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Improvement of Electrolytic Etching on Stainless Steel Grade SUS 304

Apiwat Muttamara [a*], Natta Naulpring [a] and Somsak Siwadamrongpong [b]

[a] *Department of Industrial Engineering, Faculty of Engineering, Thammasat University, Pathumtani, THAILAND*

[b] *Manufacturing Engineering, Faculty of Engineering, Suranaree University of Technology, Nakhon Ratchasima, THAILAND.*

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ABSTRACT

The objective of this study is to improve production rate for bio-medical devices stainless tube. In particular, attention is focused on parameters of the electro-etching procedure into the manufacturing process for stainless wire. The wire is used for angiography or arteriography that is a medical imaging technique used to visualize the inside, or lumen, of blood vessels and organs of the body, with particular interest in the arteries, veins, and the heart chambers, so quality of work is important. Electrolytic etching is technique which gives fine, bright and stress-free surface. This paper reports the findings of an experimental investigation on Electrolytic etching of SUS304 stainless steel. In this investigation, effect of process parameters on performance measure as diameter reduction rate was studied. The process parameters which were chosen were: current (A) and electrolysis time (min.). The relation between current and material removal was proposed. The results and the significance of controlling parameters were analyzed using analysis of variance (ANOVA). It was found that current is a significant parameter. The electrolysis time and the number of workpiece for each lot sizes were investigated.

1. Introduction

In the biomedical field, quality of work is important. Electrolytic etching is especially useful for the finish machining of stainless steel wire. The wire is used for angiography or arteriography with particular interest in the arteries, veins, and the heart chambers. Electrolytic etching does not induce surface stress, promoting the preferential removal of surface roughness [1,2]. The dissolution rate of the metallic surface is significantly increased by the application of anodic currents [3,4]. Electrolytic etching is normally performed in an appropriate solution under suitable

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conditions [4]. There are many factors that affect to the quality of the finished product. Life time of dissolute is involved to environment. The more use of dissolute may be extended. Such factors include current density, concentration and temperature of solution, polishing duration, frequency of applied current, and the method of agitation [5- 7]. The process consists of an anode (work piece), a cathode (electrode) and electrolyte. Generally, the electrolyte is composed of viscous acid fluid. During the anodic dissolution, the dissolution rate at the anode is the slowest and is the controlling factor [8]. Therefore, the electrochemical reaction is under diffusive mechanism. Due to the diffusive mechanism, a viscous film will be formed on the anode. The increase in resistance of the viscous film prevents the electrochemical reaction efficiency from increasing [6]. The surface is basic-ally etched in an acid bath and in parallel an electric field is applied to the surface which in turns causes a current flow. Because of the current enhancement, sharp tips on the surface are removed. For most metals one finds similar current density-voltage curve for the electrochemical polishing process [7]. The detailed shape of the curve depends on the ratio of surface area of cathode and anode and the initial surface roughness [9]. During electrolyte etching, positive metal ions leave the specimen surface and diffuse into the electrolyte, an equivalent number of electrons remains in the materials.

2. Experiment procedure

Specimens of SUS–304 stainless steel rod of size 120 mm length and 0.05 mm diameter were cut and their sharp edges and burrs were remove on grinder as shown in Figure 1. As the specimens have rolling marks, surface defects scratches and scales, initial mechanical polishing is needed. After mechanical polishing, specimens were clean in running tap water, dried and solvent degreased. It is then weighed. Before polishing, specimen is dipped in hot steelex 20 (alkaline soap cleaner) solution for 5-10 minutes. Its temperature was maintained approximately 70-800°C.

