Effect of Acid Pickling on Microstructure and Surface Roughness of 316L Stainless Steel

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Abstract— Hydrofluoric and Nitric acids solution with additional of water were tested as pickling solutions for 316L stainless steel before application of hydroxyapatite coatings. Pickling was explored at room temperature for 5-10-15 min. The microstructure of surface layer, surface roughness and microhardness of 316L stainless steel treated by acid pickling were investigated in this study. The microstructure and surface morphology were examined by an optical microscope (OM) and scanning electron microscope (SEM), while surface roughness was measured by using perthometer (Mahr). The experimental result showed that increment of surface roughness when longer exposed to the acids solution. Microhardness values were higher than annealed sample. It is expected that surface treated 316L stainless steel strongly adhere to the coating after applying acid pickling treatment.

Keywords—316L stainless steel; acid pickling; microstructure

1. INTRODUCTION

Austenitic stainless steel of the type of 316L is widely used as the most economical alternative for orthopaedic implants compared to Co-Cr or Ti alloys since to lower the expenses of public health services [1-3]. 316L stainless steel is widely used in orthopaedic surgeries like joint replacement and fracture due to the high corrosion resistance, high mechanical strength and excellent biocompatibility [4-6]. The provided 316L stainless steel has a low carbon content to improve corrosion resistance in body fluids [7-9]. Besides, the existence of 2 to 4wt.% Mo build-ups the resistance to pitting corrosion. Nevertheless, austenitic stainless steel has poor wear resistance and low mechanical strength based on the amount of the chemical composition and austenite microstructure [10-12], leads to the major difficulties hindering its application [13-16]. Also, burr, oil contamination and oxide scale that formed through the machining practice, are also undesirable for the following processes such as assembly and welding. Surface treatment such as acid pickling is applied to eliminate the surface impurities, such as oxidation scale, rust and stains after sectioning and annealing process [17-19]. It is an efficient method for chemical removal of surface oxides or other foreign matters by immersion in an acid aqueous solution [17, 20-22]. In the previous study, pickling treatment is one of the methods to adhere the coating onto metal substrate [23, 24]. It stated by [23], the coating layer on the pickled sample was denser than compared to the non-pickled sample. However, the coating application of the pickled stainless steel did not mention in this report. The objectives of this study were to introduce a pickling solution for 316L stainless steel and to identify the effect of this pickling treatment on microstructure and surface roughness of the substrate. Microstructure and surface

morphologies were observed by the optical microscope (OM) and scanning electron microscope (SEM). Besides that, surface roughness and microhardness have been measured by using perthometer and Micro Vickers Hardness Tester.

2. METHODOLOGY

The metal used for this study was a commercial 316L stainless steel disc of 3 mm thickness and 15.8 mm diameter. The chemical composition is shown in Table 1. The base metal was processed by wrought process. All the samples have been undergone annealing process at 1120 ^{o}C for two hours in high purity argon flow atmosphere and cooled inside tube furnace for at least four hours. Then, annealed samples were pickled into 7.5 ml nitric acid (HNO_3) with additional of 5 ml hydrofluoric acid (HF) and 37.5 ml of water for removing the oxide scales and unwanted substances onto the sample's surface. The acid pickling process was conducted room temperature with different immersion times; 5-10-15 minutes. After being pickled, all the samples were cleaned using distilled water in an ultrasonic agitation bath for 10 min and were dried in air. The microstructure of samples was visually checked by the optical microscope (OM) and scanning electron microscope (SEM). The values of surface roughness before and after acid pickling were measured using Perthometer. Also, the microhardness on the surface of every sample was measured by Micro Vickers Hardness Tester with a load of 1000 g and holding time of 30 s. The reading for every sample taken at least five times, the mean value was concluded as the final result for surface roughness and microhardness.

Table 1: Chemical composition (wt.%) of the employed 316L stainless steel.

Component	C	P	Cr	Mo	Ni	S	Si	Mn	Fe
Base metal	0.02	0.032	19.00	2.48	13.60	0.01	0.54	1.65	Bal.

