REDUCING ELECTROPOLISHING TIME WITH CHEMICAL MECHANICAL POLISHING

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Summary:

It is commonly admitted that 150-200 µm need to be removed by electropolishing on the internal surface of niobium RF cavities before reaching optimal results. The proposed reason is generally the existence of a damage layer on the Nb sheet surface. Indeed recent disorientation measurement made at Cornell [1] show that hotspots exhibit higher misorientation. Damage has also been considered in the formation of pits close to the welding seam during electropolishing.

Removing of 200 µm by electropolishing is a hazardous process, not only because of the dangerous chemicals involved in the process, but also because of the spread of results, probably due to the chemical mixture aging. Reducing the amount of electropolishing to a final light treatment would be a way to decrease both costs and risks of the RF cavities for large projects such as ILC.

We have tried to evaluate the thickness of the damage layer after various deformations steps (mainly rolling, deep drawing and chemical mechanical polishing) by observing the density of etching figures after several light chemical etches. This provides a coarse but very rapid evaluation of the thickness of the damage layer. Complementary observations with EPSB are also presented.

Finite element, orientation imaging and/or and etching figures show that the damage layer induced by rolling is noteworthy already ~150 µm thick, with a specific (001) texture that resists recrystallization.

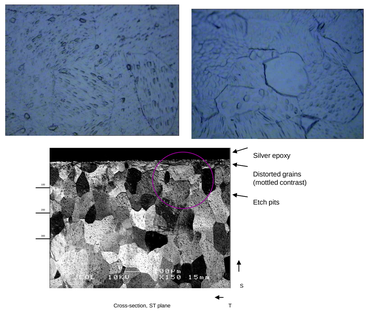
Deep drawing brings further and deeper damage in particular in the equator region where the friction against the forming dye is the highest. Welding also influences the damage distribution.

Getting rid of this damage layer is possible with BCP, but it needs another 100 µm to smoothen the surface afterwards. Mechanical polishing like tumbling obviously leaves a thick damage layer, but "chemical mechanical" polishing is a way to prepare surfaces with a very thin damage layer (< 1µm?). We think that chemical mechanical polishing of half cells before welding would be a way to decrease the thickness of electropolishing necessary for the preparation of RF Nb cavities, and reduce costs and risks.

Signs of the damage layer:

Etching figures

- Typical etching figures
  - optical microscope, surface etching
  - SEM, cross section, transverse to rolling direction

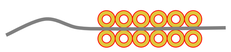


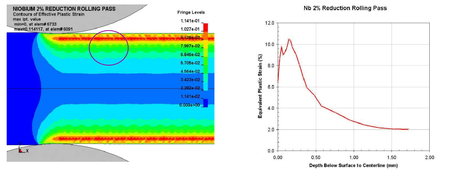
- Existence of etching figures provide a coarse but very quick estimation of the presence of remaining stress inside the material

Source of the damage layer:

Rolling : skin pass

- After rolling sheets undergo a skin pass for planarity

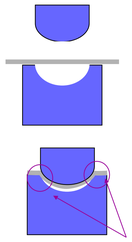


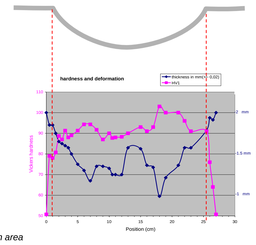


Finite element simulation of 2% reduction of 3.5 mm sheet with 1 cm diameter rolls (Courtesy Non-Linear Engineering, L.L.C.). Strain is concentrated in the near-surface region (red). Localized strain exceeds the average by a factor of 5

- Rolling leaves a damage layer ~150 µm with structure resistant to recrystallization, i.e. same order of magnitude than the necessary etching of material

Deep drawing

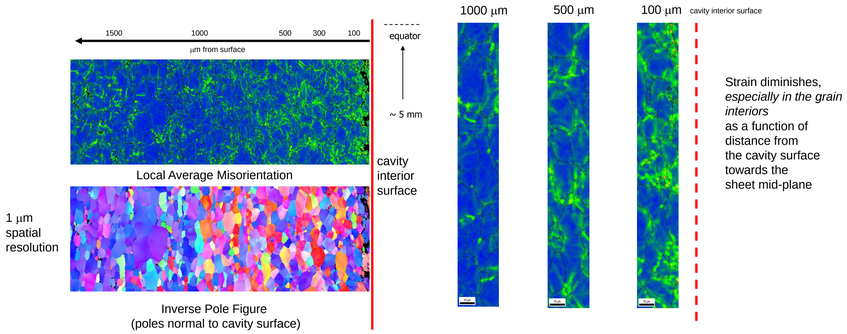




High friction area

- Additional damage (due to friction against dye) appear due to deep drawing
- The more deformed (thinner), the highest strain (hardness)
- Location of strain varies a lot with the drawing process (lab to lab variations)
- Annealing due to welding cures some damage but also induces local thermal strain

Deep drawing : orientation imaging



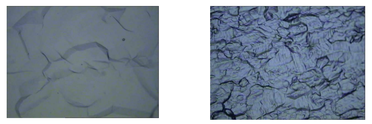
Mechanical chemical polishing principle

- This technique was initially developed for the preparation of TEM samples, but since then it has been thoroughly applied to wafer preparation and optical lenses. It allows preparing large, curved surface in an industrial way.
- first step involves the use of a series of SiC papers with decreasing grain size (Ø 100 to 5 µm), The damage layer due to each stage has been evaluated. Diamond is not recommended for smooth materials.

