LASER RE-MELTING INFLUENCE ON NB PROPERTIES: GEOMETRICAL AND CHEMICAL ASPECTS

A. Dzyuba#, FNAL, Batavia, IL 60510, USA and NSU, Novosibirsk, 630090, Russia
E. Toropov##, L. D. Cooley, FNAL, Batavia, IL 60510, U.S.A.
M. Ge, Cornell University, Ithaca, NY 14853, U.S.A.
G. Wu, ANL, Argonne, IL 60439, U.S.A.

Abstract

Repair of SRF cavities has provided improved yield at 35 MV/m. Laser melting is one technique to remove quench limitation by altering the niobium defect topography. In this paper we present a part of our recent R&D using a single laser pulse to deposit enough energy to entirely melt the defect area. The pulse produces morphology analogous to an impact. Also, the short time takes advantage of the argon cover gas to minimize oxidation. Materials characterizations are presented and discussed with regard to the advantages of the technique from the materials point of view.

INTRODUCTION

A significant fraction of cavity quench locations can be traced to surface irregularities, usually hemispherical pits and crack - like terraces. It has been suggested that magnetic field lines are enhanced on the edges of those objects, which leads to the exceeding of the local critical field and to the initiation of the thermal runaway process when flux lines then penetrate [1]. The model ties in the external magnetic field with geometrical parameters of the pit, predicting higher quench field for bigger defects [2]. Thus, it is essential to smoothen such local imperfections so field enhancement no longer imposes a limit to the accelerating field.

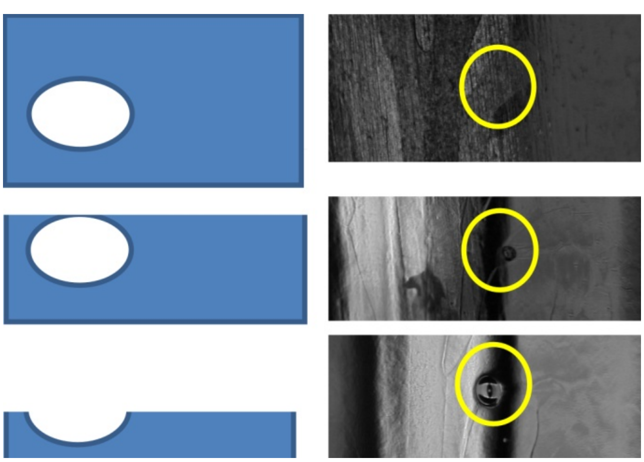

Figure 1: Defect uncovered by consequent EP and tracked by optical inspection (FNAL, D. Sergatskov).

The origin of pits is presently being debated. One explanation is that hydrogen bubbles are uncovered during chemical polishing. This suggests local repair approaches are valuable to repair pits, since bulk re-polishing might expose new irregularities (see Fig.1). Taking into account that the size of pits varies from 100 μm to 1000 μm, as well as the constraints imposed by working from the inside of the cavity, repair techniques can be highly technical. Laser melting provides a highly collimated, high intensity beam capable of altering the Nb surface. The proof of this principle has been demonstrated by G.Wu and M.Ge on a 1-cell cavity [3]. Here, we analyze in more depth the effect of various parameter choices on Nb surface morphology and composition. to better understand the advantages of the laser over other local repair techniques. We also aim to develop an appropriate post processing, if any. To achieve that goal the Electron Backscattered Diffraction (EBSD) microscopy has been used on the same level with Laser Scanning Confocal Microscopy (LSCM). In this paper we present only the results of single pulse melting, as used by Ge and Wu earlier. Multiple pulses can also be used to more gently heat the surface and produce different effects, which will be the subject of another article.

#dzyuba@fnal.gov
##etoropov@fnal.gov

EXPERIMENTAL DETAILS

Single pulse melting has been performed on the both single grain and fine grain Nb coupons with indented pits. The indentation of artificial defects is supposed to model both the geometrical perturbation (hemispherical pit, Fig.4 middle row) and subsurface microstructural perturbations (enhanced dislocation content, Fig.2 middle row) found in actual cavity pits. After remelting microscopy analysis has been performed in the FNAL Microscopy Lab by means of the EBSD (Oxford INCA 200) system built upon a scanning electron microscope (JEOL JSM-5900LV). Topography was analyzed with a LCSM (Keyence VK-9700 Color 3D).

RESULTS AND DISCUSSION

In Figure 2 the single crystal data is presented. The left column consists of electron microscope snapshots of initial surface, of indent on the surface and of remelted indent (from top to bottom respectively). Right column represents the local misorientation maps. This EBSD capability shows changes of grain orientation over a small scanning area, which can be associated with the presence of dislocations and stress. The color amplitude is significantly increased after indenting (second row, right picture) due to the plastic deformation, whereas after remelting the dislocation density drops to the initial value observed before indenting. Hence, these data imply that the laser does indeed anneal the Nb surface over the broad area of the indent. This result is of particular importance for elimination of losses that might be caused by dislocations or material located on them [4].

