DEVELOPMENT OF A HIGH-PRESSURE CHEMICAL ETCHING METHOD AS A SURFACE TREATMENT FOR HIGH-FIELD ACCELERATING STRUCTURES MADE OF COPPER

Hiromitsu Tomizawa, Tsutomu Taniuchi, Hideki Dewa, Akihiko Mizuno, Hirofumi Hanaki,
Accelerator Division, Japan Synchrotron Radiation Research Institute (JASRI/SPring-8),
Kouto, Sayo-cho, Sayo-gun, Hyogo 679-5198, Japan

Abstract

The acceleration gradient is limited by rf breakdown in accelerating rf structures, as well as by the surface condition of the inner wall. Surface treatment is an important technique for achieving the maximal acceleration gradient of accelerating rf structures. We chose chemical etching as a surface treatment method for accelerating rf structures made of copper (OFC). A maximum cathode surface field of 183 MV/m was achieved for an S-band (2856 MHz) pillbox-type single-cell rf gun cavity with this surface treatment under an rf-conditioning elapsed time of 1.9×10^7 shots (21 days) in 2004. Furthermore, we developed a high-pressure chemical etching method for complex inner structures in 2006. The results showed that the higher the pressure, the higher the cathode surface field (162 MV/m; 6 atm).

INTRODUCTION

We have been developing a surface treatment method for optimizing the surface condition of the inner wall in order to achieve a maximum cathode surface field of ~200 MV/m. The acceleration gradient is limited by the rf breakdown in accelerating rf structures, as well as by the surface condition of the inner wall. Surface treatment is an important technique for achieving the maximal acceleration gradient in accelerating rf structures. It is necessary to realize chemically noncontaminated and physically smooth surfaces. In accelerator fields, oxygen-free copper (OFC) is widely used as a base material for normal conducting accelerators. Previously, we chose chemical etching as a surface treatment method for accelerating rf structures made of copper after several failed attempts involving high-pressure pure water rinsing [1]. In order to study rf breakdown and the effects of surface treatment [2], we used an S-band (2856 MHz) pillbox-type single-cell rf gun cavity. The highest cathode surface field (183 MV/m) of the rf gun cavity was achieved with this surface treatment under an rf-conditioning elapsed time of 1.9×10^7 shots (21 days) in 2004 [3]. The SPring-8 rf gun has been operating with the world's highest gradient of 190 MV/m [4]. This indicates that our treatment is remarkably effective in improving the properties of the inner cavity surfaces, which are made of copper (OFC). Furthermore, we developed a high-pressure chemical etching method for achieving more complex inner structures in 2006 [5]. Using a cartridge-type revolver photocathode rf gun [6], high-field experiments were performed with cathode plugs subjected to chemical etching at different pressure conditions. We found that the higher the pressure, the higher the cathode surface field. Here, we report these novel results regarding the highest gradient, which were obtained by using test copper (OFC) samples treated with high-pressure chemical etching.

CHEMICAL ETCHING AND ITS OPTIMUM CONDITIONS

Chemical Etching

Chemical etching entails a chemical reaction between an acid and a metal. We used an etching solution composed of sulfuric acid (H_2SO_4) and hydrogen peroxide (H_2O_2), each at 2 wt% standard concentration, in order to make the processing time loss negligible in relation to the etching rate at room temperature.

Investigation of Optimum Conditions for Etching

Before etching, a copper test piece was dipped in Neos CM200C (5 wt%) solution at 40°C for 1.5 h as a degreasing preprocess. After rinsing in tap water for 5 min at room temperature, the copper test piece was etched with shaking to remove hydrogen bubbles from its surface. The etched copper test piece was then rinsed in pure water and then in ultrapure water for 1 min each at room temperature.

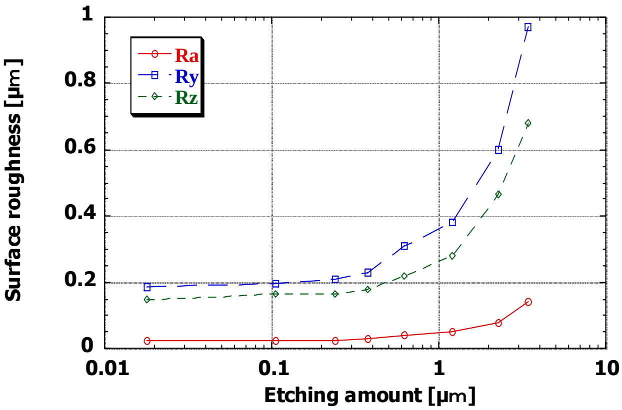

Figure 1: Dependence of the surface roughness on the etching amount in oxygen-free copper (OFC): After degreasing, Ra: 0.02 μm, Ry: 0.19 μm, Rz: 0.17 μm.

