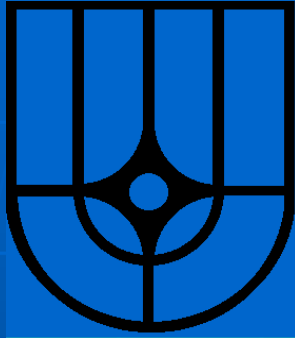
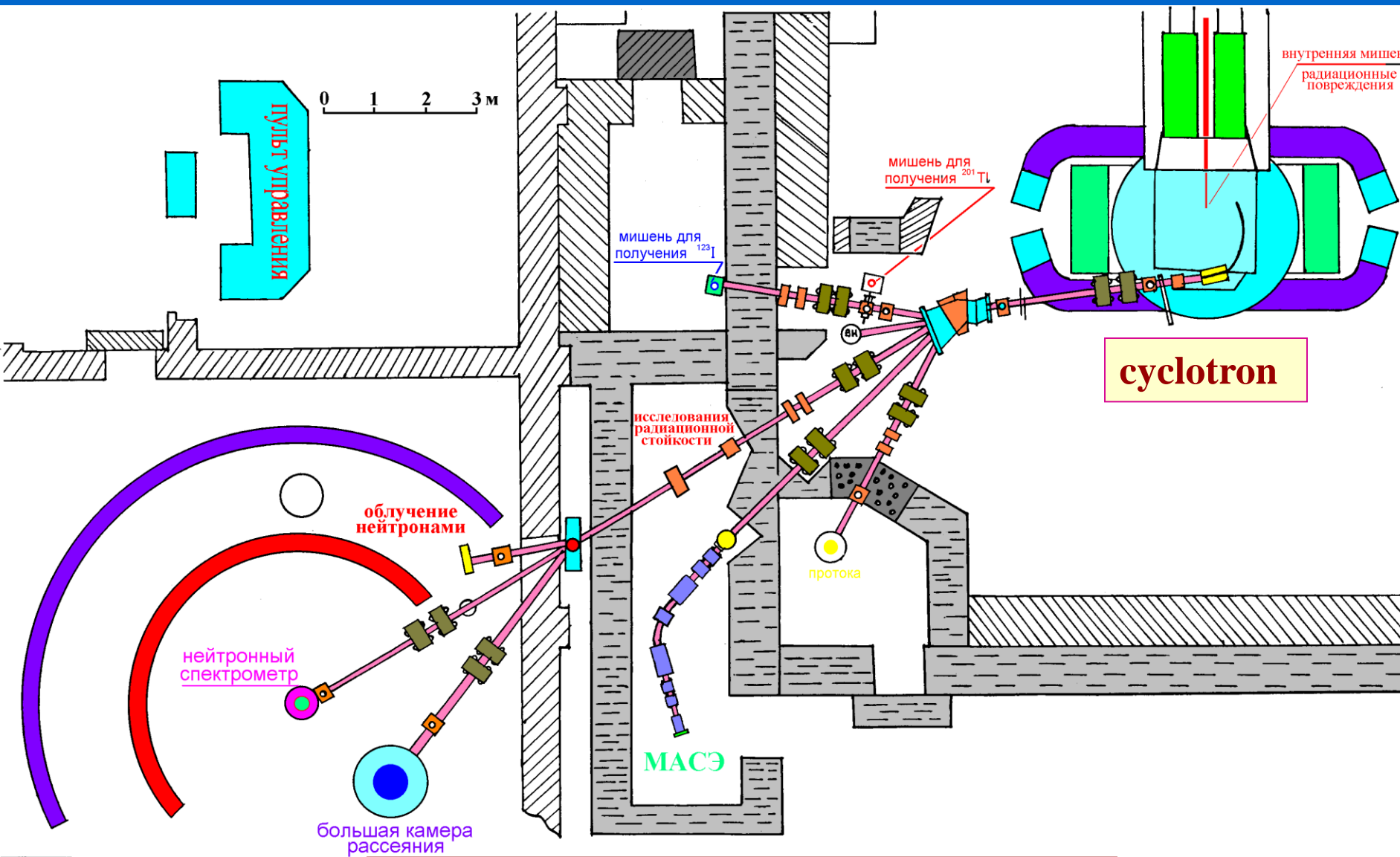


National Research Centre” Kurchatov Institute”



