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Investigation of mechanical damage produced during CBP of SRF Nb cavities by metallographic techniques

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Introduction

Many modern and proposed future particle accelerators rely on Superconducting Radio Frequency (SRF) cavities made of bulk niobium (Nb) as primary particle accelerating structures. Important metrics of SRF cavity technology are the quality factor Q_0 and the maximum electric field *Eacc* to which high values of Q_0 can be sustained. The present state of cavity fabrication and processing art places strong emphasis on attaining a very smooth surface because both Q_0 and *Eac*c depend on the surface quality (inclusions of foreign material, impurities, grain boundaries, surface defects, etc.). The processing stream for SRF niobium cavities includes a material removal from their inner surface through electropolishing (EP) and/or buffered chemical polishing (BCP). Centrifugal barrel polishing (CBP) is an alternative technique which is considered to be a candidate either for replacing a so-called "bulk EP" step in cavity production, since it not only successfully removes such defects as pits and scratches, but also foreign materials inclusions which cannot be repaired by chemical methods. It is a simple technology that could be transferred easily to industry, and could help to increase cavity yields and, moreover, requires no acid. In ILC-HiGrade Laboratory at DESY FLA group this method is actively investigated with the aim to optimize the parameters of the up-to-date CBP recipe acknowledged in the literature [1]. For this purpose, a so-called "coupon" cavity was used. The topic of my investigation is devoted to development of the preparation technique of Nb coupons in order to investigate the mechanically damaged surface layer left after CBP.

In the following sections the main results obtained in ILC-HiGrade Laboratory on the surface studies of CBP treated cavities are presented. Then, in Section 2 the basics of metallographic techniques with emphasis on the peculiarities for treatment of Nb material are outlined. In Section 3 the procedure for metallographic investigation of Nb coupons with the aim of estimation of the mechanically damaged layer produced during CBP, is described in detail. In the last section, the results of the current work are summarized.

1. Centrifugal Barrel Polishing in ILC-HiGrade Laboratory

As a starting point for investigation of CBP procedure the recipe obtained in Fermi National Laboratory, USA was chosen. The barrels and the main shaft were rotating at 100 rpm. Below the parameters of polishing media and processing time are listed:

Step 1: cutting with Soap & Ultrapure Water, ~8 hours.

Step 2: intermediate polishing with Soap & Ultrapure Water, ~15 hours.

Step 3: intermediate polishing with Water + 15 μm Alumina, ~30 hours.

Step 4: final polishing with Colloidal Silica 0.04 μm, 40^{+} hours.

Advantages of the CBP process:

- defect removal;
- repair cavities having severely damaged surface;
- create low surface roughness;
- reduce cost, industrialization;
- remove chemistry.

Despite the simplicity of the process it is not so easy to investigate Nb cavities for further improvement of CBP process. It is nearly impossible to understand the effect of each processing step on a cavity at the end of processing. So the utilization of coupon cavities is critical in understanding the effect of CBP steps. Coupons represent small samples of Nb material, which is installed in the SRF cavity. After CBP, these coupons can be taken off for performing surface studies on them.

The preliminary investigation using coupon cavities revealed the following drawbacks of the current recipe of CBP (fig. 1 and fig. 2):

- residual scratches and embedded particles;
- mechanical damage observed on the surface of the coupons.

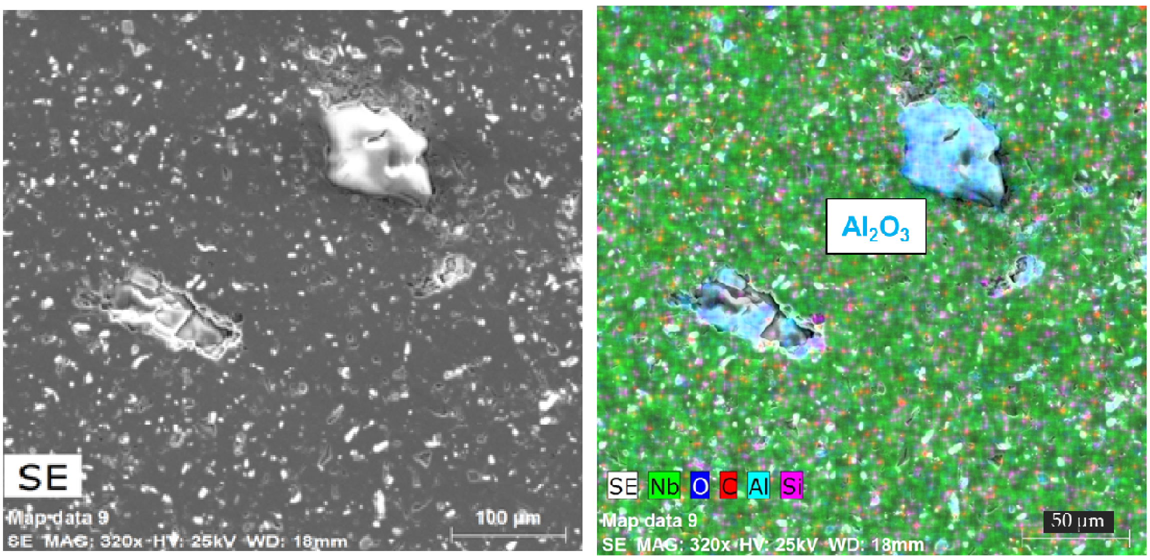

Figure 1 - The Scanning Electron Microscope (SEM) and the corresponding Energy-Dispersive X-ray spectroscopy (EDX) image of Nb coupon surface after CBP: the embedded particles of polishing materials

