C from the melting point temperature.

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