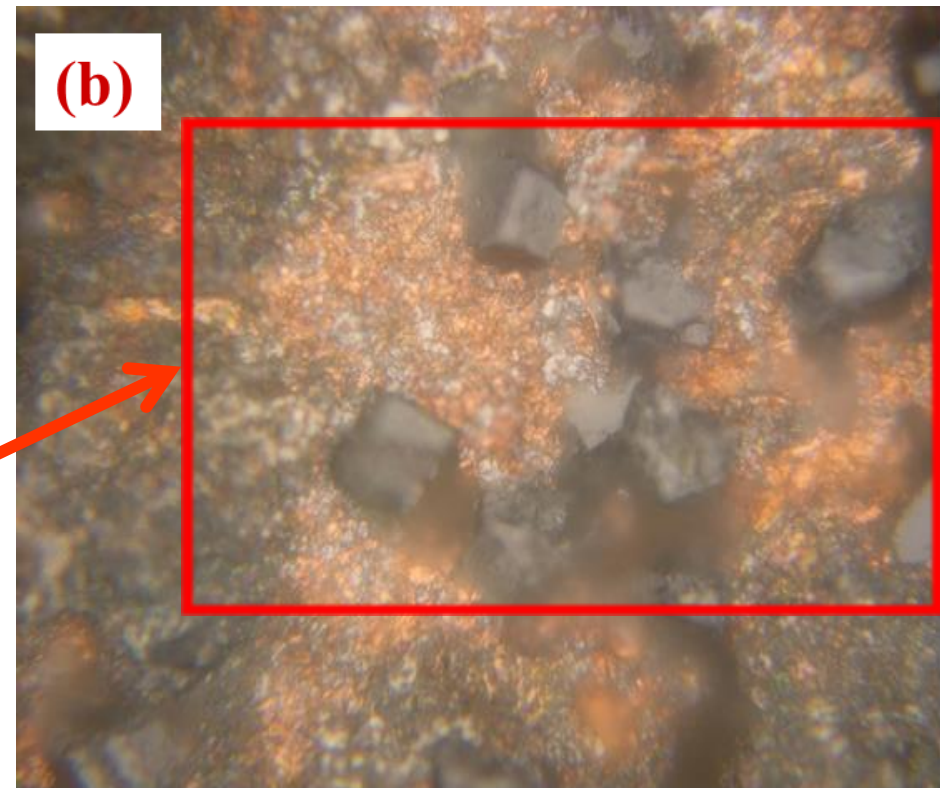
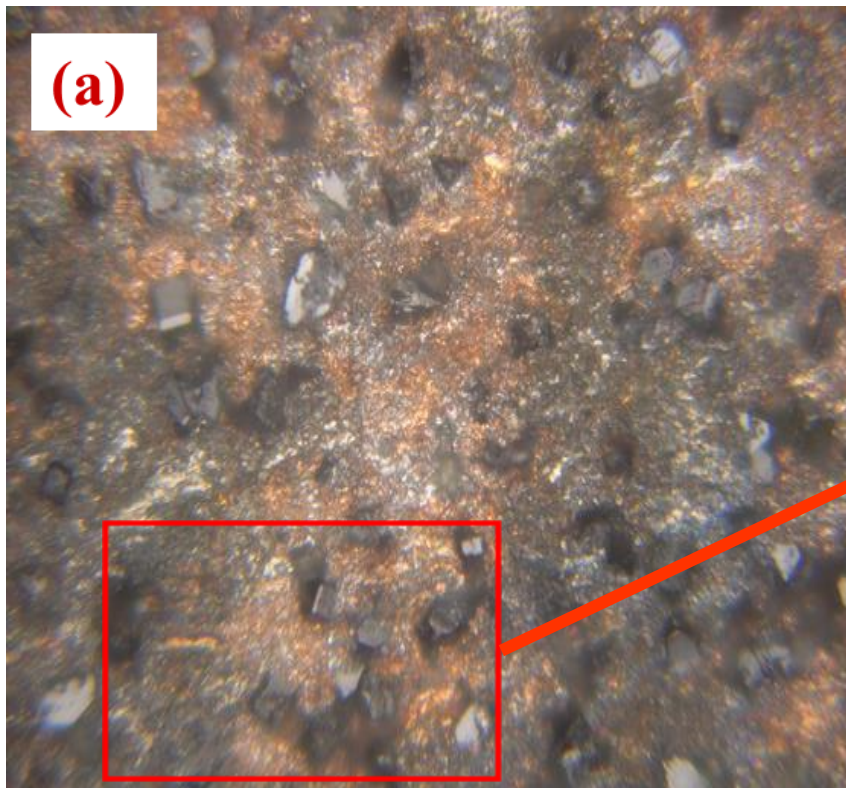
Investigations of fast particle irradiations on the properties of Molybdenum-Copper-Diamond collimator materials for the Large Hadron Collider at CERN using the NRC KI cyclotron.

Alexander Ryazanov

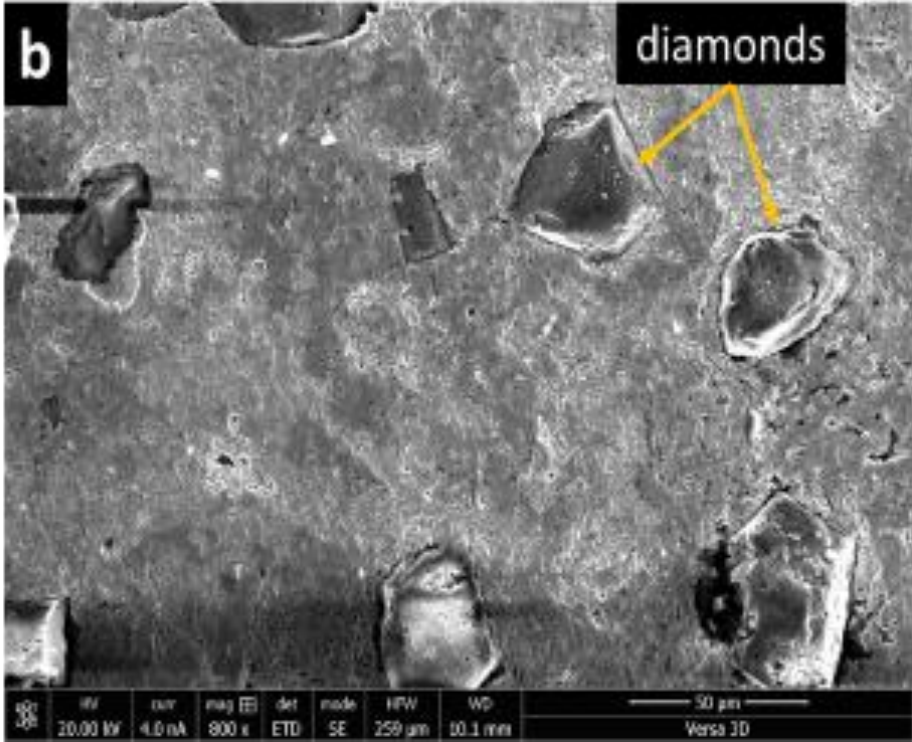
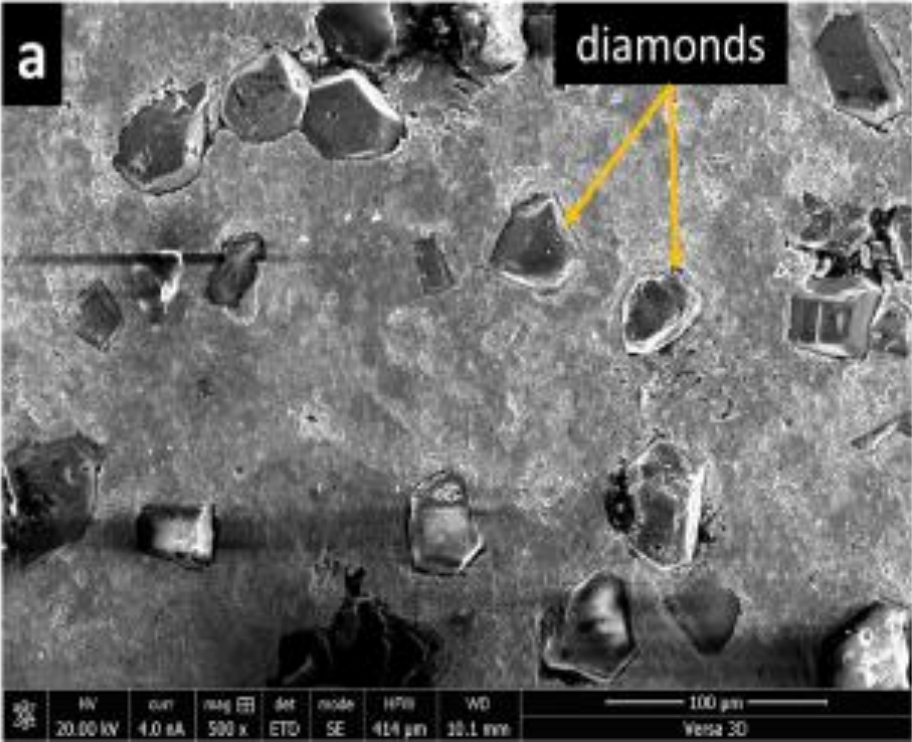


