

Development of Ultra Clean Machining Technology with Electrolytic Polishing Process

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ABSTRACT

Electrolytic polishing is the anodic dissolution process in the transpassive state. It removes non-metallic inclusions and improves mechanical and corrosion resistance of stainless steel. If there is a Bailby layer, it will be removed and the true structure of the surface will be restored. Electrolytic polishing is normally used to remove a very thin layer of material from the surface of a metal object. A new electrolyte composed of phosphoric, sulfuric and distilled water has been developed in this study. Two current density regions, high & low current density regions, have been applied in this study. In the region of high current density, there is no plateau region but excellent electrolytic polishing effect can be accomplished in short machining time because material removal process and leveling process occur simultaneously. In the low current density region, there can be found plateau region. The material removal process and leveling process occur successively. The aim of this work is to determine electrolytic polishing process for stainless steel in terms of high & low current density and workpiece surface roughness.

Keywords : Electrolytic polishing, anodic dissolution, Bailby layer, current density, electrolyte, material removal process, leveling process

1. Introduction

Recently, developments in the semiconductor industry require stainless steel STS316L (BS: 316S12, AISI: 316L) to contain the highest levels of purity and surface finish, which cannot be achieved by traditional machining. Traditional machining leaves impurities such as rust spots, scale and non-metallic inclusions on the grooves of machined surfaces that are inadequate for the semiconductor industry. To fulfil these requirements, electrolytic polishing is the most common surface finishing technique aside from the traditional methods where tools are in contact with the workpiece.

Electrolytic polishing is useful in many areas related to the semiconductor, bio-hygiene, medical, ultra-clean gas, large vessel and atomic energy industries. The first reference to electrolytic polishing was in 1912, when the German Government issued a patent for the finishing

of silver in a cyanide solution. Although further experimentation continued, the next significant advancement was not made until 1935 when copper was successfully electrolytic polished by Pierre Jacket. During World War II new formulas and results were discovered [1].

Previous research has studied the relationship of the electrolytic polished surface microstructure with current density [2-5]. This paper reports on work to analyze the machining characteristics of electrolytic polishing in terms of low, high current density and surface roughness.

2. Electrolytic polishing mechanism

Electrolytic polishing is an anodic dissolution process using an electrochemical reaction. It can provide a smooth, bright and reflective surface that exhibits superior corrosion resistance when the workpiece (+) and tool electrode (-) are charged with electricity.

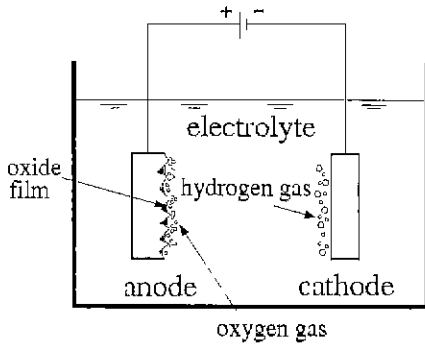


Fig. 1 Principle of electrolytic polishing. As the current is applied to the anode and cathode in an electrolyte solution, oxygen is given off at the cathode with the dissolution of workpiece and hydrogen is given off at the anode without any dissolution.

Electrolytic polishing is suitable for the polishing of both complex shapes and hardened materials that can hardly be machined mechanically because the electrode and the workpiece are not in contact with each other. The definite mechanism has not yet been determined but it is usually explained like this; the electrolytic polishing effect occurs because of differential dissolution, as the current is applied, the oxidization film covering the lower peaks of surface, which has a high specific gravity, viscosity and insulation, prevents dissolution while the higher peaks uncovered with the oxidization film, which receive greater current from the cathode, dissolve more quickly. As the current is applied to the anode and cathode in an electrolyte solution as illustrated in Fig. 1, oxygen is given off at the cathode with the dissolution of workpiece and hydrogen is given off at the anode without any dissolution. Ben Franklin [6] proved that a piece of machined metal that is charged tends to have a very pronounced concentration of charge at its edges and irregular or sharp points. Since the electrolytic polishing process by nature, is a metal dissolution process, each of the peaks that protrude into the anode film will have significantly greater charge concentration on them as compared to the valleys of the workpiece and thus will be removed faster than the valleys of the materials surface.

