

# LASER POLISHING OF NIOBIUM FOR SRF APPLICATIONS\*

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## Abstract

Smooth interior surfaces are desired for niobium SRF cavities, now obtained by buffered chemical polish (BCP) and/or electropolish (EP). Laser polishing is a potential alternative, having advantages of speed, freedom from chemistry and in-process inspection. Here we show with Power Spectral Density (PSD) measurements that laser polishing can produce smooth topography similar to that obtained by EP. We studied the influence of the laser power density and laser beam raster rate on the surface topography. These two factors need to be combined carefully to smooth the surface without damaging it. Computational modeling was used to simulate the surface temperature and explain the mechanism of laser polishing.

## BACKGROUND

Surface chemistry is needed in the fabrication process of niobium SRF cavities [1]. Buffered chemical polishing (BCP) with 1:1:2 (or 1:1:1) solution is commonly applied for fast etching and cleaning of niobium pieces. Electropolishing (EP) is optionally used as a final step to remove sharp features caused by BCP. These two techniques have become standardized and are being used routinely in large quantity production. However, pursuit of greener and/or faster treatment methods is still on-going, such as centrifugal barrel polishing (CBP) and non-HF EP. We have shown that laser polishing is able to achieve smooth topography on niobium [2, 3] that is similar to the EP produced surface. In this study we continue our efforts to further explore parameter space suitable for laser polishing and the polishing mechanism itself.

## EXPERIMENT

### Material and Preparation

The niobium samples were 49 mm diameter disks cut from 3.2 mm high RRR sheet material of the type used for cavity fabrication. The niobium samples were etched in 1:1:1 BCP solution for 1 minute, rinsed with de-ionized water and air dried. The resulting sample surface resembled typical BCP topography. The sample disks were loaded into the laser treatment system and pumped down to  $\sim 10^{-7}$  -  $10^{-8}$  Torr for polishing experiments.

### Laser Treatment System

A Spectra-Physics High Intensity Peak Power Oscillator (HIPPO) table top laser was used as the laser source. The wavelength used for the polishing experiment

was 1064 nm. The beam was directed into the UHV chamber of a PVD-5000 System. The focused spot size on the niobium samples was  $96 \mu\text{m} \times 104 \mu\text{m}$  FWHM (spot area  $7.84 \times 10^{-5} \text{cm}^2$ ). The repetition rate was 19 kHz, and the pulse length at this repetition rate was about 8 ns.

### Polishing Parameters

Table 1 summarizes the range of parameters explored in our experiments. The number of pulses overlapped within one beam width, the pulse displacement and scan speed are listed.

Table 1: Range of Parameters Covered

Fluence	Number of pulses overlapped	Pulse displacement	Scan speed
J/cm <sup>2</sup>	Within one beam width	$\mu\text{m}$	cm/s
0.18~0.61	15~960	6.4~0.1	12.16~0.19

### Characterization

After laser treatment, the topography of the niobium samples was studied by optical microscopy and atomic force microscopy (AFM). Root mean square (RMS) roughness was obtained from AFM data. Power spectral density (PSD) analysis was applied to the AFM data as we have described previously [4].

## RESULTS

Figure 1 shows the optical images of niobium surfaces after laser treatment with different combinations of fluence and number of pulses overlapped.

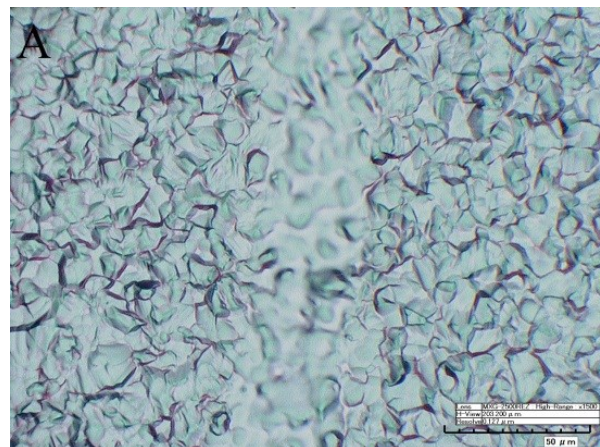


Figure 1A: Optical images of niobium surfaces after laser treatment:  $0.24 \text{ J/cm}^2$ , 53 pulses overlapped,  $1.8 \mu\text{m}$  pulse displacement, scanning speed  $3.4 \text{ cm/s}$ .