System of cyclotron transportation

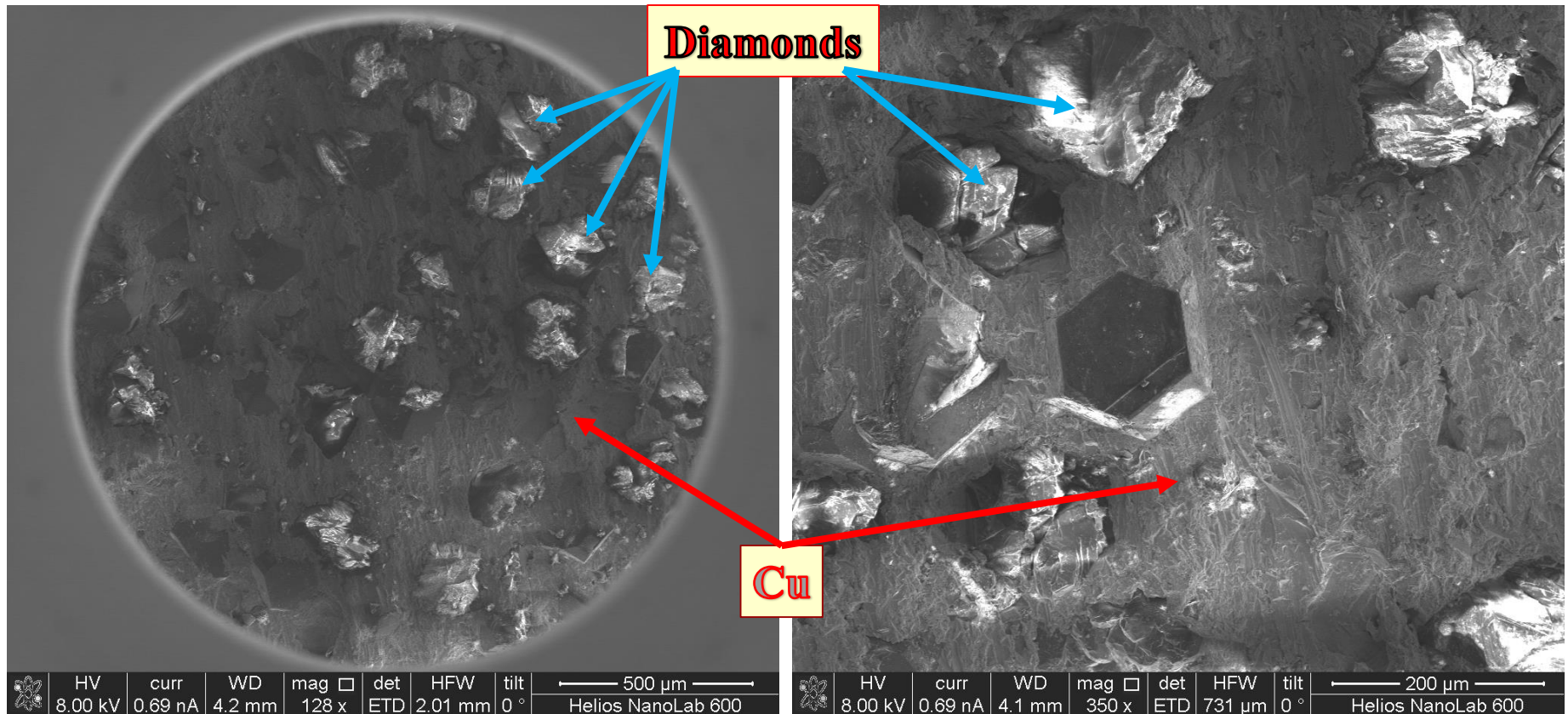
Microstructure of Mo – Cu - Diamond sample surface under different magnification (-200 (a) and -500 (b)) made on optical microscope Carl Zeiss Axio Observer D1m



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).