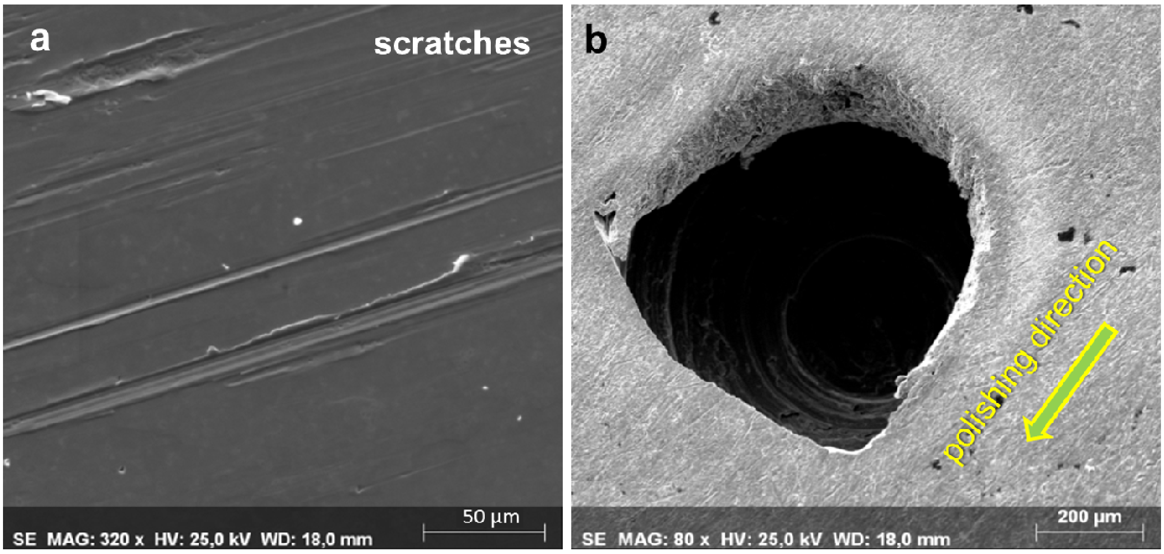

Figure 2 - Residual scratches (a) and mechanical damage (b) on the surface of the coupons (the polishing direction is pointed with an arrow).

The aim of the present study is investigation of the mechanically damaged layer produced during CBP, which was revealed by observation of shearing effect by SEM (fig. 2b). For this purpose, the metallographic technique will be used, for studying the crystal structure on the cross-section of the Nb coupons.

2. Metallographic Basics: Main Steps

A proper preparation of a metallographic specimen in order to determine the microstructure and content requires that a rigid step-by-step process to be followed. Specimen must be kept clean, and preparation procedure carefully followed in order to reveal accurate microstructure. Incorrect techniques in preparing the sample may result in altering the true microstructure and will most likely lead to erroneous conclusions. The main requirement of the metallographic preparation is that the microstructure of the specimen should not be altered. In sequence, the steps include sectioning, mounting, grinding, polishing, chemical etching (fig. 3) and microscopic examination.

2.1 Sectioning and Mounting

Sectioning (or abrasive cutting) offers the best solution to eliminate undesirable features of work piece; the resultant surface is smooth, and the sectioning task is quickly accomplished. So to obtain a specimen, sectioning from the basic material (work piece) is necessary.

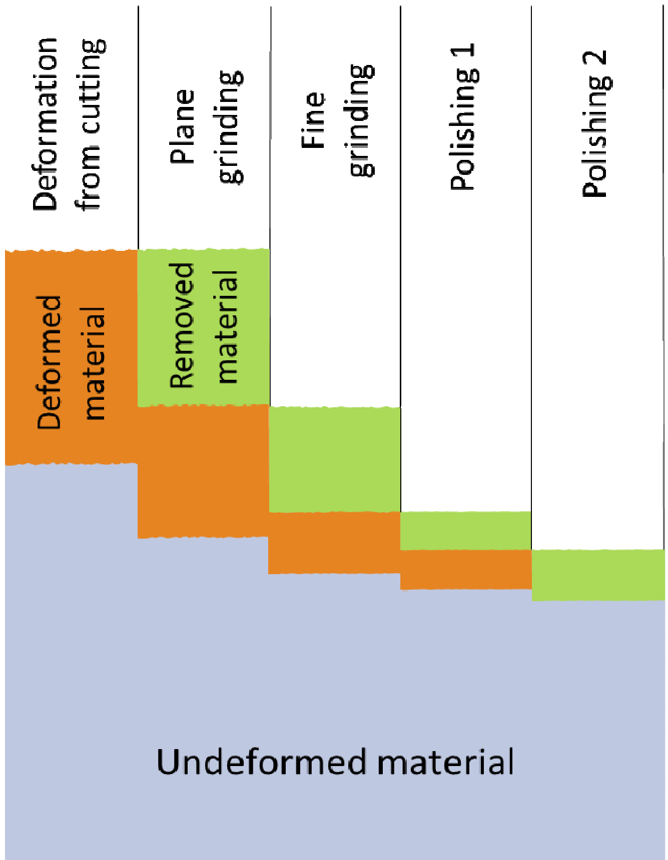

Figure 3- Step-by-step removal of deformation

Metallographic cut-off machine is applied for wet abrasive cutting. It is the standard manual laboratory cut-off machine for chop cutting with dual vices to clamp samples on both sides of the cut-off wheel. A 3 kW motor and 254 mm cut-off wheels with cut capacity 76 mm in diameter. The correct combination of abrasive and bond is important to ensure the right cut-off process. Since a soft material will not wear out the abrasive very fast, therefore a hard wheel should be used. For cut-off wheels of Nb used SiC type of abrasive. This synthetic material is hard and tough, but dulls and glazes rapidly when used with steels. It is well suited for cutting of softer materials like Nb. The work piece should be placed correctly to obtain the shortest area of contact between cut-off wheel and work piece. Due to the poor machinability and to minimize the deformation, a relatively soft wheel, as thin as possible, should be used.

