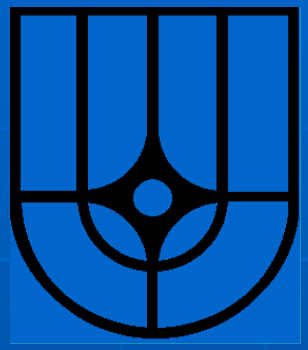
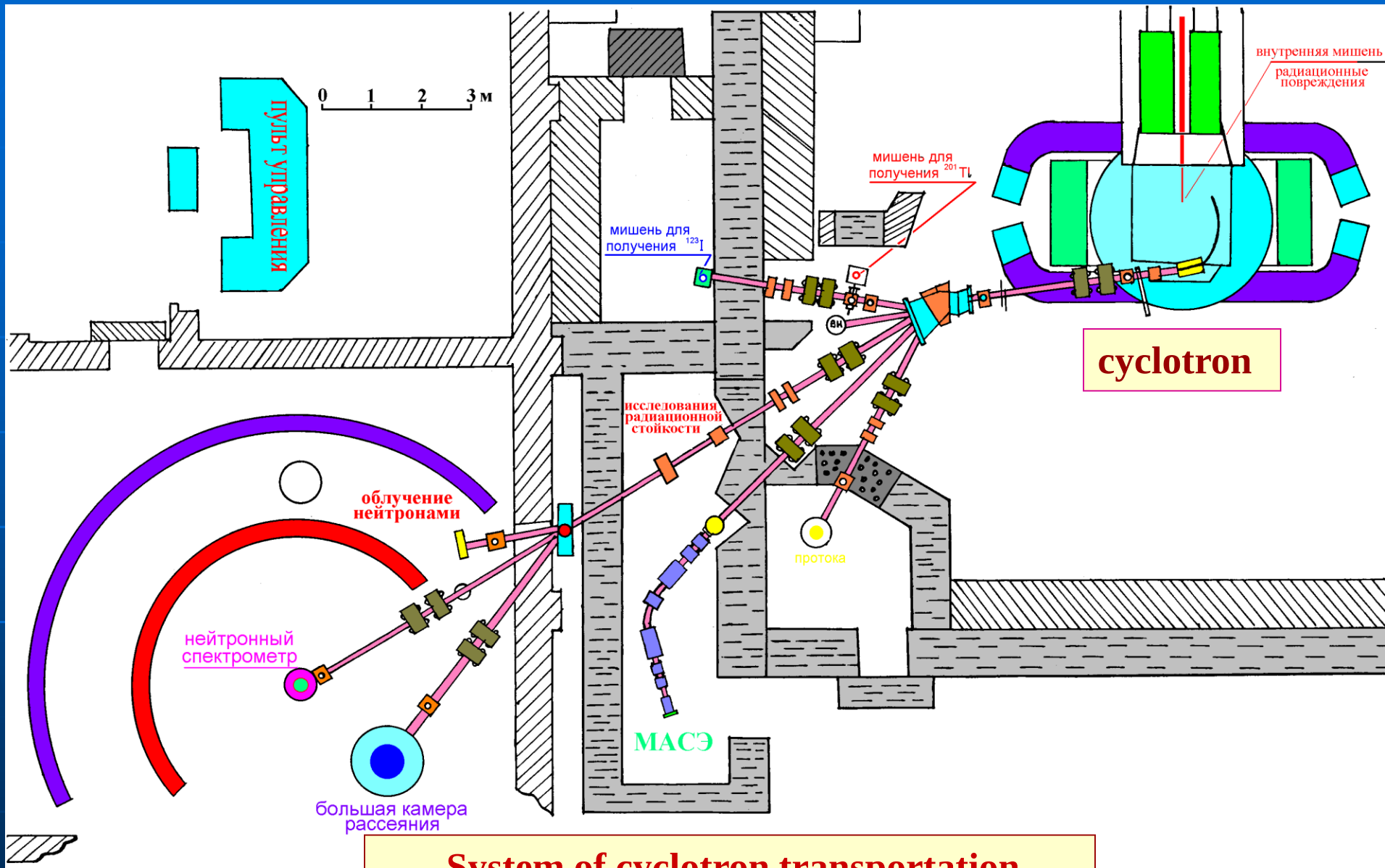
National Research Centre" Kurchatov Institute"



Investigations of fast particle irradiations on the properties of Molybdenum-Copper-Diamond collimator materials for the Large Hadron Collider at CERN using the NRC KI cyclotron.

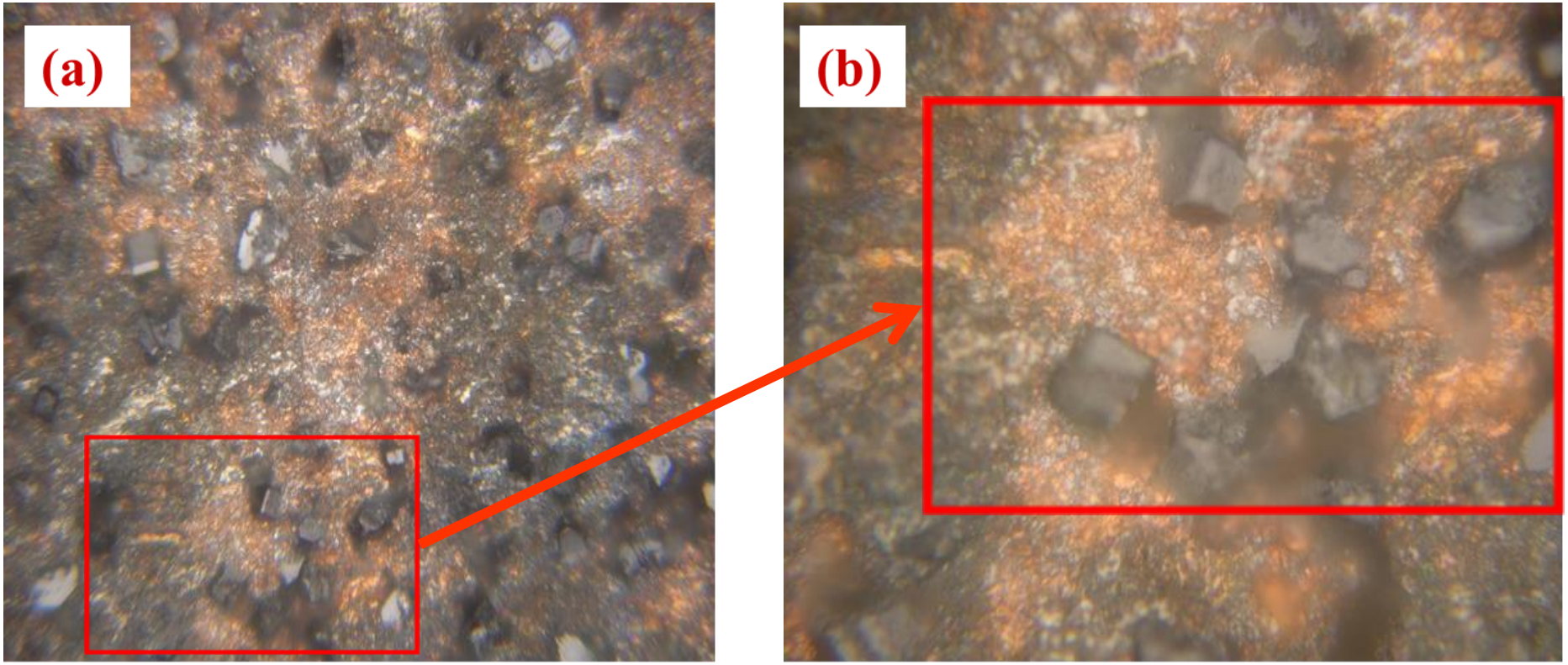
Alexander Ryazanov

27-28 November 2017, Torino, Italy

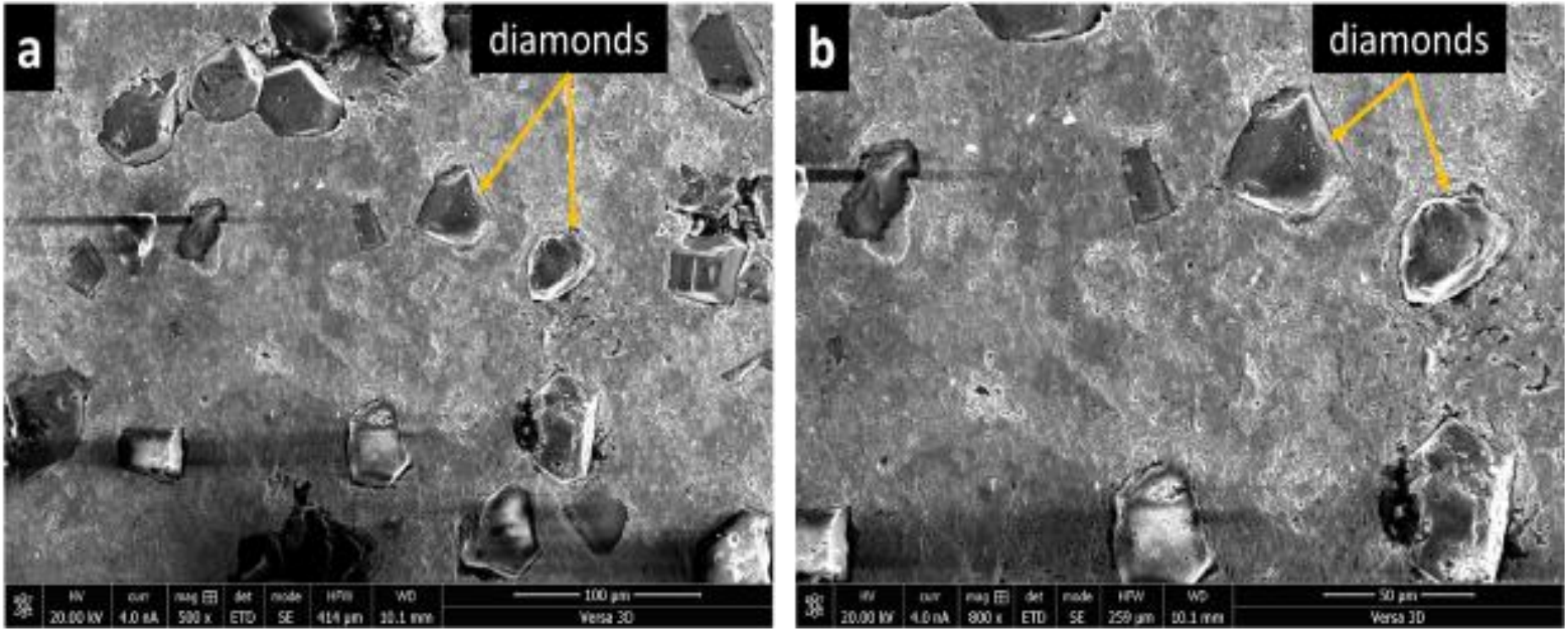


System of cyclotron transportation

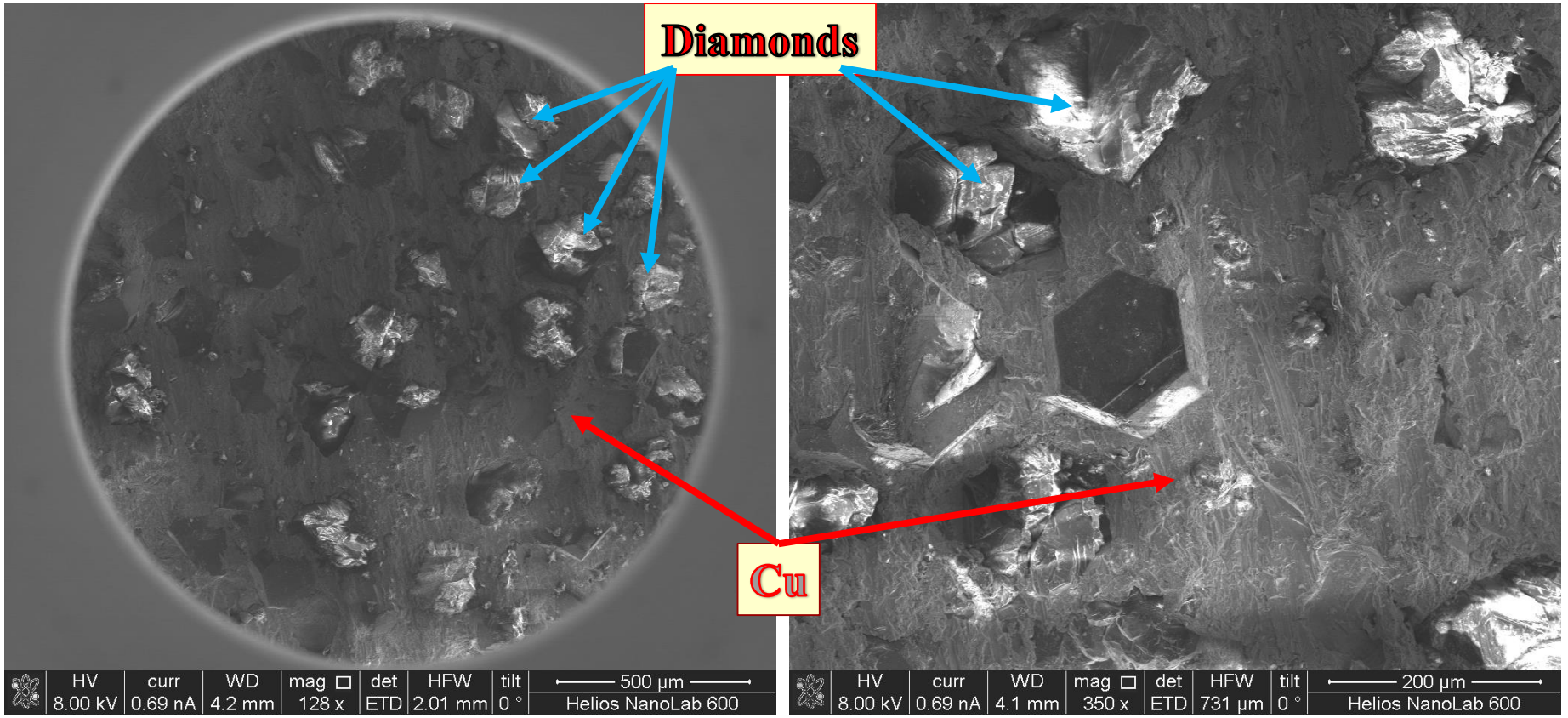
Microstructure of Mo – Cu - Diamond sample surface under different magnification (-200 (a) and -500 (b)) made on optical microscope Carl Zeiss Axio Observer D1m



SE SEM micrographs of the sample section. The microdiamonds embedded to the metal matrix of Mo-Cu-D are indicated by arrows (a) and (b).

