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EVALUATION OF ELECTROPOLISHING CHARACTERISTICS OF 316L STAINLESS STEEL TUBE IN CONTAMINATED ELECTROLYTE

In the electropolishing process, the polishing quality of the metal surface varies according to the contamination of the electrolyte. In this study, the electrolyte was evaluated according to the usage time, and the effect of each factor on electropolishing was investigated. As the electrolyte is contaminated, the concentration of metal ions in the electrolyte increases and the ion conductivity decreases. In addition, the pH and specific gravity of the electrolyte increase due to the metal sludge formed as the metal ion concentration increases. When the electrolyte usage time was more than 5 days, many scratches remained on the surface of 316L stainless steel, and relatively high surface roughness was measured. The surface roughness improvement rate compared to the initial specimen was 30% for the unused electrolyte, 26% on the 3rd day, 19% on the 5th day, and 17.5% on the 13th day. Since the low current density due to electrolyte contamination causes a decrease in polishing efficiency, initial scratches on the metal surface still exist on the polished surface. Therefore, it is necessary to manage the electrolyte to maintain the quality of electropolishing.

Keywords: Electropolishing; 316L Stainless steel; Electrolyte contamination; Surface roughness

1. Introduction

Electropolishing of stainless steel has long been used in many industries to achieve perfectly smooth surfaces. Recently, as the degree of integration of semiconductor circuits has increased, research on manufacturing and high-purity UHP (Ultra High Purity) 316L stainless steel is being accelerated [1-5]. There are many variables in the electropolishing process that affect the surface roughness of the workpiece. In particular, electrolytes have the most detailed factors that affect surface roughness, such as chemical composition, temperature, viscosity, electrical conductivity, and supply method, so thorough management is required [6].

Industrial electrolytes exposed to the electropolishing process for a long time cause (Fe, Cr, Ni)-contamination of the process bath due to the dissolution of the anode metal. During electropolishing, metal ions such as Fe, Cr, and Ni, which are the main components of stainless steel, are dissolved during the polishing process and accumulated in the electrolyte. When these metal ions are small quantities, they do not significantly affect polishing, but when a large amount of metal ions accumulate, metal sludge is formed, and the efficiency of electropolishing is

reduced [7]. Contamination of the electrolyte due to metal ions affects the quality of electropolishing, so studies on regeneration of the contaminated electrolyte have been reported [8-10]. In addition, in the stainless steel electropolishing industry, there are mentions of the replacement time based on electrolyte contamination [11,12], but there is no specific study.

In this study, the correlation between the properties of the contaminated electrolyte and the quality of electropolishing was presented. The metal ion content, ion conductivity, specific gravity, pH, etc. in the electrolyte according to the process exposure time of the electrolyte were compared. In addition, the polarization characteristics of each electrolyte were confirmed, and the effect of electrolyte contamination on the electropolishing process was investigated by comparing the surface roughness and polishing rate of the specimen after electropolishing.

2. Experimental

The 316L stainless steel used in the experiment was a commercial 316L stainless steel tube (Seah steel, KOREA). The 316L stainless steel tube with a length of 6,000 mm was cut

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into 170 mm specimens, and the surface roughness of each specimen was measured, and the average value was indicated as the initial surface roughness. As the electrolyte, an industrial electrolyte (SBR3000, SEBITCHEM, KOREA) used for electropolishing of stainless steel was used, and phosphoric acid, sulfuric acid, and water were mixed in a weight ratio of 6:3:1. According to the usage time of the electrolyte, it was classified into four types: unused electrolyte, 3 days, 5 days, and 13 days used, and the used electrolyte was based on 8 hours of use per day. To confirm the characteristics of each electrolyte, the ion content was measured using inductively coupled plasma-optical emission spectroscopy (ICP-OES, Thermofisher, iCAP pro XP, USA). The ion conductivity of the electrolyte was measured in the temperature range from room temperature to 80°C, and the pH and specific gravity were measured at room temperature. To derive an I-V curve according to the contamination of the electrolyte, a voltage was applied at a rate of 0.1 V/s and the current was measured in units of 1 A. Based on the polishing voltage derived from the I-V curve of the unused electrolyte, electropolishing was performed under constant voltage conditions with a polishing time of 30 to 120 seconds, and TABLE 1 shows the electropolishing conditions. Internal surface images after electropolishing were analyzed by scanning electron microscope (SEM, Tescan, MIRA3 LMH, Czech Republic). In addition, the surface roughness of the inner wall of the specimen was measured with a confocal laser scanning microscope (CLSM, Keyence, VK-X3000, Japan), and the average value was shown after measuring the surface roughness of the specimen 5 times

for each point. Finally, the weight loss of the specimen and the polishing time were used to confirm the polishing rate for each electrolytic polishing condition.