The following step after sectioning is *Mounting,* because a specimen is very small and is difficult to handle. Also it has an awkward shape and mounting is necessary to secure the preparation of the correct surface (relatively flat). To support the edge of the specimen, the mounting material should be in the same level as the specimen (no relief). In our case, the Nb coupon is mounted using a thermosoftening plastic (fig. 4), which has the peak

Figure 4 – Components of thermosoftening plastic (acrylic resin)

temperature of 70 °C and curing time 1 h.

The specimen is supported by metal clips and then it is placed on the bottom of the mold, and the liquid acrylic resin is poured into the mold. After curing, the mounted specimen is taken off for the following preparation (fig. 5).

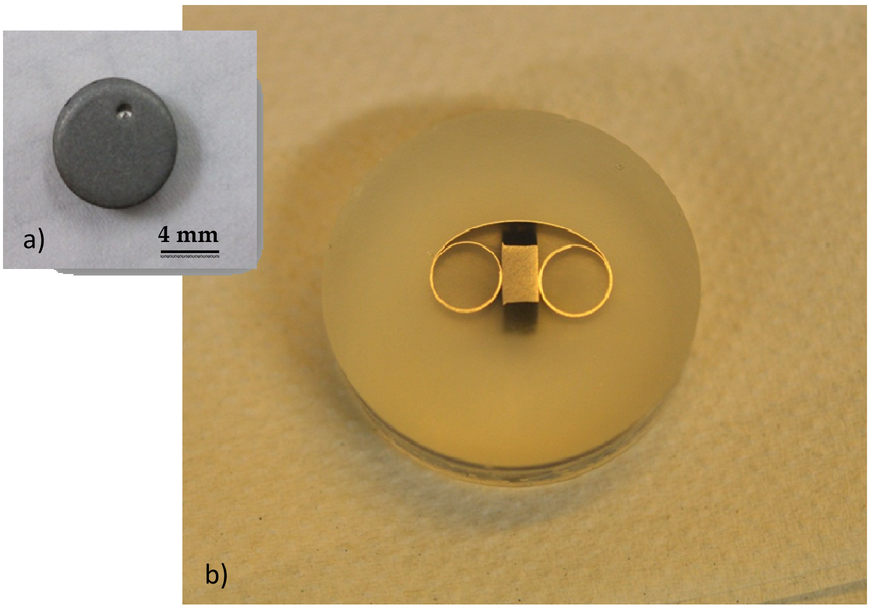

Figure 5 - Nb coupon (a) and a mounted Nb coupon (b)

Manual cleaning is used for cleaning a specimen because it is the most effective method. The specimen is held under running lukewarm water and then purified using ethanol. Drying should take place in a stream of mild air. It is important that the layer of alcohol is not dried on the prepared surface but is blown away and evaporates from the sides of the specimen. The specimen should be cleaned between every step in the grinding and polishing process. Also the grinding/polishing disks should be cleaned after use. It is important that the process is not contaminated with abrasive grains from the previous step.

2.2 Mechanical Grinding.

After sectioning and mounting, the metallographic specimen is has to be prepared to obtain the true microstructure or a structure, which, in spite of certain defects (artifacts), will give a true examination result. So the next step is the *Mechanical Grinding*, which consists of two stages:

- Plane Grinding;
- Fine Grinding.

The first step is the *Plane Grinding (PG)*. Plane grinding, also called planar grinding, is necessary for several reasons: to plane the surface after sectioning, and to remove the deformation caused by the sectioning and establish a known "start surface" of the specimen to secure a reproducible further preparation.

The next step is the *Fine Grinding (FG)*. Fine grinding is the process used for establishing a specimen surface suited for the first polishing step. This means that the relatively rough surface from plane grinding, through one or several steps with finer and finer grain sizes, is changed into a surface that can be treated by polishing. The several fine grinding steps are needed because the material removal is relatively high, opposite to the polishing steps with a low material removal. So in our case after Plane Grinding, 4 stages of FG was held. Recommended parameters for each stage of Mechanical Grinding of Nb are presented in Table 1.

Table 1 – Recommended parameters for each stage of Mechanical Grinding of Nb

Grinding	**PG**	**FG1**	**FG2**	**FG3**	**FG4**
Disc/Cloth	SiC paper	SiC paper	SiC paper	SiC paper	SiC paper
Abrasive Type	SiC	SiC	SiC	SiC	SiC

Grit or Grain Size μm	P220	P320	P500	P1200	P2400
Lubricant Type	Water	Water	Water	Water	Water
Rotation Disk/Holder rpm/rpm Comp/Contra	300/150 Comp or contra	300/150 Comp or contra	300/150 Comp or contra	150/150 Comp	150/150 Comp
Force per Specimen N (lb)	20-30 (4.5-7)	20-30 (4.5-7)	20-30 (4.5-7)	20-30 (4.5-7)	20-30 (4.5-7)
Time Minutes	Until plane	0.5-1	0.5-1	0.5-1	0.5-1

2.3 Mechanical Polishing.

After grinding to a sufficiently fine finish the preparation is continued with *Mechanical Polishing*. Mechanical polishing is defined as a material removing process with loose abrasive grains placed on a substrate like a polishing cloth. The abrasive added before or during the process and lubricant is applied during the process to avoid any thermal damage. The whole polishing process consists of several steps.

The first step is the *Rough Polishing*. It is immediately follows the last fine grinding step. The rough polishing is very important because most of material, damaged by the grinding, is removed in this step. During two steps of Polishing (P1 and P2) the last deformation is removed and a reflective surface is produced. Plane grinding, fine grinding and mechanical polishing take place on the same grinder/polisher only changing the disk used for each step. Grinder/polisher has a specimen mover for semiautomatic preparation, 200/250 mm disks and 150/300 rpm. Recommended parameters for each stage of Mechanical Polishing of Nb are presented in Table 2.