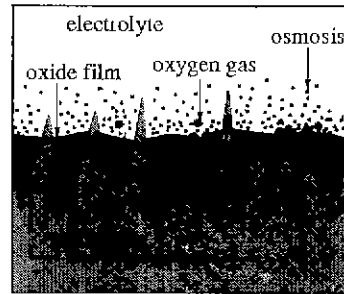


Fig. 2 Electrolytic polishing mechanism. The higher peaks of the surface are dissolved faster than the lower peaks.

By osmosis, the metal ion in solution at the edge of the anode film will naturally migrate into the main body of the solution. This loss of ions into the main body of the solution reduces the ion concentration of the electrolytic polishing solution at the surface of the anode film.

Fig. 2 shows that the outer layer of the anode film becomes more active than the inner layer. This creates a situation where metal can still be removed in the regions of the anode film with a high charge concentration farthest away from the surface of the workpiece [6,7]. As electrolytic polishing takes place, oxygen is formed as a natural part of the process. The oxygen is generated at the outermost edge of the anode film since this oxygen is generated as a gas, it will form bubbles and moves the main body of the electrolytic polishing solution along the surface of the anode film. With this movement of reducing its outer layer, the highest spots of the workpiece surface will be dissolved faster while the lower regions of workpiece are inaccessible. But in the event that a high current is applied, a great amount of oxygen bubble formation occurs and it leaves pit marks on the work surface that may contain impurities [7,8].

During anodic dissolution, a polarization curve can be obtained as both the tool electrode and the workpiece are positioned close together and the applied current is increased. For anode potentials in the AB range, the metal surface becomes etched

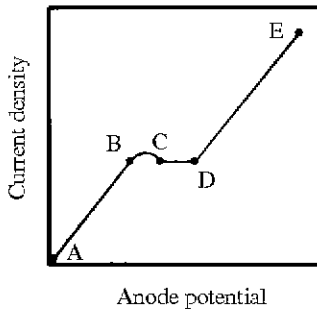


Fig. 3 Simple illustration of polarization curve of electrolytic polishing[7]. AB range ; the metal surface becomes etched. BC range , an oxide film may be formed on the anode abruptly. CD range ; the electrolytic polishing effect takes place and there is hardly any change in current density as voltage increases.

When the anode potential becomes greater than B in the BC range, an oxide film may be formed on the anode abruptly. In the CD range, named the "plateau region" where the electrolytic polishing effect takes place, there is hardly any change in current density as voltage increases. When the electrolytic polishing process is completed in a short time using a high current it leaves surface defects such as pits; hence a current in the plateau region is usually used. However, it should also be pointed out that an applied current slightly higher than one in the plateau region is actually required. Therefore the plateau region in electrolytic polishing is not the best range to achieve the best results, but actually applied current density is a more important parameter [7]. Most of materials not electrolytic polished usually have impurities and damaged layers on their surfaces, which corrode easily as a nuclear of corrosion. Once started the corrosion will continue to spread even without damaged layers.

3. Experiment

Table 1 Chemical composition of STS316L(%)

Symbols for element	Ni	Cr	Mo	Mn	Si	P	S	C
Composition (%)	12.19	17.80	2.28	0.80	0.62	0.03	0.02	0.02

The anodic workpiece was stainless steel STS316L and its chemical composition is listed in Table 1. Three tool electrodes were prepared of different diameters made of copper and the workpiece chucking apparatus was made of titanium. The experimental conditions are presented in Table 2. The quality of the polished surface was observed in the surface roughness tester, optical microscope and metallographic microscope.

