RIGA TECHNICAL UNIVERSITY

Status of Laser Cleaning and Polishing Facility, Sample Characterisation

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Riga
22.05.2018

OUTLINE

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3. Results on Nb/Cu Polishing and adhesion by Laser Radiation.

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Proposed Tasks

RF cavity is the main device in synchrotron accelerators.

Our task is to **improve the quality of RF cavity by improving adhesion of Nb thin layer on Cu substrate** and **to decrease surface resistivity of Nb layer.**

The film deposited by ARIES partners goes through the laser processing in order to increase the grain size and anneal the defects.

Afterwards we characterize the irradiated surface topography by AFM and analyze it by XRD to determine the change of grain size.

The Aim of the Work

The aim of the study is to develop laser technology for application in RF cavity.

Therefore, the tasks are following:

1. Increase the grain size of Nb;
2. Increase adhesion of Nb layer to Cu substrate.

(Anneal the defects by laser radiation).

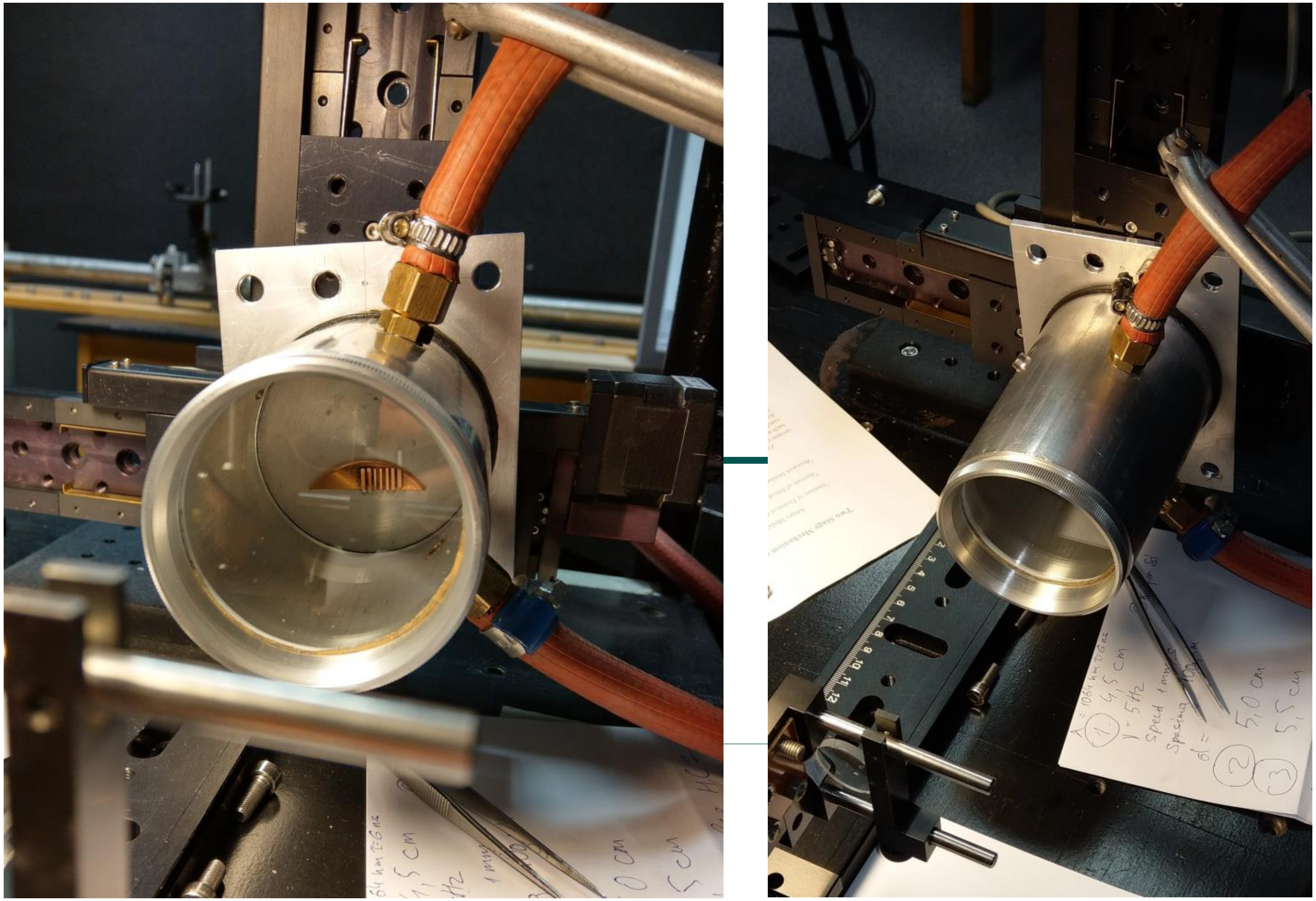


Fig.1. Laser cleaning and polishing chamber with inert gas - Argon

Cu samples received from CERN: C13, C15, C16, C17, C18, C19, C20, C23

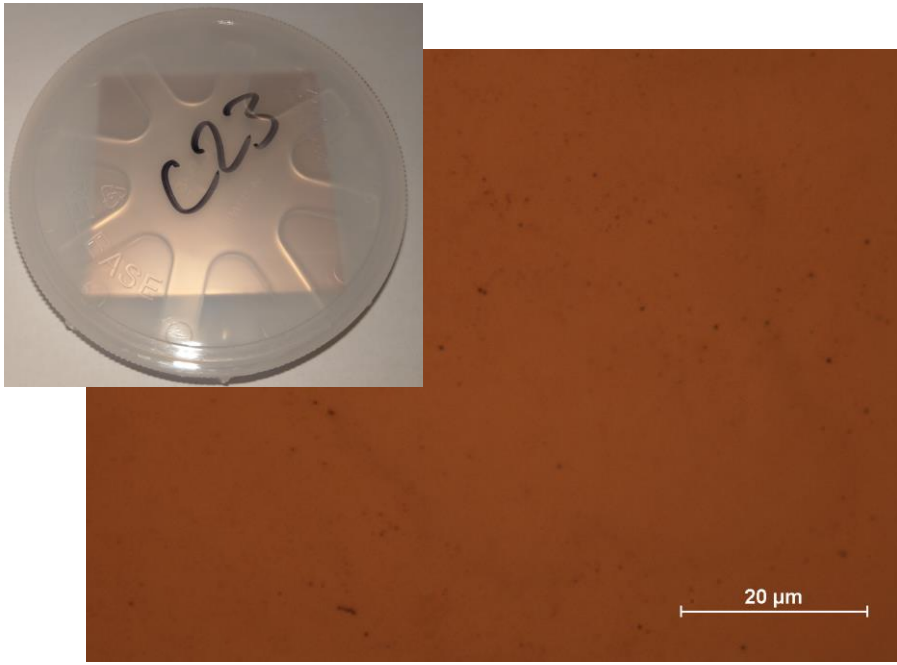


Fig.2. Optical microscope image of C19 sample

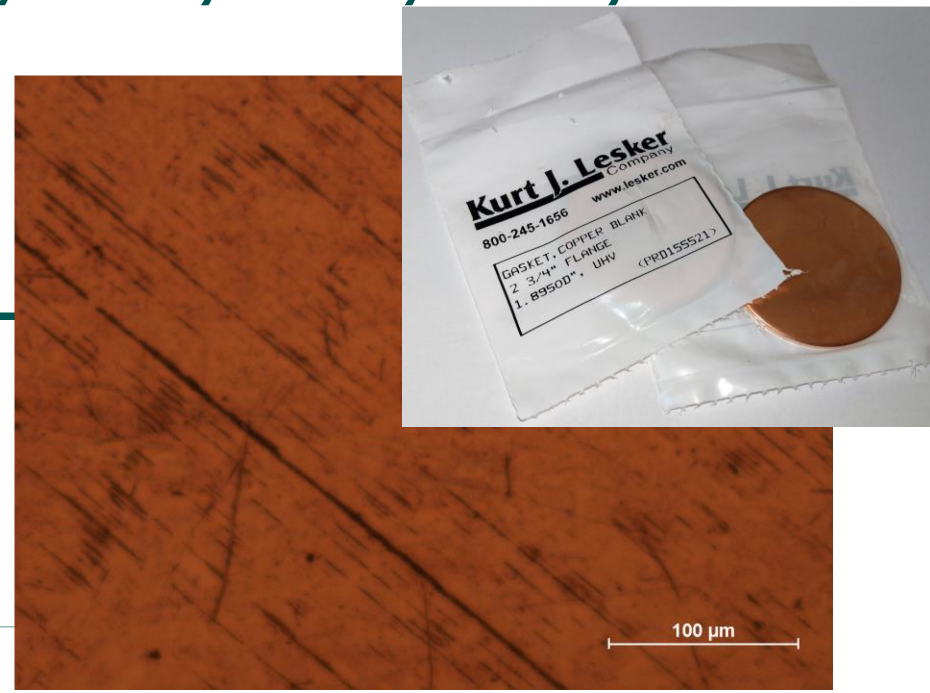
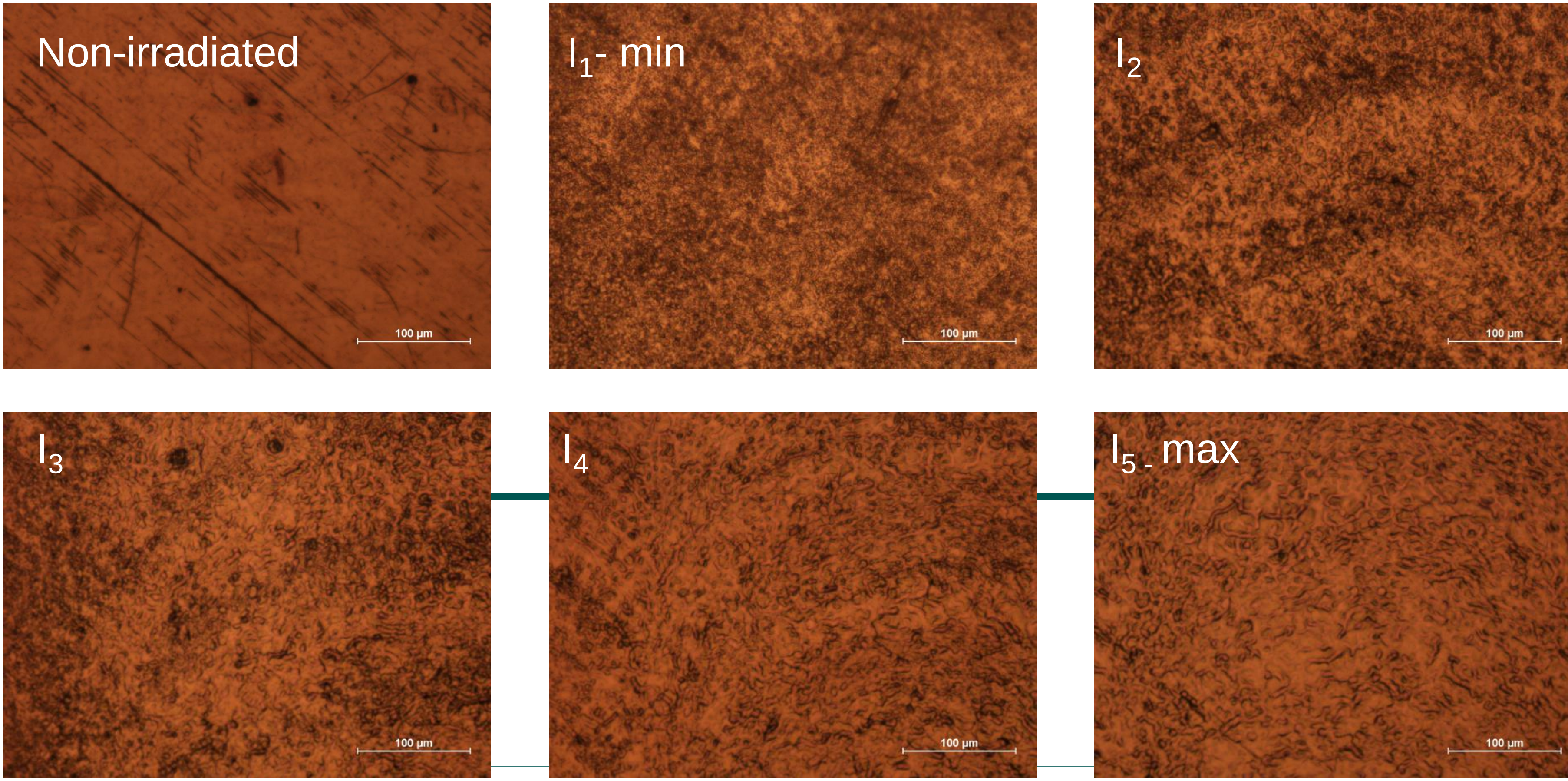


Fig.3. Optical microscope image of Kurt J. Lesker Company sample



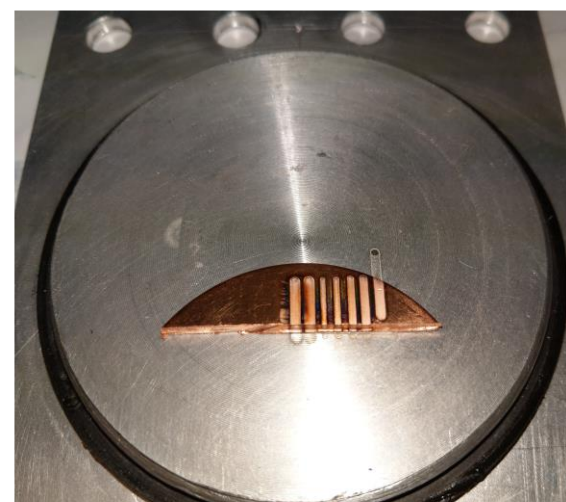
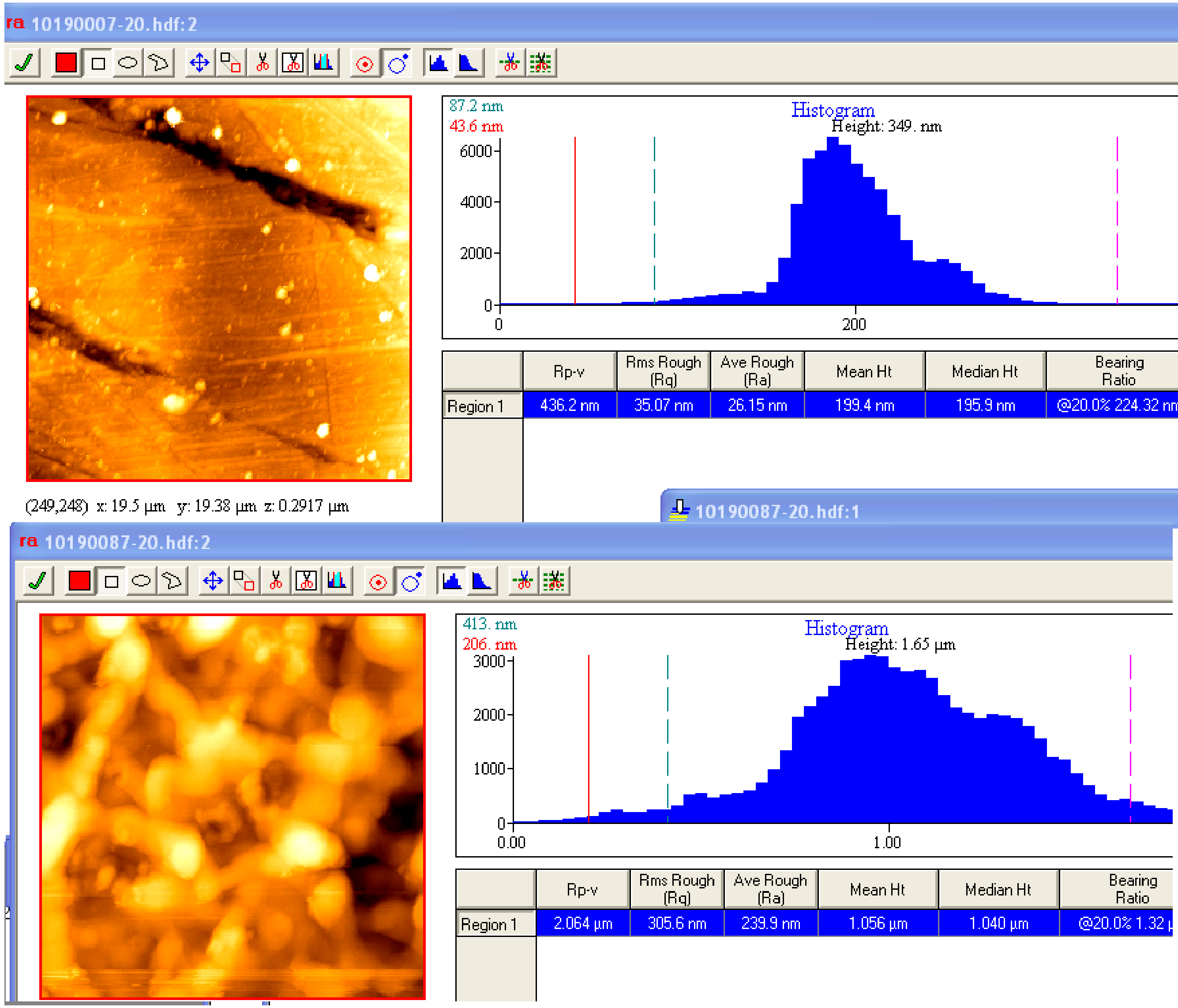


