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ANANYSIS OF BCP CHARACTERISTICS FOR SRF CAVITIES*

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Abstract

In order to achieve Eacc > 35 M/V, good surface processing is critical. BCP experiments of niobium samples were carried out with two different chemical compositions, HF (48%) : HNO_3 (60%) : H_3PO_4 (85%)=1:1:1 and 1:1:2. Two types of niobium samples were tested; as-received Nb (undeformed) and deformed Nb with same thickness in 3 mm. Surface analyses with optical photographs were performed regarding etch rates, surface morphologies depending on the temperature and the time of BCP.

INTRODUCTION

Constructing Rare Isotope Accelerator, named as RAON in Korean, by Rare Isotope Science Project (RISP) of Institute of Basic Science (IBS), requires more than 500 SRF (Superconducting Radio Frequencies) cavities to be produced, which means the same amount of cavities need to be processed chemically and mechanically [1]. A chemical polishing is a well-known process with additional subsequent heat treatment [2] to improve superconducting cavities such as Q (Quality Factor) and Eacc (Acceleration Electric Field) [3]. Buffered Chemical Polishing (BCP) has been widely used in SRF cavity processing thanks to the effectiveness and the controllability [4]. Thus, many researchers have made great efforts to develop the chemical process that improves surface states more effectively since cavity performances are largely dependent on the surface roughness [5]. Some baseline data of BCP with time and temperature for RAON will be presented.

EXPERIMENTAL

Material Preparation (Niobium)

We used RRR Niobium plates from ATI, Wah Chang Inc. for BCP tests, and Table 1 shows some important properties of Nb, which were guaranteed by ATI's certification sheets.

For experiments, two types of niobium samples having the dimension of 10 × 10 × 3 mm^3 ($W \times L \times T$) were arranged; as-received (undeformed) niobium and deformed niobium. As-received niobium plate was cut into the pieces in a specific size by Electrical Discharge Machining (EDM) cutting. And then pieces were plastically deformed by a press machine with 300 Ton. Thus, approximate pressure applied on the samples was 29 GPa (yield strength of niobium is less than 0.1 GPa) since sample area was 10 × 10 mm^2. All Nb samples were processed with ultrasonic cleaning (BRANSON 5210) for 10 min. with ethanol and DI water before an experiment.

Table 1: Specifications of Niobium Plates for RAON

Items	Specification	Values	Unit
Dimension	W×L× Thick.	635 × 1200 × 3	mm^3
Electrical Properties	RRR	300	-
Chemical Properties	Oxygen Con.	< 40	ppm
	Nitrogen Con.	< 20	ppm
	Hydrogen Con.	< 5	ppm
	Carbon Con.	< 20	ppm
Mechanical Properties	Tensile Strength	> 100	MPa
	Yield Strength	50 < Rp < 100	MPa
	Elongation (LD)	> 40	%
	Elongation (TD)	> 35	%
	Hardness	Avg. < 60	Hv10
	Grain Size	ASTM #5	μm

Chemical Preparation (BCP Solution)

Table 2 lists two BCP solutions having different ratio of chemical composition. Since the total volume of solution was set as 250 ml due to the size of BCP reactor (see Fig. 1 in the below equipment setup section). Three acids in BCP solution were mixed by using not their volume but mass to reduce errors easily occurred while measuring the volume of liquid with naked eyes.

Table 2: BCP Solution (250 ml) with Different Ratio

Ratio	HF	HNO_3	H_3PO_4	Unit
1:1:1	83.3	83.3	83.3	Volume (ml)
	95.8	114.9	140	Weight (g)
1:1:2	62.5	62.5	125	Volume (ml)
	71.9	86.2	210	Weight (g)

Equipment Setup (BCP Reactor)

Detailed design of BCP reactor and the picture is shown in Fig. 1. Inner part where a chemical reaction occurred and tubes for cooling circulation were made of Teflon. The outer cover was made of SUS. During BCP process, the reaction temperature was controlled by a chiller. And a Teflon-coated thermocouple was inserted to monitor the acid temperature. In addition, a magnetic stirrer (IKA, C-MAG HS 7) with a stirring speed at dial 2 was utilized to assist more uniform polishing during BCP experiment. Etch rates of samples were obtained by not only measuring the weight of the sample before and after BCP with a micro balance

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but also measuring dissolved Nb ions in BCP solution with ICP-AES.