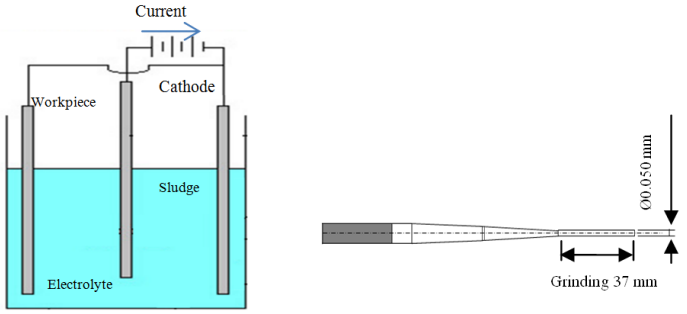

Figure 1: Experimental setup of etching process and finish product.

After soaking, steel specimen maintains approximately 70-800°C. After soaking, steel specimen cleaned in running water then dipped in 10% HCL for 10-20 seconds to remove alkaline film formed on the specimen. It is then cleaned in running water followed by cleaning in distilled. Evenly spread thin film of distilled water on specimen indicates that specimen is completely clean. Then specimen is dried and stored on clean paper. And then, specimens were immersed in

electrolyte (500ml H_2SO_4 + 340ml H_3PO_4 + 100ml HOC-COOH + 3.5gm/100ml $K_2CR_2O_7$ + 60ml distilled water per liter). The diameter of finished product is 0.028 – 0.034 millimeter. For parameters was set up on Table 1. Voltage is fixed at 18 volts. It has been reported that the voltage for electro-polishing affect to surface's quality [10]. The influence of temperature, applied current and time on surface roughness reduction were investigated by the analysis of variance (ANOVA) and the obtained results were followed by scanning electron microscopy measurements.

Table 1: Experimental conditions

Parameters	Value
Electrode	SUS304
Workpiece	SUS304
Workpiece height	120 mm
Etching area (Ø 0.05 x 37 mm)	5.81 mm^2
Current density	0.2 – 0.4 A
Number of workpiece / etching	4 pcs

3. Results and Discussions

For electrolytic etching, current density is a function of the process. The current density is a function of the ohm resistance. Figure 2 shows relationship between current and material removal rate (MRR). The highest MRR value occurs at the highest current density. However, if the current is set more than 0.4 ampres the surface of workpiece was burned. Moreover, increasing the current density makes result in a smoother and more polished surface while more material loss, due to an increase in the mass transport rate [10].

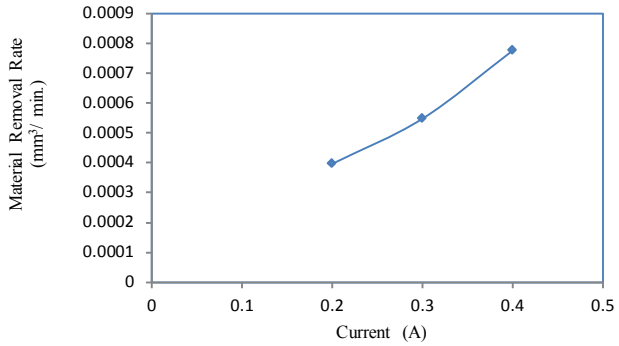

Figure 2: Relationship between current and material removal rate (MRR).

ANOVA (Analysis of Variance) is a statistical tool for obtaining the contribution of variable parameters. The purpose of the analysis of variance (ANOVA) is to reveal the process parameters that significantly affect the performance characteristics. Using the experimental design module in the MINITAB, one obtains the results as shown in Table 2. A statistical tool called a P value can be used to identify the factors that significantly affected on the quality characteristic. In performing the P value is less than 0.05. In this study by using ANOVA the contribution of current shows its interaction towards MRR. In the table DF is for degree of freedom, SS for sum of squares, MS for

mean of square and F value is the ratio of regressions mean square and mean square error. The standard deviation of errors in the modelling (s=0.0005774) indicates that the model is capable of predicting the response with a high response.

Table 2: Results of ANOVA.

Source	DF	SS	MS	F	P
Current	3	0.003091	0.0010303	3091	0.000
Error	8	0.0000027	0.0000003		
Total	11	0.0030937			

S = 0.0005774 R-Sq = 99.91% R-Sq(adj) = 99.88%

The influence of electrolyte concentration on the precision process is highlighted using empirically adjusted formulae. In Industrial practice optimum concentration is found by trial and error to give the desired specification. The solution should have ion concentration. Electrode reaction kinetics are affected by the electrode surface cleanliness, surface microstructure, and surface chemistry.