Table 2 – Recommended parameters for each stage of Mechanical Polishing of Nb

Polishing	**Rough Polishing**	**P1**	**P2**
Disc/Cloth	Cloth, napless, hard, wov, syn	Cloth, napless, hard, wov, syn	Cloth, napless, soft, porous, syn
Abrasive Type	Dia, spr or susp	Dia, spr or susp	Dia, spr or susp
Grain Size μm	6	3	1
Lubricant Type	Water-based suspension	Water-based suspension	Water-based suspension
Rotation Disk/Holder rpm/rpm Comp/Contra	150/150 Comp	150/150 Comp	150/150 Contra
Force per Specimen N (lb)	20 (4.5)	20 (4.5)	20 (4.5)
Time Minutes	3-5	3	1

2.4 Chemical Etching (Microetching-Contrast)

Microscopic examination of a properly polished, unetched specimen will reveal only a few structural features such as inclusions and cracks or other physical imperfections. The prepared surface often looks as a mirror when examined in the microscope, not showing the microstructure. For this purpose, the surface of specimen is etched chemically using Buffered Chemical Polishing (BCP) to discriminate between phases, grains, grains boundaries and other details (fig. 6).

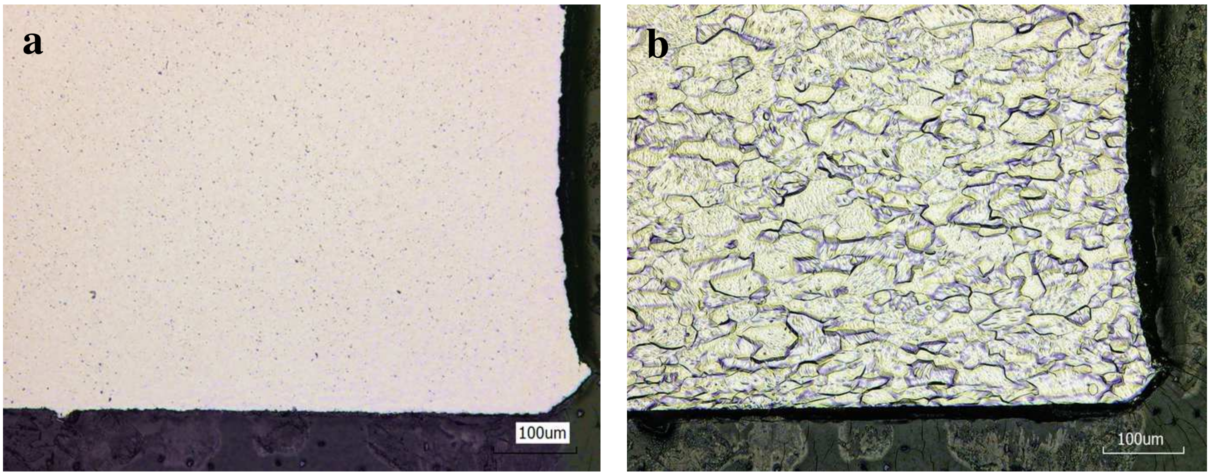

Figure 6 - 3D Laser Microscopy image of specimen surface (20x): a) unetched specimen, only the inclusions can be seen; b) etched specimen, revealing the grains.

In case of niobium, BCP solution represents a mixture of hydrofluoric (HF), nitric (HNO_3) and phosphoric acids (H_3PO_4), respectively, in the ratio 1:1:2. The process of etching takes 4 minutes. Nitric acid plays the role to oxide the niobium surface. Hydrofluoric acid transforms the niobium pentaoxide into a salt that is soluble in water. Phosphoric acid acts as a moderator for the chemical reaction giving rises to a less turbulent and more controllable reaction. That’s why often 1:1:2 is preferred to 1:1:1 for treatment.

For revealing the general structure the following composition of etchants can also be used:

- $HF:HNO_3:HCl$=2:1:2 (immerse to 2 min);
- $HF:HNO_3:C_3H_8O_3$=1:1:2 (swab 5-15 s.);
- $HNO_3:HF$=5:1 (immerse 5-120 s.).

For identifying grain boundaries apply composition of etchants such as:

- $HF:HNO_3:C_3H_6O_3$=1:3:6 (swab 10-20 s.);
- $HF:H_2SO_4:HNO_3:H_2O$=4:3:1:25 (immerse to 5 min.);
- $HNO_3:C_2H_60_2:C_2H_60$=1:10:4 (electrolytic at 0.05 A/cm^2 for 2 min, using stainless steel cathode).

After chemical etching the specimen is immediately washed under running water, rinsed with alcohol and dried in an air blast. Do not touch, wipe or swab the specimen following etching. After drying, move on to the microscopic examination stage. But if further etching is required you may return and proceed through all the steps varying the parameters of the polishing depending on the results.

3. Analysis of CBP-treated Nb coupons

The optical microscope is a very useful tool for the observation of materials and can be used to gain valuable information about a large variety of specimens. Sample preparation is a critical part of microscopy, as this determines the quality of the images produced. Many techniques, when correctly applied to a specimen, can enhance the information present. One of the limitations of the optical microscope is its resolution. High resolution imaging is more commonly carried out in a Scanning Electron Microscope (SEM). In addition, polarized light microscopy can offer large benefits, with high contrast possible. The data in Table 1 and Table 2 are approximate; for particular cases they can be varied. Since Nb is a very soft material, to get a good specimen and hold all the preparing steps are difficult. So in the next section an algorithm for preparing the cross-section of Nb coupon revealing the microstructure in order to estimate a mechanically damaged layer of SRF Nb cavities produced during CBP process.

3.1. Results

Step 1: Mechanical grinding

Mechanical grinding was held with the following parameters: disk - SiC, grit - P400, lubricant type - water, rotation disk/holder - 150 rpm, force per specimen - 25 N, time - 2 min. Obtained result you can see in fig. 5 made by optical microscopy. Specimen surface has a lot of scratches of different width and depth.