Chemical etching cleans the surface using a chemical reaction, resulting in physically rough metal surface. Therefore, we investigated the optimum etching conditions for copper test pieces. After a drying postprocess, the surface roughness of each test piece was measured with a chemical balance for the etching amount and Surftest SJ-301 (Mitutoyo). A laser microscope (VK-9500; KEYENCE) was used for measuring the grain size (see Figure 3) and for cross-checking surface measurements. Figure 1 shows that surface roughness does not increase in the region where the etching amount is less than 0.3 μm. Figure 2 indicates that the dipping time for our standard etching solution should be less than 2.5 min in order to keep the etching amount at less than 0.3 μm (Ra) at 20°C. It was ensured that the copper test pieces are comparable base materials in terms of grain size, impurities, and other properties. We observed the surface structures of each test piece with a laser microscope, and the obtained images are shown in Figure 3. For the etched copper test pieces, FTIR (IFS120HR; BRUKER) at SPring-8 BL43IR showed no significant chemical contamination (molecular bonding on the surface).

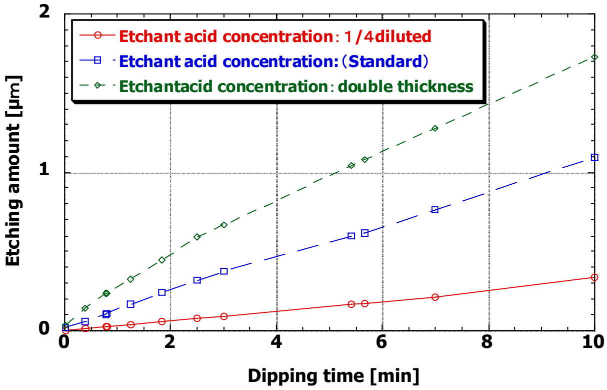

Figure 2: Dependence of the dipping time on the chemical etching amount at 20°C.

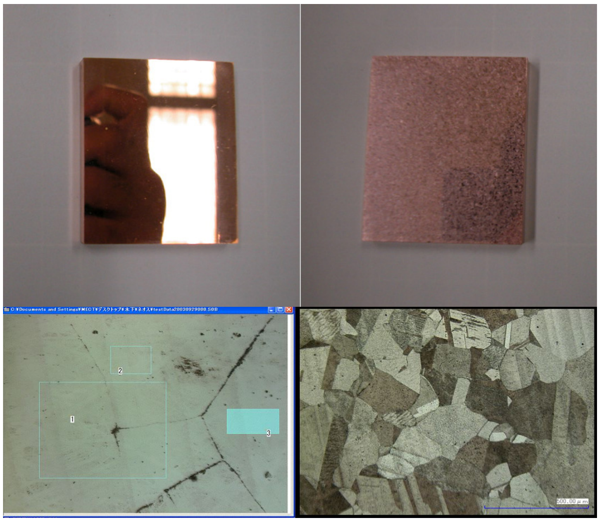

Figure 3: Effect on the structure of OFC due to chemical etching (Left: before etching; Right: after etching).

FEASIBILITY TEST ON HIGH-FIELD ACCELERATION USING A CHEMICALLY ETCHED RF CAVITY

We performed experiments on high-field acceleration with an rf gun cavity using the etching process mentioned above. After the degreasing preprocess, the whole gun cavity made of copper (OFC) was dipped for 2.5 min in the etching solution of standard concentration at room temperature with shaking. Through a drying postprocess with nitrogen gas flow, the rf gun cavity was evacuated and installed in an aging test stand at SPring-8.

In the rf processing for 21 days (1.9×10^7 shots), the maximum field gradient on the cathode reached 183 MV/m, as shown in Figure 4 [3]. Spark light spectrography showed that no significant rf breakdown occurred during this rf conditioning [2].

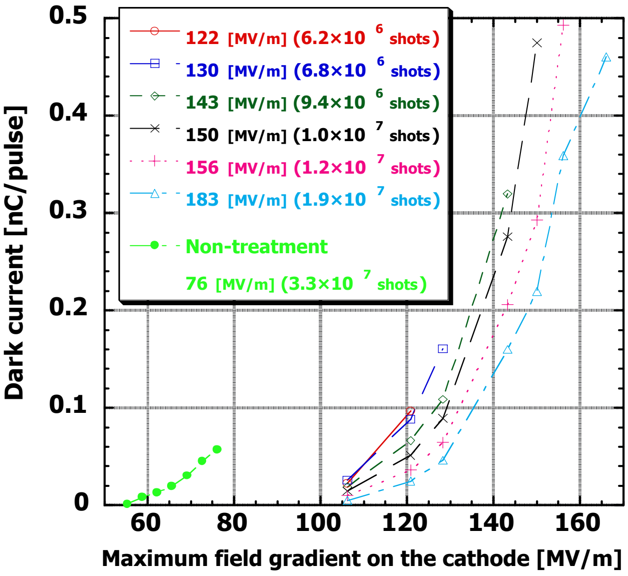

Figure 4: Dark current reduction during rf conditioning of an S-band pillbox-type single-cell rf gun cavity: In the figure, both the highest cathode surface field and the elapsed time of the rf conditioning are shown.

HIGH-PRESSURE CHEMICAL ETCHING AND A TEST OF ITS FEASIBILITY

Using a simple single cell rf gun cavity, we proved that chemical etching is remarkably effective in improving the inner surface of cavities. Since the cavity structure was simple and relatively small, it was possible to simply shake the cavity during the process of dipping in the etching solution in order to remove hydrogen bubbles from the copper surface. However, in the case of accelerating structures longer than 1 m, the application of this method is not straightforward. In some parts of complex structures, hydrogen bubbles might serve as a masking layer against etching on the inner wall surface.