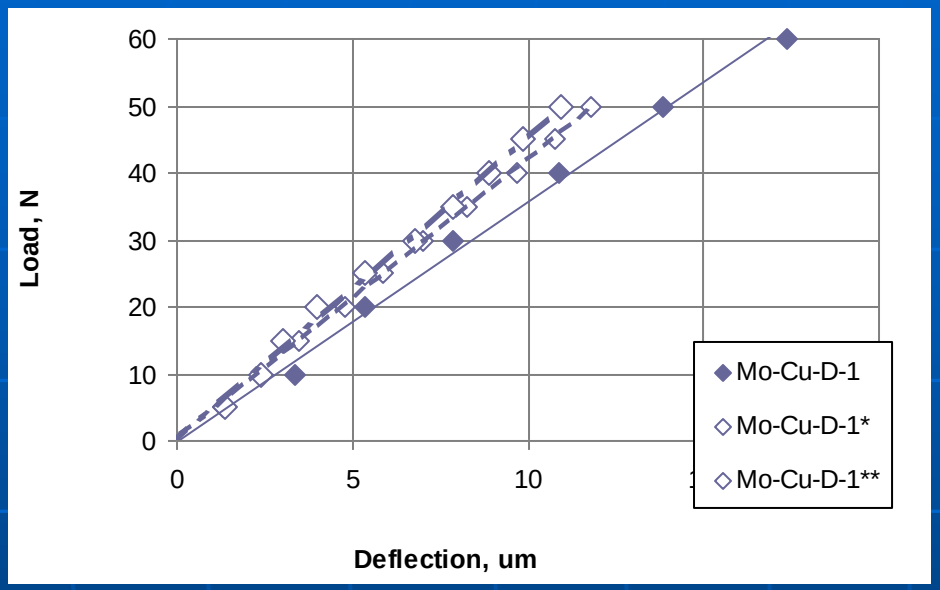


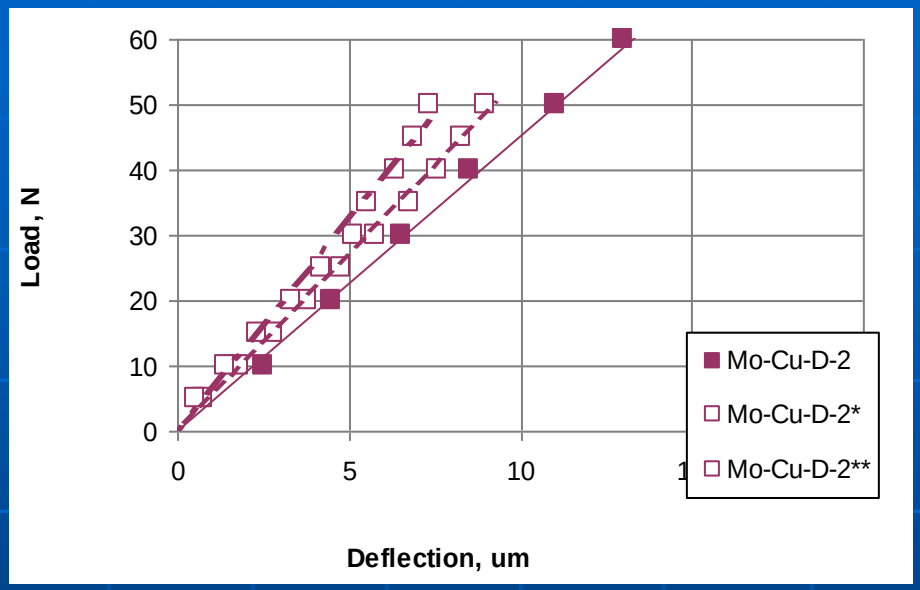
The SEM study and sample preparation

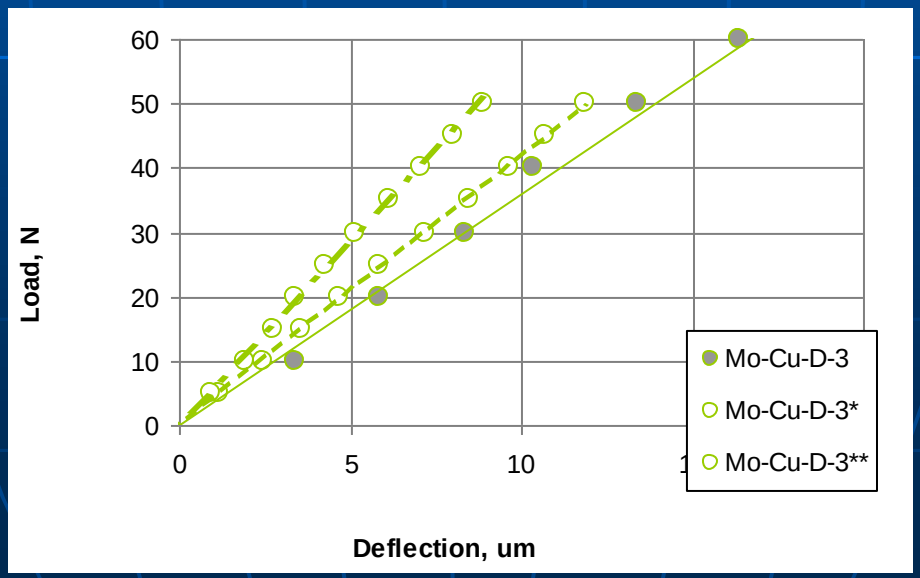


SEM (secondary electrons -SE) images of the Cu-Diamond composite

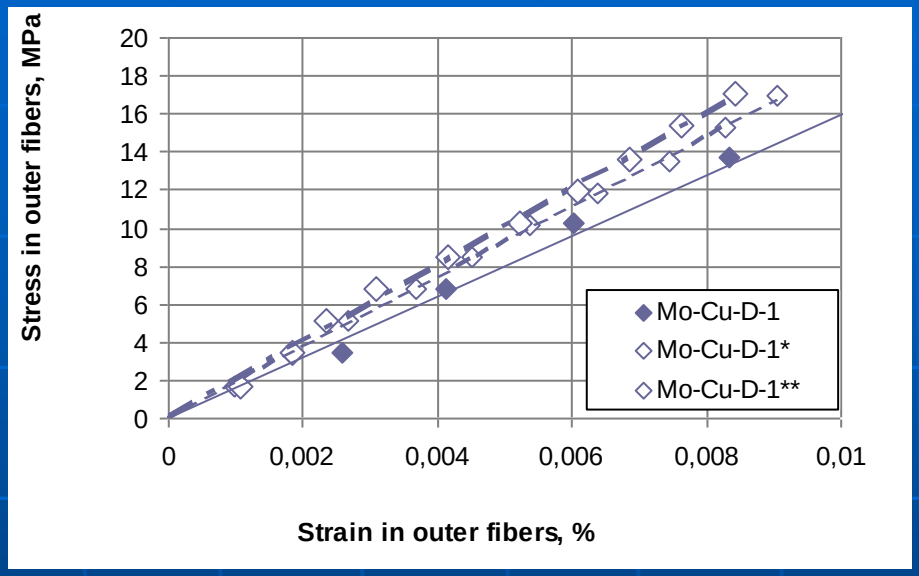
«Molybdenum-Copper Diamond» measurements of elastic modulus ($E^{И}$, $E^{И*}$, $E^{И**}$) on non-irradiated samples, after 1st irradiation by 30 MeV protons with doses 10E17p/cm2 and 2nd irradiation with doses 10E18p/cm2

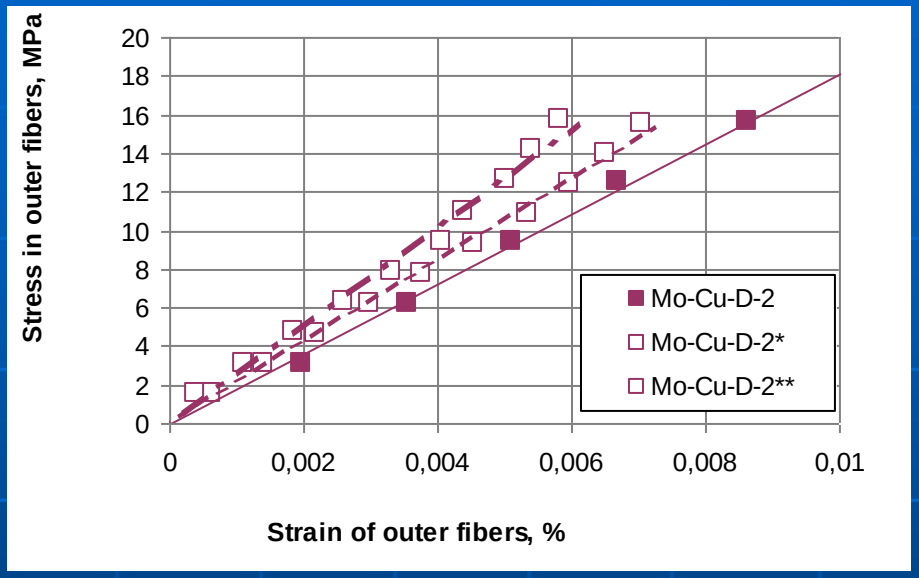


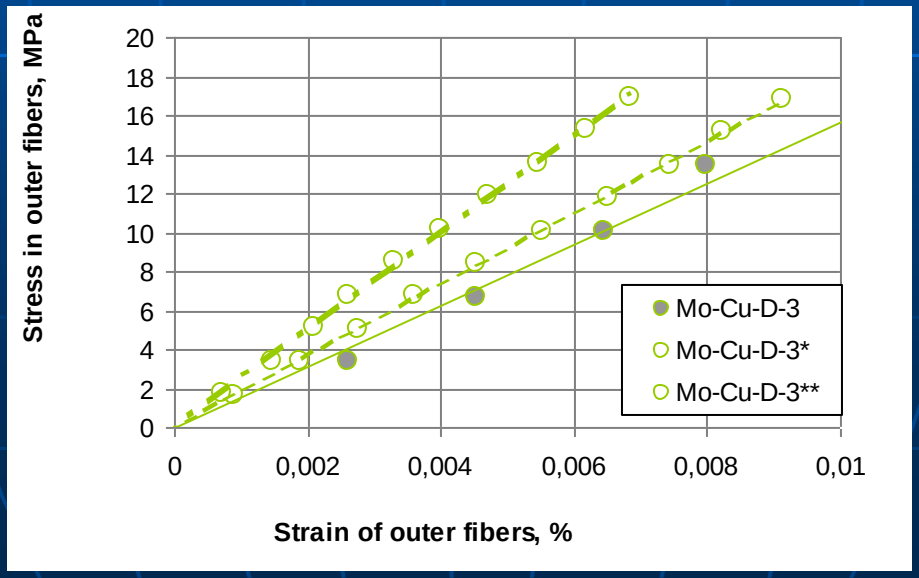




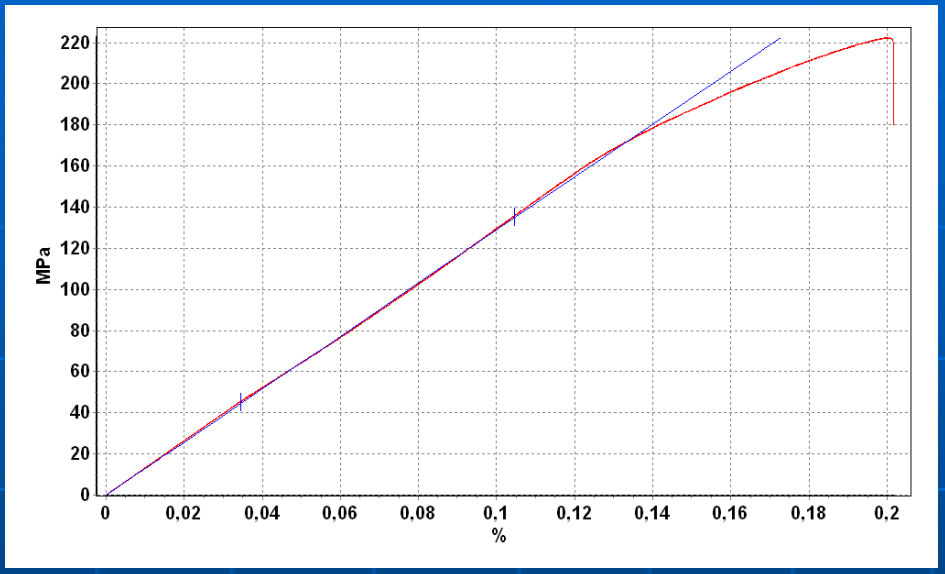
Deformation curves in outer fibers of the samples versus strain in non-irradiated, after 1st irradiation by 30 MeV protons with doses 10E17p/cm2 and 2nd irradiation with doses 10E18p/cm2