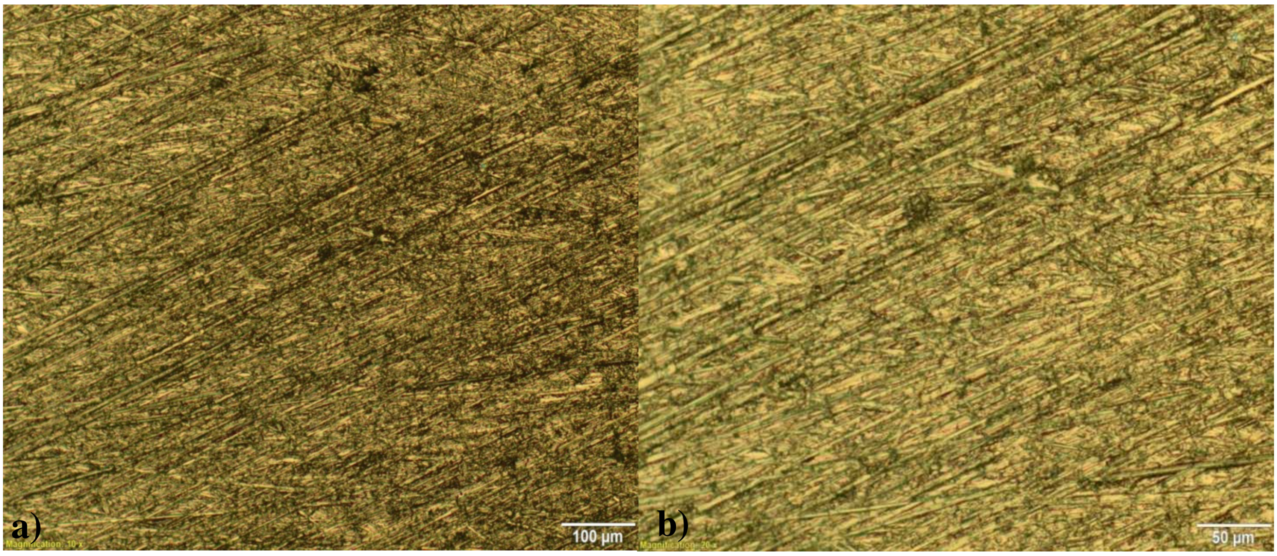

Figure 7 - Specimen surface after mechanical grinding:
a) objective 20x; b) objective 10x.

Step 2 – Mechanical polishing

Mechanical polishing will always leave a layer of disturbed material on the surface of the specimen, if the specimen is particularly susceptible to mechanical damage, as in our case. So for milder polishing we used 4 steps:

- *Polishing 1:* cloth - MD: Largo, abrasive type - diamond suspension 9μm, rotation disk/holder - 150 rpm, force per specimen - 25 N, time - 4 min. In fig. 8 a specimen surface can be seen, which is still has a lot of scratches.

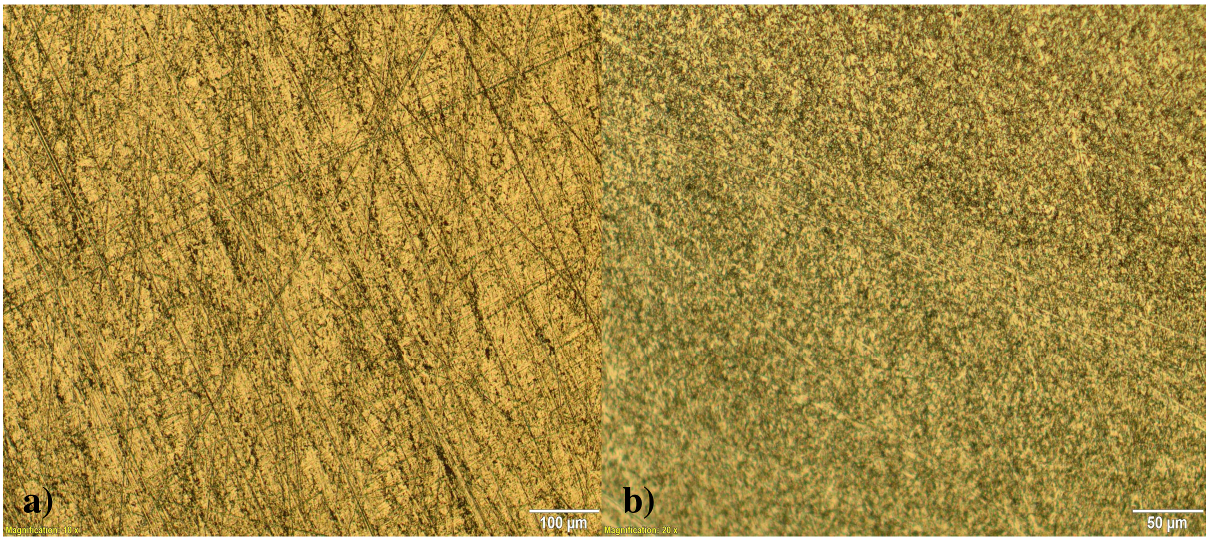

Figure 8 - Specimen surface after mechanical Polishing 1:
a) objective 10x; b) objective 20x.

- *Polishing 2:* cloth - MD: Largo, abrasive type - diamond suspension 9μm, rotation disk/holder - 150 rpm, force per specimen- 25 N, time - 4 min.