Fig.4. Optical microscope images and a photo of sample from «Kurt J. Lesker Company» irradiated by nanosecond Nd:YAG laser radiation with different intensities.

Fig.5. AFM images of sample from «Kurt J. Lesker Company»:

a) Non-irradiated

b) Irradiated by nanosecond Nd:YAG laser radiation with intensity I_1.



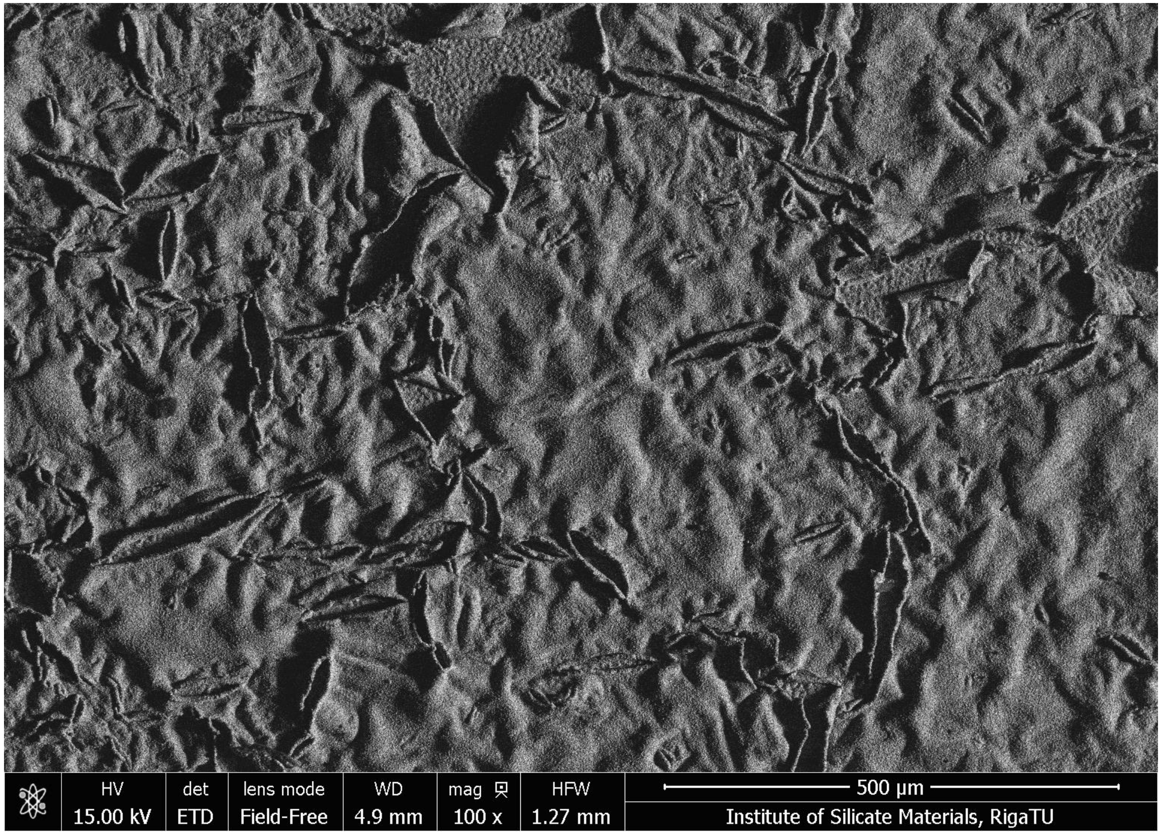
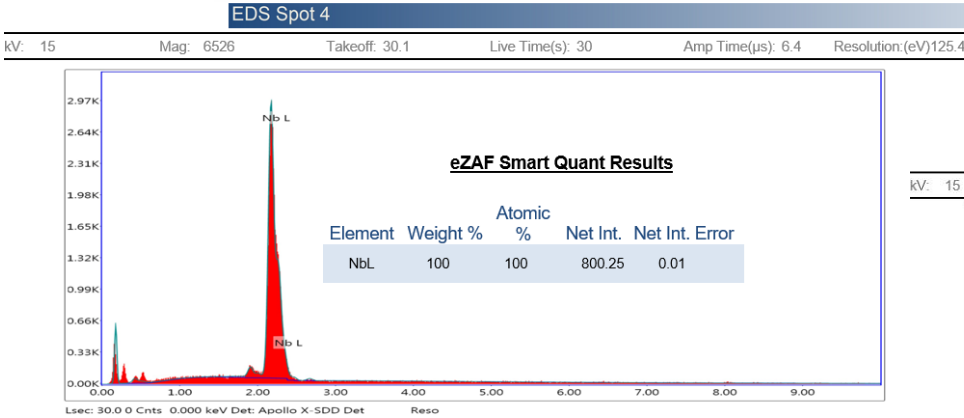
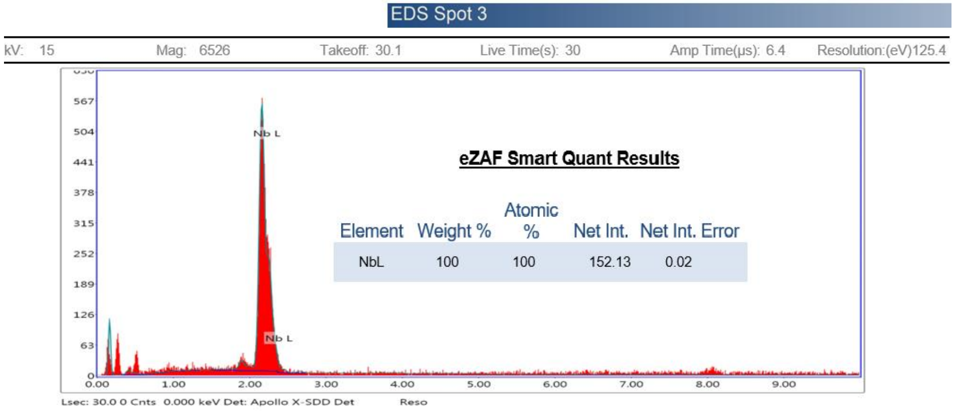


Fig.6. SEM image of Nb/Cu, non-irradiated, with a delaminated Nb layer

It can be seen that Nb film is delaminated in some places.
Probably, such effect takes place due to mechanical stresses after deposition of thick Nb film on Cu substrate.
For solving this problem, we propose to split the deposition technology into **three steps**.
The first step is **the deposition of thin Nb film with thickness about 20-40 nm on Cu substrate ,** the second step is **the laser-irradiation of the sample for interdiffusion of Nb and Cu** and the third stage is **additional deposition of thick Nb film with thickness about 1 mkm on the treated surface**.
This procedure is very important to increase adhesion of the Nb film.