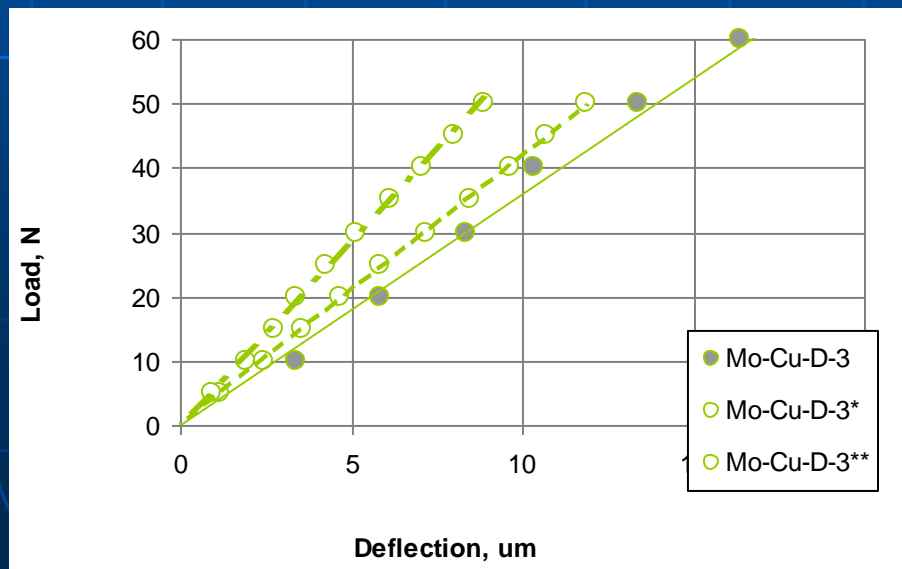
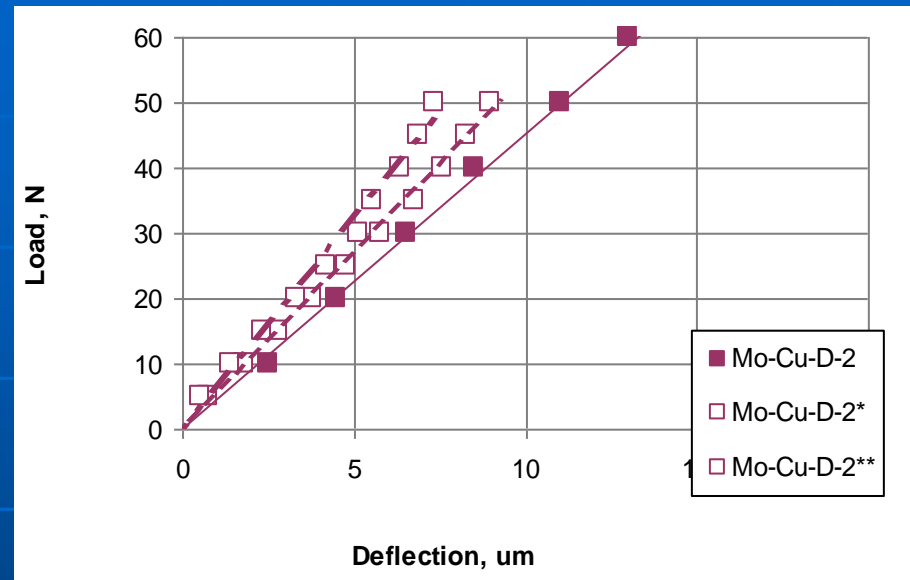
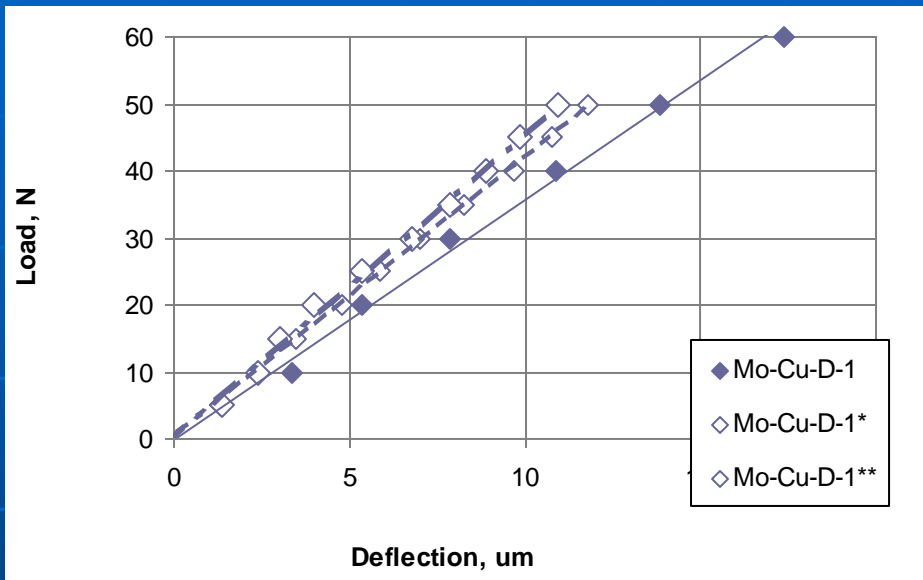


The SEM study and sample preparation

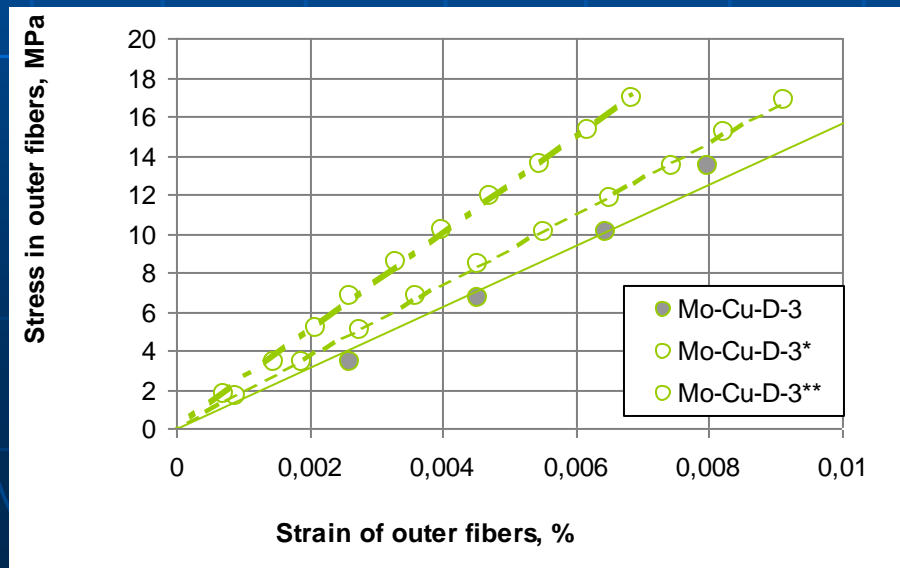
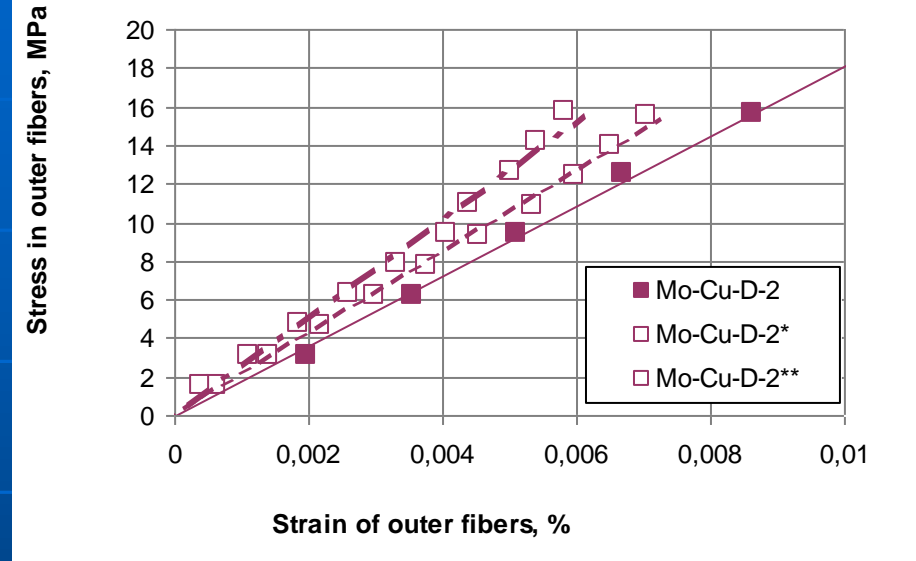
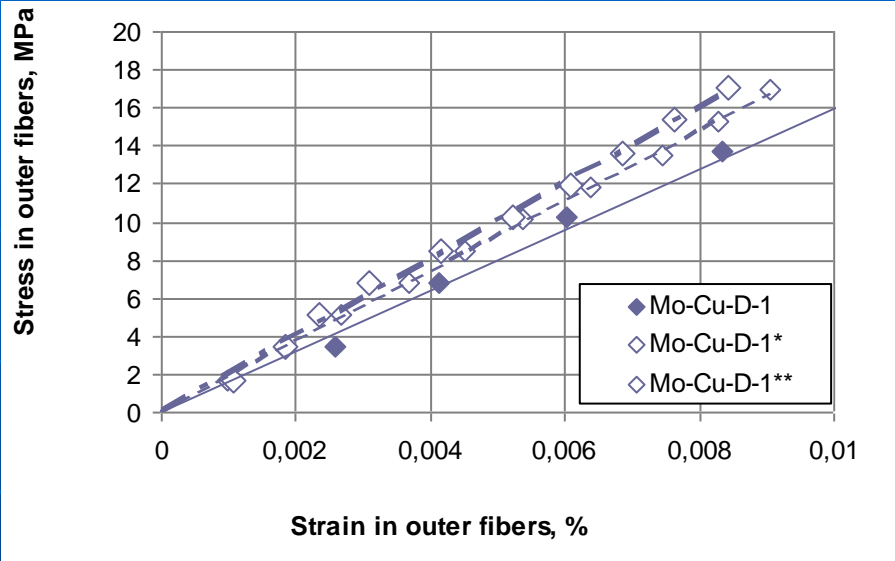