TABLE 1

Electropolishing conditions of 316L Stainless steel tube

Working electrode (anode)	316L Stainless steel tube (diameter : 10.47 mm, length : 170 mm)
Counter electrode (cathode)	Copper rod (6 ϕ)
Electrolyte	Unused electrolyte, 3 days, 5 days, 13 days used electrolyte (4 types)
Polishing time	30, 60 ,90, 120 sec
Electrolyte temperature	65°C
Electrolyte flow rate	3.5 LPM

3. Results and discussion

Fig. 1(a) is the result of ICP analysis of the metal ion concentration of each electrolyte. As the usage time of the electrolyte increases, the ion concentration of the electrolyte increases in the order of Fe, Cr, and Ni. In the electrolyte used for 13 days, about 20,000 mg/L of Fe, 5,000 mg/L of Cr, and 3,000 mg/L of Ni were detected. The increase rate of metal ions in the electrolyte used for 3 days was the highest, and a similar level was maintained thereafter. Fig. 1(b) shows the ion conductivity of each electrolyte, and the conductivity decreases as the

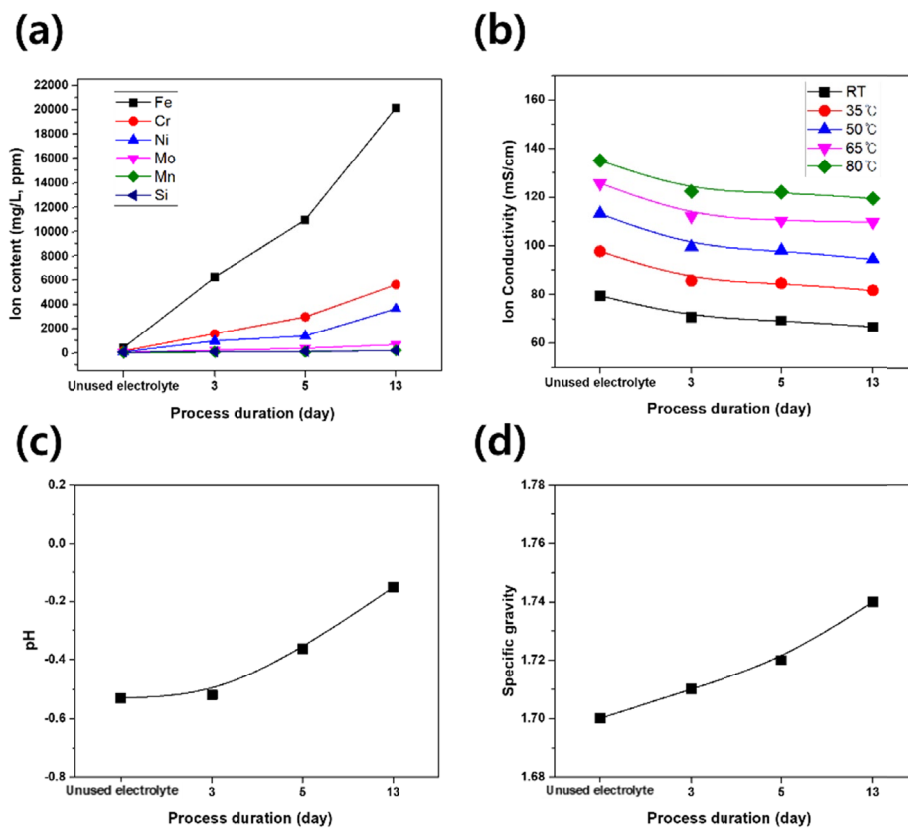


Fig. 1. Characteristic change according to the usage time of electrolyte; (a) Change of metal ion concentration in each electrolyte, (b) Change in ion conductivity of each electrolyte, (c) Change in pH of each electrolyte (d) Change in specific gravity of each electrolyte

usage time of the electrolyte increases [13,14]. In addition, as the temperature of the electrolyte increases, the movement of ions becomes more active, and the conductivity increases [11]. However, this is not a permanent recovery of the electrolyte, and it is important to perform electropolishing by selecting an appropriate temperature according to the electrolyte during the process due to the temporary improvement in conductivity according to the temperature. Fig. 1(c) and (d) show the pH and specific gravity of each electrolyte, and it is confirmed that the pH and specific gravity of each electrolyte increase as the usage time of the electrolyte increases. Since the electrolyte used for a long time has a high metal ion concentration, the amount of hydrate formed during the electropolishing process also increases [15]. These hydrates were accumulated in the electrolyte in the form of sludge, and the pH and specific gravity increased as impurities in the specimen were mixed with the electrolyte during electropolishing. As a result of analyzing the sludge recovered from the electrolyte used for 13 days, it was analyzed as nickel sulfate monohydrate. Such metal sludge hinders the flow of the electrolyte between the specimen and the counter electrode during electropolishing, and increases the electrical resistance, thereby lowering the electropolishing efficiency.