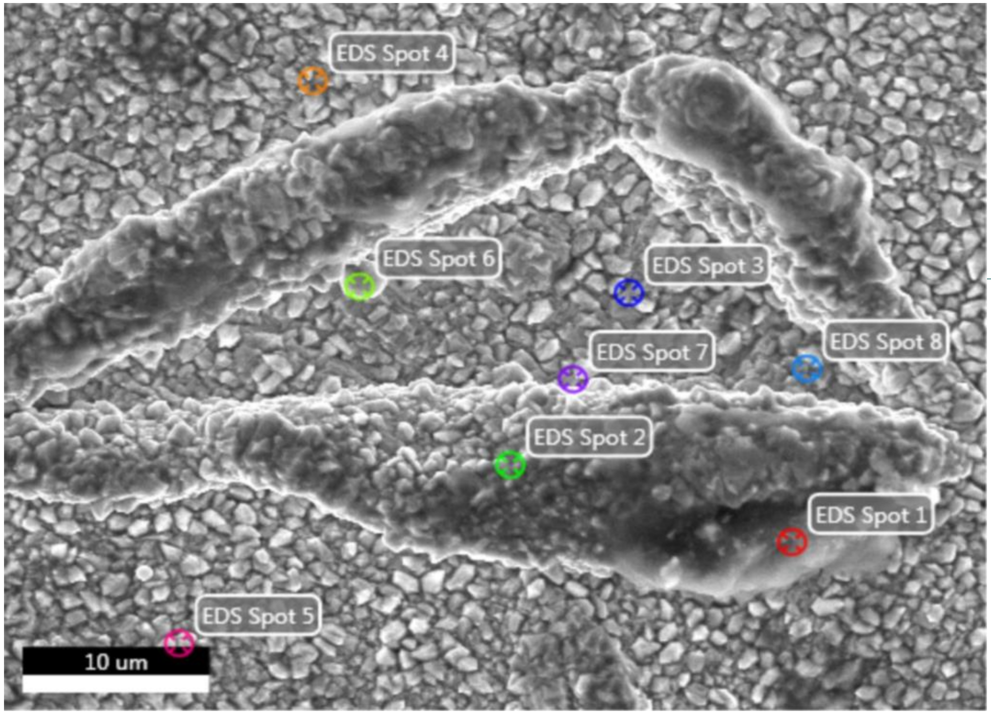
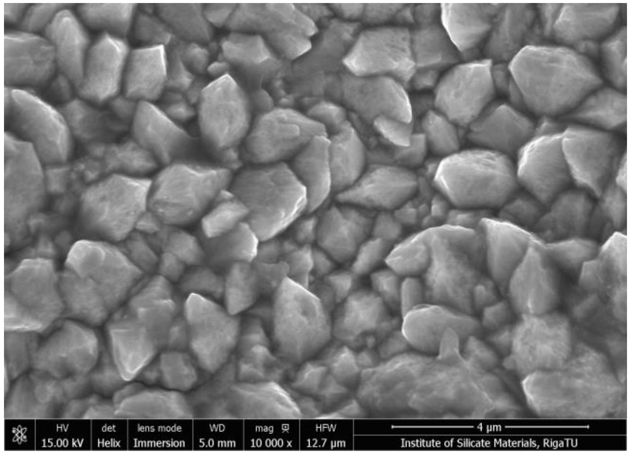
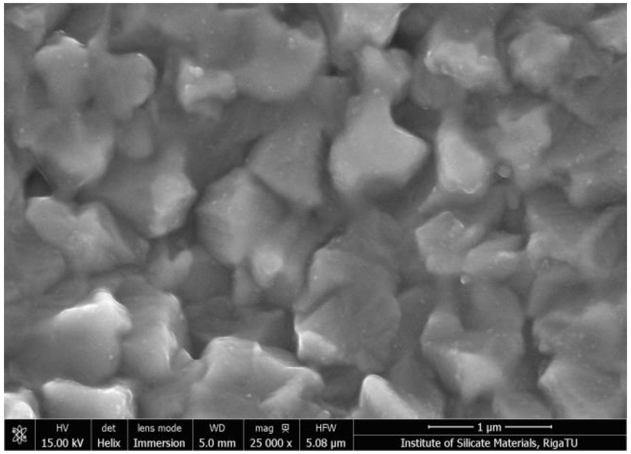


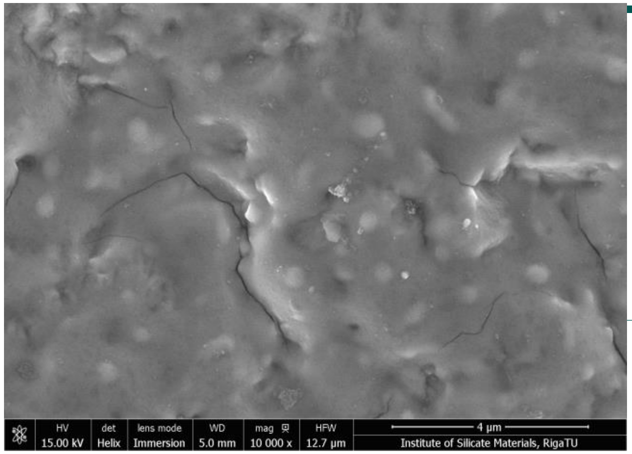
Fig.7. SEM image of Nb/Cu, non-irradiated, with the delaminated Nb layer



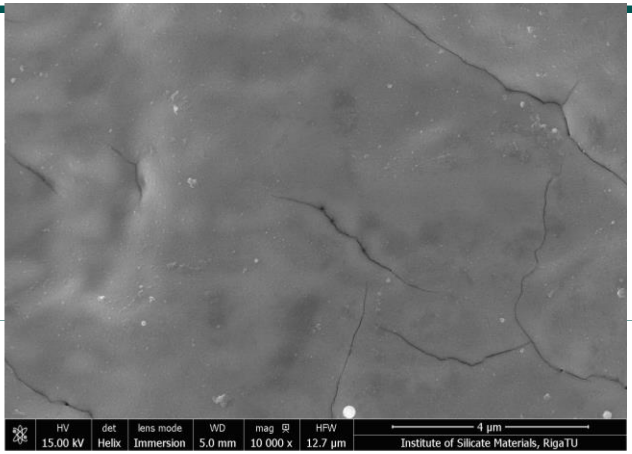
a)



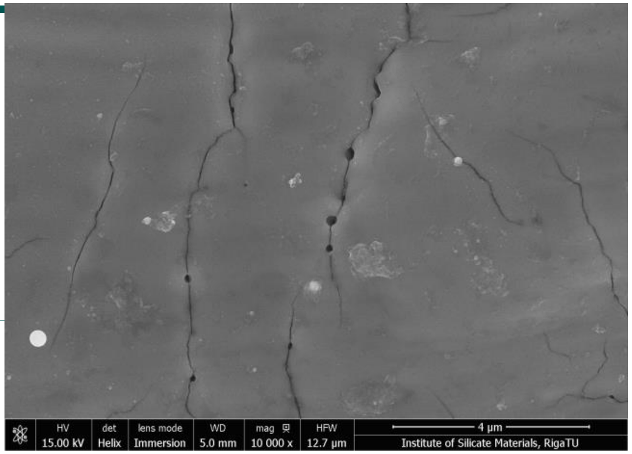
b)



c)