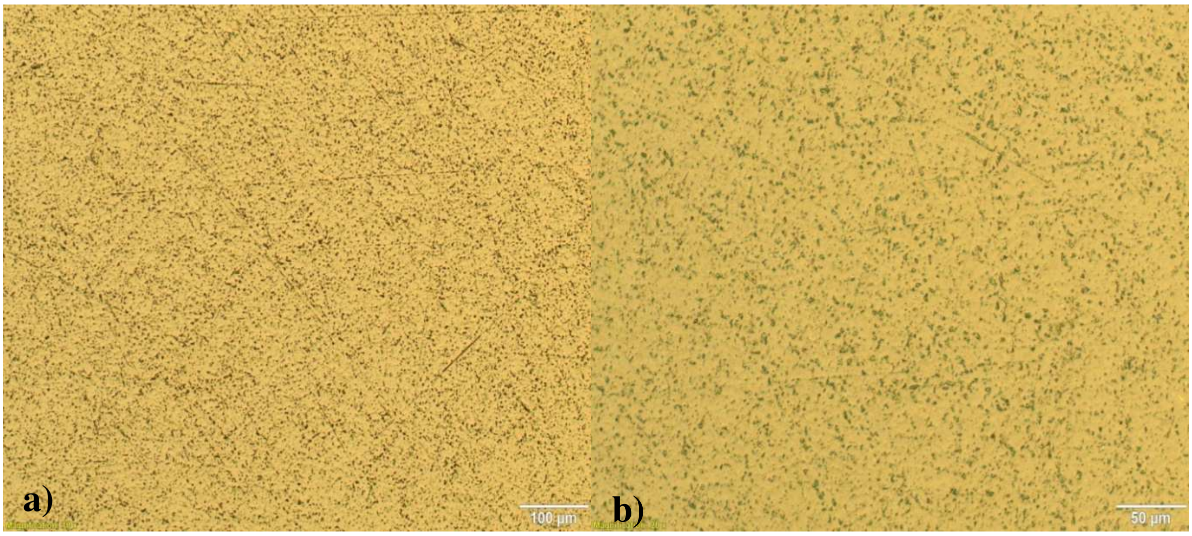

Figure 9 - Specimen surface after mechanical Polishing 2:
a) objective 10x; b) objective 20x.

- *Polishing 3:* cloth - MD:Dac, abrasive type - diamond suspension 1μm, lubricant type - water and oil-based, rotation disk/holder - 150 rpm, force per specimen - 20 N, time - 2 min. After the third step of mechanical polishing a surface becomes smoother, but there are a lot some inclusions (fig.10).

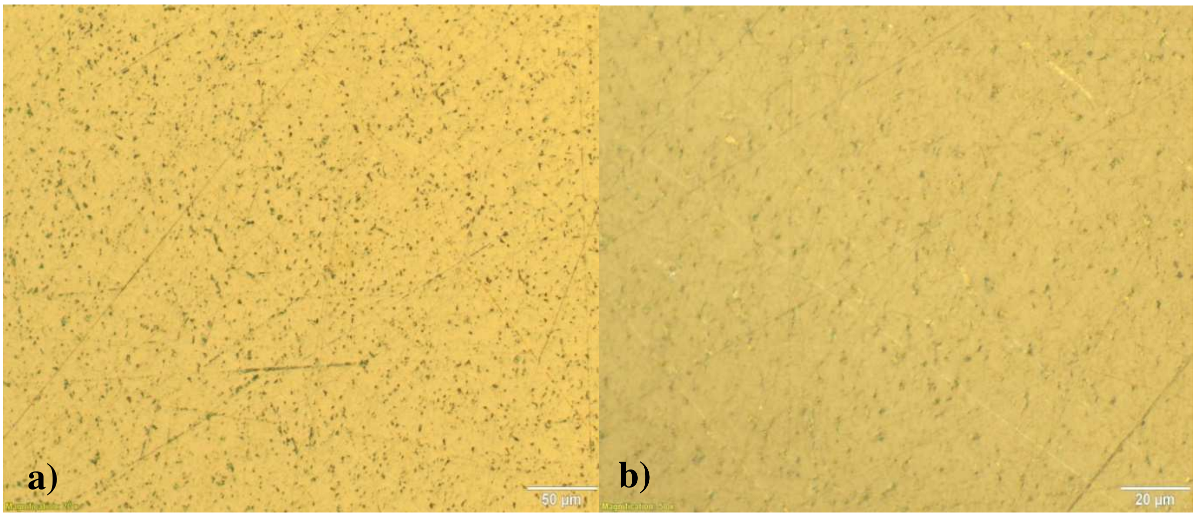

Figure 10 - Specimen surface after mechanical Polishing 3:
a) objective 20x; b) objective 50x.

- *Polishing 4:* cloth - Md:Chem, abrasive type - silica 40 nm, lubricant type - water and oil-based, rotation disk/holde r - 150 rpm, force per specimen - 20 N, time - 4 min. It is the last polishing step; specimen surface almost hasn't scratches, only inclusions can be seen (fig. 11).

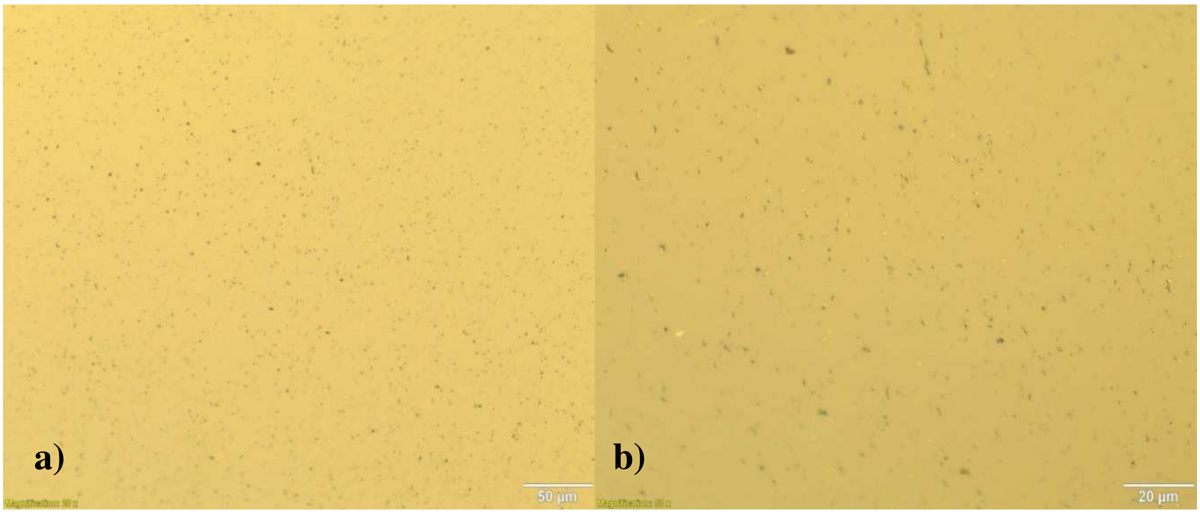

Figure 11 - Specimen surface after mechanical Polishing 4:
a) objective 20x; b) objective 50x.