Table 2 Experimental conditions

Power supply	50V, 50A DC
Workpiece (anode)	STS316L tube, Φ 10.0mm
Electrolyte	Phosphoric acid(H_3PO_4) Sulphuric acid(H_2SO_4) Distilled water(H_2O)
Electrode (cathode)	Cu
Chucking	Titanium Chucking
Surface roughness tester	Mitutoyo Surftest SV400
Surface measurement	Olympus BH2-UMA
Metallographic measurement	Olympus DME3-312B
Chemical composition measurement	SEM-EDS

For the experiment of machining characteristics of electrolytic polishing, an apparatus illustrated in Fig. 4 was used: the cathode was fixed by an electrode holder, the anode was submerged in electrolyte in the process bath, and the current was applied next. The applied power source was max 50V, 50A variables. The electrolytic polished workpiece was cut with a wire-cutting machine, and the inside surface of the workpiece was investigated. The anodic workpiece was stainless steel STS316L and its chemical composition is listed in Table 1.

The workpiece was polished mechanically on lapping abrasives no. 600, 1800, 3000. The solution was mixed with phosphoric acid, sulfuric acid and distilled water.

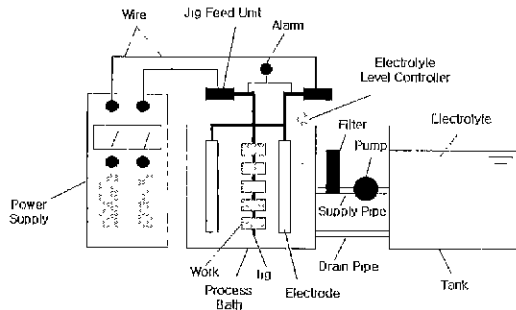


Fig. 4 Experimental setup

Phosphoric acid ensures a slow and uniform dissolution at the appropriate working conditions without corrosion, erosion and oxidation whereas sulfuric acid increases current density efficiency by causing initial dissolution of the workpiece surface.

4. Results and discussion

4.1 Evaluation of the low current density

Fig. 5 presents the low current density-voltage curve obtained through the electrolytic polishing of stainless steel STS316L. The applied electrode gap was 1mm, the range of current density was $0.04 \sim 1.0 \text{ A/cm}^2$, and the electrolyte temperature was 60°C . The electrolyte was composed of phosphoric acid, sulfuric acid and distilled water. This curve shows that the current density has a tendency to increase as the voltage increases.

In the region, including current density variances of $0.04 \sim 0.2 \text{ A/cm}^2$, the observations of current density-voltage curve shows the current density increases slightly as the voltage increases.

In the region, including current density variances of $0.2 \sim 0.4 \text{ A/cm}^2$, the observations of current density-voltage curve show the current density increases abruptly as the voltage increases.

In the region, supposed to be plateau region, including current density variances of $0.4 \sim 0.6 \text{ A/cm}^2$, the observations of current density-voltage curve show the current density increases slightly as the voltage increases.

In the region, including current density variances over 0.6 A/cm^2 , the observations of current density-voltage curve show the current density increases abruptly as the voltage increases.

The material removal rates, surface roughnesses and

metallographic micrographs of work surfaces were analysed to find the region of electrolytic polishing on this curve, because the exact region of electrolytic polishing couldn't be justified from Fig. 5.

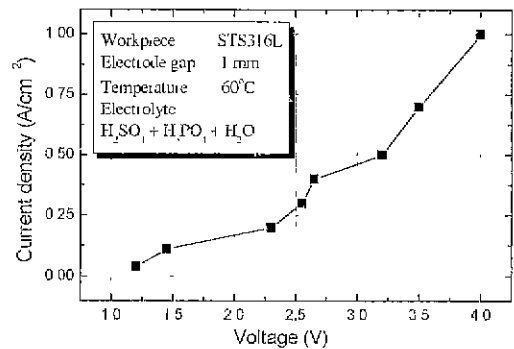


Fig. 5 Low current density-voltage curve obtained through the electrolytic polishing of stainless steel STS316L.

Fig. 6 presents the Relationship between material removal rate and current density curve obtained the electrolytic polishing of stainless steel STS316L in the low current region. This curve shows that the material removal rate increases from $0.2 \times 10^{-3} \mu\text{m/sec}$ to $35 \times 10^{-3} \mu\text{m/sec}$ as the current density increases.