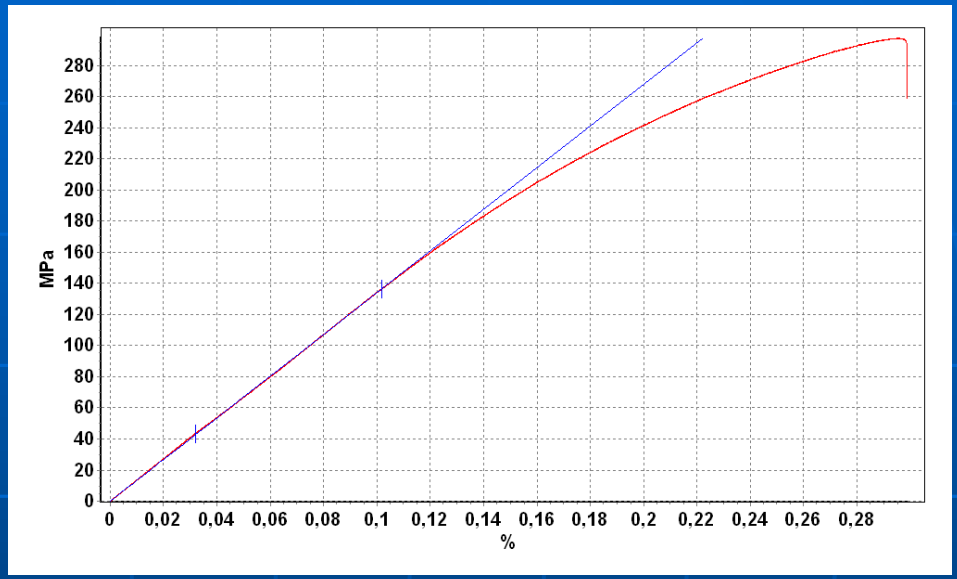


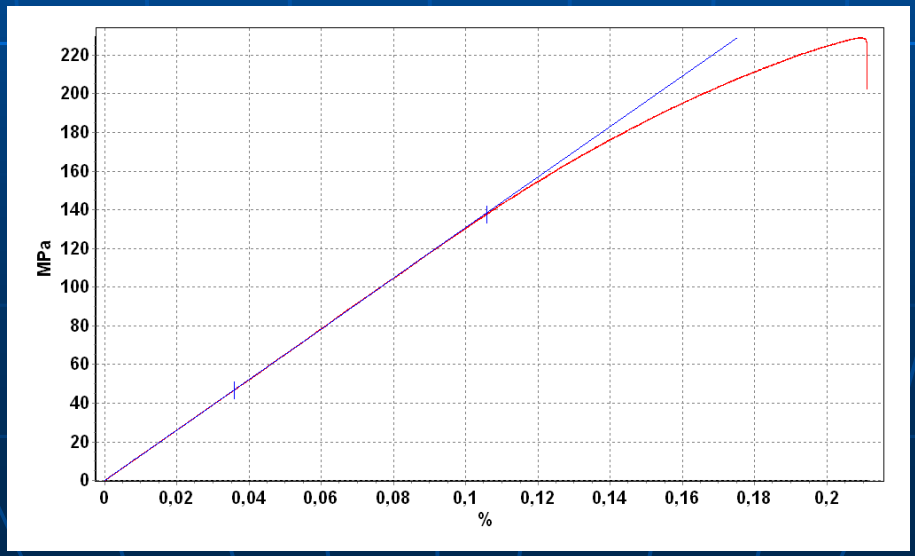




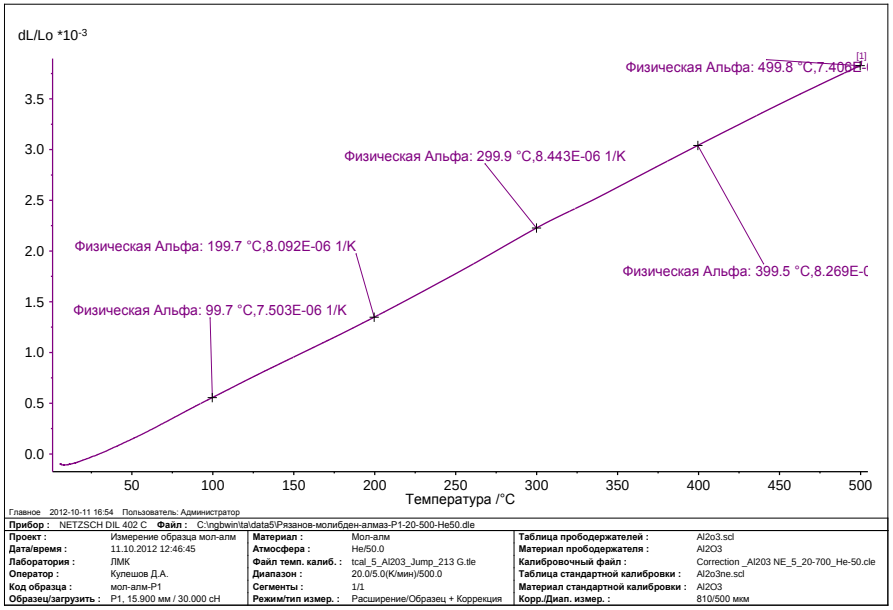
Deformation curves in outer fibers of three Mo-Cu-D samples versus strain at higher stresses after 2st irradiation by 30 MeV protons with doses 10E18p/cm2

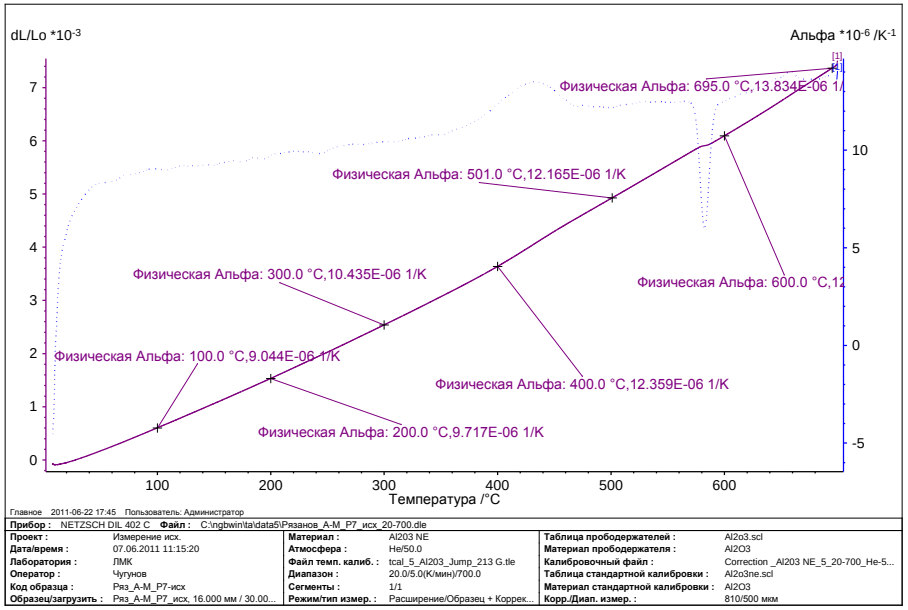




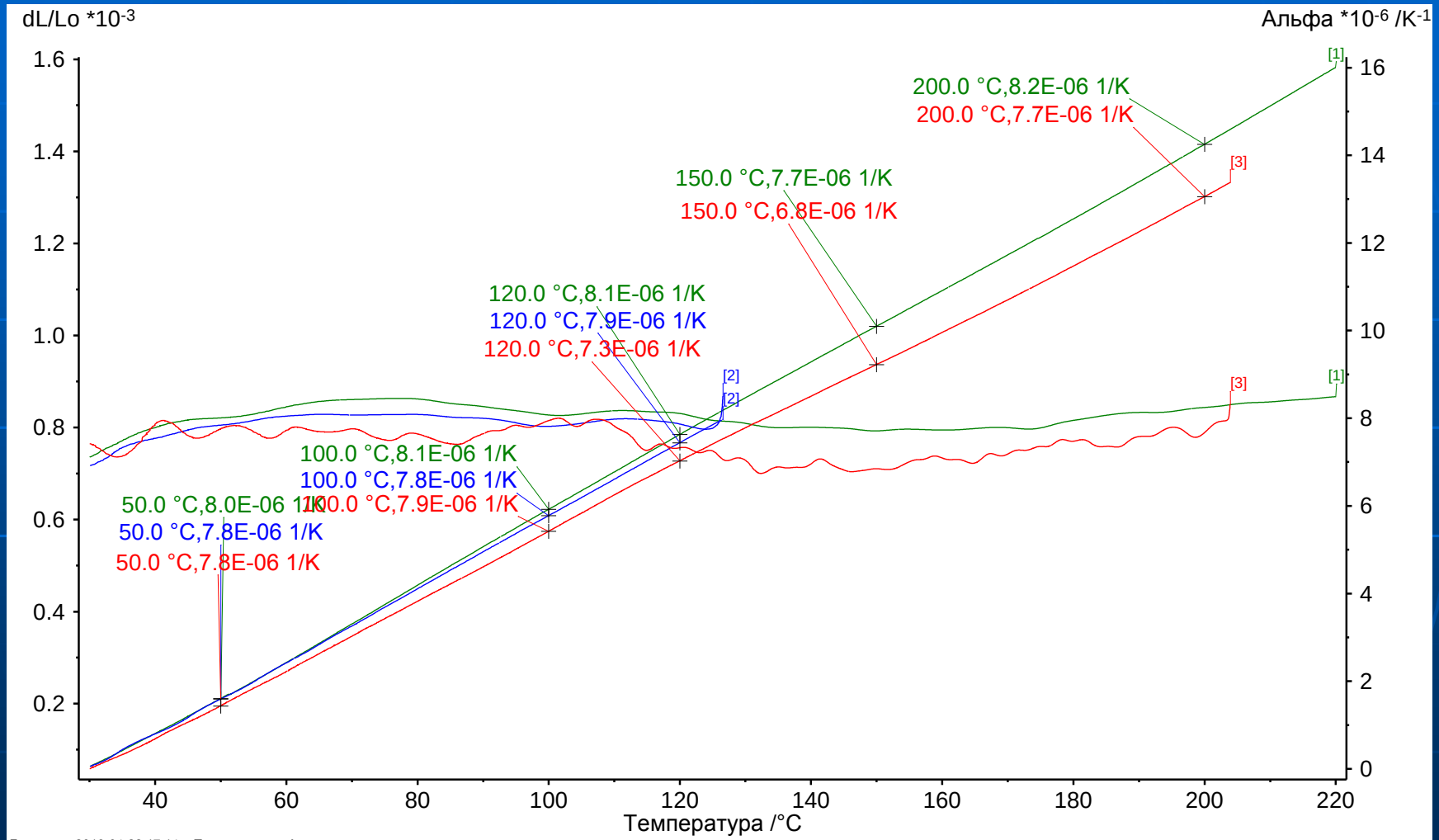


Temperature dependence of the thermal expansion coefficient (CTE) for Mo-Cu-Diamond material (P1, P7 samples) before irradiation