In electropolishing, the I-V curve represents the electrical characteristics, and the I-V curve of each electrolyte is shown in Fig. 2(a). The dashed box indicated in the graph means the plateau region in the I-V curve, and electropolishing is performed under the electrical conditions of the plateau region in the process. The voltage range that stabilizes current density appeared gradually increased as the use time of the electrolyte increased, and the slope of the current density decreased compared to the unused electrolyte. As a result of analyzing the ion conductivity of each electrolyte according to the use time, the electrical conductivity of the electrolyte decreases as the metal ion concentration increases, and higher electrical conditions are required during electropolishing. In the electropolishing process, the counter electrode releases only hydrogen gas due to a reduction reaction, and no other reaction occurs. However,

electrolyte contamination increases the resistance of the counter electrode, lowering the current density and increasing the voltage range in which electropolishing occurs. Fig. 2(b) shows the behavior of the I-V curve before and after contamination of the counter electrode. As a result of analyzing the surface of the contaminated counter electrode, Fe and Ni were observed on the copper electrode surface. During electropolishing, Fe and Ni ions accumulated in the electrolyte may be reduced and coated on the surface of the counter electrode. As such, the surface contamination of the counter electrode increases the resistance and decreases the current efficiency. Therefore, as the use time of the electrolyte increases, washing to prevent deterioration of the counter electrode is essential.

Fig. 3 shows the inner surface of the 316L stainless steel tube after electropolishing under each electrolyte conditions. There are scratches and molding marks on the inner surface of the initial stainless steel specimen in the drawing direction. This is caused by friction with the mold during the drawing process of the tube. As the polishing time increases, the amount of metal ions dissolved in the surface increases and the smoothing of the surface proceeds. Since the scratch valley formed by plastic working is far from the counter electrode, it dissolves slowly, and electropolishing proceeds relatively slowly compared to the region without scratch valley. Scratches remained on the surface electropolished for 120 seconds with the electrolyte used on the 5th and 13th, and many pits were found in the scratch valleys on the surface polished with the electrolyte used on the 13th. As shown in Fig. 2(a), as the usage time of the electrolyte increases, it moves to a higher voltage range than the plateau range, and the current density appears relatively low. When electrolytic polishing is performed with the electrolyte used in this study at the same voltage as the unused electrolyte, it can be confirmed that it has been performed in the low voltage range out of the plateau range. According to Faraday's law, the amount of charge and the amount of reactant are proportional. Therefore, since the polishing amount is relatively small according to the application of a low current density, the polishing effect on the inner surface