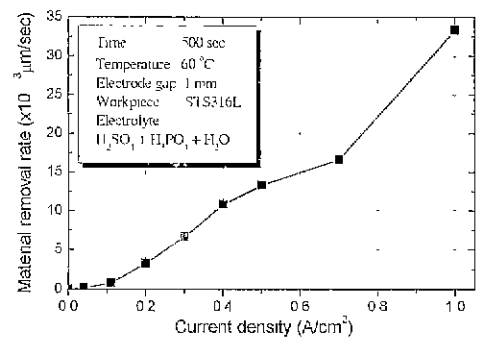


Fig. 6 Relationship between material removal rate and current density curve obtained the electrolytic polishing of stainless steel STS316L in the low current region.

When the applied current increases, the material removal rate increase because the higher the current increases, the higher the chemical reaction activates.

Especially, in the region including current density variances of $0.4 \sim 0.6 \text{ A/cm}^2$, the observations of material removal rate and current density curve show a slight increasement of material removal rate.

The reason of these phenomena can be drawn as follows. The leveling process starts and oxidization film starts to be stacked on the surface of workpiece in this region, so the selective dissolution of higher peaks uncovered with oxidization film starts. Thus the leveling process occurs more than the material removal process.

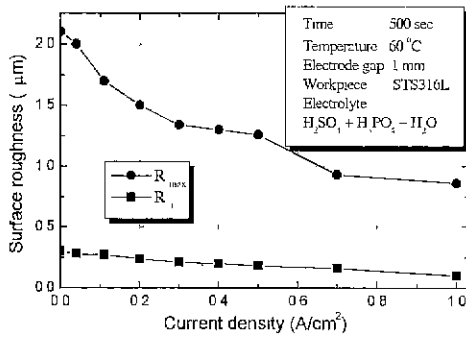


Fig. 7 Relationship between surface roughness and low current density. The surface roughness of the workpiece has a tendency to become smoother and more leveled as the current density increases.

Fig. 7 presents the relationship between the surface roughness of the workpiece and low current density. The surface roughness of the workpiece before electrolytic polishing was $0.3 \mu\text{m Ra}$, $2.1 \mu\text{m Rmax}$. In this test, applied polishing time was 500sec, the electrolyte temperature was 60°C and the electrode gap was 1.0mm. The electrolyte was composed of phosphoric acid, sulfuric acid and distilled water. A long polishing time was used to clarify the differences of machining characteristics because the maximum value of the current density was relatively low. The surface roughness of the workpiece has a tendency to become smoother and more leveled as the current density increases. In the region of current density $0.4 \sim 0.5 \text{ A/cm}^2$, a slight improvement rate of surface roughness is obtained without abrupt improvement. The leveling process has just started in this region.

In the region of current density $0.5 \sim 0.6 \text{ A/cm}^2$, an abrupt improvement rate of surface roughness is obtained. The leveling process and selective dissolution has just

finished in this region.

Thus, in the region of current density $0.4 \sim 0.6 \text{ A/cm}^2$, leveling process occurs more than material removal process.

In the region of current density over 0.6 A/cm^2 , a slight improvement rate of surface roughness is obtained. The material removal process occurs more than leveling process.

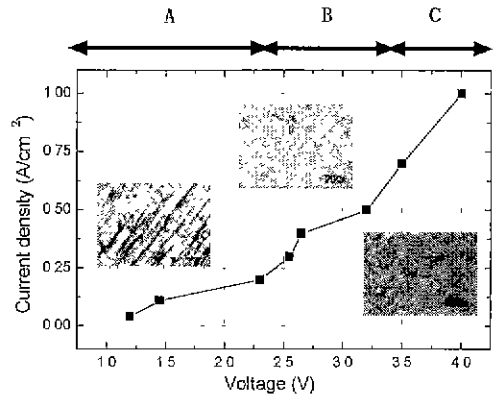


Fig. 8 Surface investigation of low current density regime.

Fig. 8 presents surface investigation of low current density regime showing metallographic micrographs in each current density region marked A, B and C.