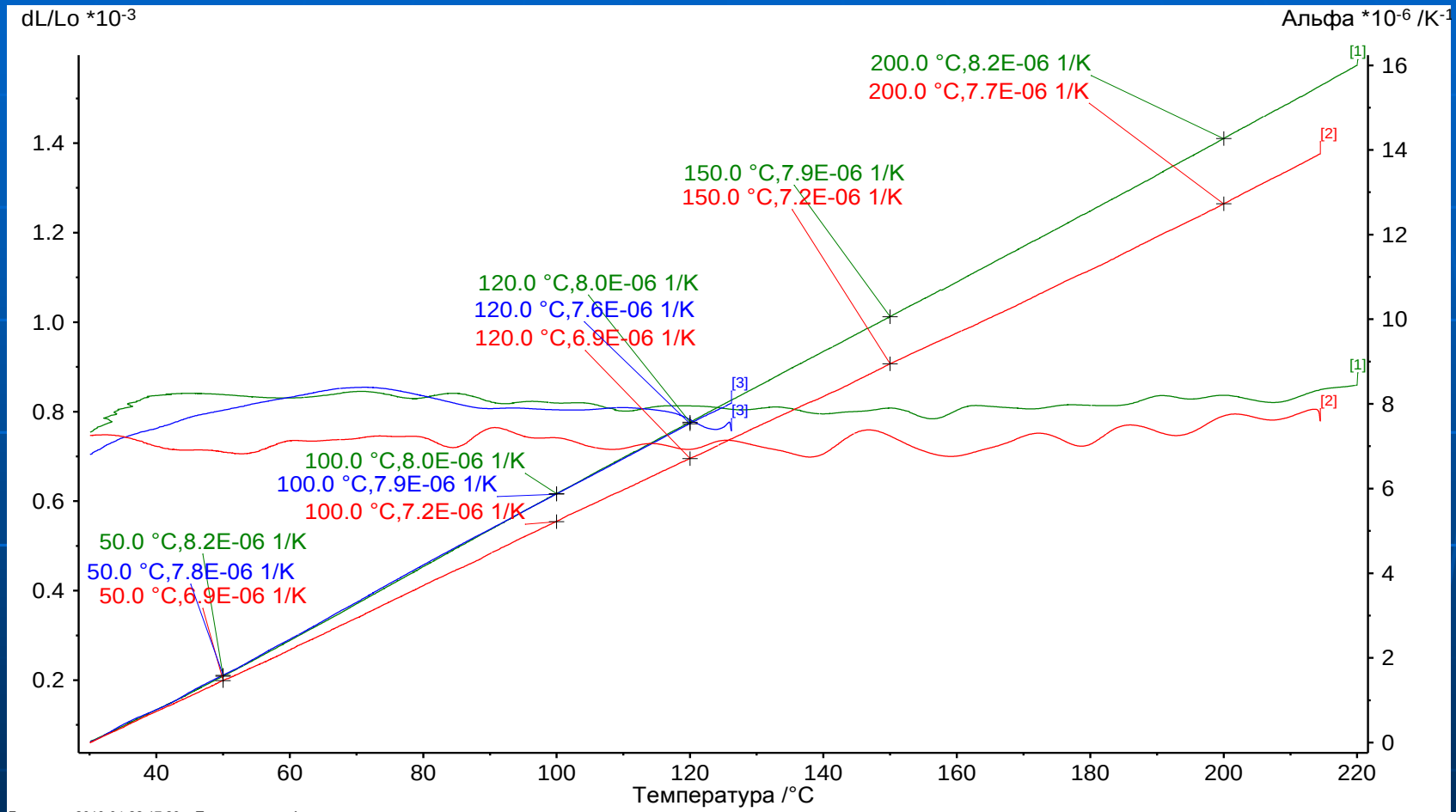
Temperature dependence of the CTE for Mo-Cu-Diamond material (P-1 sample) before irradiation (green line), after first irradiation (blue line), and after second irradiation (red line).



Главное 2016-04-22 17:14 Пользователь: Администратор

[#] Приб...	Файл	Дата	Код образца	Образец	Дл...	С..	Диапазон	Атм...	Корр.
[1] DIL 40...	Рязанов-молибден-алмаз-P1-20-500-He50.dle	2012-10...	мол-алм-P1	P1	15....	1...	20.0/5.0(K/мин)/5...	He/5...	dL:013, Аль...
[2] DIL 40...	Разан_Мол_Алм-P1-1 обл-120-He.dle	2014-04...	Ряз_Мол_Ал_P1_1обл	Ряз_Мол_Алм_P1-1обл	16....	1...	20.0/5.0(K/мин)/1...	He/5...	dL:013, Аль...
[3] DIL 40...	Ряз_мол-медь-алм_P-1_2-обл_20-5K-215-40K-20_H...	2016-04...	Ряз_Мол-медь-алм_P-1_2...	Ряз_Мол-медь-алм_P-1_2...	16....	1...	20.0/5.0(K/мин)/2...	He/5...	dL:213, Аль...

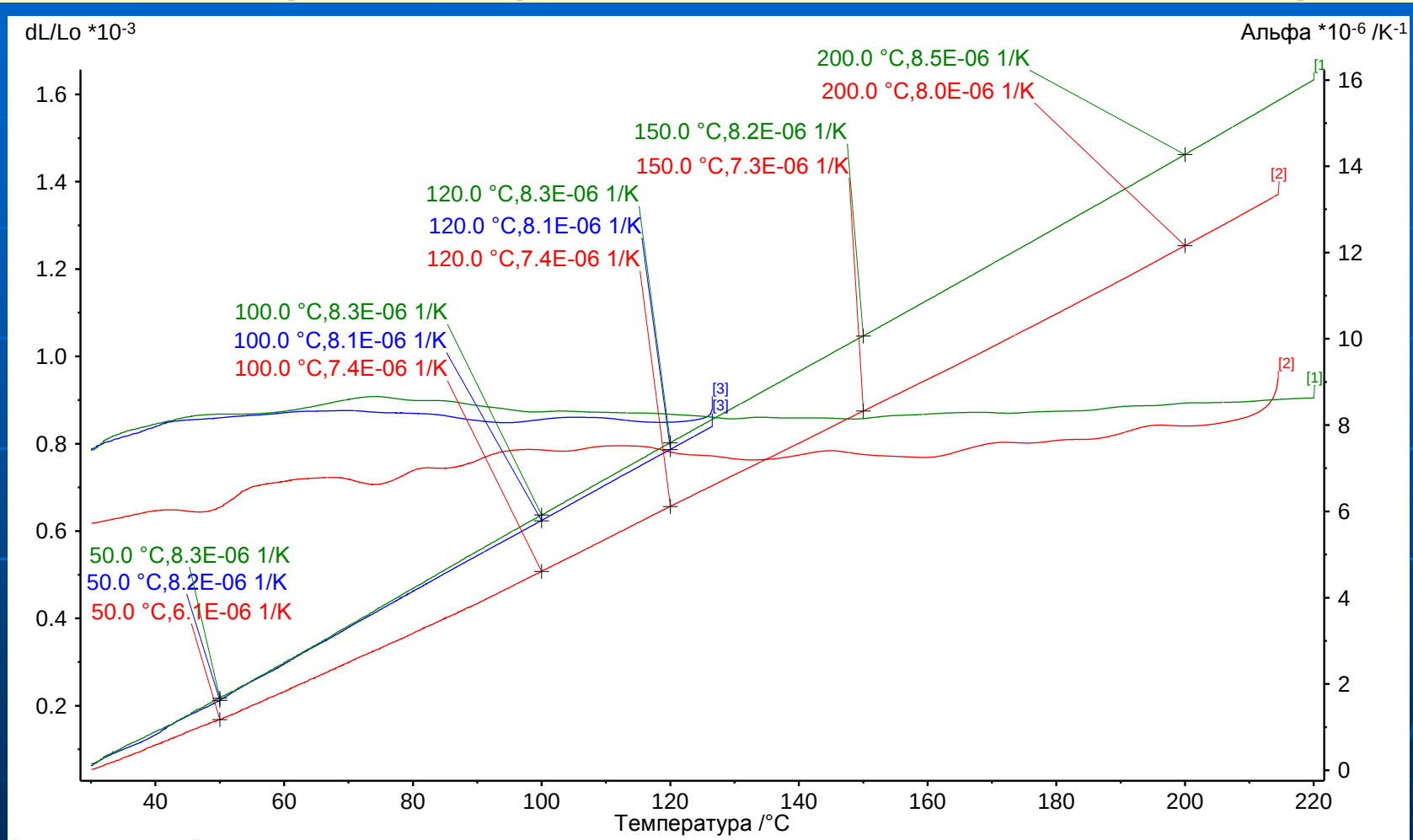
Temperature dependence of the CTE for Mo-Cu-Diamond material (P-2 sample) before irradiation (green line), after first irradiation (blue line), and after second irradiation (red line).



Главное 2016-04-22 17:23 Пользователь: Администратор

[#] Приб...	Файл	Дата	Код образца	Образец	Дл...	С..	Диапазон	Атм...	Корр.
[1] DIL 40...	Рязан-Р2-мол-медь-ал-20-500-He.dle	2013-03...	Рязан-Р2-Мо-медь-алма	Рязан-Р2-мол-медь ал	16....	1...	20.0/5.0(К/мин)/5...	He/5...	dL:013, Аль...
[2] DIL 40...	Ряз_мол-медь-алм_Р-2_2-обл_20-1К-215-40К-20_Н...	2016-04...	Ряз_Мол-медь-алм_Р-2_2...	Ряз_Мол-медь-алм_Р-2_2...	16....	1...	20.0/1.0(К/мин)/2...	He/5...	dL:013, Аль...
[3] DIL 40...	Рязан_Мол-Алм_Р2-1 об120-He.dle	2014-04...	Рязан_Мол-Алм-Р2-1обл	Рязан_Мол-Алм_Р2-1об	16....	1...	20.0/5.0(К/мин)/1...	He/5...	dL:013, Аль...

Temperature dependence of the CTE for Mo-Cu-Diamond material (P-3 sample) before irradiation (green line), after first irradiation (blue line), and after second irradiation (red line).



Главное 2017-02-07 16:12 Пользователь: Администратор

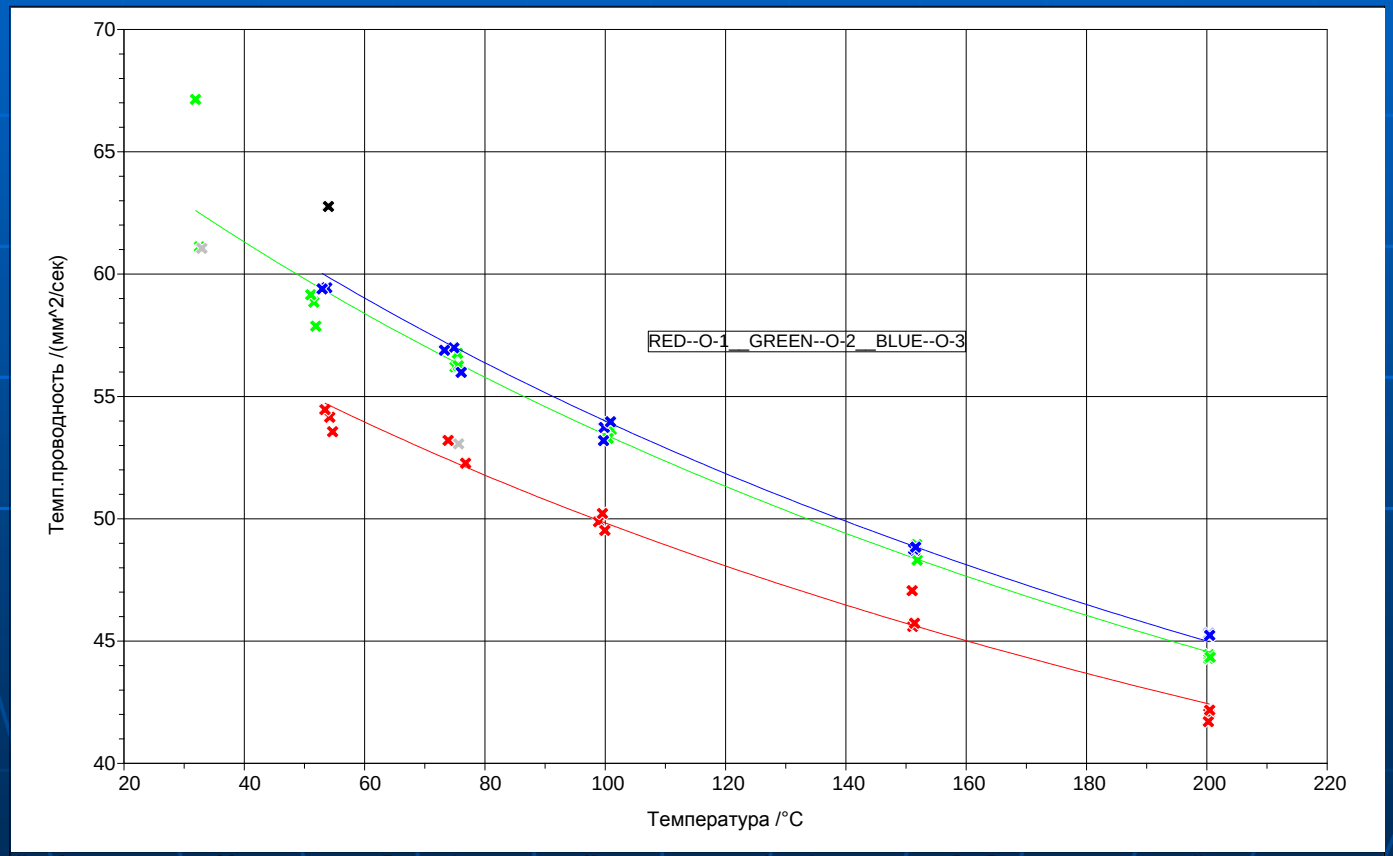
[#] Приб...	Файл	Дата	Код образца	Образец	Дл...	С..	Диапазон	Атм...	Корр.
[1] DIL 40...	Рязан-Р3-Мол-медь-алм_20-500_5_He-50.dle	2013-03...	Рязан-Р3-Мол-медь-алмаз...	Рязан-Р3-Мол-медь-алмаз...	16....	1...	20.0/5.0(К/мин)/5...	He/5...	dL:813, Аль...
[2] DIL 40...	Ряз_мол-медь-алм_Р-3_2-обл_20-1К-215-40К-20_H...	2016-04...	Ряз_Мол-медь-алм_Р-3_2-...	Ряз_Мол-медь-алм_Р-3_2-...	15....	1...	20.0/1.0(К/мин)/2...	He/5...	dL:813, Аль...
[3] DIL 40...	Рязан_Мол-Алм_Р3-1 обл-20-120.dle	2014-04...	Рязан-Мол-Алм-Р3-1 обл	Рязан-Мол-Алм_Р3-1 об	16....	1...	20.0/5.0(К/мин)/1...	He/5...	dL:013, Аль...