\*Work supported by DOE Office of High Energy Physics under grant DE-FG02-12ER41838.  
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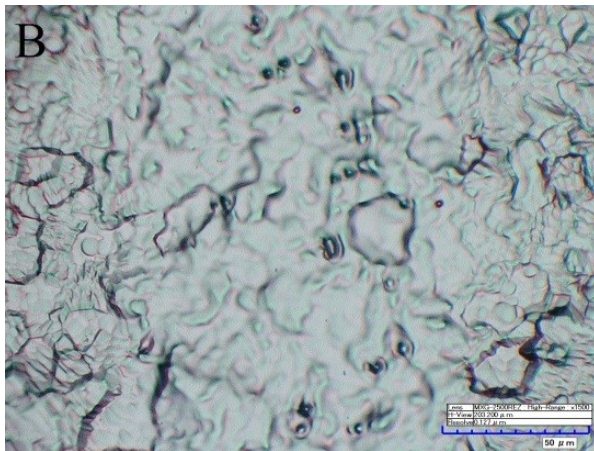


Figure 1B: Optical images of niobium surfaces after laser treatment:  $0.61 \text{ J/cm}^2$ , 32 pulses overlapped,  $3 \text{ μm}$  pulse displacement, scanning speed  $5.7 \text{ cm/s}$ .

Both  $0.24 \text{ J/cm}^2$  and  $0.61 \text{ J/cm}^2$  cases showed a smoothed surface. But the scanning speed was  $3.4 \text{ cm/s}$  and  $5.7 \text{ cm/s}$  respectively. Therefore, proper combination of fluence and scanning speed is important to achieve polishing effects. The benefit of higher fluence is that it enables a higher polishing rate.

Figure 2 A, D shows the AFM image of the sample surface in Fig. 1A. Figure 2 A is the surface on the same sample without laser treatment, which is a typical BCP topography. Figure 2 D is the BCP surface after laser polishing. Figure 2 B, C are typical EP and CBP surfaces of niobium. The CBP surface was produced based on the standard recipe as described in reference [5]. It is easy to notice the wavy and smooth shape on EP surface (despite the relatively large  $R_q$  value due to larger scanned area than the other three), the ridges on BCP surface and the special pattern on CBP surface. The different characteristics of these surfaces result from their different polishing mechanisms. The laser polished surface most closely resembles the EP surface.

The PSD of surface height is a useful measure of topography from a spatial frequency point of view. Larger PSD value on the curve means larger contribution from corresponding spatial frequency. Sharp "fractal" edges on the surface will produce a straight line "power law" on such a log-log plot. Figure 3 shows the PSD analysis result of the four types of surfaces in Fig. 2. The BCP surface shows a straight line between  $10^4 \text{ nm}$  and  $10^2 \text{ nm}$ . The laser polished BCP surface shows a curve with lower PSD value than BCP line from  $0.25 \times 10^4 \text{ nm}$  to  $10^2 \text{ nm}$ , meaning the sharpness on BCP surface is reduced. The EP curve shows two steps: one from  $0.5 \times 10^5 \text{ nm}$  to  $0.25 \times 10^4 \text{ nm}$  and another from  $0.25 \times 10^4 \text{ nm}$  to  $0.2 \times 10^3 \text{ nm}$ . The CBP curve shows lower PSD than other curves over most of the range except the slight hump around  $10^3 \text{ nm}$ . This could relate to the features observed in Fig. 2 C, which are attributed to the polishing media.