In the A region, including current density variances of $0.04 \sim 0.4 \text{ A/cm}^2$, the observations of metallographic micrographs show that many protuberances formed during the process of melting-fusion are located on the workpiece surface with only slight improvement of the surface roughness. In the B region, including current density variances of $0.4 \sim 0.6 \text{ A/cm}^2$, the observations of metallographic micrographs show that a microstructure with obscure grain boundaries can be obtained because lots of protuberances and damaged layers start to be removed. In the C region, including current density variances of $0.6 \sim 1.0 \text{ A/cm}^2$, especially good results are obtained owing to the leveling effect of electrolytic polishing accompanied by the selective dissolution of higher peaks though some pits can be observed.

The observations of the metallographic microstructure show the best result too. The microstructure is uniformly polished with clear grain boundaries.

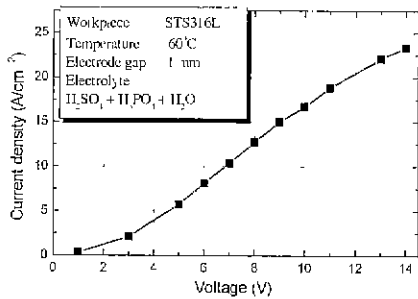


Fig. 9 Current density-voltage curve obtained through the electrolytic polishing of stainless steel STS316L. Current density has a tendency to increase as the voltage increases.

From above studies, the electrolytic polishing effect can be drawn as follows. When the current density range is in $0.4 \sim 0.6 \text{ A/cm}^2$, the oxidization film starts to be stacked on the surface of workpiece and leveling process starts, so the selective dissolution of higher peaks uncovered with oxidization film starts. Thus in the current density region above 0.6 A/cm^2 , the surface roughness improves and microstructure of work surface is uniformly polished with clear grain boundaries.

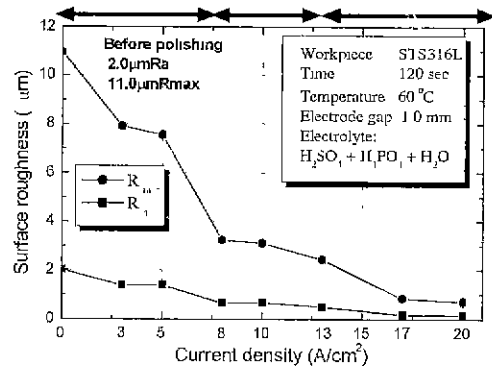
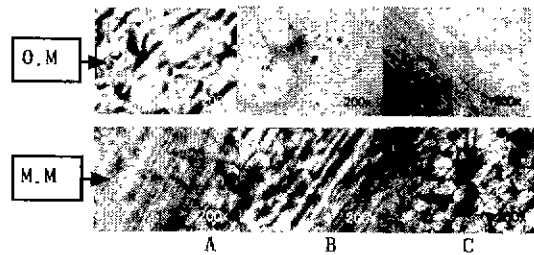
4.2 Evaluation of the high current density

Fig. 9 presents the current density-voltage curve obtained through the electrolytic polishing of stainless steel STS316L. The applied electrode gap was 1mm and the electrolyte temperature was 60°C . The electrolyte was composed of phosphoric acid, sulfuric acid and distilled water. This curve shows that the current density has a tendency to increase as the voltage increases. But the plateau region could not be found in high current density region.

Fig. 10 presents the relationship between the surface roughness of the workpiece and current density, and shows optical, metallographic micrographs in each of the current density regions marked A, B and C. The surface roughness of the workpiece before electrolytic polishing was $2.0 \mu\text{mRa}$, $11.0 \mu\text{mRmax}$. In this test, applied polishing time was 120sec, the electrolyte temperature was 60°C and the electrode gap was 1.0mm.

The electrolyte was composed of phosphoric acid, sulfuric acid and distilled water. A short polishing time was used to clarify the differences of machining

characteristics because the maximum value of the current density was relatively high.



O.M : Optical micrographs
M.M : Metallographic micrographs

Fig. 10 Relationship between surface roughness and current density. The surface roughness of the workpiece has a tendency to become smoother and more leveled as the current density increases.