SEM (secondary electrons -SE) images of the Cu-Diamond composite

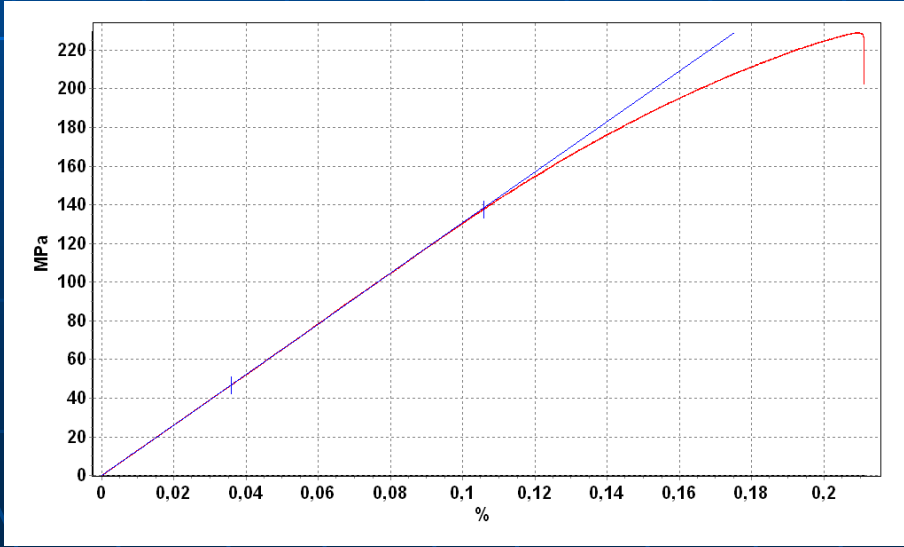
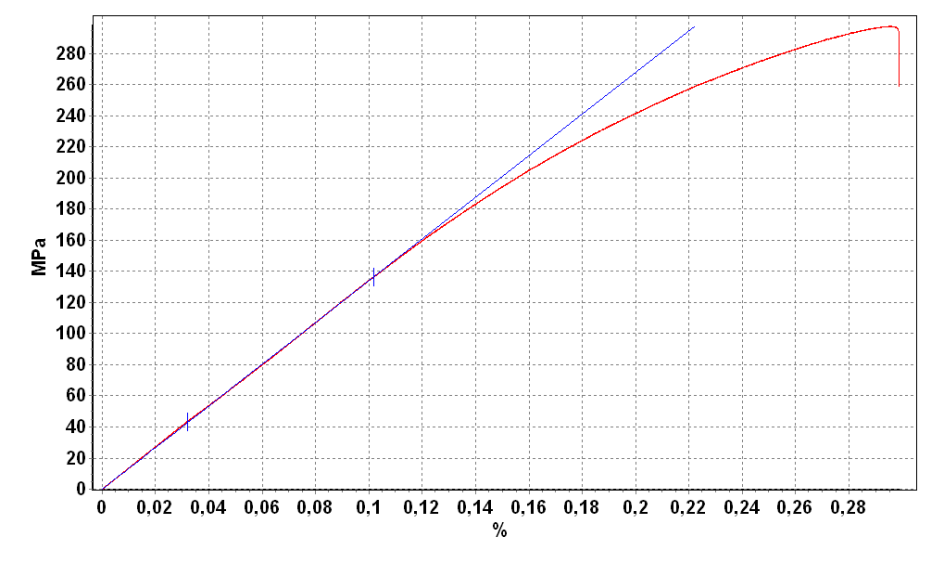
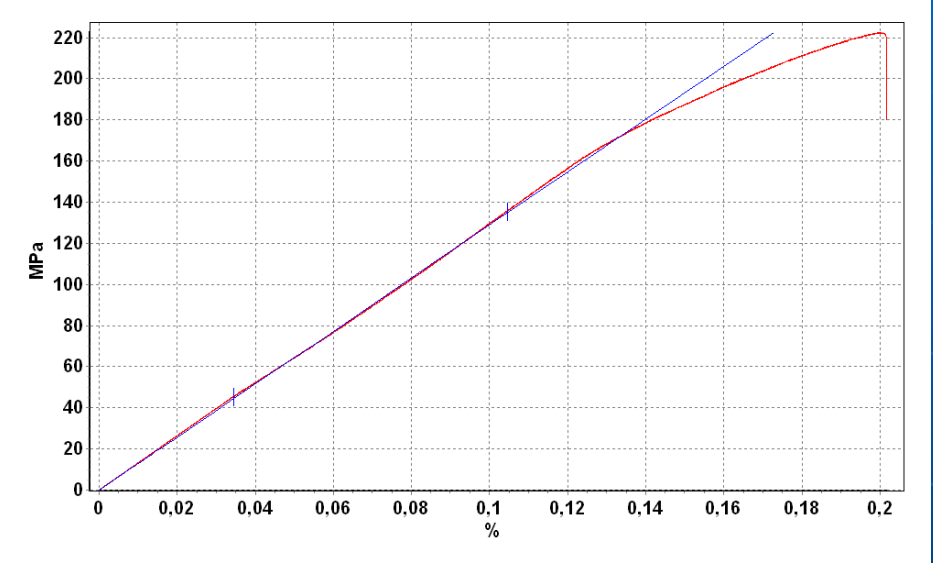
«Molybdenum-Copper Diamond» measurements of elastic modulus (E^N , E^{N*} , E^{N**}) on non-irradiated samples, after 1st irradiation by 30 MeV protons with doses $10E17p/cm^2$ and 2nd irradiation with doses $10E18p/cm^2$