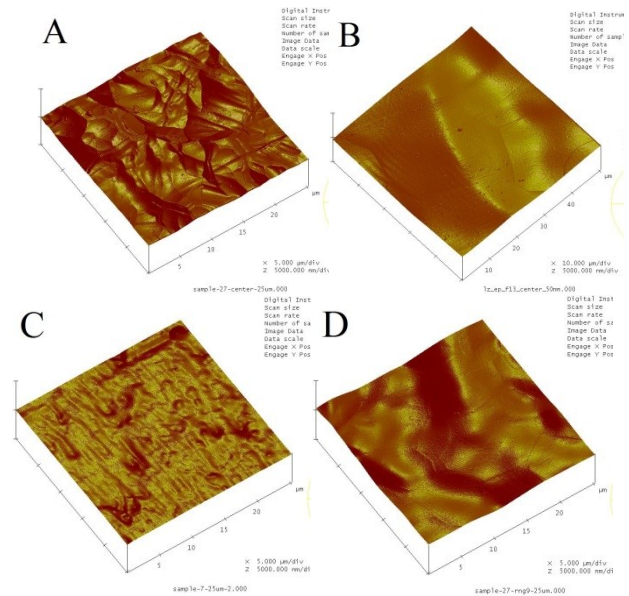


Figure 2: AFM images of niobium surface after: A) BCP,  $R_q=177 \text{ nm}$ ; B) EP,  $R_q=271 \text{ nm}$ ; C) centrifugal barrel polishing (CBP),  $R_q=31 \text{ nm}$ ; D) laser polishing (LP),  $R_q=170 \text{ nm}$ . Scanned areas are  $25 \text{ μm} \times 25 \text{ μm}$  in A, C, D, and  $50 \text{ μm} \times 50 \text{ μm}$  in B.

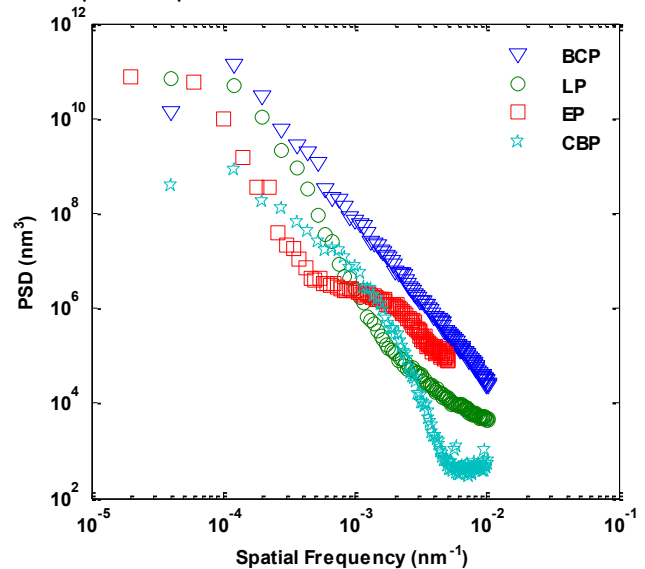


Figure 3: PSD analyses of the four types of surface in Figure 2.

## SIMULATION AND DISCUSSION

As a guide to experiment and for preliminary understanding, a one-dimensional conduction heat transfer equation with initial and boundary conditions below [6] can be used to simulate laser polishing of niobium, if the surface does not undergo severe ablation process. Here we used constant thermal properties. No radiant heat loss or melting was considered.

Figure 4 shows the simulation result of niobium temperature as a function of time during laser treatment at fluence of  $0.24 \text{ J/cm}^2$  and  $0.61 \text{ J/cm}^2$ . The temperature rise due to the first laser pulse is shown in the figures. The pulse starts from time zero and ends at  $8 \text{ ns}$  (the dotted

vertical line). Initial temperature is room temperature 20°C. The temperature drop after the pulse is simulated to 100 ns. Both the surface temperature and the melt-depth temperature profiles are shown in each plot. The calculated melt depth due to the first pulse in the two cases is 50 nm and 520 nm respectively. And we noticed that the surface temperature dropped below the melting point a few nanoseconds after the pulse ending, so the melted part solidified quickly after the pulse. For the 0.24 J/cm<sup>2</sup> case, only a shallow layer of the surface was in the melting region as expected. For the 0.61 J/cm<sup>2</sup> case, a thicker surface layer was melted, and the calculated surface temperature even exceeded the boiling point of niobium. Surprisingly, we didn't observe ablation on the sample surface. One reason for this discrepancy could be, fluence this high is beyond the scope of our assumptions. The actual temperature vs. time curve could be different, because the thermal properties of liquid niobium are different from solid niobium and the latent heat of phase change needs to be considered as well.

Another factor not included in the simulation is the influence of earlier pulses on later pulses. And this may well relate to the pulse accumulation factor we have been using in the experiments. In fact, the temperature of sample does not return to the initial temperature when the next pulse arrives. As the heat from earlier pulses accumulates on the sample disk, the starting temperature for each pulse becomes higher. And the initial temperature could make a difference on the temperature profiles shown in Figure 4 [3].

Despite these limitations, the simulation indicates that, if we scan the laser fast enough, the surface could still be polished instead of damaged, as seen in the topography results above.