In order to apply our etching method even in complex rf structures, we developed a high-pressure chemical etching method [5]. In this method, remove hydrogen bubbles is performed by using high pressures and low temperatures during the etching process in order to dissolve hydrogen gas into the solution, in accordance with Henry's law. The first test was performed with a high-pressure chamber complex by using high-pressure argon gas, as shown in Figure 5. We performed experiments for high-field acceleration with a revolver-type of S-band rf gun system using a cartridge-type electric tube [6]. This system can accommodate 12 cathode tip plugs simultaneously, which can be replaced in vacuum. We performed high-pressure etching under different conditions on the OFC-cathode tips in the high-field acceleration test in comparable rf conditioning states. A high-pressure etching test stand was fabricated with multiple monitors in order to check the condition of the cathode tip surface (e.g., thermograph, manometers, and microscope cameras).

Figure 5: High-pressure (1~6 atm) chemical etching test stand for OFC-cathode tip plugs: the surface of the cathode tips was evaluated with a laser microscope.

Table 1: Maximum Surface Fields After rf Conditioning Under Different Conditions

HIP-processed cathode

Condi-tion	Final highest cathode surface field achieved	Total elapsed time for rf conditioning to achieve highest field
1 atm	137.61 MV／m	119,727sec（33.3hr）
3 atm	147.04 MV／m	123,614sec（34.3hr）
4 atm	150.66 MV／m	260,478sec（72.4hr）
5 atm	158.83 MV／m	287,094sec（79.7hr）
6 atm	162.09 MV／m	80,565sec（22.4hr）
without etching	134.66 MV／m	263,639sec（73.2hr）
without etching	133.39 MV／m	94,969sec（26.4hr）
without etching	129.90 MV／m	72,751sec（20.2hr）
without etching	131.66 MV／m	82,032sec（22.8hr）

Non-HIP-processed cathode

Condi-tion	Final highest cathode surface field achieved	Total elapsed time for rf conditioning to achieve highest field
1 atm	134.58 MV／m	310,994sec（86.4hr）
3 atm	140.60 MV／m	171,720sec（47.7hr）
4 atm	150.66 MV／m	230,802sec（64.1hr）
6 atm	156.34 MV／m	258,837sec（71.9hr）
without etching	125.07 MV／m	82,375sec（22.9hr）
without etching	128.31 MV／m	83,611sec（23.2hr）
without etching	129.47 MV／m	152,306sec（42.3hr）

Higher pressure etching is significant from a statistical point of view.

In Table 1, 1 atm indicates atmospheric pressure without additional pressure. We conducted a feasibility study of the effect of high-pressure chemical etching on the non-HIP-treated OFC-cathode surface condition with high-field acceleration. Before testing the etched cathodes, rf conditioning was performed on non-etched cathode plugs. Table 1 shows that the highest cathode surface field could not exceed 135 MV/m without etching. The test was performed with a blind check in order to avoid biased interpretation. The results show that the higher the pressure, the higher the cathode surface field. The effect of HIP-processed base materials on the realization of the highest cathode field is not highly significant from a statistical point of view.

SUMMARY

We chose chemical etching as a surface treatment method for accelerating rf structures made of copper (OFC). In this paper, we studied the effects of etching on the copper surface by using OFC test samples subjected to chemical etching under different pressures. In order to study the effects of the surface treatment through high-field acceleration, we first used an S-band pillbox-type single-cell rf gun cavity. After rf processing for 21 days (1.9×10^{7} shots), the maximum field gradient on the cathode reached 183 MV/m [3]. This indicates that chemical etching is remarkably effective in improving the inner surface of cavities made of OFC. The result of high-pressure chemical etching indicates that the higher the pressure, the higher the cathode surface field. The highest field achieved with the above method was 162 MV/m in the case of the HIP-processed sample subjected to chemical etching under a pressure of 6 atm.

We are currently preparing a high-pressure chemical etching test facility for rf accelerating structures (Figure 6). In this system, a 1-m-long accelerator tube can be directly etched under a pressure of up to 10 atm.

Figure 6: High-pressure chemical etching test facility for rf accelerating structures made of OFC.

REFERENCES

[1] H. Tomizawa, et al., Proc. of the 28th Linear Accelerator Meeting in Japan, 2003, p. 132.

[2] H. Tomizawa et al., Applied Surface Science, 2004 235/1-2, 2003, p. 214.

[3] H. Tomizawa, et al., Proc. of the 29th Linear Accelerator Meeting in Japan, 2004, p. 147.

[4] H. Tomizawa, the 3rd PASJ Prize Award for Young Scientists, Proc. of the 4th Annual Meeting of Particle Accelerator Society of Japan, 2007, p. 130.

[5] H. Tomizawa and H. Ohtsuka, Japan Patent Application No. 2007-84245.

[6] J. Sasabe, et al., NIM A 528, 2004, p. 382.