It is seen that with every polishing step the surface becomes smoother and scratches disappear. Only some inclusions are visible, which can be removed by

Buffered Chemical Polishing. All parameters for each stage are summarized in the Table 3.

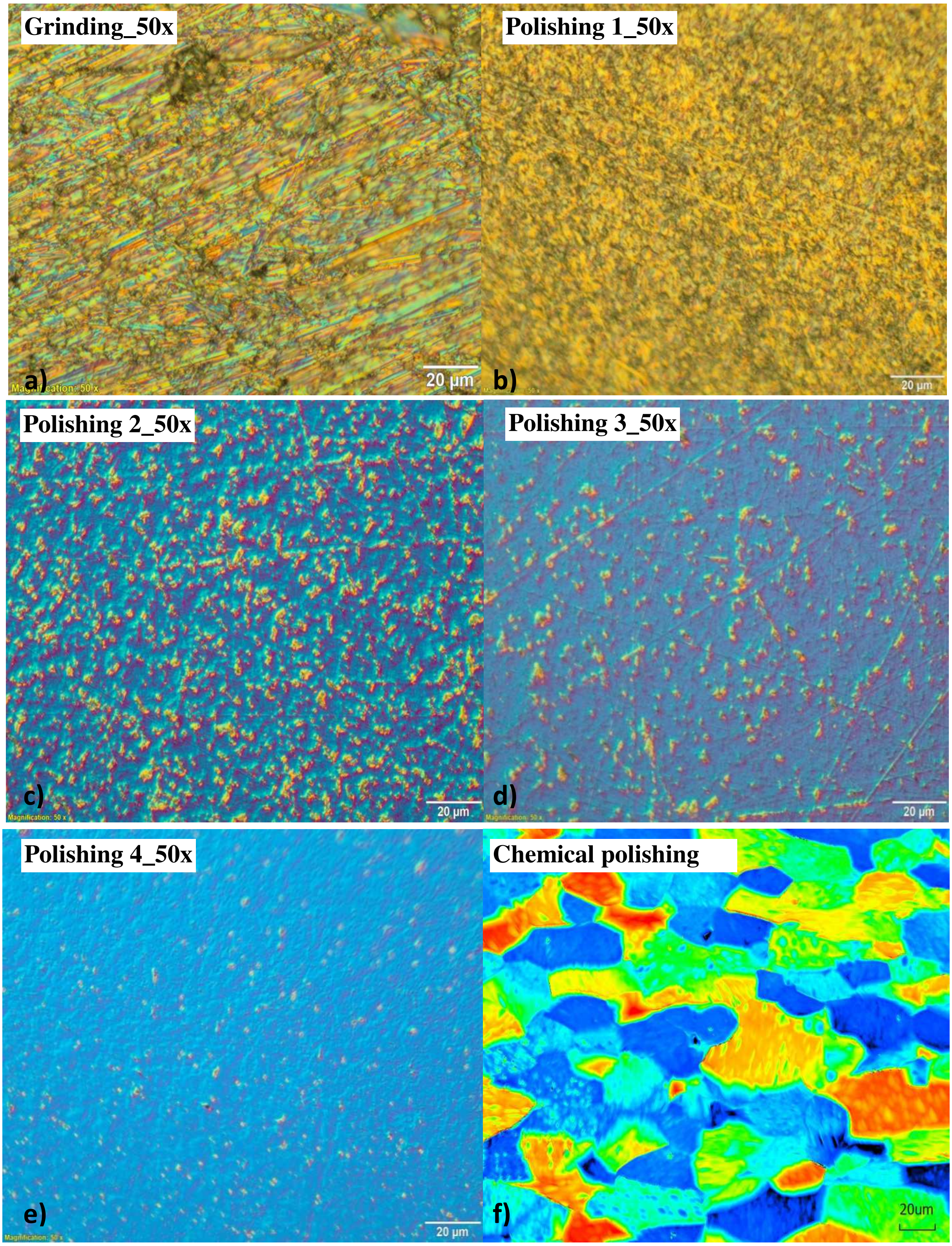

Figure 12 – Images of the specimen surface obtained by optical microscopy in polarized light (a-e); "Height" image made by Keyence VK-X100K 3D laser microscope (f).

Table 3- The optimal parameters of grinding/polishing of Nb coupons

Grinding/Polishing	G	P1	P2	P3	P4
Disc/Cloth	SiC paper	MD:Largo	MD:Mod	MD:Dac	Md:Chem
Abrasive Type	SiC	diamond suspension	diamond suspension	diamond suspension	silica
Grit or Grain Size, μm	P400	9μm	3μm	1μm	40 nm
Lubricant Type	water	-	water and oil-based	water and oil-based	water and oil-based
Rotation Disk/Holder, rpm/rpm Comp/Contra	150/150 Comp	150/150 Comp	150/150 Comp	150/150 Comp	150/150 Comp
Force per Specimen, N	25	25	20	25	20
Time, min	2	4	2	2	4

Also, the influence of force per specimen on the polishing result was investigated during Polishing 3. As it is seen from fig. 13, applying 25 N a better surface quality is obtained, as compared to 20 N.