It would be useful to continue this study with more precise control of the parameters and a wider range of variables accessible; for example, changing the pulse width, lower the repetition rate and changing the sample temperature. It would also be very helpful if the sample temperature could be monitored during the polishing process.

### CONCLUSION

- Laser polishing of niobium is achievable, if a proper combination of fluence and pulse displacement is applied.
- Comparison of topography and PSD analysis of laser polishing with other polishing methods shows that laser polishing smoothens sharp features in a way similar to EP.
- One dimensional heat conduction model shows that a melting depth from 50nm to 520nm can be achieved.

### ACKNOWLEDGMENT

The experiment was conducted at Jefferson Lab. Authored by Jefferson Science Associates, LLC under U.S. DOE Contract No. DE-AC05-06OR23177. The U.S. Government retains a non-exclusive, paid-up, irrevocable, world-wide license to publish or reproduce this manuscript for U.S. Government purposes. Thanks to staff at the College of William and Mary characterization lab, especially Olga Trofimova for the AFM images. Thanks to Chen Xu for the PSD program, and Ari Palczewski for helping with the CBP samples.

### REFERENCES

- [1] C. E. Reece et al., Reviews of Accelerator Science and Technology 5 (2012) 285.
- [2] L. Zhao et al., "Parameter Optimization for Laser Polishing of Niobium for SRF Applications," WEPWO087, p2498 (2013), Proceedings of IPAC2013, Shanghai, China.
- [3] S. Singaravelu et al., J. Vac. Sci. Technol. B 30 (2012) 051806.
- [4] C. Xu et al., Physical Review Special Topics – Accelerators and Beams 15 (2012) 043502.
- [5] A. D. Palczewski et al., "Exploration of Material Removal Rate of SRF Elliptical Cavities as a Function of Media Type and Cavity Shape on Niobium and Copper Using Centrifugal Barrel Polishing (CBP)," TUP063, Proceedings of SRF2013, Paris, France.
- [6] E. M. Breinan et al., Laser Materials Processing, (North-Holland, 1983), 241.

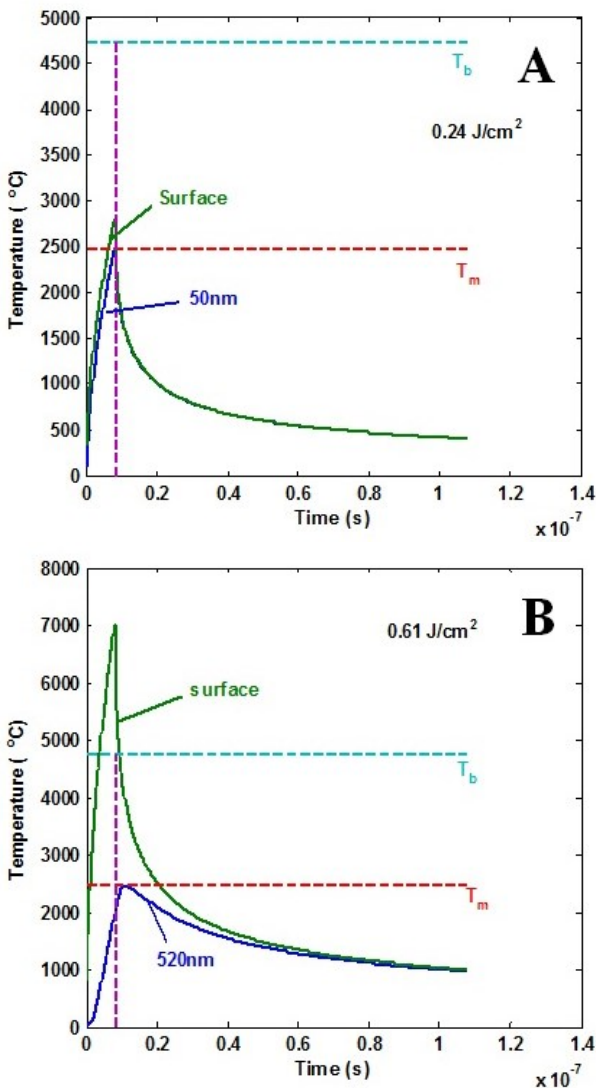


Figure 4: Temperature as a function of time during laser treatment at fluence of A) 0.24 J/cm<sup>2</sup> and B) 0.61 J/cm<sup>2</sup>.