It is interesting to notice that the EBSD data could not be obtained until after a light HF rinse of the sample after the remelting procedure. Since the EBSD software acquires information from the top 40 nm of surface and indexes according to crystallographic reflections, this suggests that an amorphous region existed on the as-melted surface. It is most probable that an amorphous oxide layer is formed, as discussed shortly.

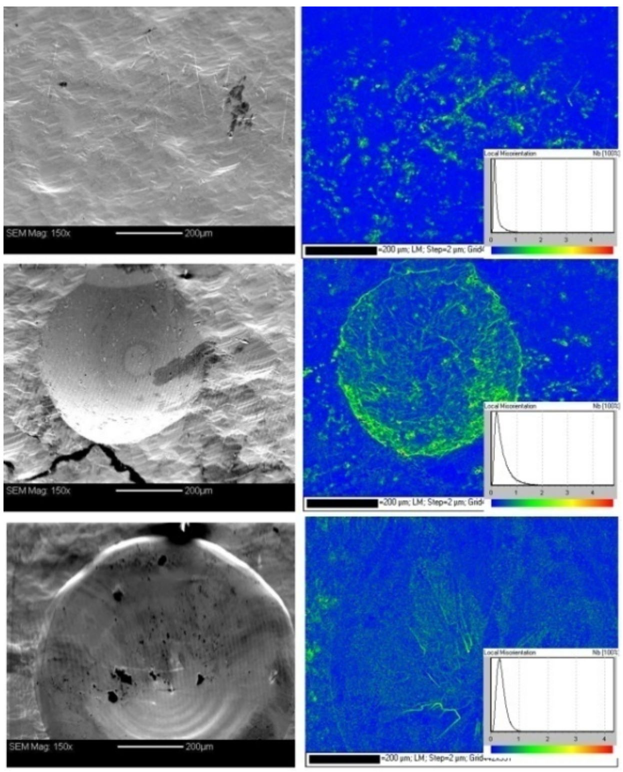

Figure 2: Electron microscope snapshots of initial surface, indent on the surface, remelted indent(left column). And concurrent local misorientation maps, showing stress amount persisting during each of the steps obtained by means of EBSD (left column).

On Fig.3 the orientation and local misorientation maps are presented for fine grain niobium surface in the absence of the pit (i.e. purely due to the heat-affected zone HAZ of the laser remelt spot). Orientation mapping is another of the EBSD capabilities. Each color corresponds to a certain atomic plane orientation, with the direction normal to the surface given by the legend insert on Fig.3. The technique allows a visualization of the grain pattern. The orientation map of the Fig.3 exhibits recrystallization behaviour with radial pattern. Origin of the radial signature could arise from either thermal gradient or shock wave propagation. Such re-solidified zones exhibit improved RRR in electron beam welds [5] and perhaps laser melting gives a similar improvement in that respect.

However, from the misorientation map registered for the same heat affected zone, another pattern is observed. Green color within the HAZ corresponds to dislocations concentration at the grain boundaries. Further, the green background outside the blue circle suggests that there is an increase in contamination, which reduces the EBSD signal from unperturbed grains outside the HAZ. These data imply that stress tends to concentrate in between re-solidified grains. In analogy with HAZ of cavity welds [6], this stress might increase pitting due to etching treatments as post processes. This prediction is confirmed for the sample etched after the melting procedure. EBSD contrast map (Fig.4) reveals deep terraces within the HAZ. The formation of those could be easily understood by the local preferential etching effect. Thus it’s clearly seen that every BCP – like treatment should be avoided after laser melting.

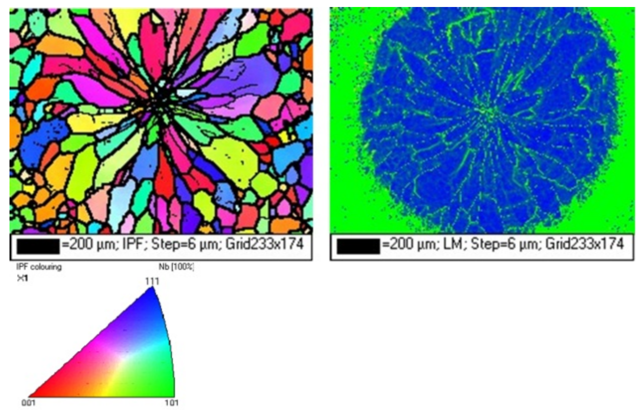

Figure 3: Orientation map and misorientation map for remelted zone in the absence of the artificial pit. Legend inclusion describes the color scheme for OM.

