Acid Etch Rate Measurement

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1 Purpose

This procedure describes the methods to determine the etch rate of niobium in BCP solution.

2 Scope

Master Procedure:
This Master Procedure is intended to be generalized such that it could apply to most items. The Project Manager or Scientific Lead for the project is encouraged to provide project specific instructions to supplement this procedure, which are to be attached to the applicable Traveler or in a project specific procedure.

3 Terms and Definitions

The following terms have specific meanings within this procedure.

Term	Definition
DI	Deionized (DI) and Ultra-Pure Water (UPW) are used interchangeably in this procedure and may also be referred to as simply water.
BCP	Buffered Chemical Polish, a mix of nitric acid, hydrofluoric acid (HF), and phosphoric acid, typically in a 1:1:1 or 1:1:2 ratio.

4 Roles and Responsibilities

The following roles have responsibilities described in this document.

Role	Responsibility
Technician	A trained, qualified person who will execute this procedure.
PI/PM/TR/SL/WCL	Principal Investigator, Project Manager, Technical Representative, Scientific Lead, and Work Center Lead (supervisor). Someone knowledgeable of or in charge of the project or item in question who can provide guidance if questions arise.

5 Safety

The individual must keep safety as the first priority in the process; before beginning any job, the user must assure they have the correct PPE for the individual job. Maintaining the level of safety and secure nature of the work area is paramount. Assure personal safety by using caution in movement and taking necessary steps to avoid unnecessary personnel in the immediate area.

6 Procedure

6.1 INFORMATION

The etch rate changes significantly depending on:

- Acid flow (agitation)
- Acid temperature
- Amount of Nb dissolved in the acid

The following dependencies can be used as a guide:

- The etch rate of Nb in fresh BCP 1:1:1 with moderate agitation depends on temperature as:

  $$ER = 4.8472*\exp(0.030417*T)$$

  Where ER is the etch rate in µm/min and T is the acid temperature in ˚C.

- The etch rate of Nb in BCP 1:1:2 at 20 ˚C (68 ˚F) with moderate agitation depends on the amount of Nb dissolved as:

  $$ER = 3.6871*C^{-0.321}$$

  Where ER is the etch rate in µm/min and C is the concentration of Nb in the BCP solution in g/liter.

- The etch rate of Nb in fresh BCP 1:1:2 with moderate agitation depends on temperature as:

  $$ER = 1.966*\exp(0.031859*T)$$

  Where ER is the etch rate in µm/min and T is the acid temperature in ˚C.

6.2 ACID ETCH RATE MEASUREMENT

1. The following steps allow measuring the etch rate with two methods:
   a. The Nb sample should be cut such that the width and the length are much larger than the thickness. (Typically, about 1" x 3".)
   b. Measure the dimensions (width, length, thickness) of the Nb sample (typically, in inches).
   c. Weigh the Nb sample on the digital balance (typically, in grams).
   d. Measure the thickness of the sample with a digital micrometer at a location marked on the sample (typically, in mm), as shown in Fig. 1.

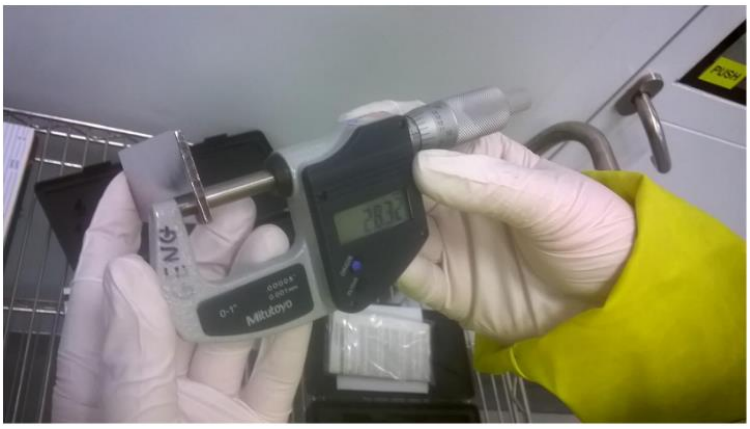

Figure 1: measurement of the thickness of the Nb sample with a digital micrometer at a fixed, marked location

   e. Pre-cool the BCP solution to 15-17 ˚C (59-63 ˚F) with an ice bath, similar to Fig. 2.

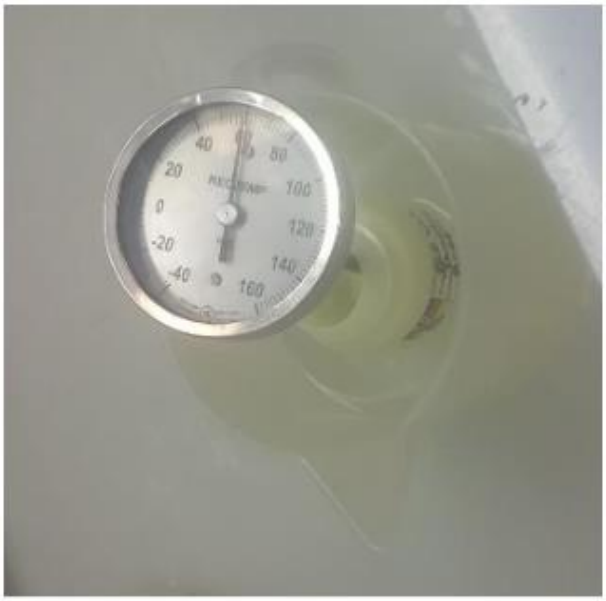

Figure 2: BCP solution pre-cooled in cold water ice bath.

f. Place the Nb sample in a suitable container and immerse into the container with the acid, placed into a container with chilled water, similar to Fig. 3.