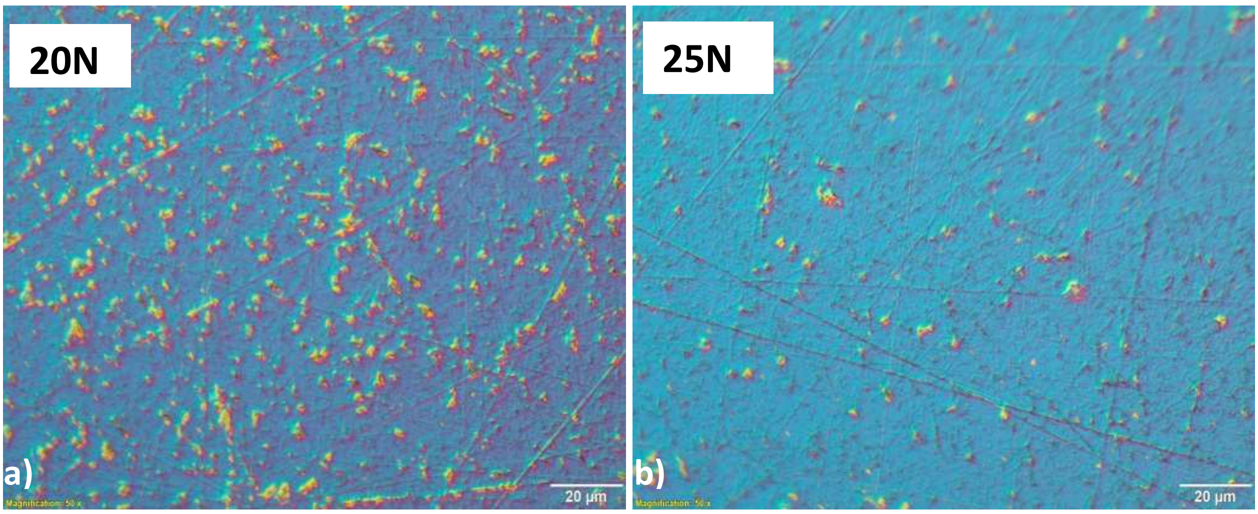

Figure 13 – Influence of force per specimen during Polishing 3.

Step 3 - Buffered Chemical Polishing

BCP was applied to reveal the microstructure of the metal through chemical attack (fig. 12-f). It also removes a thin, highly deformed layer introduced during grinding and polishing.

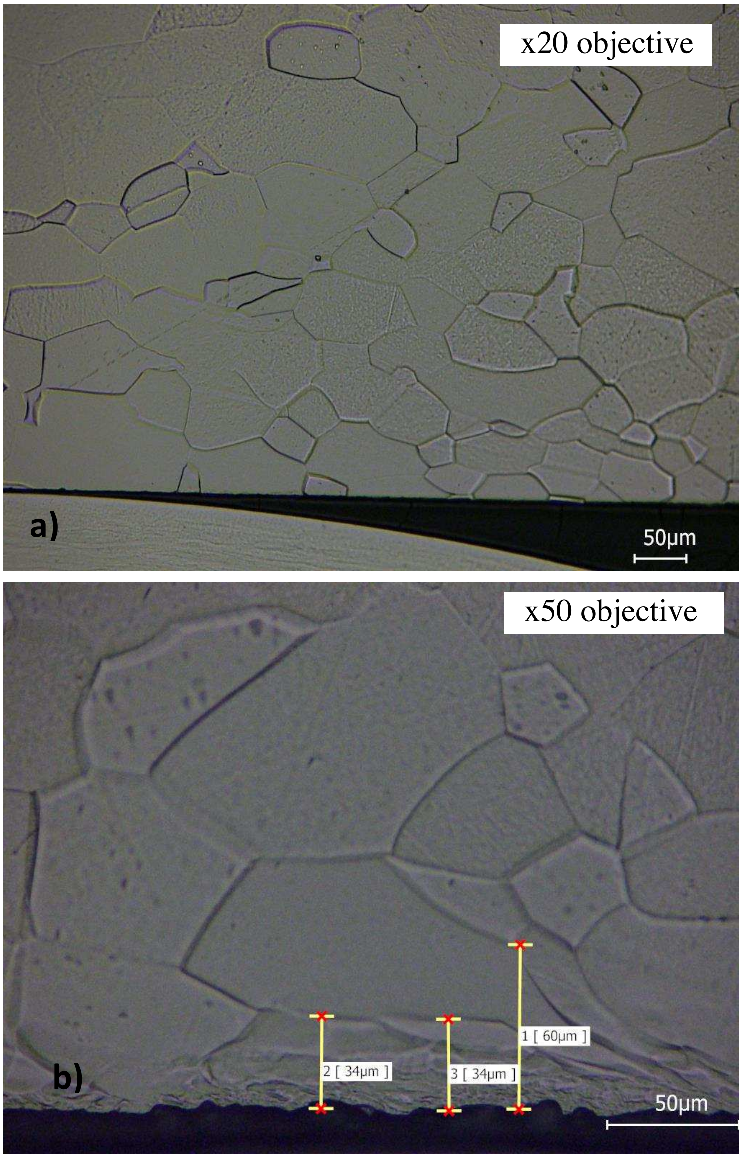

Figure 14 – A comparison of specimen's cross-section before (a) and after centrifugal barrel polishing (b) made by Keyence VK-X100K 3D laser microscope

In figure 12 the obtained by the described metallographic technique cross-section of Nb coupon which surface was treated with the 1st step of CBP, is shown. From this picture a damaged layer can be estimated, and it has the thichness of $34\,\mu$m - $60\,\mu$m. Therefore, the metallographic preparation has been successfully applied to Nb material, and the goal of this investigation was achieved.

Conclusions

The metallographic technique has been successfully applied to Nb material. The existing recipe has been updated by changing such parameters as abrasive type, grit, grain size and time. Based on the analysis of crystalline structure of Nb, the damaged layer produced by CBP was estimated.

In this way the metallographic technique procedure can be used for future studies on improvement of CBP for SRF Nb cavity process.

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