The quality of the surface obtained by electrochemical polishing strongly depends on the electrolyte properties[1-2]. However, appropriate use affect to health hazards and environmental. Irritation, corrosive injuries and burns have a direct impact on the workers in the field. Improper disposal of chemical etchants changes the level of acidity and alkalinity affect the flora and fauna in soil and water. Normally, electrolyte can be used for 46 times (each time sets for 4 pieces). It means that 184 of workpieces were used for etching. The test was carried out to investigate reused electrolyte. The second lot was carried out with the same electrolyte. Figure 3 shows relationship between etching time to diameter of workpiece with first electrolyte and second electrolyte using current 0.4 Amperes.

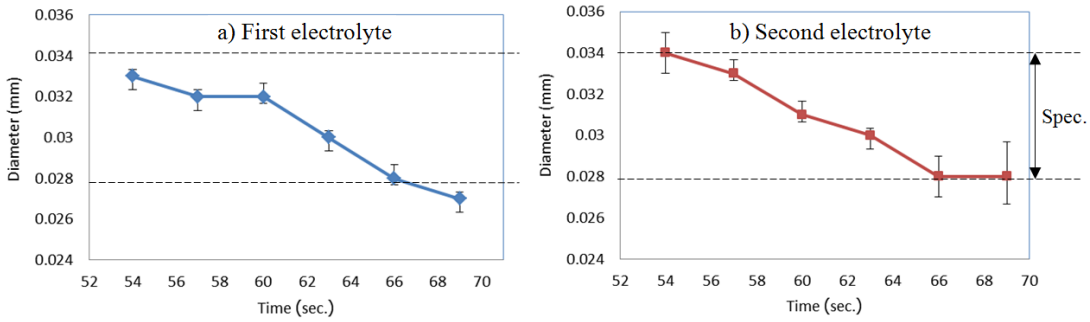

Figure 3: Relationship between etching time to diameter of workpiece with first electrolyte and second electrolyte.

The workpiece can thus be predicted and controlled to improve the dimensional accuracy of the process. The result shows that first electrolyte_can control the specification of 0.028–0.034 mm. From the results, etching time = 66 seconds make workpiece lower specification. So, suitable

values of processing time are 56-62 seconds. From Figure 3(b), when the test were carried out in second electrolyte. The processing time was used longer than that of new electrolyte. Lifetime of electrolyte was reduced with ion concentration of the etching liquid. The new electrolyte enables higher accuracy to be realised than with lower ion concentration electrolyte. For second electrolyte, etching time can be used during 57–61 seconds. Figure 4 shows SEM of finished surface after the process compared with first electrolyte and second electrolyte. Figure 4(b) displays the etched surface of specimen with second electrolyte. The result show that etched surface after second electrolyte is rougher than that of finished surface with first electrolyte. When a process is stably done and in control, it displays common cause variation, which is inherent to the process. A process is in control when based on past experience it can be predicted how the process will vary within limits. When a process operates in this condition, that process is in control and produces 100 percent conformance [11].

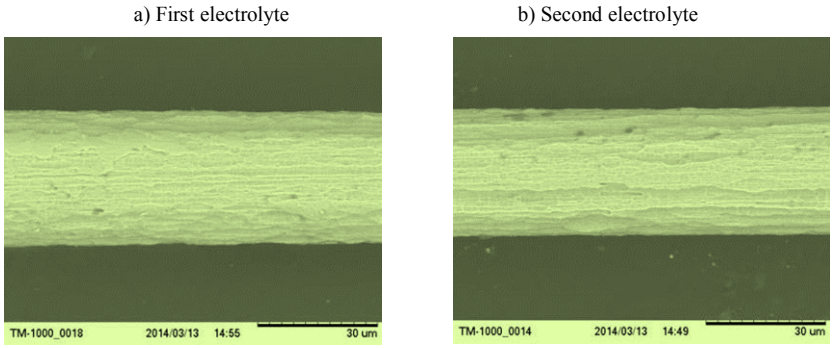

Figure 4: SEM of finished surface after the process with a) First electrolyte and b) Second electrolyte