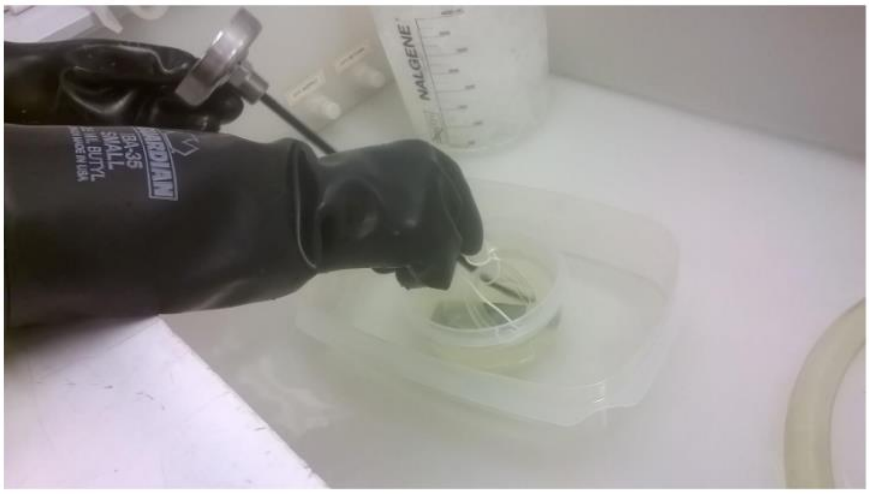

Figure 3: Nb sample in container with BCP solution, placed in a chilled water bath.

g. Etch the sample:
    i. **2 min for BCP 1:1:1**
    ii. **5 min for BCP 1:1:2**
h. It is important that:
    i. The acid temperature is monitored for the duration of the etching and should be maintained in the range 15-17 ˚C (59-63 ˚F). Add ice to the water bath to lower the temperature or DI water to warm it up as necessary, similar to Fig. 4.
    ii. The top and bottom faces of the sample must be constantly immersed and in contact with the acid so that both surfaces are equally etched.
    iii. Gently agitate the sample in the acid continuously.

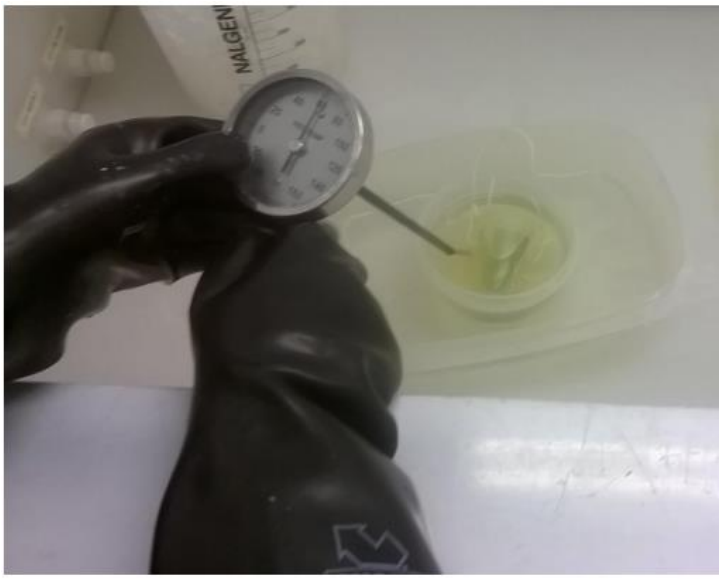

Figure 4: Assure that the acid temperature is kept between 15-17 ˚C (59-63 ˚F) during the etch rate test and that all surfaces of the sample are in contact with the acid throughout the test.

i. Rinse the Nb sample in DI water.
j. Don clean gloves and dry the sample completely with compressed air or N_2.
k. Weigh the Nb sample on the digital balance.
l. Measure the thickness of the sample with a digital micrometer at the same location marked on the sample from step d.
m. Enter the collected measurements and the etch time in the spreadsheet, Acid Usage & Etch Rate.
n. The etch rate by weight is calculated as:

$$ER1 = \Delta w/(\rho * A)$$

Where ER1 is the etch rate in μm/min, Δw is the difference in weight before and after etching in grams, Δ is the density of Nb (8.57 g/cm^3) and A is the sample surface area in cm^2.

o. The other method to calculate the etch rate by thickness is given by:

$$ER2 = \Delta d * 1000/2$$

Where ER1 is the etch rate in μm/min, Δd is the difference in thickness before and after etching measured with the micrometer in mm.

p. If the values of ER1 and ER2 do not agree within **20%** the measurements should be repeated.
q. Record the average value of the etch rate (ER1+ER2)/2 in the logbook.
r. Store the Nb sample protected in a sealed plastic bag.

7 References

Document No.	Title
SRF-01-ML-001	SRF Quality Manual
SRF-20-102692-OSP	Production Chemistry Room OSP

8 Release and Revision History

Rev #	Major Changes	Effective Date:
1	Initial version	03 OCT 2022

9 Approvals

Approved by:	Name:	Signature:	Date:
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