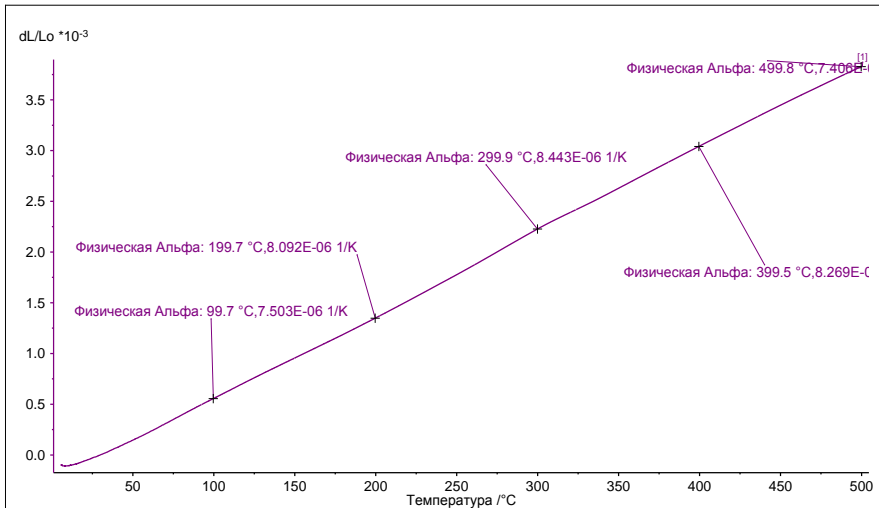
Deformation curves in outer fibers of the samples versus strain in non-irradiated, after 1st irradiation by 30 MeV protons with doses 10E17p/cm² and 2nd irradiation with doses 10E18p/cm²



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



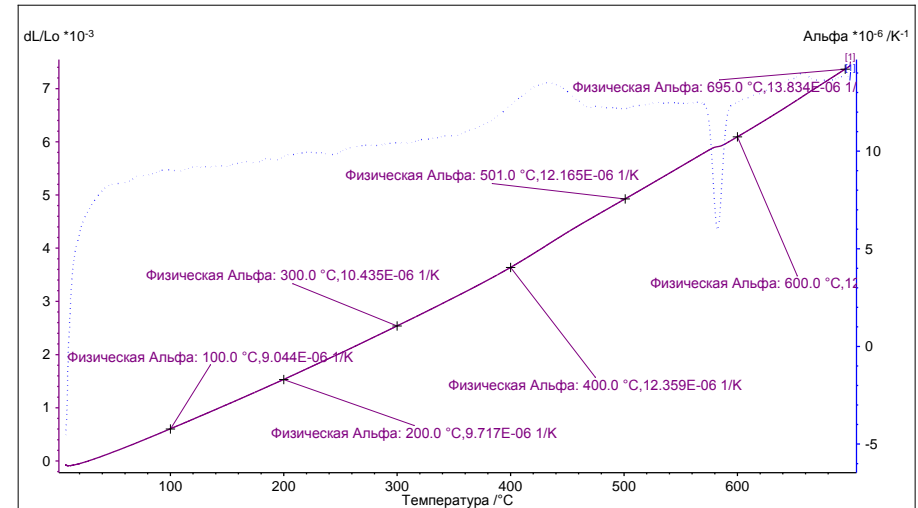
Temperature dependence of the thermal expansion coefficient (CTE) for Mo-Cu-Diamond material (P1, P7 samples) before irradiation



Главное: 2012-10-11 16:54 Пользователь: Администратор

Прибор: NETZSCH DIL 402 C Файл: C:\ngbw\inta\data\9\Рязанов-молибден-алмаз-P1-20-500-He50.dle

Проект: Измерение образца мол-алм	Материал: Мол-алм	Таблица прободержателя: AI203.scl
Дата/время: 11.10.2012 12:46:45	Атмосфера: He50.0	Материал прободержателя: AI203
Лаборатория: ЛМЖ	Файл темп. калиб.: tot_5_AI203_Jump_213 G.dle	Калибровочный файл: Correction_AI203_NE_5_20-700_He-50.dle
Оператор: Кулешов Д.А.	Диапазон: 20.0/5.0(К/мин)/500.0	Таблица стандартной калибровки: AI203ne.scl
Код образца: мол-алм-P1	Сегменты: 1/1	Материал стандартной калибровки: AI203
Образец/загрузить: P1, 15.900 мм / 30.000 сН	Режим/тип измер.: Расширение/Образец + Коррекция	Корр. Диап. измер.: 810/500 мм