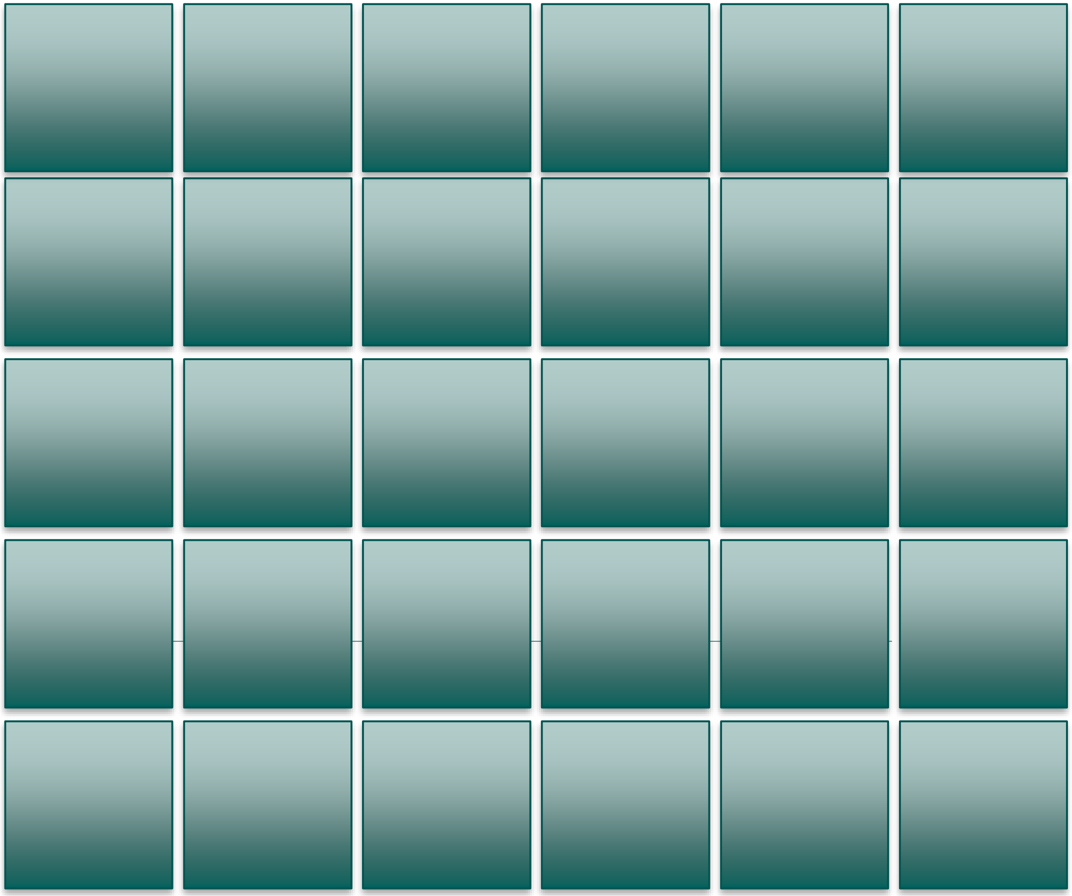
d)



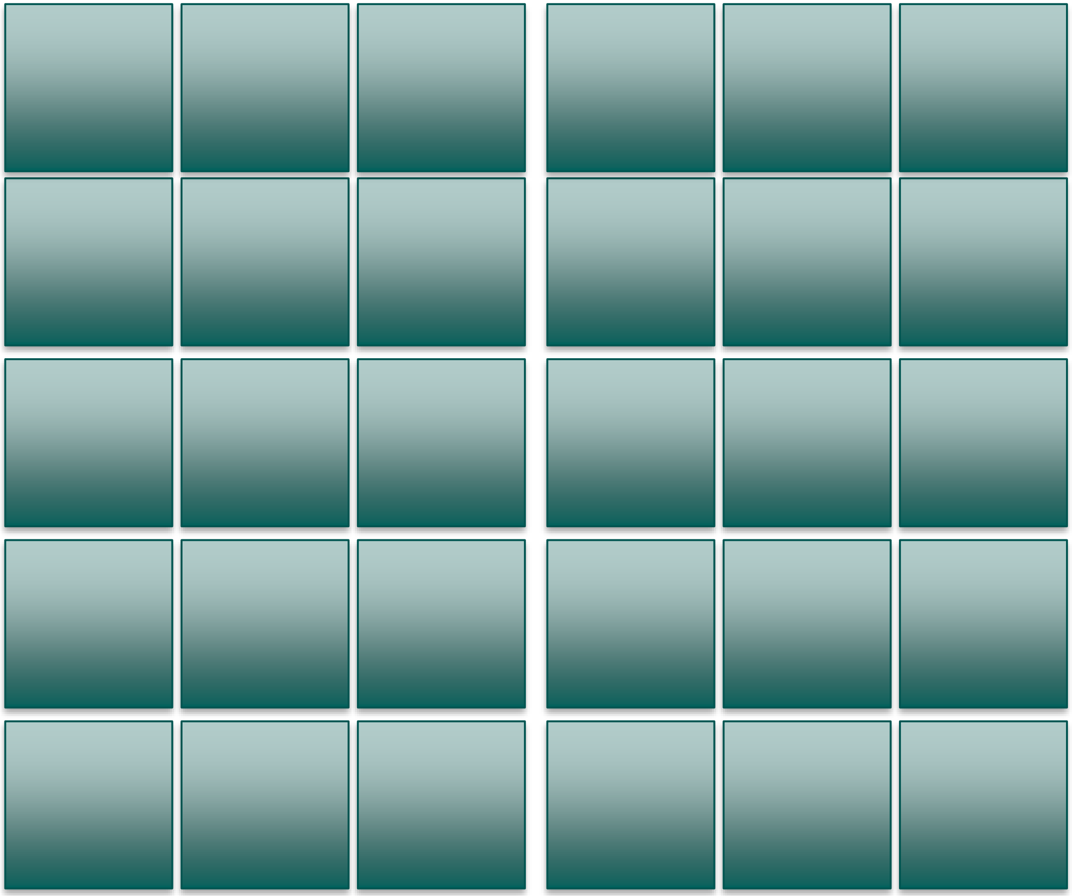
e)

Fig.8. SEM images of Nb/Cu structure before irradiation (a) and after irradiation by Nd:YAG laser with I_1 = 140 MW/cm^2 (b); I_2 = 170 MW/cm^2 (c); I_3 = 253 MW/cm^2 (d); I_4 = 320 MW/cm^2 (e)

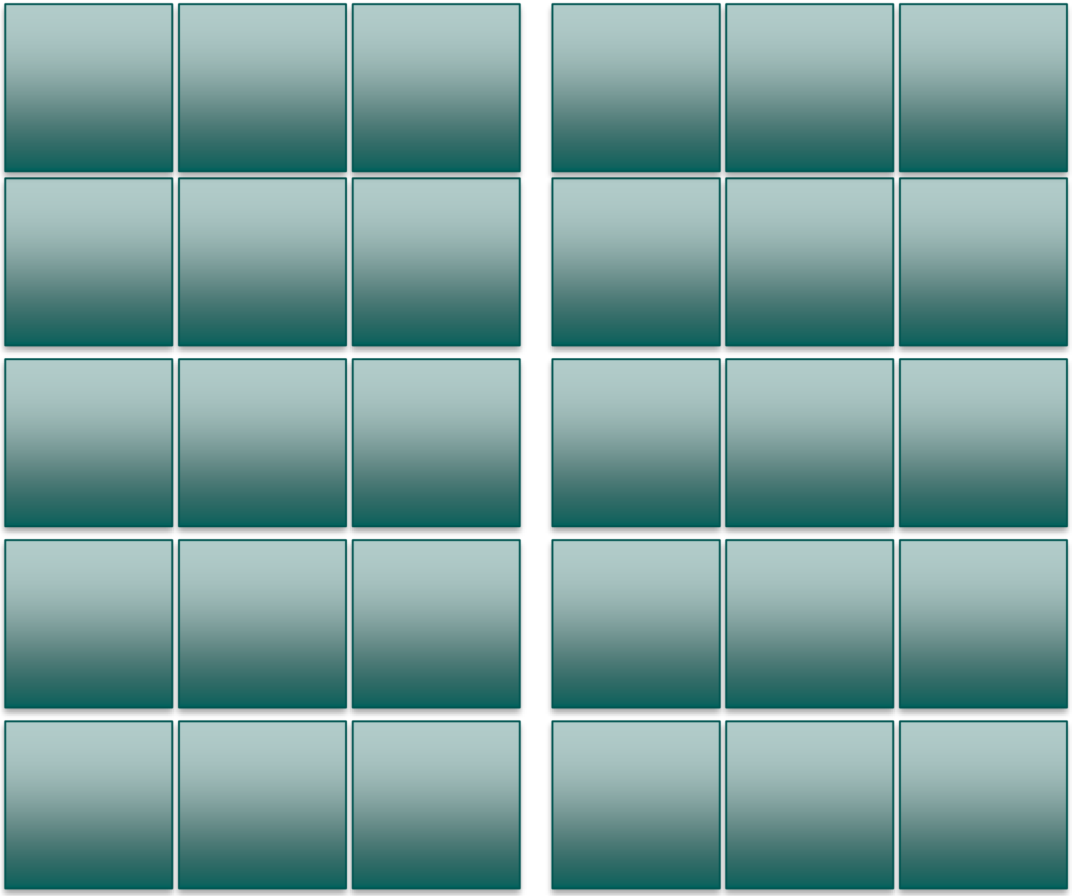
The Animation of cracks formation in Nb by laser radiation