Density measurements of “Molybdenum-Copper-Diamond” material samples

Sample, shape	Mass, g	Dimensions, mm	Density d, g/cm^3, hydrostatic	Density change $(d_{irr}-d_{init})/d_{init}$, %
O-1, cylinder	2.225/2.254/ 2.252*	D=10.115 H=4.225	6.569/6585/6580	0.2/0.2
O-2, cylinder	2.093/2.090/ 2.084	D=10.00 H=4.185	6.285/6.289/6.253	0.1/-0.5
O-3, cylinder	2.515/2.485/ 2.464	D=10.225 H=4.505	6.745/6.681/6.667	-0.9/-1.2
P-1, parallelepiped	1.918/1.903/ 1.934	L=16.0	6.686/6.737/6.562	0.8/-1.9
P-2, parallelepiped	- /1.982/1.979	L=16.0	6.686/6.705/6.615	0.3/-1.1
P-3, parallelepiped	1.970/1.944/ 1.899	L=16.0	6.665/6.668/6.679	0.05/0.2

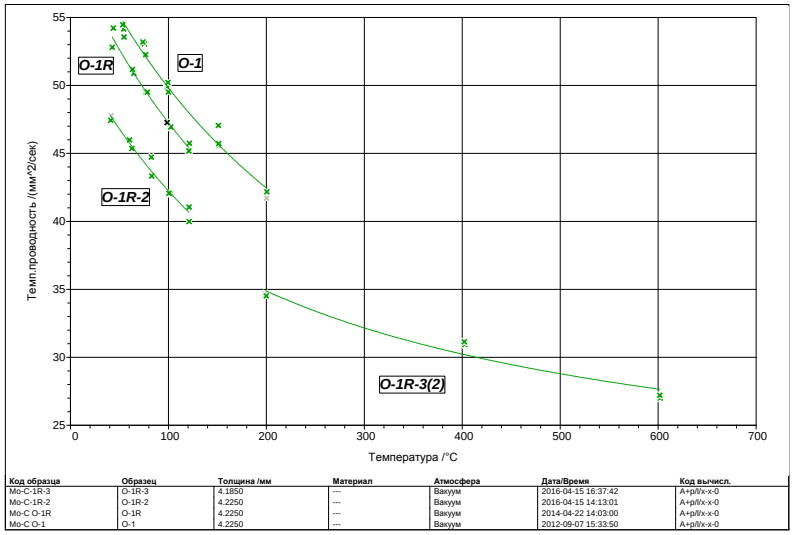
*** - values are given in the nominator before irradiation and in the denominator after irradiation.**

Temperature dependence of thermal conductivity for 3 non-irradiated Mo-Cu-Diamond samples up to 200ºC. Red dots correspond to the sample O-1; green dots correspond to the sample O-2; blue dots correspond to the sample O-3.

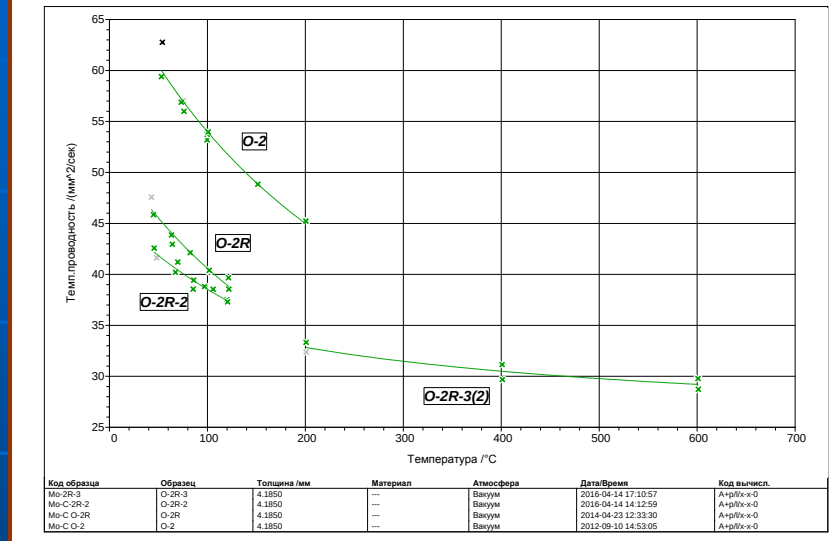


Код образца	Образец	Толщина /мм	Материал	Атмосфера	Дата/Время	Код вычисл.
Mo-C O-3	O-3	4.4900	---	Вакуум	2012-09-14 11:50:26	A+p/l/x-x-0
Mo-C O-2	O-2	4.1850	---	Вакуум	2012-09-10 14:53:05	A+p/l/x-x-0
Mo-C O-1	O-1	4.2250	---	Вакуум	2012-09-07 15:33:50	A+p/l/x-x-0

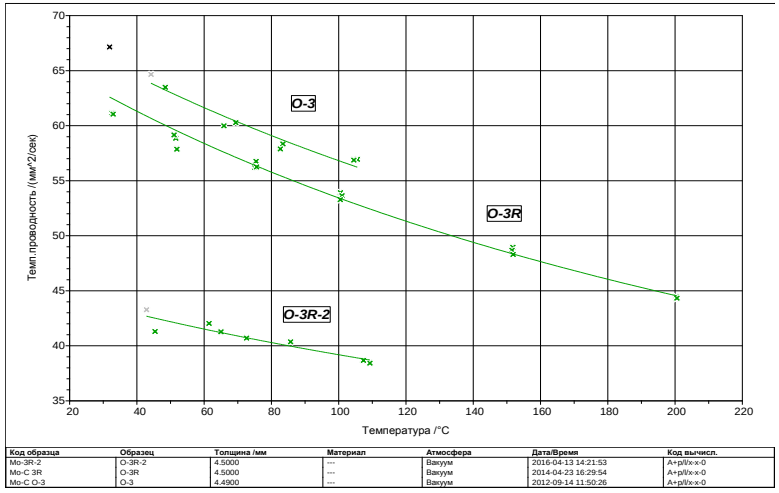
Temperature dependence of thermal conductivity for three Mo-Cu-Diamond samples (O-1, O-2, and O-3) up to 200ºC (initial), up to 120ºC (after first irradiation (marked as R), after second irradiation (marked as R2)), and up to 600ºC (after second irradiation (marked as R3))



Код образца	Образец	Толщина /мм	Материал	Атмосфера	Дата/Время	Код вычисл.
Mo-C-1R-3	O-1R-3	4.1850	---	Вакуум	2016-04-15 16:37:42	A+p/l/x-x-0
Mo-C-1R-2	O-1R-2	4.2250	---	Вакуум	2016-04-15 14:13:01	A+p/l/x-x-0
Mo-C O-1R	O-1R	4.2250	---	Вакуум	2014-04-22 14:03:00	A+p/l/x-x-0
Mo-C O-1	O-1	4.2250	---	Вакуум	2012-09-07 15:33:50	A+p/l/x-x-0



Код образца	Образец	Толщина /мм	Материал	Атмосфера	Дата/Время	Код вычисл.
Mo-2R-3	O-2R-3	4.1850	---	Вакуум	2016-04-14 17:10:57	A+p/l/x-x-0
Mo-C-2R-2	O-2R-2	4.1850	---	Вакуум	2016-04-14 14:12:59	A+p/l/x-x-0
Mo-C O-2R	O-2R	4.1850	---	Вакуум	2014-04-23 12:33:30	A+p/l/x-x-0
Mo-C O-2	O-2	4.1850	---	Вакуум	2012-09-10 14:53:05	A+p/l/x-x-0

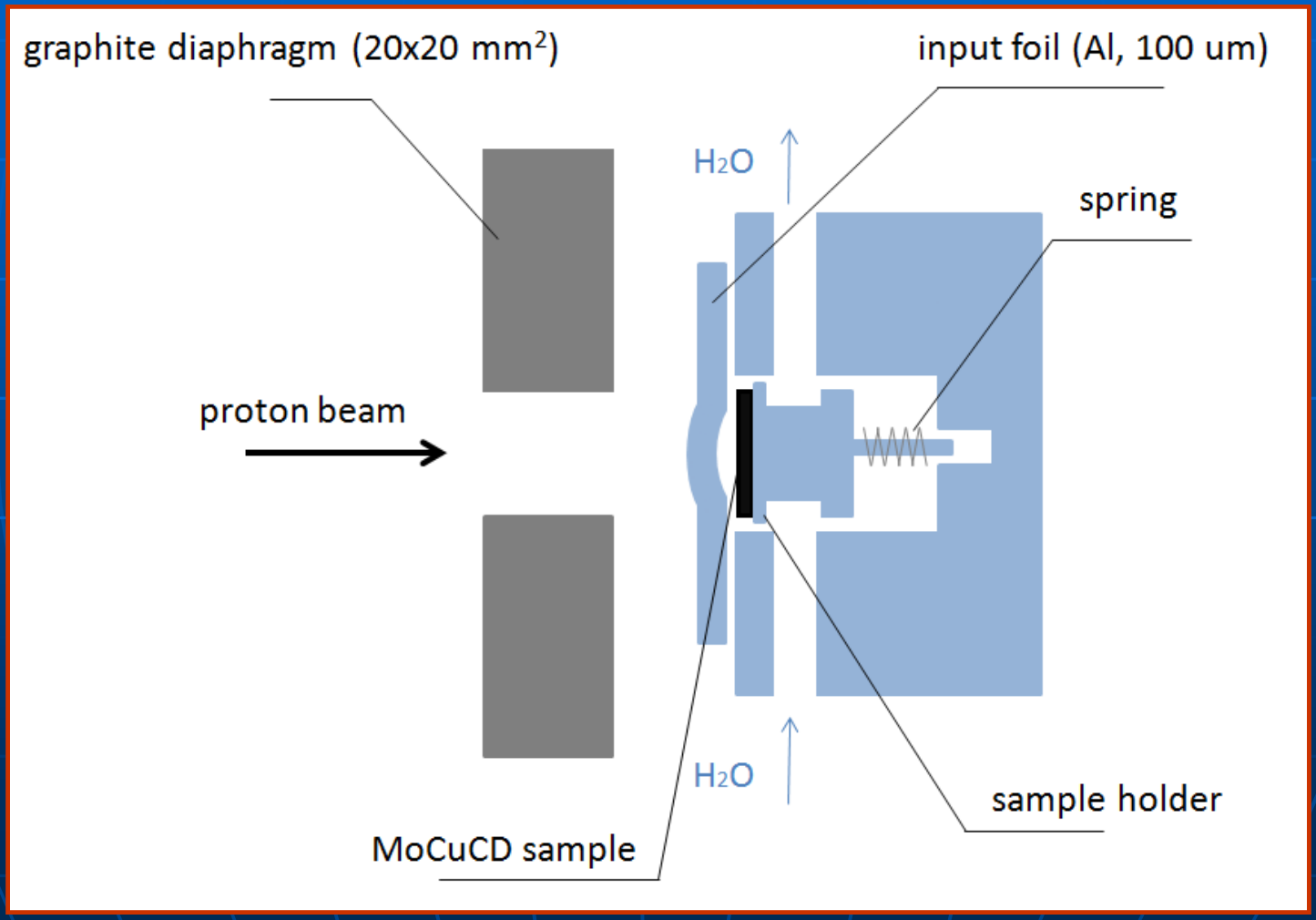


Код образца	Образец	Толщина /мм	Материал	Атмосфера	Дата/Время	Код вычисл.
Mo-3R-2	O-3R-2	4.5000	---	Вакуум	2016-04-13 14:21:53	A+p/l/x-x-0
Mo-C 3R	O-3R	4.5000	---	Вакуум	2014-04-23 16:29:54	A+p/l/x-x-0
Mo-C O-3	O-3	4.4900	---	Вакуум	2012-09-14 11:50:26	A+p/l/x-x-0