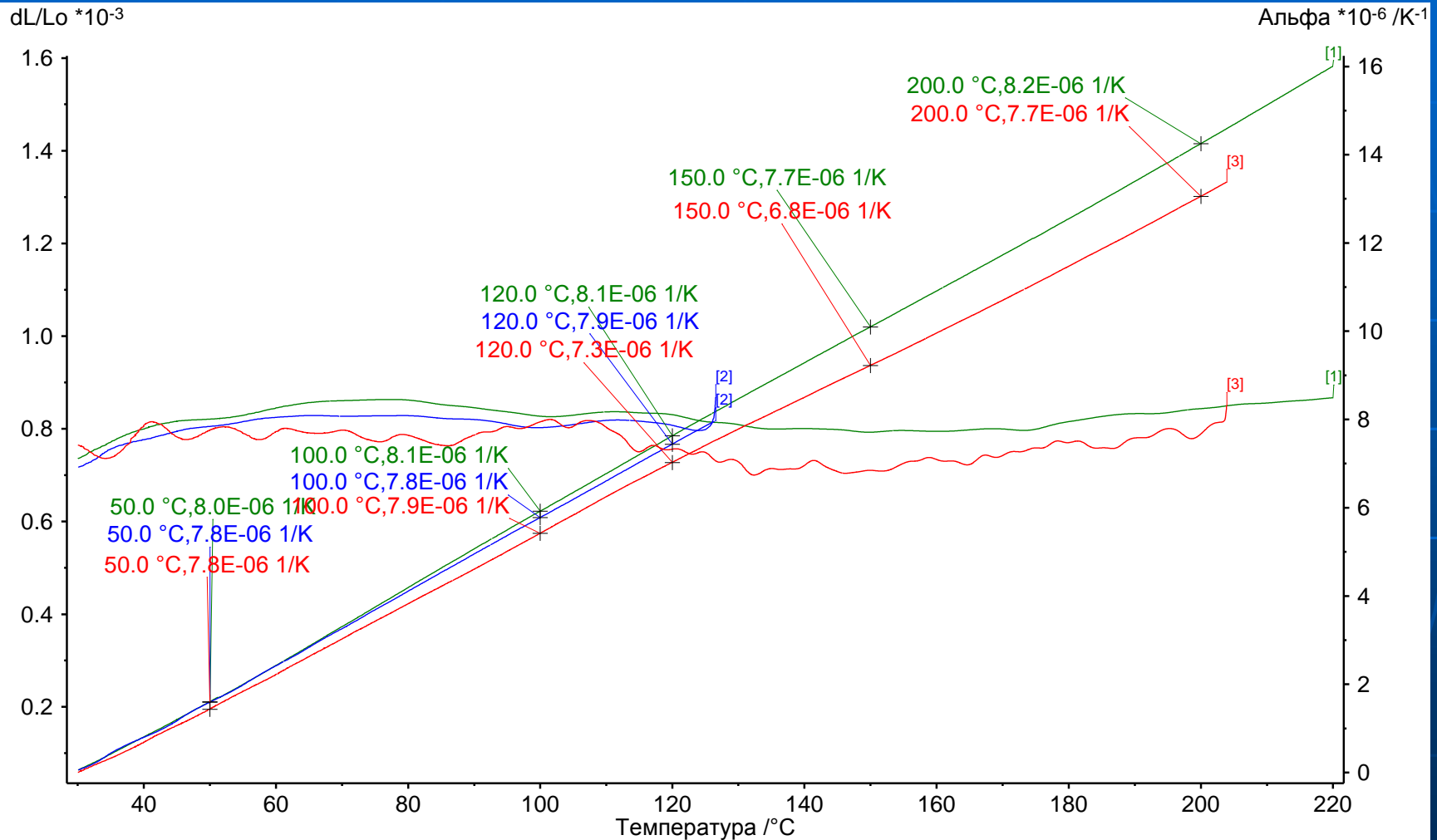


Главное: 2011-06-22 17:45 Пользователь: Администратор

Прибор: NETZSCH DIL 402 C Файл: C:\ngbw\inta\data\9\Рязанов_A-M_P7_исх_20-700.dle

Проект: Измерение исх.	Материал: AI203 NE	Таблица прободержателя: AI203.scl
Дата/время: 07.06.2011 11:15:20	Атмосфера: He50.0	Материал прободержателя: AI203
Лаборатория: ЛМЖ	Файл темп. калиб.: tot_5_AI203_Jump_213 G.dle	Калибровочный файл: Correction_AI203_NE_5_20-700_He-5...
Оператор: Чунов	Диапазон: 20.0/5.0(К/мин)/700.0	Таблица стандартной калибровки: AI203ne.scl
Код образца: Ряз_A-M_P7-исх	Сегменты: 1/1	Материал стандартной калибровки: AI203
Образец/загрузить: Ряз_A-M_P7_исх_16.000 мм / 30.00...	Режим/тип измер.: Расширение/Образец + Коррек...	Корр. Диап. измер.: 810/500 мм

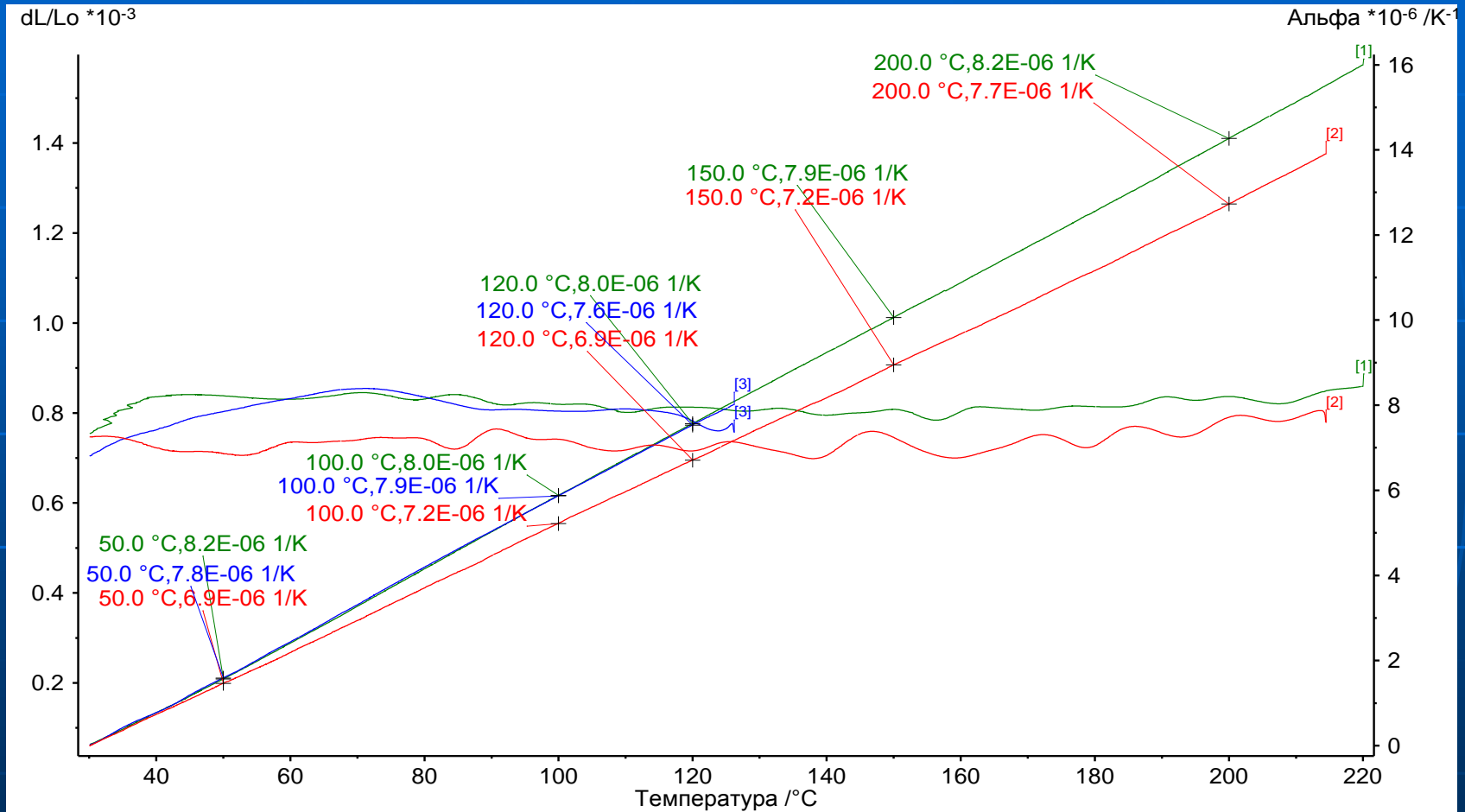
Temperature dependence of the CTE for Mo-Cu-Diamond material (P-1 sample) before irradiation (green line), after first irradiation (blue line), and after second irradiation (red line).