Relatively surface roughness resulted normal electrolyte and used electrolyte are shown in SEM Figure 4. The craters on surface etched in used electrolyte (Figure 4b) are larger than the craters on that etched in new electrolyte (Figure 4a). The surface roughness is declined with diluted electrolyte concentration. This is because the higher electrolyte concentrations make the stronger activation. Then, the oxide film will have very little effect on ECM as the concentration increases. The dissolution of the elements in the alloy will be uniform, and the surface roughness is better. The present study deals with the objective of finding process parameters to yield the machining time by the design of experiment. The quality of the surface obtained by electrochemical polishing strongly depends on the electrolyte properties. Appropriate care has to be taken when using this solution due to environmental and health hazards. Number of workpiece can be used with 370 pieces. It is possible that more workpieces may accept, due to quality of surface obtained within specifications.

Table 4: Analysis of Variance for S/N ratios for MRR

Source	DF	SS	MS	F	P
Concentration	2	0.0000021	0.0000011	1.78	0.183
Etching time	5	0.0002429	0.0000486	81.98	0.000
Interaction	10	0.000003	0.0000003	0.51	0.874
Error	36	0.0000213	0.0000006		
Total	53	0.0002693			

S = 0.0007698 R-Sq = 92.08% R-Sq(adj) = 88.34%

The Two-Way ANOVA was used for analyze of the parameters (electrolysis time and concentration of electrolysis). The table showed the P-value under 0.05 in case of electrolysis time but P-value of validated electrolysis is over 0.05. It is comprehended that electrolysis time is noticeable significant factor. However, electrolytic concentration is not significant. The total sum of the squared deviations was decomposed into two sources as sum of the squared deviations due to each process parameters. The percentage of contribution of each process parameter in the total sum of the squared deviations can be used to evaluate the importance of each process parameters over the performance characteristic. The F value test was also used to investigate the degree of significance of each process parameter over the performance. Usually, the largest value of F recorded the most significant process parameter.

4. Conclusions

The paper presented improvement of Electrolytic Etching on Stainless Steel Grade SUS 304 by experiment. As a result, the target performance characteristics can be concluded.

- The quality of the surface obtained by electrochemical polishing strongly depends on currents and the etching time that is verified by experiment and analysis of variance.
- The quality of the workpiece that are included precision dimension and surface roughness, preliminarily assessed by visual inspection, could be confirmed by scanning electron microscopy.
- Processing time is 56-62 seconds for normal electrolyte.
- Processing time is during 57 – 61 seconds for the second electrolyte.

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Dr.Apiwat Muttamara is an Assistant Professor of Department of Industrial Engineering at Thammasat University. He received his B.Eng. from Kasetsart University and the D.Eng. in Materials Science from Nagaoka University of Technology, Japan. Dr. Muttamara is interested involve Electrical Discharge Machining of insulating materials.

Natta Naulpring obtained her Master of Engineering degree in Industrial Work Development from Thammasat University. Her research focuses on efficiency improvement of electrolytic etching on stainless steel.

Dr.Somsak Siwadamrongpong is teaching in Manufacturing Engineering, Faculty of Engineering, Suranaree University of Technology, Nakhon Ratchasima, Thailand. He obtained his B.Eng. (Mechanical Engineering) from Chulalongkorn University, Thailand. He got his master degree in Environmental System Engineering from Nagaoka University of Technology, Japan, and his PhD in Energy and Environment Science also from Nagaoka University of Technology, Japan. Dr.Siwadamrongpong research encompasses Material Characteristics, Reliability of Product, Processes and Parts, and Jig & Fixture Design for Manufacturing / Maintenance.