The surface roughness of the workpiece has a tendency to become smoother and more leveled as the current density increases. In the region of current density over 8 A/cm^2 , a slightly improved rate of surface roughness is obtained without abrupt improvement. In the A region, including current density variances of $0 \sim 8 \text{ A/cm}^2$, the observations of optical and metallographic micrographs show that many protuberances formed during the process of melting-fusion arc located on the workpiece surface with only slight improvement of the surface roughness. In the B region, including current density variances of $8 \sim 13 \text{ A/cm}^2$, the observations of optical and metallographic micrographs show that a microstructure with obscure grain boundaries can be obtained because lots of protuberances and damaged layers start to be removed. In the C region, including current density variances of $13 \sim 20 \text{ A/cm}^2$, especially good results are

obtained owing to the leveling effect of electrolytic polishing accompanied by the selective dissolution of higher peaks though some pits can be observed. The observations of the metallographic microstructure also show the best result. The microstructure is uniformly polished with clear grain boundaries in a short time.

These results show that the electrolytic polishing effect can be obtained in a short time if a high current density is applied and improvement of the surface roughness can be achieved at a current density over a certain value. Therefore, current density is an important parameter that influences the characteristics of electrolytic polishing for stainless steel STS316L.

From above results, the leveling process and the material removal process occur simultaneously because there is no plateau region though electrolytic polishing effect is completed.

Fig. 11 presents a comparison of before and after electrolytic polishing through metallographic micrographs. The observations of the surface show distinct grain boundaries without any scratch marks or damaged layers. Fig. 12 illustrates the reflected photos of the inside surfaces before and after electrolytic polishing. Fig. 13 illustrates other applications of electrolytic polishing, for example kitchen utensils (STS304). The Electrolytic polished one shows an excellent glazed surface rather than the untreated one owing to the effect of smoothing, leveling and passivation. In addition, higher corrosion resistance can be achieved owing to the passivation effect.

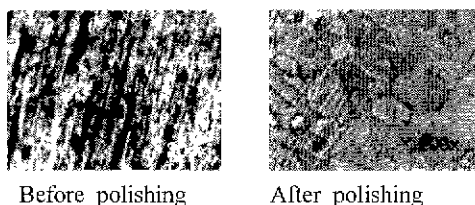


Fig. 11 Comparison of before and after electrolytic polishing. A surface treated with electrolytic polishing exhibits not only a smooth, level surface finish but distinct grain boundaries without any scratch marks or damaged layers as compared with the untreated one.

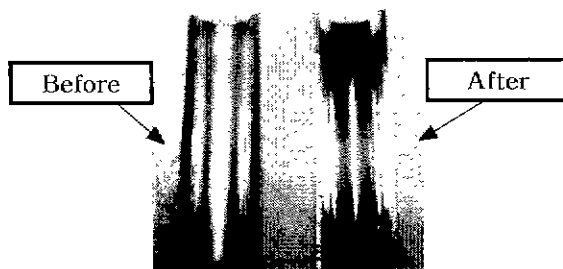


Fig. 12 Comparison of inside surface

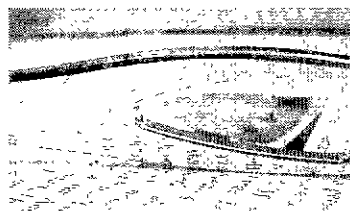


Fig. 13 Other applications of electrolytic polishing -kitchen utensils(STS304).

5. Conclusion

Based on the investigations of electrolytic polishing characteristics for stainless steel STS316L carried out, the following conclusions may be drawn.

In low current density region, the electrolytic polishing process can be drawn as follows. When the current density range is in $0.4 \sim 0.6 \text{ A/cm}^2$, the oxidation film starts to be stacked on the surface of workpiece and leveling process starts, so the selective dissolution of higher peaks uncovered with oxidation film starts. Thus in the current density region above 0.6 A/cm^2 , the surface roughness improves and microstructure of work surface is uniformly polished with clear grain boundaries.

In high current density region, the leveling process and material removal process occur simultaneously because there is no plateau region though electrolytic polishing effect is completed.

Applied current density is an important parameter that influences the characteristics of electrolytic polishing for stainless steel STS316L. The electrolytic polishing effect can be obtained in a short time through the observations of metallographic and optical micrographs when a high current density at least over 8 A/cm^2 is applied.

Acknowledgement

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