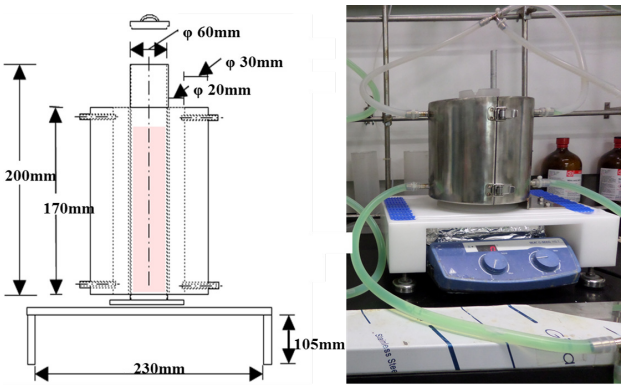

Figure 1: BCP Reactor (a) left: cross-section view of the reactor (b) right: picture of the reactor

RESULTS

BCP data of undeformed (as-received) and deformed niobium samples versus temperature for fixed 50 min. etching time are shown in (a) and (b) of Fig. 2. Red horizontal dot lines in (a) and (b) represent calculated etch rates. For example, 3 μm/min-etch lines mean 150 μm thickness of one surface was etched for 50 min. Since total surface area of each niobium sample equals to the sum of 2×(top and bottom area) and 4×(side area), the total etched mass is 0.411 g by using mass density (ρ) of niobium as 8.57 g/cm^3 when 150 μm thickness is etched, which means, in turn, the weight decrement of the sample is 16% because the total mass of each sample is the product of volume of a sample and mass density ρ. In addition, both weight decrement and dissolved amount of samples during BCP at the fixed temperature of 15°C with time are shown in (c) and (d) of Fig. 2. Similarly, red circles represent calculated etch rates, for instance, 6 μm/min-etch circle represent 32% weight decrement occurred for 50 min, which means doubles of 150 μm thickness was etched for that time, and in turn, the dissolved amount of Nb is 2×0.411 g in (d). Except for 1:1:1 solution case in (a) of Fig. 2 , in most cases, undeformed Nb samples showed higher etch rate than deformed samples. Also, in all cases 1:1:2 BCP solution showed lower etch rate than 1:1:1 solution. Furthermore, etch rate of 1:1:2 solution was relatively uniform than that of 1:1:1 solution from 10°C to 15°C at a given time (see (a), (b)). Except for 1:1:2 solution in (c), etch rate in most cases decreased with time at a given temperature (see (c), (d)). SEM images and optical photographs (by Hirox KH-7700) of a surface and a cross-section of the niobium sample are shown in Fig. 3 and Fig. 4. As one can see, deformed samples showed a texture along certain direction in (c) and (d) of Fig. 3. RMS surface roughness from AFM are plotted in Fig. 5. As-received Nb has around 0.041 μm of RMS$_{9\mu m\times 9\mu m}$ while deformed Nb has 0.101 μm of RMS$_{9\mu m\times 9\mu m}$ by deformation. As described above, etch property was not only uniform but also controllable less than 15°C and it was confirmed in Fig. 5. On the contrary, BCP treatment above 15°C aggravated the surface roughness.

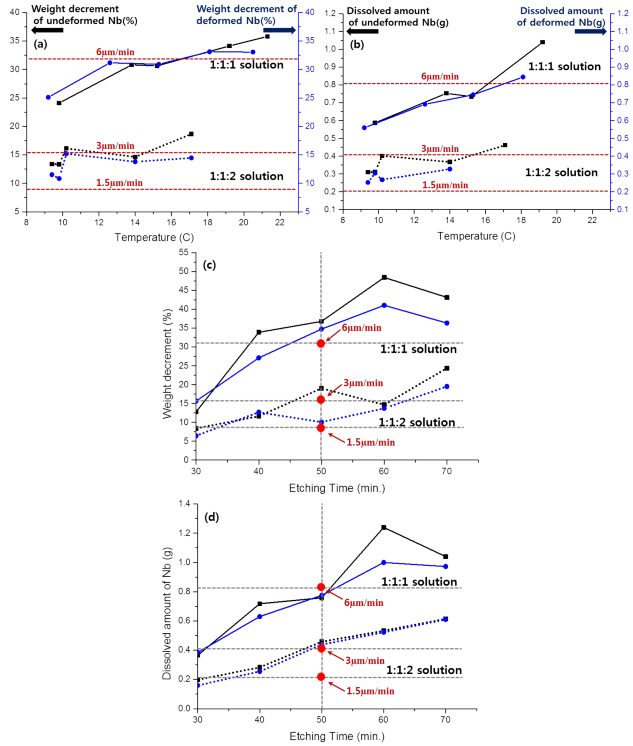

Figure 2: Curves of etch rate vs temperature or time. Black lines represent undeformed Nb sample and blue lines represent deformed Nb sample. (a) weight decrement vs temperature for 50 min. BCP, (b) dissolved Nb amount vs temperature for 50 min. BCP, (c) weight decrement vs time at 15°C, and (d) dissolved Nb amount vs time at 15°C.