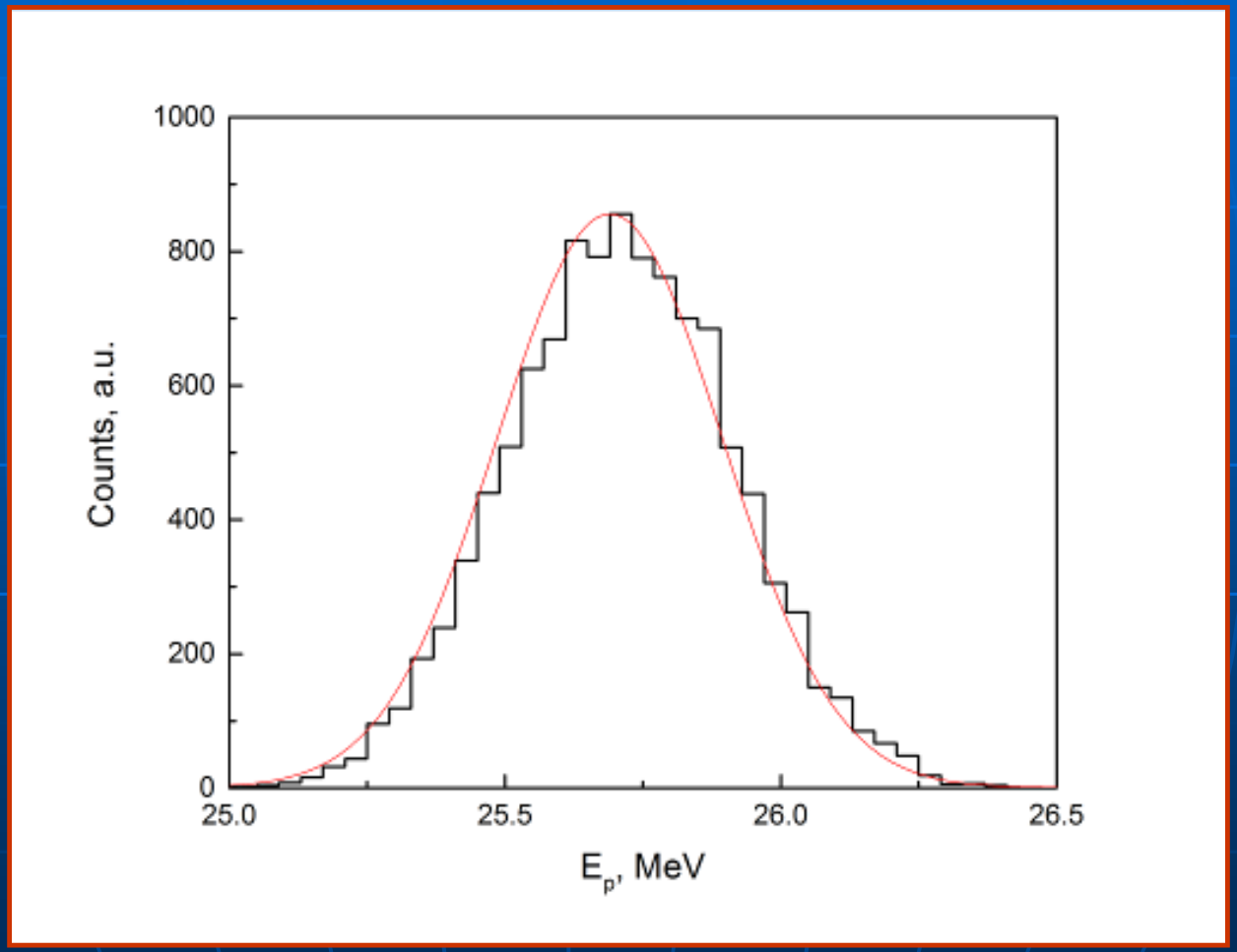
Measurement results of specific resistivity for Mo-Cu-CD collimator materials before and after two irradiations by fast protons with the energy 30 MeV at two doses: Φ1=10e17 p/cm2 and Φ2=10e18 p/cm2

Initial	Mo-Cu-D-1	Mo-Cu-D-2	Mo-Cu-D-3
ρ_{init}, 10^{-8}, Ohm·m	11.0±0.7	8.5±0.5	9.1±0.5
1st irradiation	Mo-Cu-D-1*	Mo-Cu-D-2*	Mo-Cu-D-3*
ρ_{irr}*, 10^{-8}, Ohm·m	14±1	11±1	13.7±0.9
$(\rho_{irr}$*- $\rho_{init})/\rho_{init}$, %	28	29	51
2nd irradiation	Mo-Cu-D-1**	Mo-Cu-D-2**	Mo-Cu-D-3**
ρ_{irr}**, 10^{-8}, Ohm·m	15±1	11±1	14.1±0.9
$(\rho_{irr}$**- $\rho_{init})/\rho_{init}$, %	36	29	55

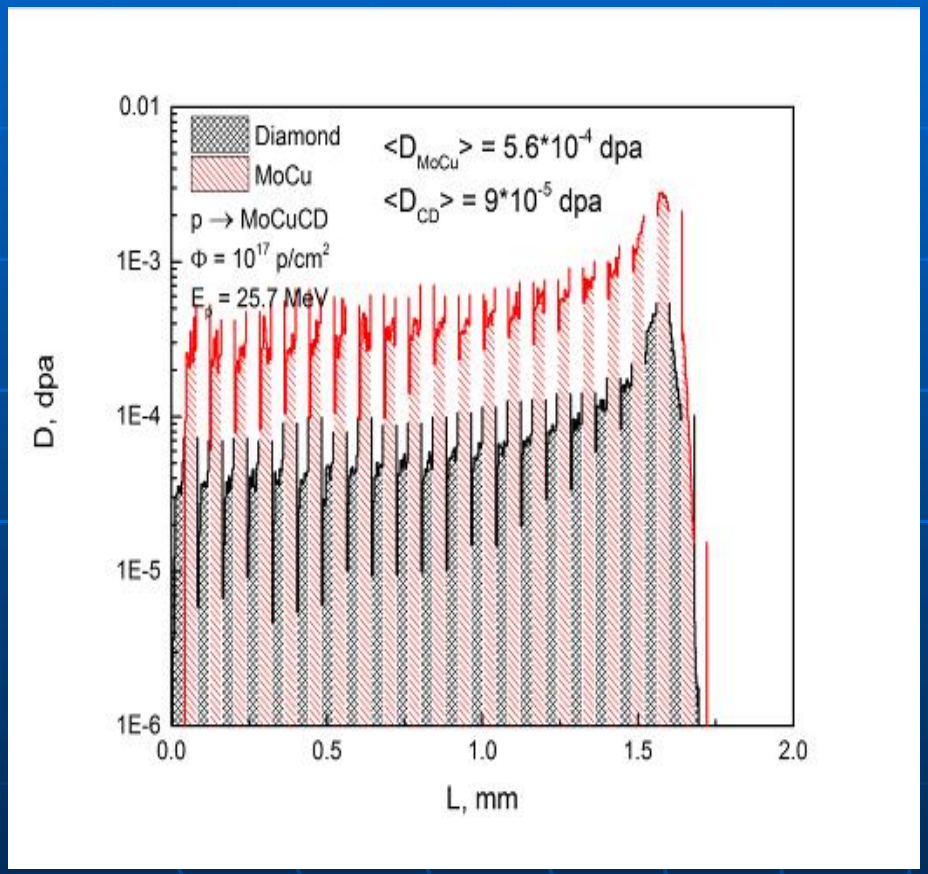
Simulation of radiation damage in Mo-Cu-CD materials taking into account water-cooling circuit.



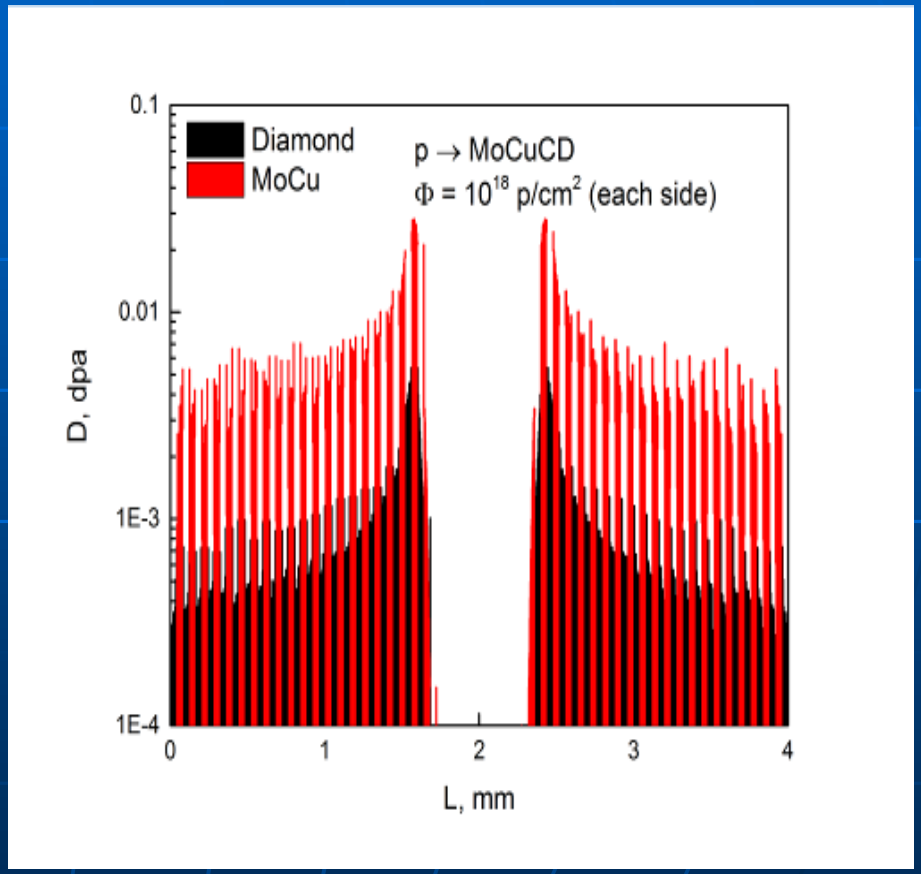
Energy spectrum of 32 MeV protons after passing through 100 um of aluminium and 3 mm of water.



Radiation damage profiles for proton energy 25.7 MeV in the layered structure of Mo-Cu-CD material for a dose up to 10^{17} p/cm^2 (a) and for a dose up to 10^{18} p/cm^2 for both sides of the sample irradiation (b).

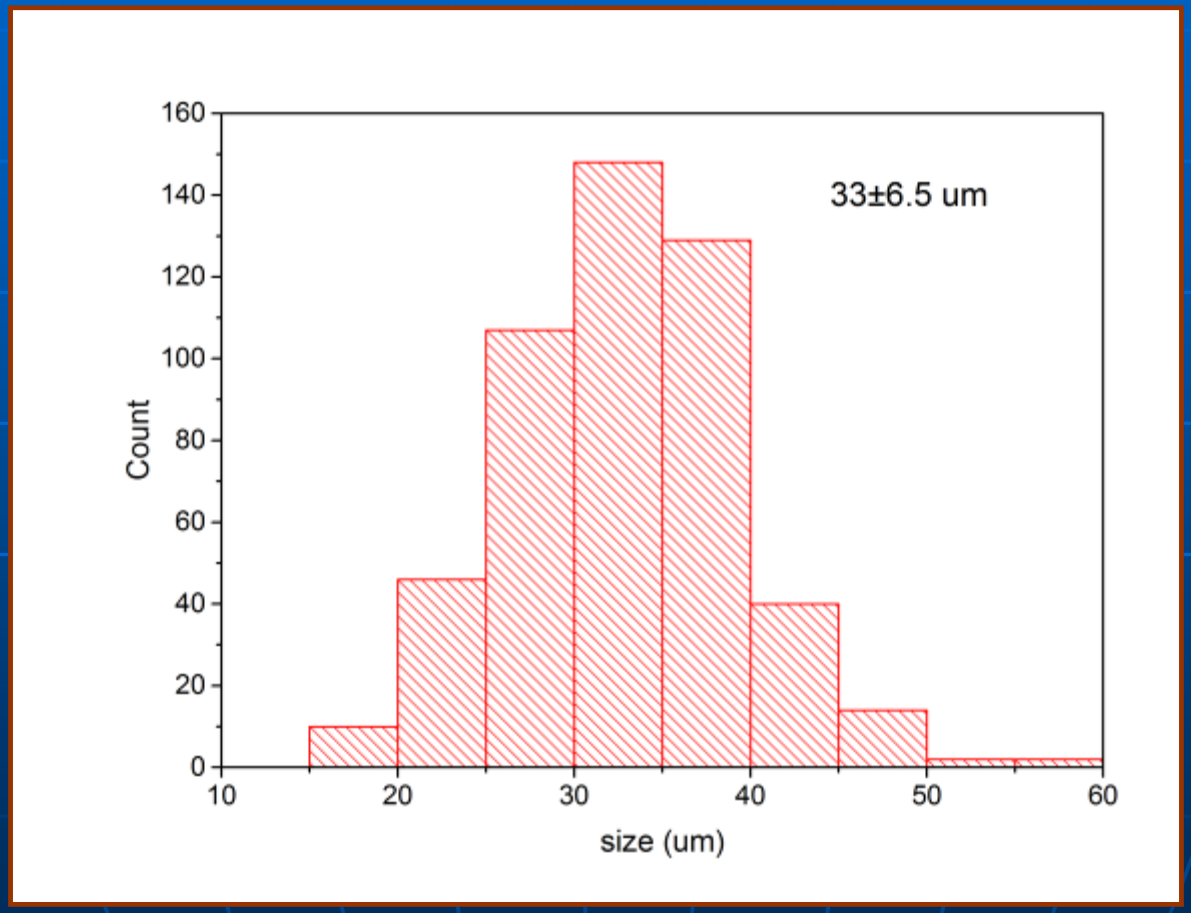


(a)