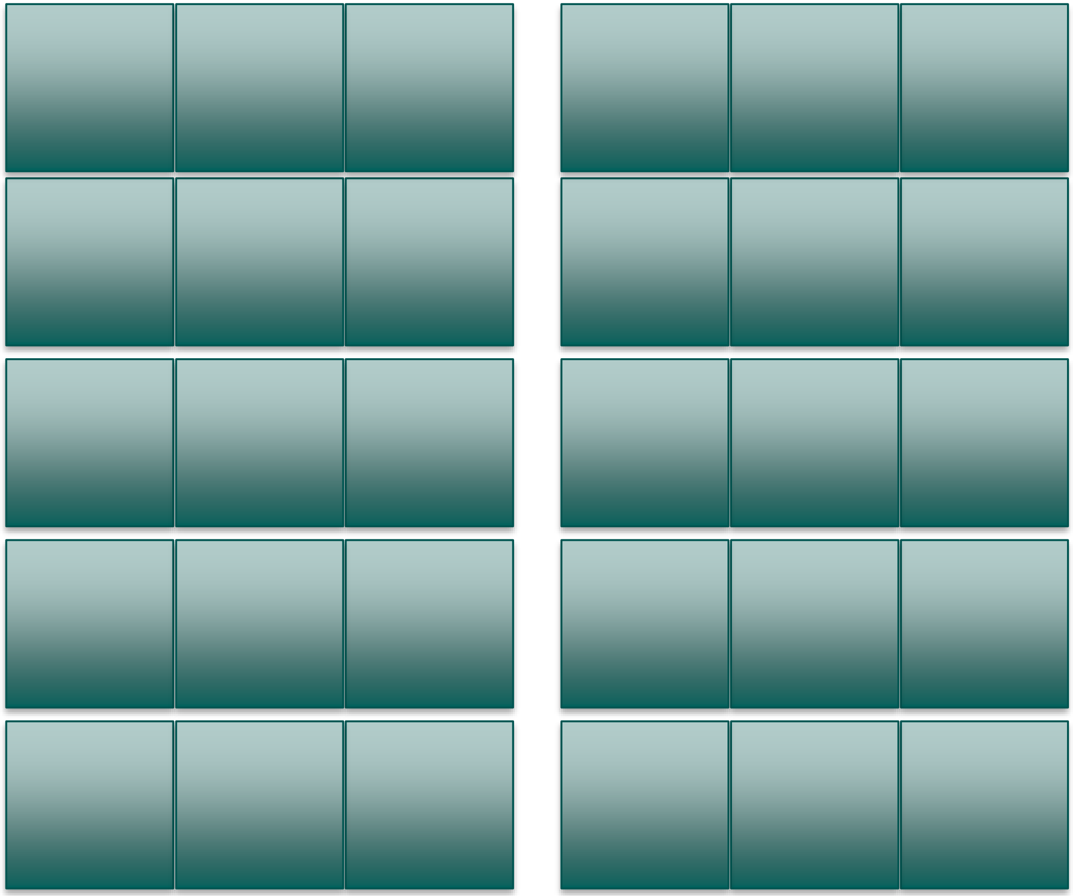
The Animation of cracks formation in Nb by laser radiation



The Animation of cracks formation in Nb by laser radiation



The Animation of cracks formation in Nb by laser radiation



Influence of Laser Radiation on Adhesion of Nb layer to Cu Substrate

Experimental

Nb films were deposited by HiPIMS (magnetron sputtering) with pulse width of 100 μs, repetition rate of 200 Hz, average plasma current of 600 mA, peak current of 40 A and a krypton pressure of 500 mTorr. The variable deposition parameters:

- 22/3/16 - 500 ˚C deposition temperature with -80 V DC substrate bias deposited for 240 mins onto as received copper substrate
- 4/5/16 – 700 ˚C deposition temperature with -80 V DC substrate bias deposited for 480 mins onto as received copper substrate

The samples were irradiated by Nd:YAG laser (λ= 1.064 μm, τ =6 ns and intensity I=200.0 MW/cm^2) in scanning mode with step 5μm in Ar atmosphere. Such intensity of the laser radiation is smaller than the ablation threshold of the structure. Four areas, D1=1.6 J; D2=2.5J; D3=4.9 J; D4=49.0 J, of the structure were irradiated with different doses.

Resistance to delamination was investigated by scratching method (CSM Micro-Combi Tester) using conical diamond indenter (Rockwell). Scratch force on the indenter was gradually increased from 10 mN to 3.0 N.

Optical microscope Nicon Eclipse 100M was used for imaging the scratch path and surface structure during experiments.

Influence of Laser Radiation on Adhesion of Nb layer to Cu Substrate

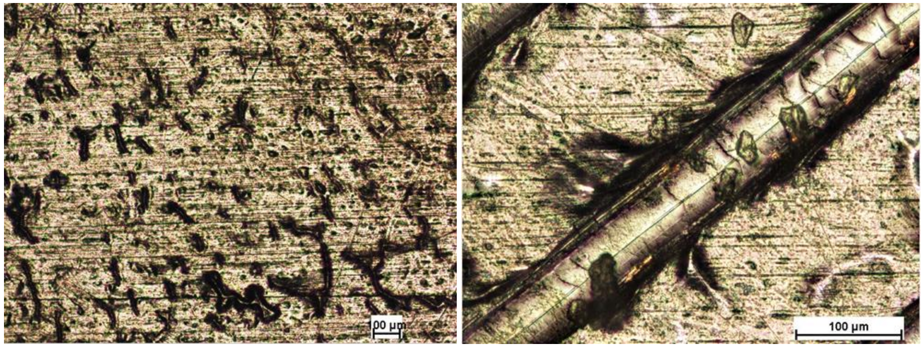


Fig.9. Optical microscope images of Nb/Cu structure sample Nr.4/5/16: (a) before irradiation and (b) after irradiation by laser with D2.