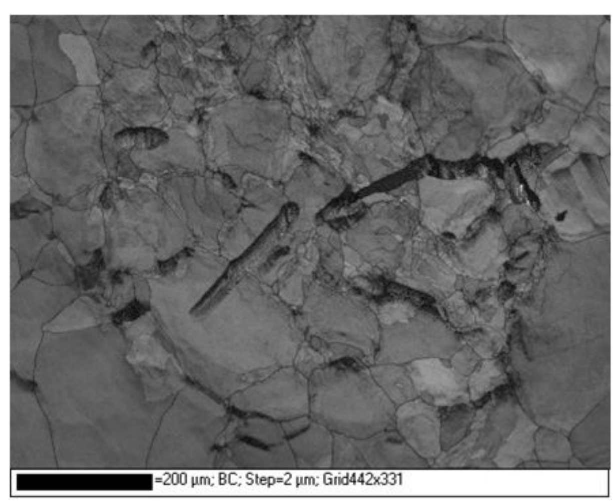

Figure 4: Contrast map of the HAZ underwent light etching obtained by means of EBSD.

Taking into account the theory of the magnetic field enhancement [2], we studied the impact of the remelting on the geometry of the pit as well. On Fig.4 one can see 3d and 2d profiles of the indented pit and of the HAZ after melting. Improvement of the topography is observed: the depth of the pit decreased and the slope of the pits edges decreased at the same time single pulse melting has been performed on the both single grain and fine grain Nb coupons with indented pits.

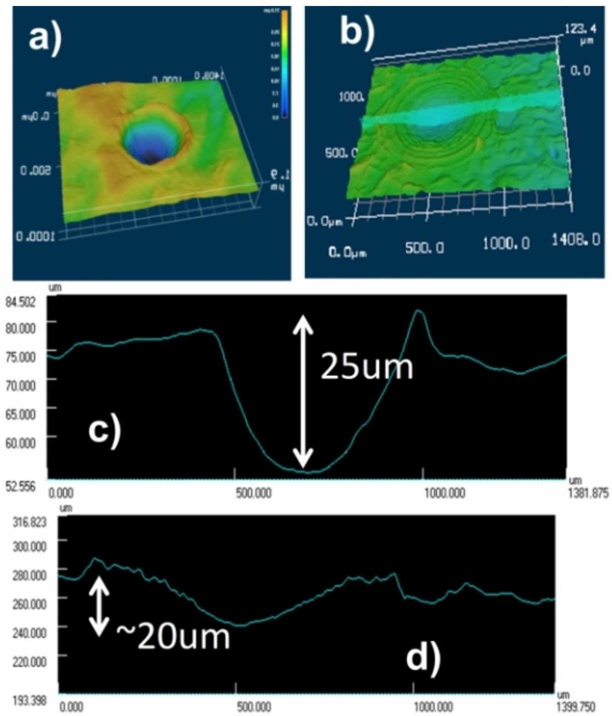

Figure 5: Laser confocal microscopy data. (a) 3D - indented pit, (b) HAZ after remelting. (c) 2D - indented pit, (d) HAZ after remelting.

CONCLUSION

Therefore results of the HAZ analysis after single pulse Laser treatment show explicit improvement in both geometrical and microstructural aspects. The investigation will be continued by means of EDX and Auger analysis as well by investigation of the multi pulse impact.

ACKNOWLEDGMENTS

The authors would like to thank Superconducting Materials Department (FNAL) members for samples preparation and fruitful discussions.

The research is supported by Fermi Research Alliance, LLC under Contract No. DE-AC02-07CH11359 with the United States Department of Energy

REFERENCES

[1] C. Reece, “Magnetic field enhancement at an inside corner of a cavity: Results of SUPERFISH calculations”, LEPP Report SRF-840302.

[2] V. Shemelin, H. Padamsee, “Magnetic field enhancement at pits and bumps on the surface of superconducting cavities”, TTC Report SRF – 080903-04.

[3] M. Ge, G. Wu, J. Ruan, J. Ozelis, T. Nicol, D. Sergatskov, D. Hicks, L. Cooley, “Laser re-melting technique at FNAL”, TTC meeting 2010.

[4] A. Romanenko and H. Padamsee, 2010 Supercond. Sci. Technol. 23 045008.

[5] A. Aizaz, P. Bauer, T .L. Grimm, N.T. Wright, and C. Z. Antoine, “Measurements of thermal conductivity and Kapitza conductance of niobium for SRF cavities for various treatments”, IEEE transactions on applied superconductivity, vol. 17, no. 2, June 2007.

[6] T. R. Bieler, N. T. Wright, F. Pourboghrat, C. Compton, K. T. Hartwig, D. Baars, A. Zamiri, S. Chandrasekaran, P. Darbandi, H. Jiang, E. Skoug, S. Balachandran, G. E. Ice, and W. Liu, “Physical and mechanical metallurgy of high purity Nb for accelerator cavities”, Phys.Rev.Spec.Topics – Acc. And Beams. 13, 031002 (2010).