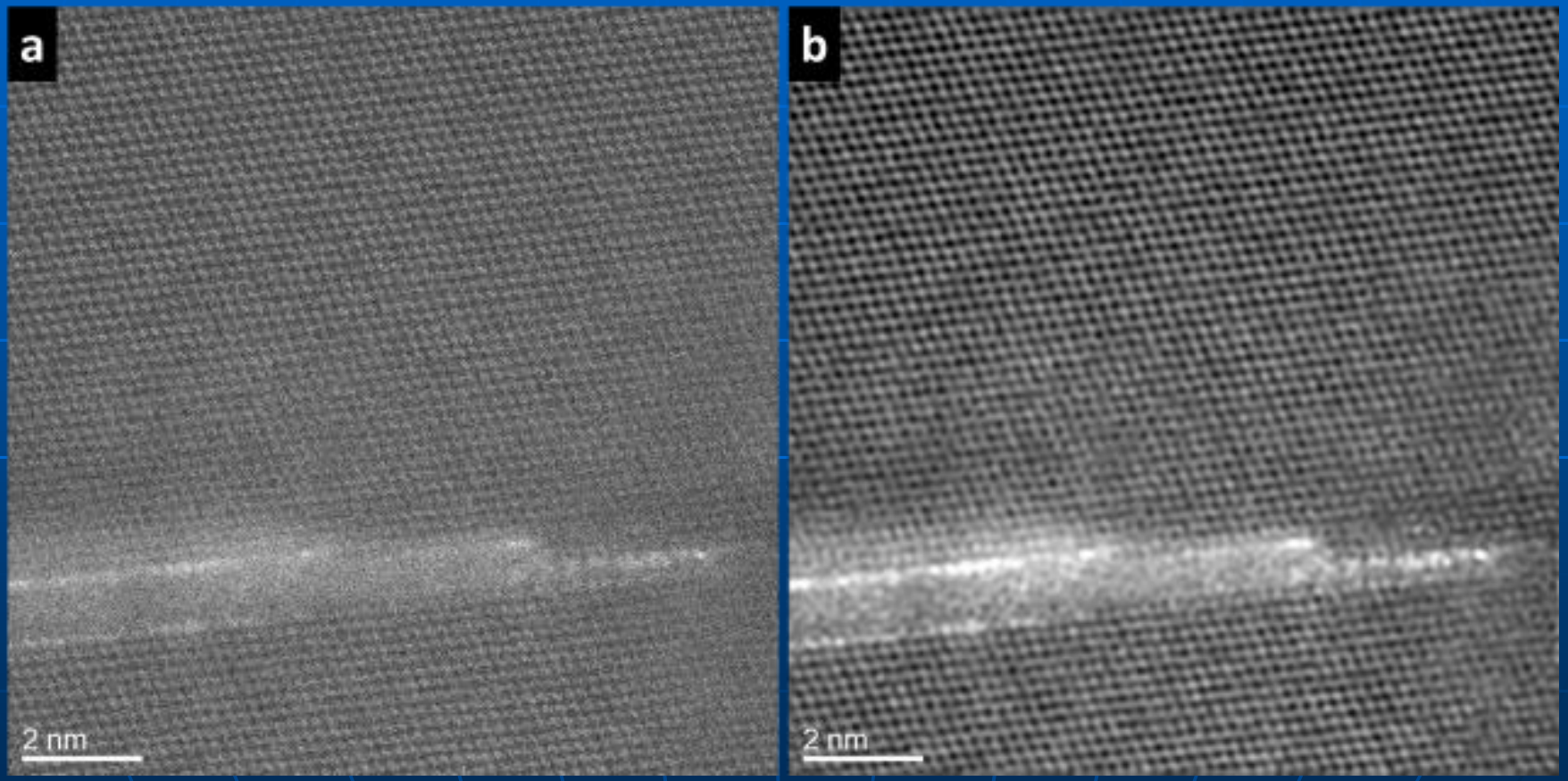


(b)

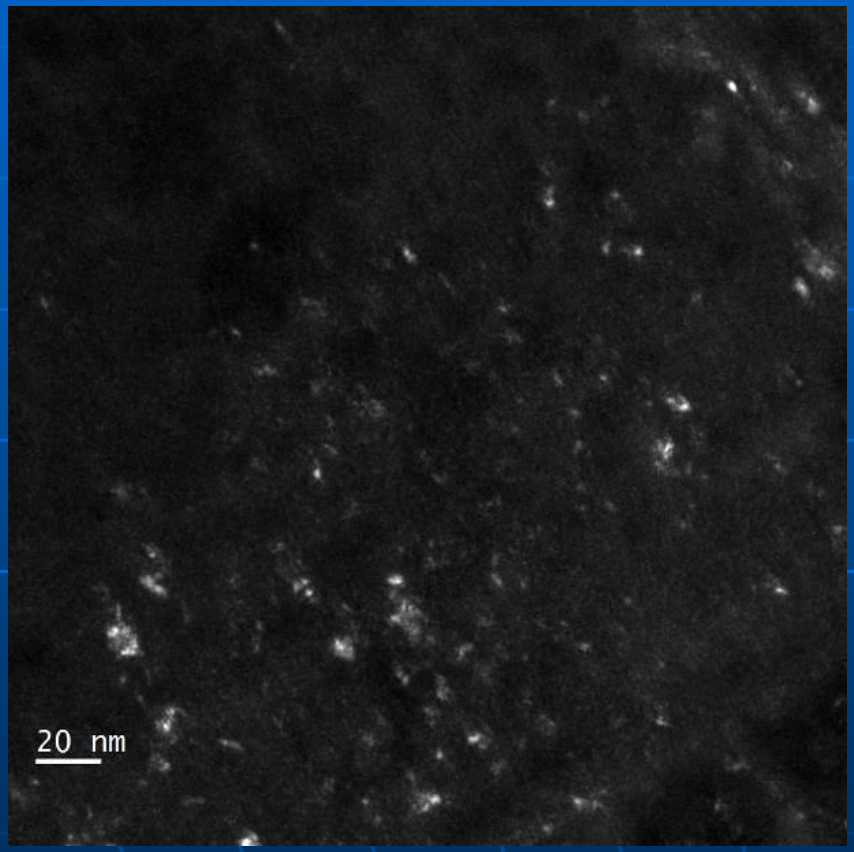
Diamond size distribution in Mo-Cu-CD material



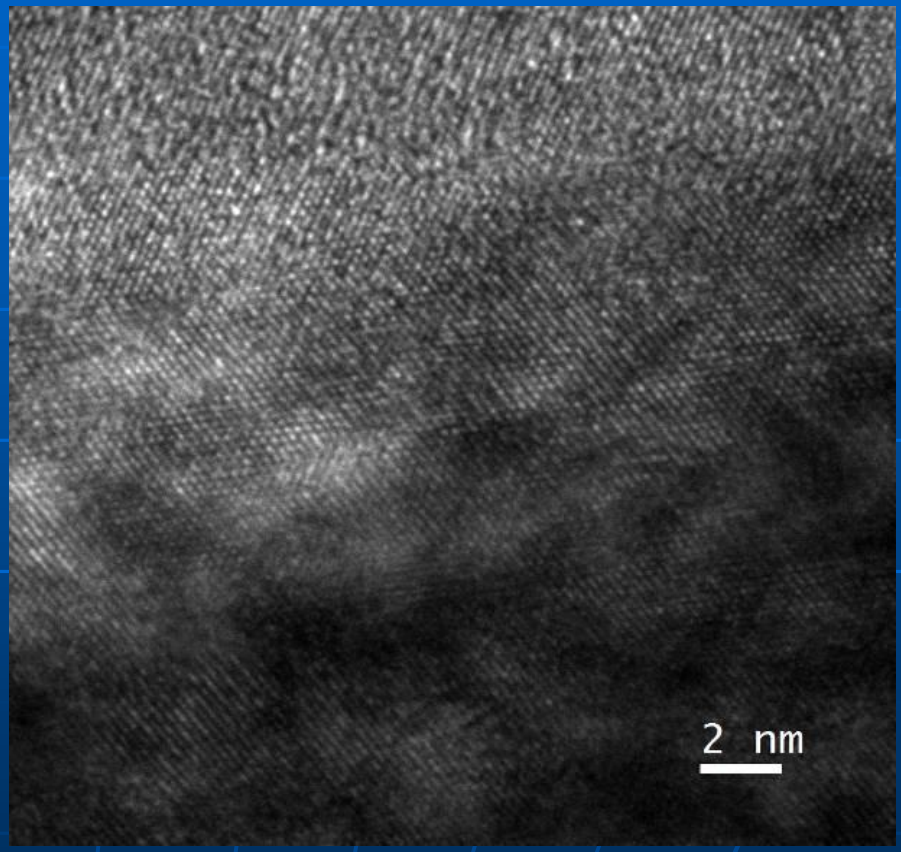
a) HR HAADFSTEM image of the crack in Mo.
b) The same image after the filtering.



Investigations of Mo-Cu-D microstructure changes after fast proton irradiation with the energy up to 30 MeV on the NRC-KI cyclotron at the temperature (T=100°C) and at the proton dose $\Phi_1 = 10^{17}$ p/cm^2 and at the radiation damage level (D=3*10^{-3} dpa).

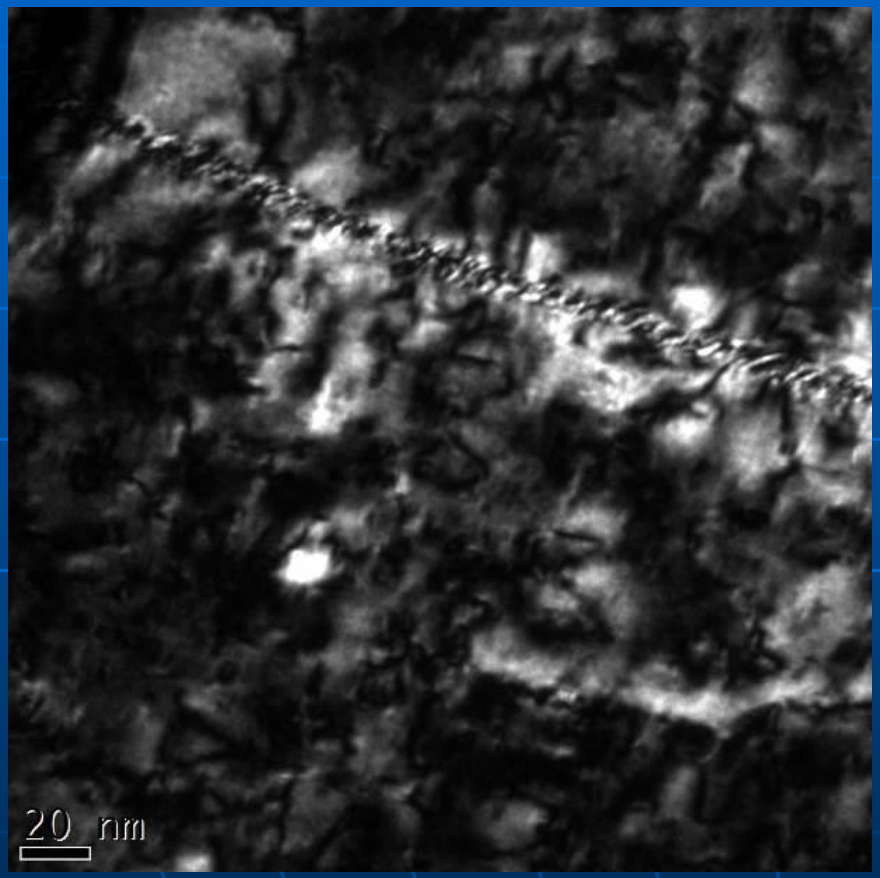


Weak Beam Dark Field image of dislocation loops on Mo metal matrix.