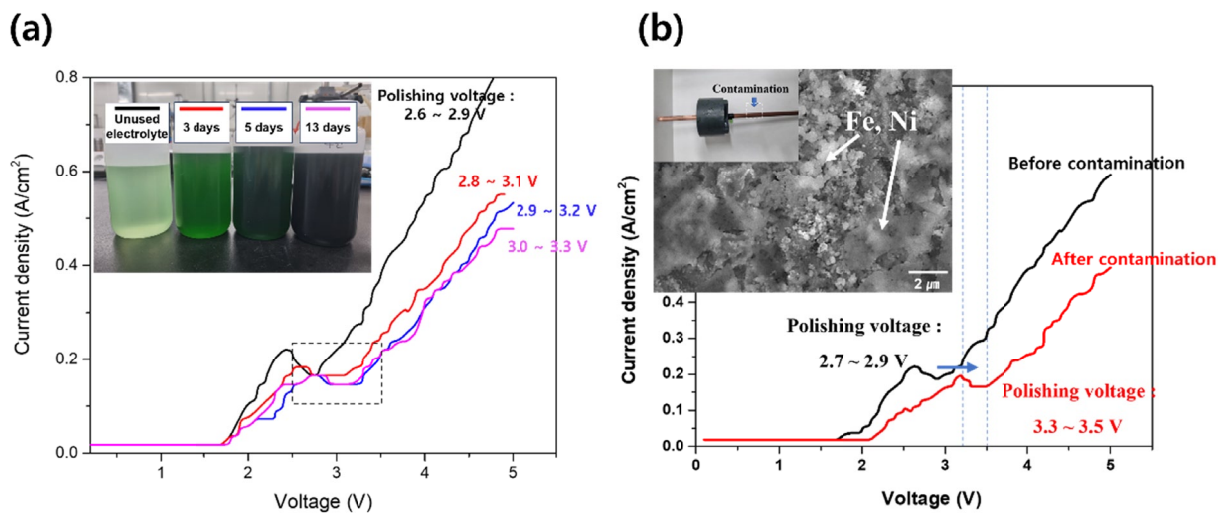


Fig. 2. Results of electrical properties of each electrolyte; (a) I-V characteristics of each electrolyte by DC rectifier, (b) Current density behavior by electrode contamination in I-V curve

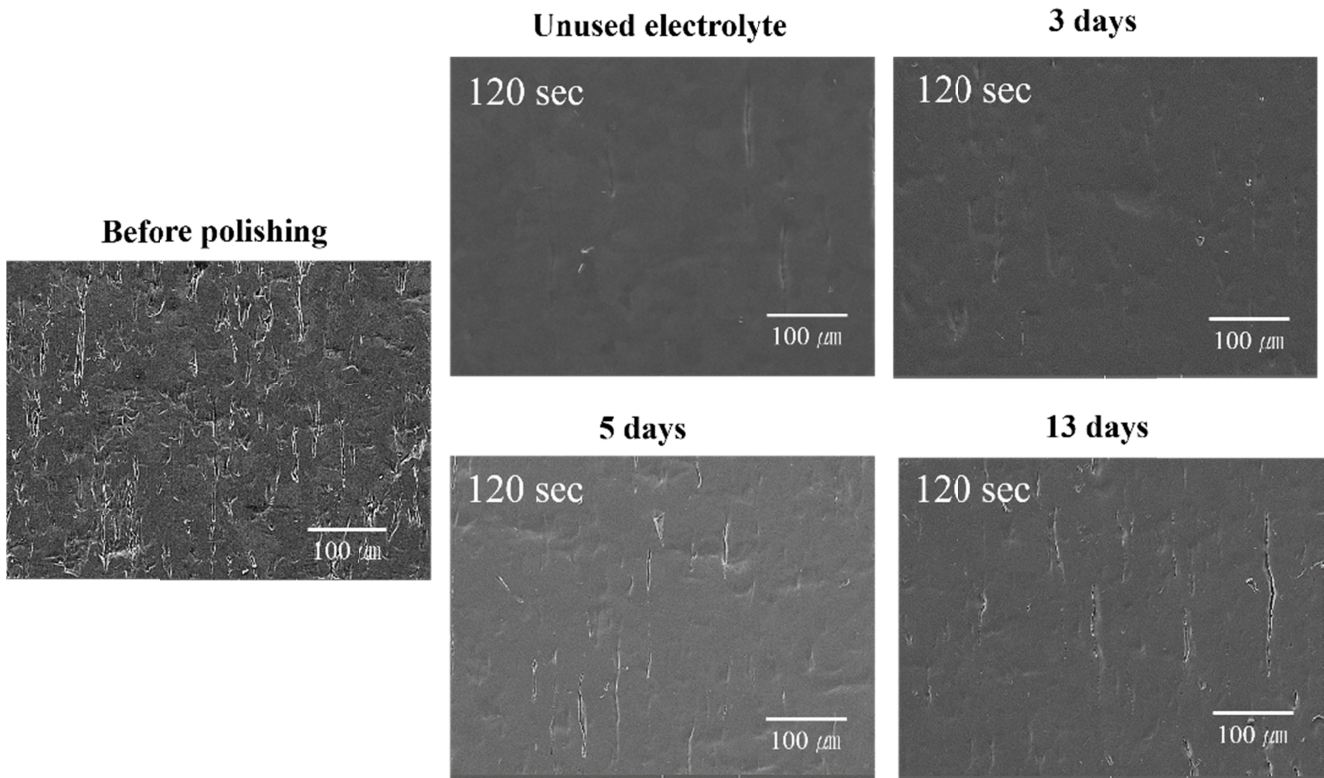


Fig. 3. SEM image of the inner surface of a 316L stainless steel tube after electropolishing

of the tube is reduced. In addition, oxygen bubbles formed on the surface of the anode during electropolishing serve to shield the electrical flow in the electrolyte [16]. When these bubbles are removed from the surface by the flow of the electrolyte, current density is momentarily concentrated at the point, causing excessive dissolution to form pits [17]. It is judged that the longer the use time of the electrolyte, the insufficient polishing of the surface, and the more oxygen bubbles stay in the remaining processing scratches, resulting in the formation of many pits in the scratch valleys.

