

## Procedure for Polishing Niobium Sheets in Pucks

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With technical contributions from Donna Hicks and Alex Romanenko

### Introduction

This procedure was developed for the purpose of polishing niobium sheets to observe pieces of cavities with pitting problems. It was developed and employed for the polishing of niobium sheets in December 2011 and January 2012, using some experimentation with a procedure similar to that already in use for polishing Nb<sub>3</sub>Sn, and another, developed by Donna Hicks, already in use for manual polishing of niobium sheets, to find the most preferable outcome. It was decided after viewing of the samples by Alex Romanenko that the procedure described below is the most appropriate for the polishing of niobium sheets.

### Sanding Steps (Developed by Donna Hicks with modifications by Marianne Bossert)

Sanding, or grinding, steps recommended depend on the amount of epoxy covering the sample to be viewed under the microscope. If any visible epoxy is covering the area to be viewed under the microscope, beginning with 180 grit sandpaper is best in order to maximize material removal. In this case, the sample should be sanded until all areas to be viewed are exposed. If no visible epoxy is covering the sample, the 180 grit step can be omitted and sanding can begin on 240 grit paper. Subsequent sanding steps are 320 grit paper, 400 grit paper, and 600 grit paper. In order to maximize flatness, adhesive-backed sandpaper should be placed on a granite slab or table, although a Buehler Handimet roll grinder can be used when available. These steps should not be done on an automatic polisher, as this is normally too abrasive to achieve the grinding steps effectively. Distilled water should be used during sanding to lubricate sandpaper, but only enough water to keep the paper damp should be used – the paper should not be dripping wet.



Buehler Handimet Roll Grinder

Samples are to be sanded until the surface to be viewed appears uniform to the naked eye. This is the most important factor to consider in the sanding steps; if the surface does not appear uniform to the naked eye, the step is not complete and the next step will take much longer to achieve. Samples can be checked under the microscope, and direction and size of scratches can be viewed, but these are not necessarily a definite indicator of a sample that is polished adequately during the sanding steps. The sample must be uniform in color and texture on a macroscopic level in order for the next step to be effective.

### **Cleaning**

When a grinding step is believed to be complete, the sample should be washed with a generous amount of Dawn dish detergent and allowed to dry completely. The best method for drying the sample is to use a clean room wipe in order to avoid scratching the sample, but Accuwipes or Kimwipes can be used as well as long as great care is taken to avoid scratching the sample. This care becomes more important as grinding and polishing progress; scratches occurring from abrasive drying are less likely to be removed on finer steps. Air drying is not recommended as water spots can change the appearance of uniformity. The sample should be checked again for uniformity after washing, as residue remaining from sandpaper can mask issues in the grinding steps.

The sample should then be put in an ultrasonic cleaner for 5-10 minutes, using a 5:256 ratio of Micro cleaning solution and distilled or ultrapure water. This recommended ratio can be varied or approximated greatly before any ill effects to the sample occur. The sample should then be dried in the aforementioned way and checked once again to ensure that the surface still looks uniform. This step should be repeated every time a new grinding step is completed, and replacement of water and Micro solution is recommended.



Ultrasonic Cleaner

**Automatic Polishing Steps** (Developed by Marianne Bossert)

Automatic polishing was done on a Leco GPX 300 Automatic Polisher. Using automated polishing for these steps appears to produce the best results with the least amount of operator involvement, as it eliminates variations in pressure and speed and the need to hold the sample on a polishing wheel for extended periods of time. Manual polishing of these samples, while not impossible, is very labor intensive as these steps can take several hours to complete.

For this step, Texmet C pads from Buehler are recommended, as these are hard enough to ensure material removal and keep edges intact but soft enough to avoid scratching the sample. 6 micron diamond oil base suspension is also to be used liberally on the Texmet pad. A new Texmet pad should be used for every new set of samples, and the pad should be saturated with the suspension when polishing begins. More suspension should be added every time the pad appears dry, or there are no visible oil swirls.



Leco GPX 300 Polisher with Buehler Texmet Polishing Pad and Buehler 6 Micron Oil Base Suspension

The polisher should be used in single force mode only. The polisher should never be used in central force mode – this is ineffective especially for finer polishing steps, which is the only kind of step recommended to be polished on the automatic polisher. The holder with the appropriate puck size (either 1.5” or 1.25” diameter) should be inserted into the head of the

polisher while the head is up, above the polishing pad, and the head should then be lowered to check the clearance above the polishing pad. If the holder touches the polishing pad or comes close, it should be adjusted to avoid contact between the holder and the polishing pad and especially to avoid contact between the fingers and the pad in empty places for samples, as this could damage the polisher. The polishing pad should also be changed during the process any time it begins to appear opaque black, because this is the point at which pullout and contamination may begin to occur.

The 6 micron polishing step is a repetition of four steps, each of which is five minutes long. The pressure, rotation directions, and rotation speeds are represented here:

Step	Pressure	Pad Rotation	Head Rotation
1	0 pounds	40 RPM Clockwise	30 RPM Counterclockwise
2	1 pound	110 RPM Counterclockwise	30 RPM Clockwise
3	2 pounds	120 RPM Clockwise	45 RPM Counterclockwise
4	3 pounds	150 RPM Counterclockwise	45 RPM Clockwise

These steps should be repeated multiple times in order to ensure an effective polish. Samples should be checked after every set of four steps by washing with a generous amount of Dawn dish detergent, drying with a clean room wipe, an initial eye inspection, and a microscope inspection. Samples will first appear to be uniform in that the entire surface to be viewed becomes shiny. At this point, the samples must be checked under the microscope to determine if any deep scratches remain. Often, deep scratches still exist even after the samples initially appear shiny to the naked eye. The best way to identify if the sample is polished effectively at this level is to check it under the microscope – at this level, all scratches should appear to be the same size. If any scratches are significantly larger in width or apparent depth (which can be observed to some degree by using the polarized light feature available on several microscopes) than other scratches, the sample must be put back on the polisher and the polishing steps should continue until all the scratches are uniform.

This collection of steps takes, in practice, anywhere from three hours to one day to complete. Quality of the grinding steps greatly affects the amount of time and the efficiency of polishing at this level, which is why taking care to observe uniformity during the grinding steps is important. Overpolishing at this step is also discouraged, because pullout can occur.

### **Vibromet Polishing** (Developed by Donna Hicks)

The recommended final polishing step for niobium sheets in pucks uses the Vibromet II Buehler vibrating polisher. 0.04 micron colloidal Silica and an Allied Ultragam pad were used, with care taken to ensure the entire pad was covered with polishing solutions. The samples were then polished for eight hours at 20-40% amplitude. Each sample was fixed in a holder

specifically designed for the Vibromet polisher, with care taken to avoid scratching of the sample and to ensure all surfaces of the sample were outside of the holder to allow them to have contact with the polishing pad. One weight was added to each of the holders. The samples were then left on the Vibromet polisher for eight hours and checked approximately every hour. Operating the machine overnight when no one is available to check the progress is not recommended, as drying of the pad or other problems may occur. Following polishing, an additional step of etching with hydrofluoric acid allows for the best analysis. After Donna Hicks performed this step, the samples were observed in a scanning electron microscope by Alex Romanenko, who confirmed that the polishing was adequate.



Buehler Vibromet Polisher



Vibromet holder with weight