Paper grit •*Europe* •*US*	*320 240/280*	*500 320/360*	*800 400*	*2400 800*	*4000 1000*
SiC Ø µm	*200*	*30*	*22*	*10*	*5*
Damage layer	*110 µm*	*95 µm*	*70 µm*	*50 µm*	*40 µm*

- The finishing step is done with colloidal SiO_2, with small addition of H_2O_2 and NH_4OH, which are known to de-passivate and complex niobium oxide. Colloidal suspension tends to break into smaller parts with time, giving a very good surface finish.

Mechanical chemical polishing on Nb

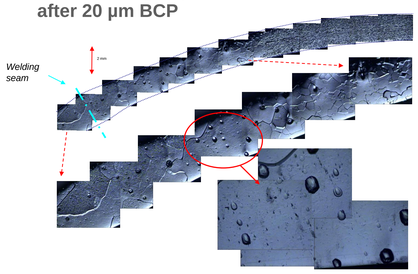


Surface state of a niobium sheet after 20 µm BCP etching with (left) or without (right) mechanical chemical polished. The high density of etching figures on the sheet without surface preparation shows that much more than 20 µm are necessary to remove the damage layer.

- The damage layer has been reduced from 150 to < 20µm after "manual" MC polishing !
- The surface state presented here results from manual polishing. It can further be optimized with an automated set-up so that the amount of damage layer is further reduced [2].

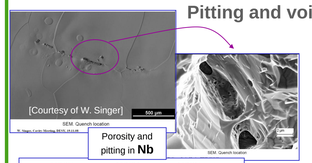
Example of automated polisher

After welding : optical observation after 20 µm BCP

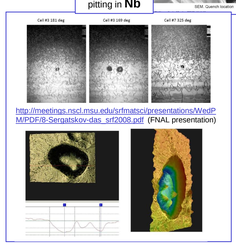


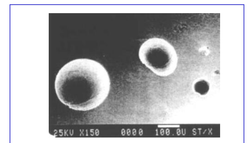
- Deep etching pits (aligned with crystallographic direction ?) are found in the heat affected area.
- Careful exploration of the remaining stress due to welding is necessary (i.e. with orientation imaging)

Pitting and voids in HAZ: a general feature of BE welding

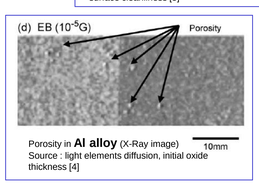


Porosity and pitting in **Nb**

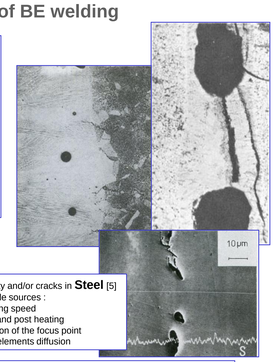




Bubbles in **Ta6V (Ti alloy)**
Source : light elements diffusion, pre-weld surface cleanliness [3]



Porosity in **Al alloy** (X-Ray image)
Source : light elements diffusion, initial oxide thickness [4]



Porosity and/or cracks in **Steel** [5]
Possible sources :
- welding speed
- pre- and post heating
- position of the focus point
- light elements diffusion

Welding speed and pre- and post heating influence on void development also observed in **Ti** and **Ta** [6]

Conclusions:

- Rolling and deep drawing are the main source of the damage layer.
- Mechanical-chemical polishing applied to Nb sheets on half-cells is a way to reduce electropolishing time after cavity completion.
- Annealing after deep drawing could be considered to reduce deep damage inside the material
- Effect of welding and cooling condition need further exploration in order to reduce the amount of thermal strain embedded inside the material.
- General information on welding well documented in the 70's80's

References:

[1] http://web5.pku.edu.cn/srf2007/download/proceedings/TU103.pdf

[2] Metallographic polishing by mechanical methods, fourth edition, L.E. Samuels, ASM international Ed.(2003)

[3] Assessment of the Origin of Porosity in Electron-Beam-Welded TA6V Plates, N. GOURET, et al., Metallurgical and material transactions A - 35A, (2004), p.879

[4] Bubble formation in aluminum alloy during electron beam welding, H. Fujii et al., Journal of Materials Processing Technology 155–156 (2004) 1252–1255

[5] Proc. Of the 2nd international colloquium on EB welding and melting, Avignon, (1978) ; Proc. Of the 3rd international colloquium on welding and melting by electrons or laser beams, Lyon, (1983) ; following edition of the same colloquium…

[6] E. Herms, Laboratoire d'Etude de la Corrosion Acqueuse, CEA, personnal communication

Keywords:

Particle accelerators, RF cavities, niobium, damage layer, mechanical chemical polishing,