Fig. 4(a) shows the surface roughness measurement results of the inner wall of the 316L stainless steel tube after

electropolishing according to the polishing time and electrolyte conditions. Even if the electrolyte was used for 5 and 13 days, the surface roughness gradually decreased when the polishing time was increased under the same electropolishing conditions, but relatively higher surface roughness was measured compared to the unused electrolyte and the electrolyte used for 3 days. Under the electrolytic polishing conditions of 120 seconds, the surface roughness of each electrolyte was measured as 0.168 μm , 0.176 μm , 0.193 μm , and 0.198 μm , respectively. The surface roughness improvement rates, compared to the initial surface roughness, were approximately 30%, 26%, 19%, and 17.5% for each electrolyte. However, to improve the surface roughness

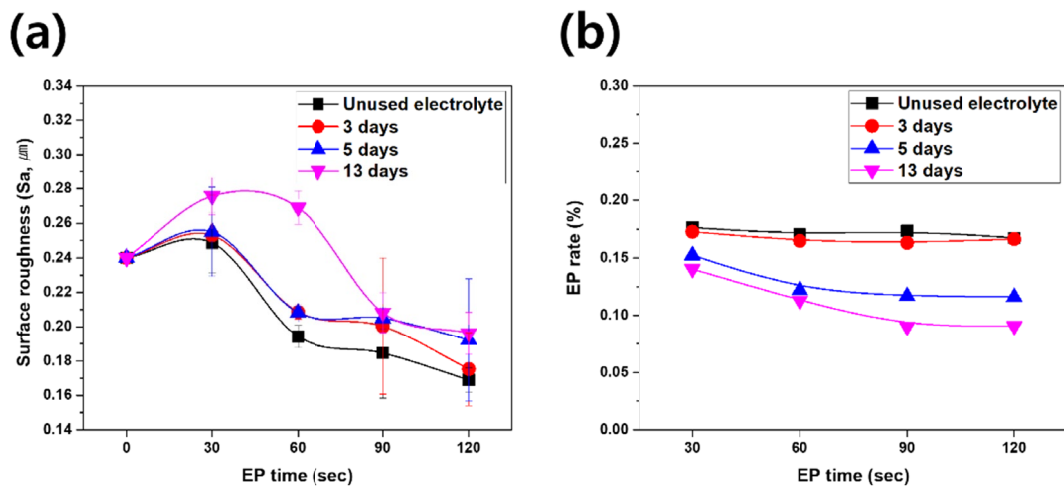


Fig. 4. Surface roughness and polishing rate after electropolishing by each electrolyte; (a) Surface roughness with polishing time, (b) Polishing rate according to weight change before and after electropolishing

and prevent the formation of pits on the surface, it is essential to replace the electrolyte or add a new electrolyte. Fig. 4(b) shows the polishing rate according to the electrolyte usage time. A specimen with a large weight loss at the same polishing time means a high polishing rate because the polishing amount is large. In the case of the unused electrolyte and the electrolyte used on the 3rd day, the polishing rate was kept constant over polishing time. However, the polishing rate of the electrolyte used for 5 and 13 days decreased according to the polishing time, and the polishing rate decreased by about 28% or more compared to the unused electrolyte under the polishing condition of 120 seconds.

4. Conclusions

In this study, the effect of contamination of the electrolyte due to metal ions such as Fe, Cr, and Ni on the electropolishing of 316L stainless steel was confirmed. As the usage time of the electrolyte increases, the content of metal ions such as Fe, Cr, and Ni gradually increases, forming metal sludge in the electrolyte. Contamination of the electrolyte reduces the electrical characteristics of the electrolyte, so that a higher voltage range and current density are required. Due to these characteristics, marks of processing on the surface remain due to an insufficient amount of polishing compared to the fresh electrolyte, and the improvement rate in surface roughness decreases. In this study, scratches remain on the surface of the 316L stainless steel specimen when the electrolyte usage period is 5 days or more, and it has a relatively high surface roughness. Although the target surface roughness can be obtained by increasing the polishing time, the polishing efficiency is low, and pits occur due to the characteristics of the contaminated electrolyte. Therefore, to prevent surface defects of stainless steel in the electropolishing process, it is essential to maintain the quality of the electrolyte by adding or replacing the electrolyte.

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