3. RESULT AND DISCUSSION

Fig. 1 (a, b, c and d) show the images of the samples before acid pickling (after annealing), after immersion in an acid solution for a 5-10-15 min. From Fig. 1 (a), annealed samples had discoloration phenomena when the samples turned to dark greenish after undergone annealing process in high purity argon atmosphere [25-28]. The annealing process was conducted in an argon atmosphere to prevent oxidation happened to the samples. Oxide scales were formed onto the surface after the annealing. The scanning electron microscope (SEM) images as shown in Fig. 2 (a and b) compared the cross section view of before and after annealed samples. It supported the statement that after annealing process or known as residual stress treatment, oxide scales formed on the surface. As shown in these Fig. 1 (b, c and d), samples immersed in the acid solution for three different immersion time (5-10-15 min). The oxide scales formed during the annealing is subsequently removed by pickling in acid solution [29-31]. From Fig. 1 (d), immersion in an acid solution for 15 min was able to remove the oxide scales fully. Microscopic images in Fig. 3 (a, b, c and d) were captured by using an optical microscope (OM) proved that immersion in acid pickling up to 15 min could remove oxide scales. The effect of acid pickling treatment on stainless steel samples, slightly roughen the surface. Prolong the immersion time in the acidic environment had slightly increased the surface roughness as shown in Fig. 4. This is probably due to longer exposure to an acid solution which cause sample became eroded. The slightly increment of surface roughness by pickling treatment would significantly improve coating layer and corrosion resistance [23, 32]. Fig. 5 shows a graph of the microhardness in the surface layer at different condition. From the Fig. 5, the microhardness for annealed samples are lower compared to as-received samples due to stress relieve during annealing process [33, 34]. While pickled samples at different time immersion had almost the equal microhardness, which was significantly increased compared to as-received sample. This is due to values of microhardness that are very sensitive to dislocation density which enhanced after plastic deformation [35-38]. Enhancement of microhardness for pickled samples indicated the higher dislocation density on the surface compared to annealed sample.

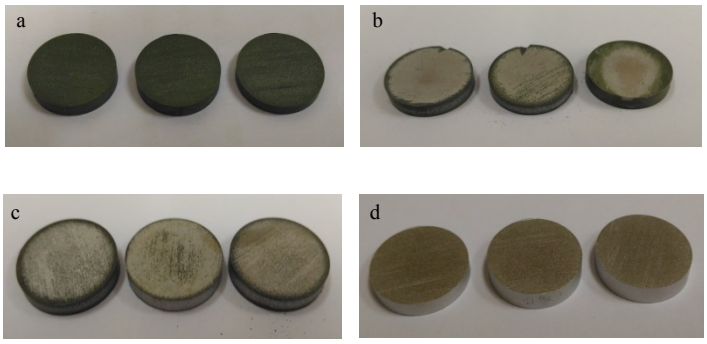

Figure 1: Images of samples (a) before acid pickling (after annealing), (b) after immersion in an acid solution for 5 min, (c) after immersion in an acid solution for 10 min, (d) after immersion in an acid solution for 15 min.

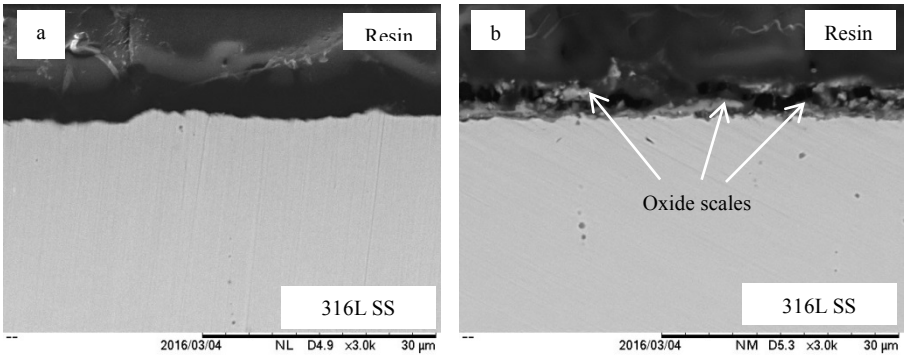

Figure 2: Cross section view of samples (a) before annealing process, (b) after annealing process at 1120 ^{o}C.