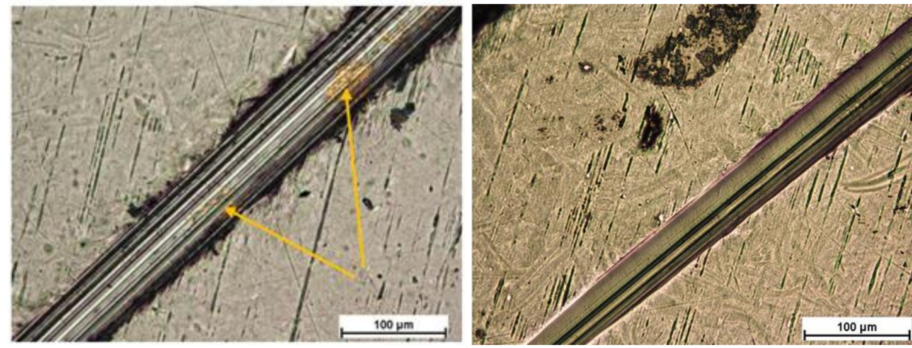


Fig.10. Optical microscope images of Nb/Cu structure sample Nr.22/3/16: (a) before irradiation and (b) after irradiation by laser with D4.

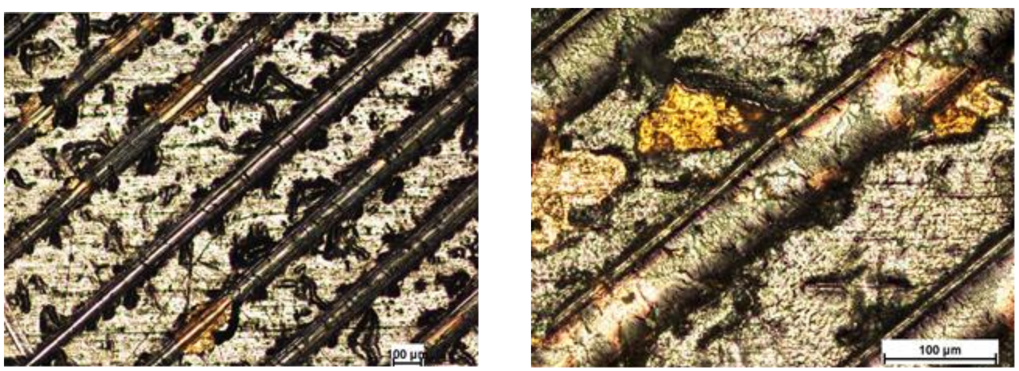


Fig11. Optical microscope images of Nb/Cu structure sample Nr.4/5/16 after scratch: (a) before irradiation and (b) after irradiation by laser with D4.

The combination of scratch test results of non-irradiated (fig. 11a) and irradiated (fig. 11b) sample Nr.4/5/16 shows that the coating has defects, such as areas of coating, which are not adhered with the cooper base. After high irradiation, these areas of coating were fused out, but areas, which were adhered with the base, were not delaminated during the scratch test. So, summarizing the results of sample Nr.4/5/16, we can say that the laser irradiation not only affects the adhesion of coating, but also increases its hardness. The worst results were obtained for Nb/Cu structure sample Nr.4/5/16 after laser irradiation D4. Surface was delaminated already after laser treatment (see fig. 11).

Influence of Laser Radiation on Adhesion of Nb layer to Cu Substrate

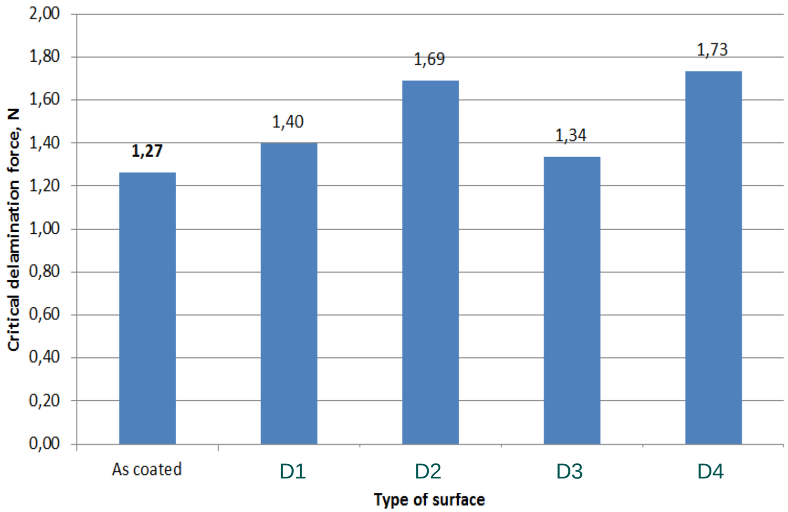


Fig.12. Critical force for the coating delamination. Sample 22/3/16.

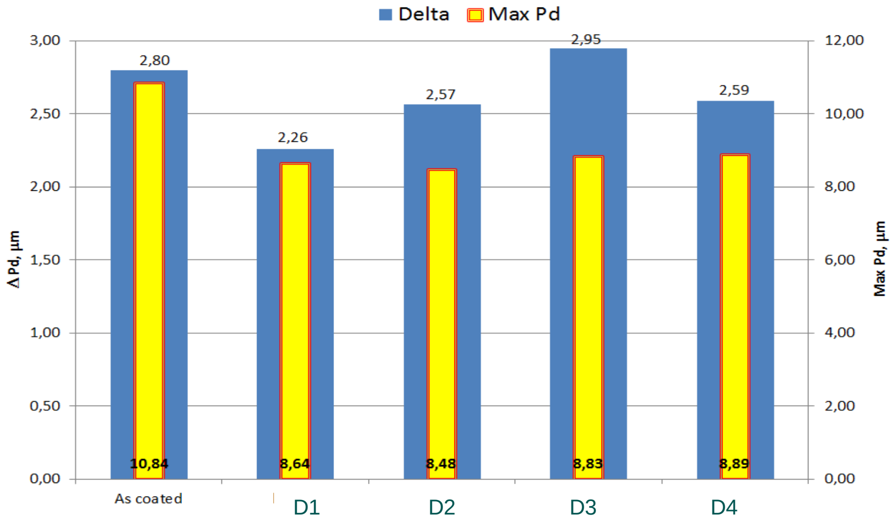


Fig.13. Maximal depth of indentation (scratch) Pd max. when load was 3N, and difference DPd between the Pd max. and Rd (residual depth). DPd shows how much surface relaxes after indentation. Sample 22/3/16.

Results

It was found that the critical delamination force for Nr.22/3/16 increased with the laser dose and was maximal 36% for the laser dose D4. Minimal indentation depth was 8.5 µm for laser dose D2 and maximal for non-irradiated area – 10.84 µm. It means that Nb cower thin layer on Cu of sample Nr. 22/3/16 is better than that of sample Nr.4/5/16 because the delamination force and the hardness increased more after laser irradiation.

The same measurements were carried out with sample Nr.4/5/16. Penetration depth and residual depth were measured depending on laser irradiation dose. Minimal indentation depth is 9.0 µm for D2 and maximal is 12.6 µm for non-irradiated area. It means that the hardness of the structure was increased after laser irradiation. Critical delamination force is maximal for laser D2 and increased by 10% as compared with non-irradiated area.

Influence of Laser Radiation on Surface Roughness of Nb/Cu Structure

Experimental

Nb films were deposited by HiPIMS (magnetron sputtering) with pulse width of 100 µs, repetition rate of 200 Hz, average plasma current of 600 mA, peak current of 40 A and a krypton pressure of 500 mTorr. Sample Nr. 1/7/16 – 500 ˚C deposition temperature with 0V DC substrate bias (grounded) for 240 mins onto copper substrate which was pre-annealed to 700˚C for 12 hours prior to deposition.

The sample was irradiated by Nd:YAG laser (λ= 1.064 µm, τ =6 ns and intensity I=193.7 MW/cm^2) in scanning mode with step 5µm in Ar atmosphere.