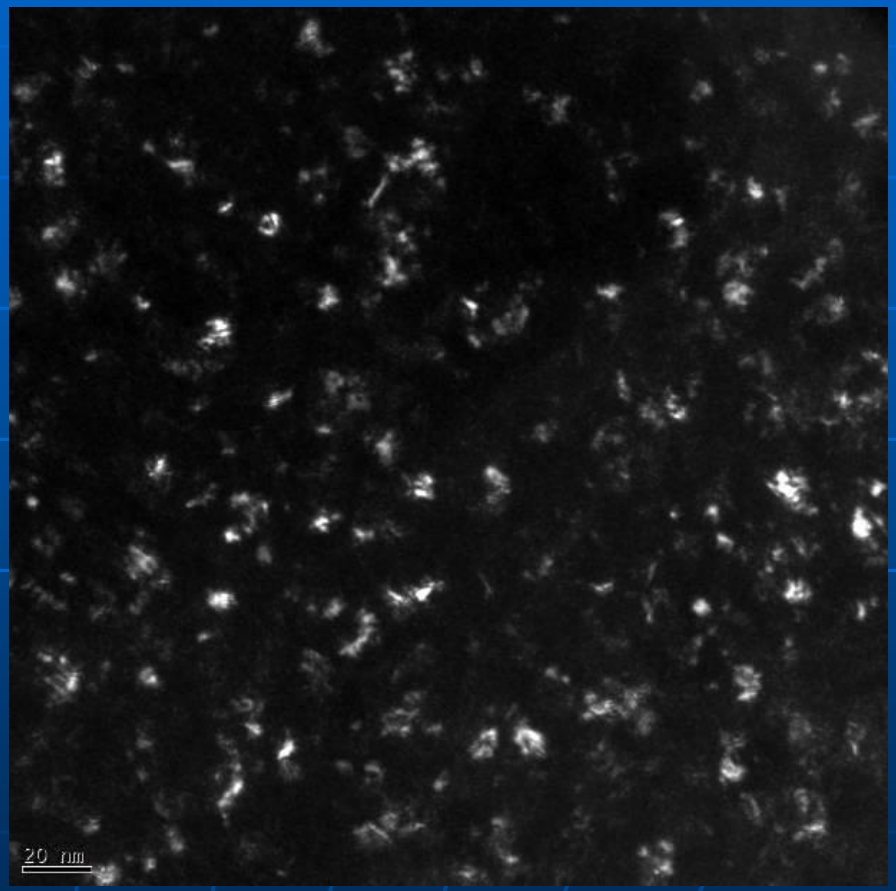


HR TEM bright field image of the Diamond grain after fast proton irradiation.

Investigations of Mo-Cu-D microstructure changes after fast proton irradiation with the energy up to 30 MeV on the NRC-KI cyclotron at the temperature (T=100°C) and at the proton dose $\Phi_1 = 10^{18}$ p/cm^2 and at the radiation damage level (D=3*10^{-2} dpa).

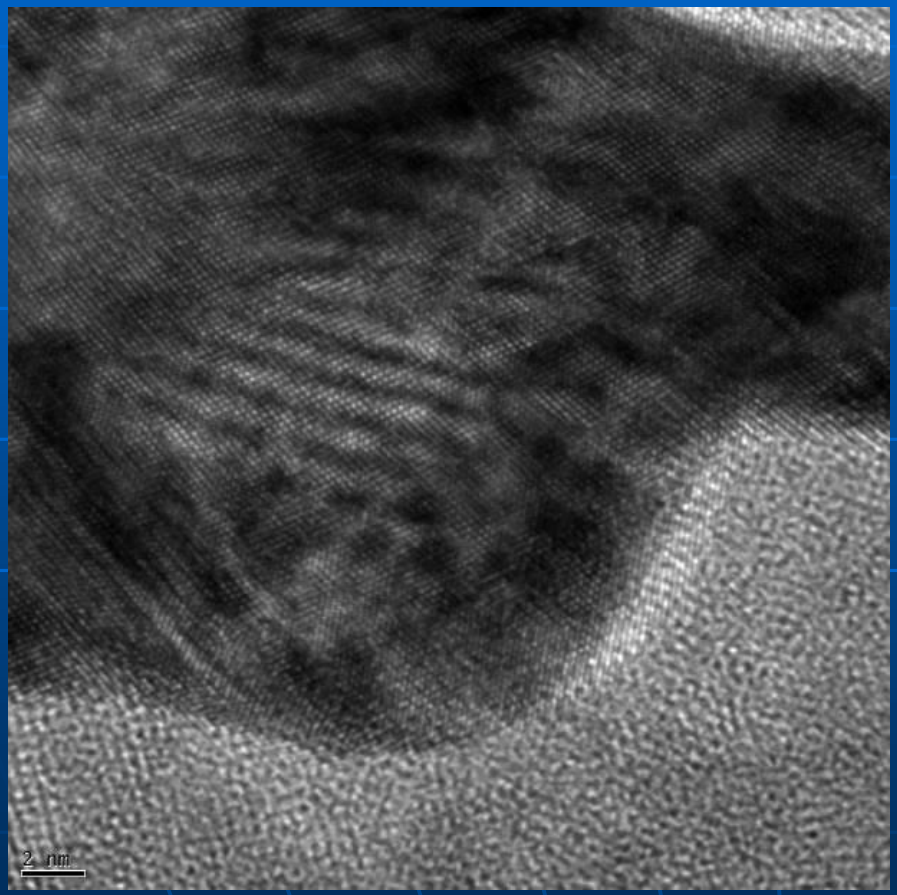


Dark Field image of dislocation loops on Mo metal matrix at 1.5 mm from the surface.

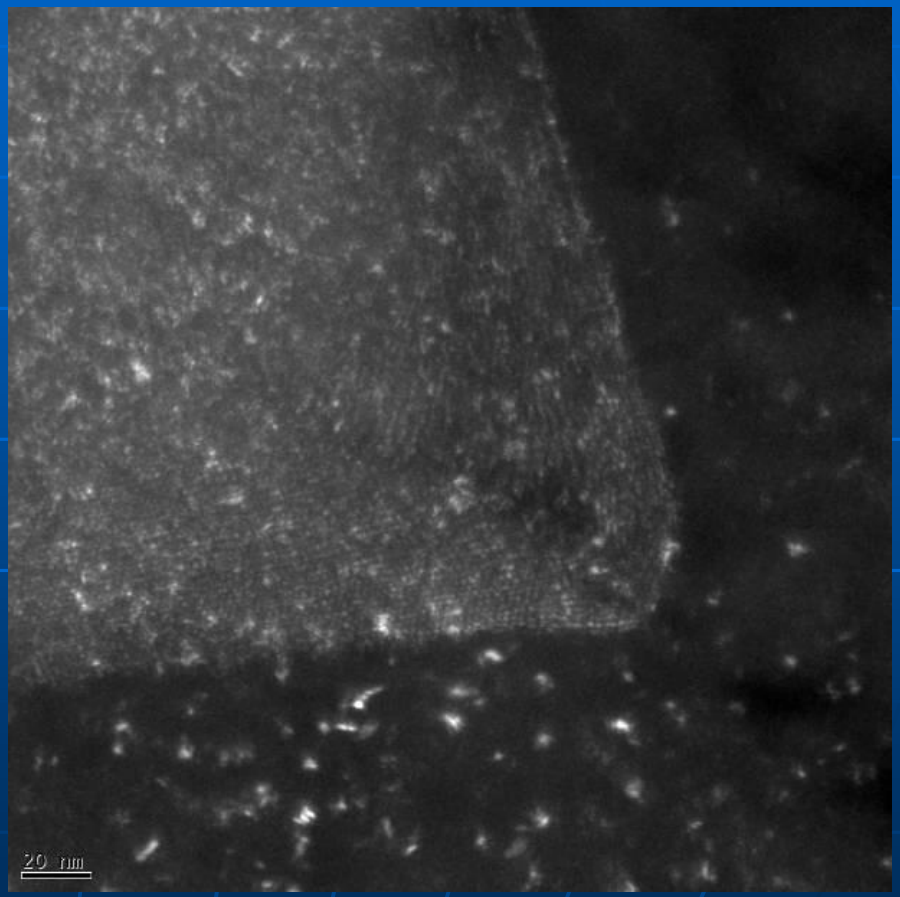


Weak Beam Dark Field image of dislocation loops in Mo metal matrix.

Investigations of Mo-Cu-D microstructure changes after fast proton irradiation with the energy up to 30 MeV on the NRC-KI cyclotron at the temperature (T=100°C) and at the proton dose $\Phi_1 = 10^{18}$ p/cm^2 and at the radiation damage level (D=3*10^{-2} dpa).



HRTEM image of stacking faults in Mo metal matrix (up) at the interface with diamond (down).



Weak Beam Dark Field image of dislocation loops on the interface between Mo metal matrix (down-right) and diamond (up-left).

Conclusions

1. A large number of radiation defects (dislocation loops with sizes 1-2 nm to 10-15 nm) are formed under 30 MeV fast proton irradiation by at depth 1.5 mm from surface of Mo-Cu-Diamond collimator material.

- 2. Dislocation loops are formed in both metallic grains and diamond inclusions.
- Density of dislocation loops is an order in diamond grains than in metallic ones.
- 3. A process of blurring in reciprocal lattice sites can be seen. Disoriented micro blocks of small size (20-30 nm) is observed, which can be a result of direct radiation defect production in diamond or micro strain due to a large number of dislocation loops.
- 4. The total number of radiation defects in both metallic and diamond grains cut from depth 0.5 mm is 2-5 times lower than corresponding sample cut from depth 1.5 mm. It's a result of diminishing of radiation dose.
- 5. There were no defects on the grain boundaries between metal and diamond indicating a beginning of destruction of interboundaries due to irradiation. A contact of different grains in composite material after irradiation corresponds to the qualitative contact in unirradiated material as TEM investigation shows.
- 6. A large number of distortions (mainly stacking defects) in metallic lattice were found in regions of metallic grains adjacent to the diamond grains. Presence of stacking defect could be a result of micro strain due to the different volumetric expansion of metal and diamond produced by radiation defects under proton irradiation.

Thank you very much to S.Redaelli and A.Bertarelli for many scientific discussions and all for your attention !

Temperature dependence of thermal expansion coefficient in Mo-Cu-Diamond collimator materials

T, oC	100	200	300	400	500
	$\alpha_{исх}$, 10^{-6} 1/K				
Cu-Diamond	9,0	9,7	10,4	**12,4**	12,2
Mo-Cu-Diamond (Sample P1)	7,5	8,1	8,4	**8,3**	7,4
MoCu-Diamond (Sample P2)	8,1	8,2	8,5	**8,1**	7,7
Mo-Cu-Diamond (Sample P3)	8,3	8,5	9,0	**8,7**	7,6

Temperature dependence of thermal expansion coefficient in Mo-Cu- Diamond collimator materials

T, oC	20	50	100	120
Unirradiated	α, 10^{-6} 1/K			
Sample 1			7,5	
Sample 2			8,1	
Sample 3			8,3	
Irradiated	α, 10^{-6} 1/K			
Sample 1	6,02	6,36	7,70	8,23
Sample 2	4,40	7,33	7,49	7,26
Sample 3	7,74	8,72	8,11	7,36

Measurements of electrical resistivity in Mo-Diamond collimator materials before and after 30 MeV proton beam irradiation on NRC KI cyclotron up to dose 10E17p/cm2

Sample	I		II		III	
Condition	Initial	Irradiated	Initial	Irradiated	Initial	Irradiated
ρ, 10^{-6}, Om·m	110	142	85	113	91	137
$(\rho_{irr} - \rho_{in})/\rho_{in}$, %	29		33		51	

The increasing of ER in Mo-D after proton irradiation is large

Measurements of electrical resistivity in Cu-Diamond collimator materials before and after 30 MeV proton beam irradiation on NRC KI cyclotron up to dose 10E17p/cm2

Sample	I		II		III	
Condition	Initial	Irradiated	Initial	Irradiated	Initial	Irradiated
ρ, 10^{-6}, Om·m	9,0	8,8	10,2	10,2	10,1	10,5
$(\rho_{irr} - \rho_{in})/\rho_{in}$, %	-2		0		4	

The increasing of ER in Cu-D after proton irradiation is negligible

Comparison of temperature dependencies of thermal expansion coefficients in Mo-Diamond and Cu-Diamond collimator materials

T, °C	100	200	300	400	500
Cu – Diamond, $\alpha_{исх}$, 1/K, 10^{-6} 1/K	9,0	9,7	10,4	12,4	12,2
Mo-Diamond, $\alpha_{исх}$, 1/K, 10^{-6} 1/K	7,5	8,1	8,4	8,3	7,4