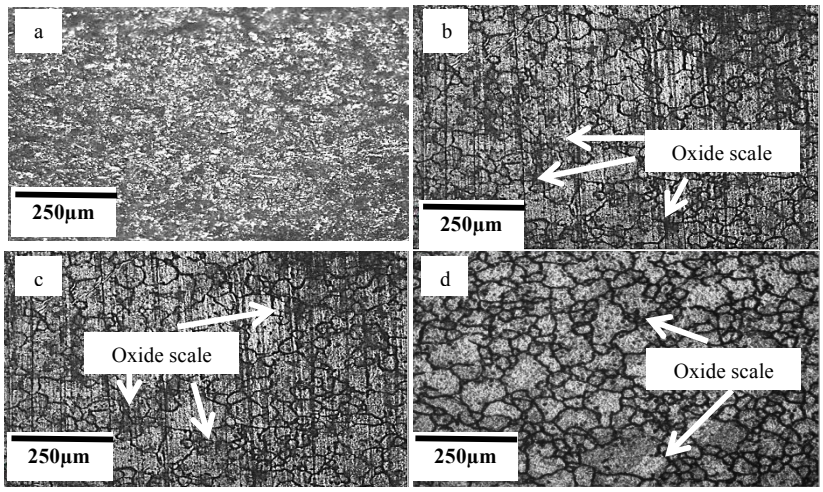

Figure 3: Microscopic images of samples (a) before acid pickling, (b) after immersion in an acid solution for 5 min, (c) after immersion in an acid solution for 10 min, (d) after immersion in an acid solution for 15 min.

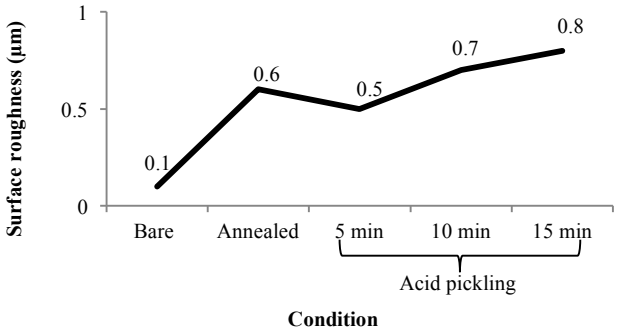

Figure 4: Surface roughness on different samples.

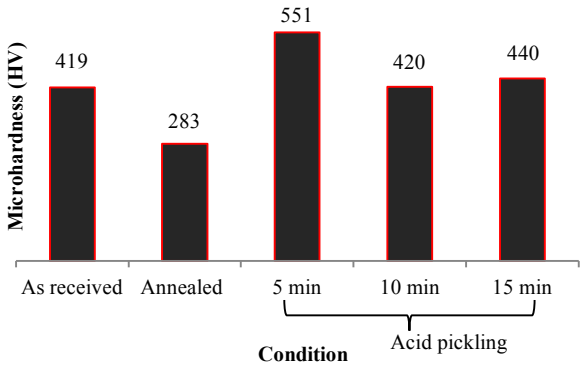

Figure 5: Microhardness on the surface in different condition.

4. CONCLUSION

In this present work, microstructure, surface roughness and microhardness were studied when a surface treatment acid pickling treated onto annealed 316L stainless steel. From the result, the pickling solution with the composition of 7.5 ml HNO_3, 5 ml HF and 37.5 ml of H_2O and suggested the time for application of the pickling solution is15 min at room temperature. OM observations showed that the oxide scales formed after annealing removed after 15 min immersion in acid solution. Other than that, the surface roughness has slightly increased while microhardness showed higher values after pickling treatment. Introducing pre-treatment as acid pickling would be able to adhere coating formation on stainless steel.

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