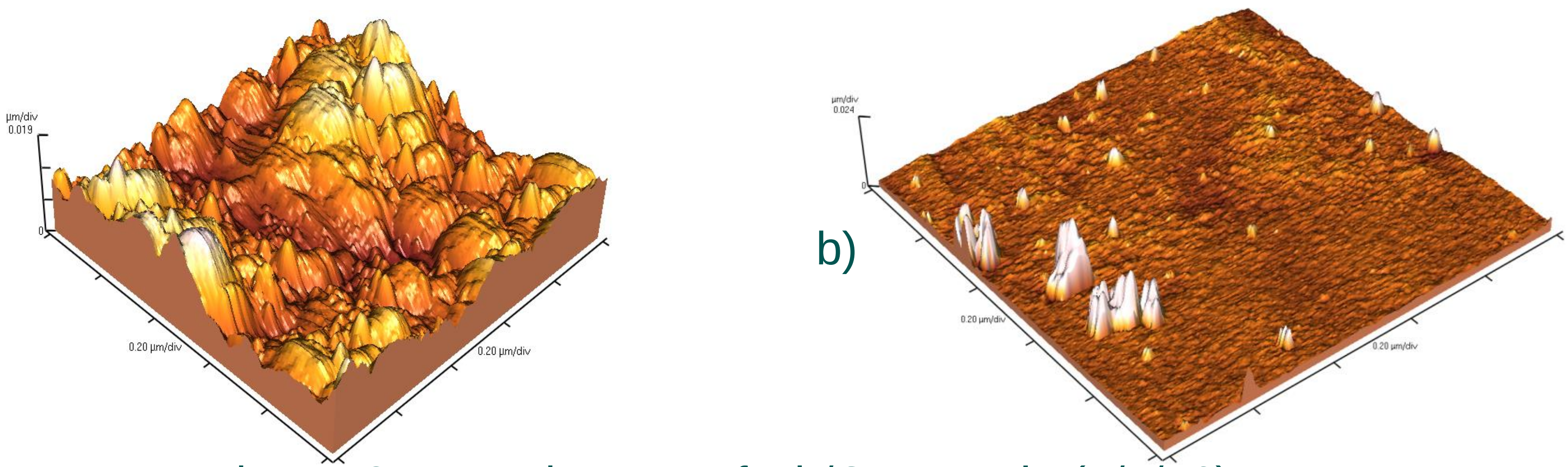
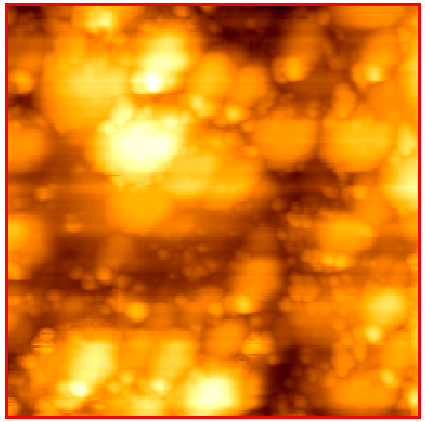
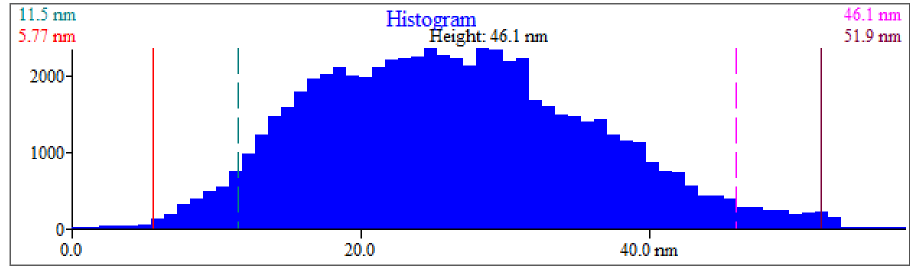


Fig.14. 3D AFM images of Nb/Cu sample (1/7/16) :
non-irradiated (a) and irradiated by Nd:YAG at intensity I=193.7 MW/cm^2 (b).

a) non-irradiated

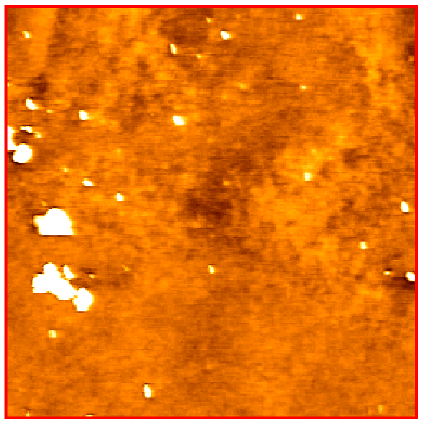


(254,256) x: 0.992 µm y: 1.000 µm z: 0.09502 µm

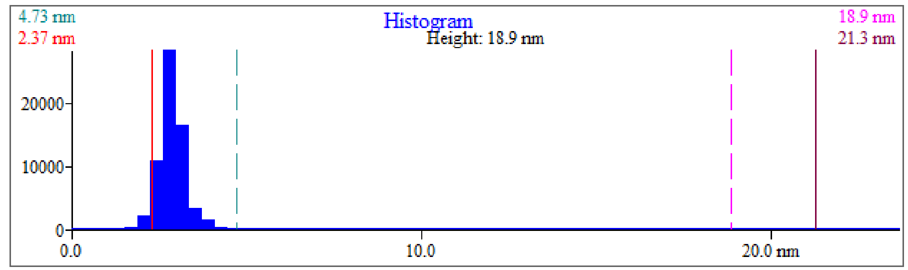


	Rp-v	Rms Rough (Rq)	Ave Rough (Ra)	Mean Ht	Median Ht	Bearing Ratio	Be F
Region 1	57.67 nm	9.528 nm	7.759 nm	26.37 nm	25.84 nm	@20.0% 34.65 nm	@80.0

b) irradiated



(255,256) x: 0.996 µm y: 1.000 µm z: -0.2818 µm



	Rp-v	Rms Rough (Rq)	Ave Rough (Ra)	Mean Ht	Median Ht	Bearing Ratio	Be F
Region 1	23.66 nm	1.233 nm	0.4612 nm	2.992 nm	2.908 nm	@20.0% 3.17 nm	@80.0

Fig.15. 2D AFM images of the non-irradiated (a) and the irradiated (b) Nb/Cu sample (1/7/16) and region analyses. The Nb surface roughness RMS (Root Mean Square Roughness) of the **non-irradiated is 9.528nm**; for laser **irradiated 1.233 nm**.

Conclusions

1. The critical delamination force for irradiated sample Nr.22/3/16 increased with the laser dose by maximally 36%. The results can be explained by interdiffusion of Cu and Nb.
2. Laser radiation leads to decrease of Nb surface roughness (RMS) from 8 nm till 1 nm.
3. The sizes of Nb crystals can be increased and defects between grains (pinholes) can be eliminated by laser radiation.
4. The best hardness results were achieved for the sample Nb/Cu Nr. 22/3/16, which was deposited at temperature 500 ˚C.

Thank you very much for your attention!

Proposal of Working Plan:

Increase the grain size and adhesion, and anneal the defects

With the aim to understand adhesion(delamination) mechanism of Nb we propose to study the following samples

1. Nb thin layer on Cu substrate after thermal annealing at 120°C, 24 hours - 4 samples 10x10x 1- 2 mm^3;
2. Nb thin film on Cu substrate after cooling down the samples below Tc - 4 samples 10x10x 1- 2 mm^3;
3. Nb thin film on Cu substrate after irradiation by RF at maximal used intensity - 4 samples 10x10x 1- 2 mm^3.

We are planning to improve the adhesion between Nb and Cu substrate by increase Cu solubility [1] in Nb crystal by more than 3.2% by laser radiation. We are planning the following steps:

1. Deposition of Nb on Cu substrate with thickness 20 nm [2] and thermal annealing at 120°C, 24 hours;
2. Irradiation of the Nb/Cu structure by Nd:YAG laser for inter-diffusion of Cu and Nb;
3. Deposition of Nb film with thickness up to 1 mkm on Nb/Cu structure at T = 120°C for Nb crystal grow.

[1] A.Medvid', A.Mychko, E.Dauksta, „Enhancement of resistivity of CdZnTe crystal by laser radiation", Advanced Materials Research, Vol. 1117, pp. 19-22, 2015.

[2] Weaver, J. H. and Lynch, D. W. and Olson, C. G., Optical Properties of Niobium from 0.1 to 36.4, PhysRevB.7.4311,1973},