DISCUSSION

BCP solution of 1:1:2 ratio showed controllable etch rate below 15°C which is in good agreement with the reference [6]. Measured etch rate of BCP experiments are 1.5 - 3 μm/min for 1:1:2 ratio and 3 - 6 μm/min for 1:1:1 ratio solution. The etch rate we report here is higher than V. Palmieri's work [6], which showed the etch rate was 1 μm/min for 1:1:2 ratio. This might be explained because the record temperature during BCP was lower than the actual reaction temperature. Since the cooling system with a chiller, we might think, did not control a reaction temperature quick enough to precise temperature, this limitation in temperature control induced high etch rate. Decrease of etch rate with time might be understood because active ions (especially F^- ions) in the BCP solution decreased with time. With regard to two exceptions in the results, we might think it was due to the different measurement system by comparing the discrepancy between the weight decrement and the dissolved ions (ICP-AES) graphs. ICP-AES data always showed consistent tendency in etching with temperature and time (see (b), (d) in Fig. 2) while weight measuring data did not (see (a), (c)

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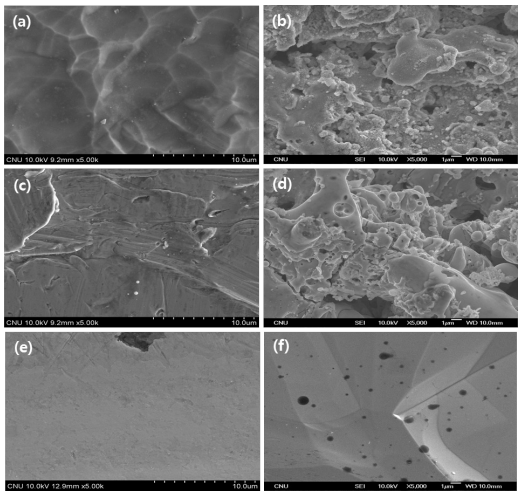

Figure 3: SEM Images of Nb samples (×5000), (a) surface of undeformed Nb, (b) cross-section of undeformed Nb, (c) surface of deformed Nb, (d) cross-section of deformed Nb, (e) surface of undeformed Nb with 1:1:1 BCP treated for 50 min, and (f) cross-section of undeformed Nb with 1:1:1 BCP treated for 50 min.

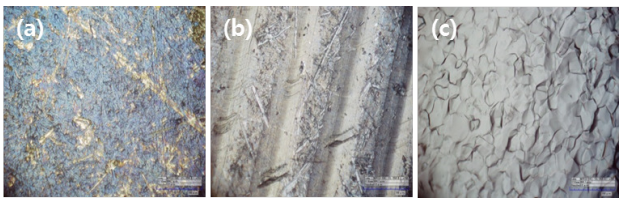

Figure 4: Surface Optical Images (×350), (a) undeformed Nb sample, (b) deformed Nb sample, (c) BCP treated sample with 1:1:1 solution for 50 min.

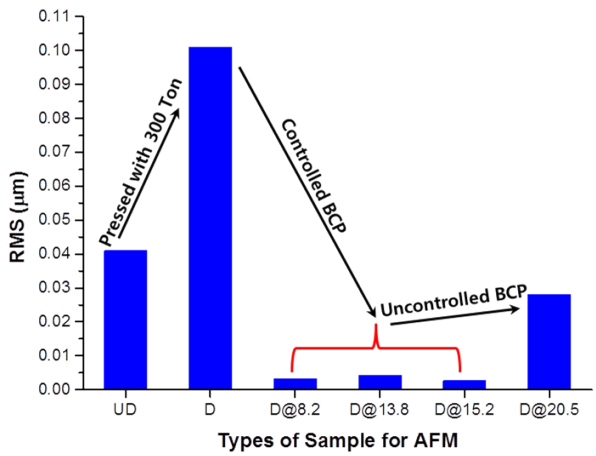

Figure 5: $RMS_{9\mu m \times 9\mu m}$ of Nb samples, UD=undeformed, D=deformed, D@8.2=deformed sample of 1:1:1 BCP treated at 8.2°C for 50 min.

in Fig. 2). Thus, more experiments need to be carried out to understand results. Regarding etch rates of undeformed and deformed samples, we might think that it was due to the texturing of the sample because etch rates can be affected by grain orientations, however still we need more following experiments to understand this phenomena.

SUMMARY

We have performed BCP tests with different Nb samples and BCP solutions. We confirmed BCP solution of HF (48%) : HNO_3 (60%) : H_3PO_4 (85%)=1:1:2 showed relatively consistent etch rate, which is uniform below 15°C. In addition, we have shown that undeformed niobium samples have relatively higher etch rate than deformed samples regardless of solution's ratio, etching temperature and etching time.

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