Condition of electron beam welding toward a high gradient application

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Surface defects of superconducting rf cavity cause locally enhanced electric and magnetic field, and trigger electron field emissions and thermal magnetic break down. Therefore surfaces must be smooth.
On and around electron beam welding (EBW) seams, however, defects are often found. An optimum parameter of EBW to minimize a number of defects should be studied.
motivation

As a first step, we are now studying

Surface condition of Nb before EBW

In this talk, I will focus on the recent study about surface condition before EBW, namely, just after the pre-weld etching by buffered chemical polishing (BCP).
I. Existences of carbon including contaminants after BCP
II. What is the origin of contaminants?
III. What is the problem?
IV. Removal of contaminants
V. Summary
Existences of carbon including contaminants after buffered chemical polishing

In the process of observing the surface of HOM antenna cut by water jet, carbon including contaminants were found.

HF rinse (30min) + BCP (200μm) + HF rinse (30min)
Example of a surface condition just after BCP + pure water rinsing

Around the same time, similar contaminants were found on Nb pieces just after BCP.
Example of a surface condition just after BCP + pure water rinsing
A contaminant is transparent and like a membrane.
EDX analysis indicates the contaminants contain “carbon”, “oxygen” and sometimes “nitrogen”.
Origin of “carbon including contaminants” have not been understood.

1. BCP solution container (PTFE, PFA)?
2. BCP solution contamination?
3. From environment?
4. Carbon in Nb (<10ppm)? → likely
• For the case that 40μm of materials are removed from Nb piece (5mm × 10mm), the volume of dissolved Nb is $2 \times 10^{-3}$ cm$^3$. Its weight is about $2 \times 10^{-2}$ g. This means that $2 \times 10^{-7}$ g carbons (10 ppm contaminants in Nb) are dissolved or drifting in BCP solution. This corresponds to $1 \times 10^5$ μm$^3$ in terms of graphite volume, which is equivalent volume of 1000 pieces of contaminants with 1μm × 10μm × 10μm!

• For the case that 40μm of materials are removed from Nb half cell (inner diameter: 56mm, outer diameter: 260mm), the total weight of carbon from Nb corresponds to $2 \times 10^{-4}$ g. This corresponds to $1 \times 10^8$ μm$^3$ in terms of graphite volume, which is equivalent volume of $10^6$ pieces of contaminants with 1μm × 10μm × 10μm!
This means that **EBW processes have been carried out under the presence of “carbon including contaminants” so far!**

Contaminated EBW environment might be a part of reasons generating pits found on and around EBW seams.

→ **Effect on pit formation should be studied.**
Pit formation mechanism?

Carbon including contaminants

Molten Nb with contaminants flood into hall

Electron beam

Contaminants and Nb evaporate

Some contaminants on the molten Nb remain

Molten Nb

Some contaminants on the molten Nb remain

Molten Nb with contaminants flood into hall
Way of removing the contaminants is under study. The following methods are effective at present:

- Ultrasonic cleansing with degreaser (FM20)
- H$_2$O$_2$ with UV (decomposition by “OH radical”)

As we will see later, these methods are effective but not perfect.
Ultrasonic cleansing with degreaser (FM20)
Example of a surface condition just after

**BCP + pure water rinsing**
Example of a surface condition just after

**BCP + pure water rinsing**
A surface condition just after

Ultrasonic cleansing with FM20 (60min)
A surface condition just after

Ultrasonic cleansing with FM20 (60min)

Effective, but not perfect
A surface condition just after Ultrasonic cleansing with FM20 (60min)

Effective, but not perfect
A surface condition just after Ultrasonic cleansing with FM20 (165min)

Additional 105 min $\rightarrow$ null effect
$\text{H}_2\text{O}_2$ with UV (decomposition by “OH radical”)
Example of a surface condition just after BCP + pure water rinsing (Before $\text{H}_2\text{O}_2$ with UV)
Example of a surface condition just after BCP + pure water rinsing (Before H$_2$O$_2$ with UV)
Example of a surface condition just after decomposition by $\text{H}_2\text{O}_2$ with UV irradiation (50min)
Example of a surface condition just after decomposition by H$_2$O$_2$ with UV irradiation (50min)

Effective, but not perfect
Example of a surface condition just after decomposition by $\text{H}_2\text{O}_2$ with UV irradiation (50min)

Effective, but not perfect
Ultrasonic cleansing with degreaser (FM20) + H$_2$O$_2$ with UV (decomposition by “OH radical”)
Example of a surface condition just after **BCP + pure water rinsing**
Example of a surface condition just after **BCP + pure water rinsing**
A surface condition just after

Ultrasonic cleansing with FM20 (60min)
A surface condition just after

Ultrasonic cleansing with FM20 (60min)

Effective, but not perfect
A surface condition just after \( \text{H}_2\text{O}_2 \) with UV (60min) in addition to Ultrasonic cleansing with FM20 (60min). Not enough
1. We found that a lot of contaminants adhere on Nb pieces after BCP.
2. Contaminants include carbons. However we have not identify their chemical structure.
3. The origin of carbon has not been identified yet. Carbons in Nb (< 10ppm) are likely candidates.
4. Way of removing “carbon including contaminants” is under study. “Ultrasonic cleansing with FM20” and “decomposition by H₂O₂ with UV irradiation” are effective, but a complete removal has not been successful.
5. Effects on pit formation at EBW seam should be studied.
note
We prepared Nb pieces as clean as possible in order to check that “carbon including contaminants” are produced in the process of BCP.

**procedure**

1. **Cut**: Cut Nb plate into several pieces by wire-EDM or shear etc.

2. **BCP-1**: Chemically polish a Nb piece in order to remove contaminations by cut process such as machine oil, wire-derived copper, and so on.

3. **Remove contaminants after BCP1**: There are a lot of carbon including contaminants on the Nb surface. But we do not know whether these contaminants are originated from BCP-1 itself or machine oil before BCP-1. Therefore we remove these contaminants and consider a obtained piece as a standard Nb sample.

4. **BCP-2**: BCP is applied to the standard Nb sample obtained through the above processes.

5. **Observe**
Way of removing the contaminants is under study.
So far we have tested the following method:

- Ultrasonic cleansing with acetone
- Ultrasonic cleansing with pure water
- HF rinse
- Ultrasonic cleansing with HF
- EP solution rinse
- Phosphoric acid rinse
- brushing
- Etching by $\text{H}_2\text{O}_2$ including BCP solution
- $\text{H}_2\text{O}_2$ with UV (decomposition by $\cdot$OH radical)
Ultrasonic cleansing with acetone
A surface condition just after BCP + pure water rinsing
A surface condition just after

**BCP + pure water rinsing**
A surface condition just after

**Ultrasonic cleansing with Acetone (35min)**
We can not remove “carbon including contaminants” by ultrasonic cleansing with acetone.
A surface condition just after

**Ultrasonic cleansing with Acetone (35min)**

Carbon and oxygen

Carbon and oxygen

Carbon and oxygen

Carbon and oxygen
A surface condition just after Ultrasonic cleansing with Acetone (35min)

Carbon and oxygen

20μm

Nb