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(К/мин)/5...	He/5...	dL:013, Аль...
[2] DIL 40...	Разан_Мол_Алм-P1-1 обл-120-He.dle	2014-04...	Ряз_Мол_Ал_Р1_1обл	Ряз_Мол_Алм_Р1-1обл	16...	1...	20.0/5.0(К/мин)/1...	He/5...	dL:013, Аль...
[3] DIL 40...	Ряз мол-медь-алм P-1 2-обл 20-5К-215-40К-20 Н...	2016-04...	Ряз Мол-медь-алм P-1 2...	Ряз Мол-медь-алм P-1 2...	16...	1...	20.0/5.0(К/мин)/2...	He/5...	dL:213, Аль...

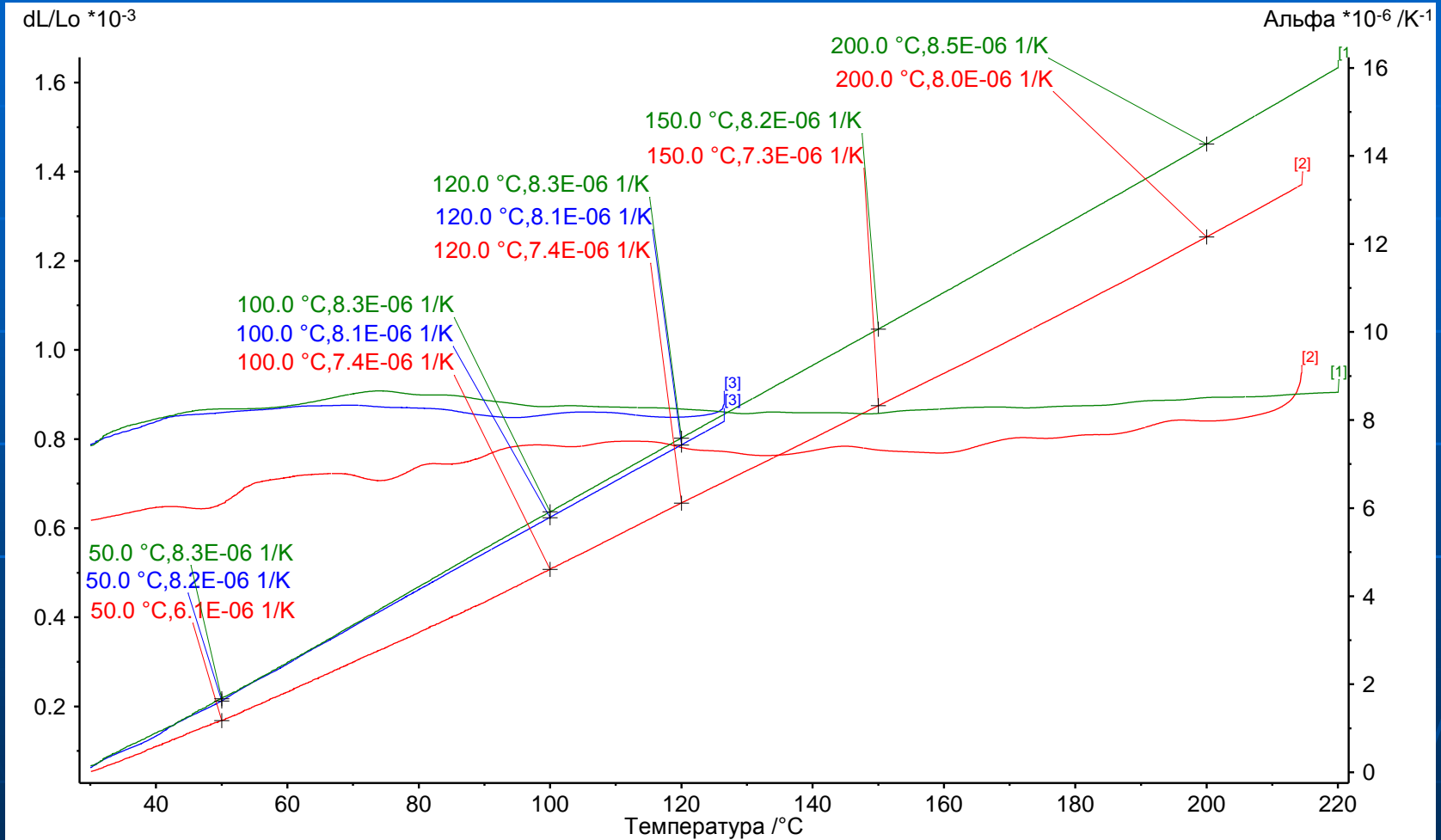
Temperature dependence of the CTE for Mo-Cu-Diamond material (P-2 sample) before irradiation (green line), after first irradiation (blue line), and after second irradiation (red line).



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

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[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 обл20-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

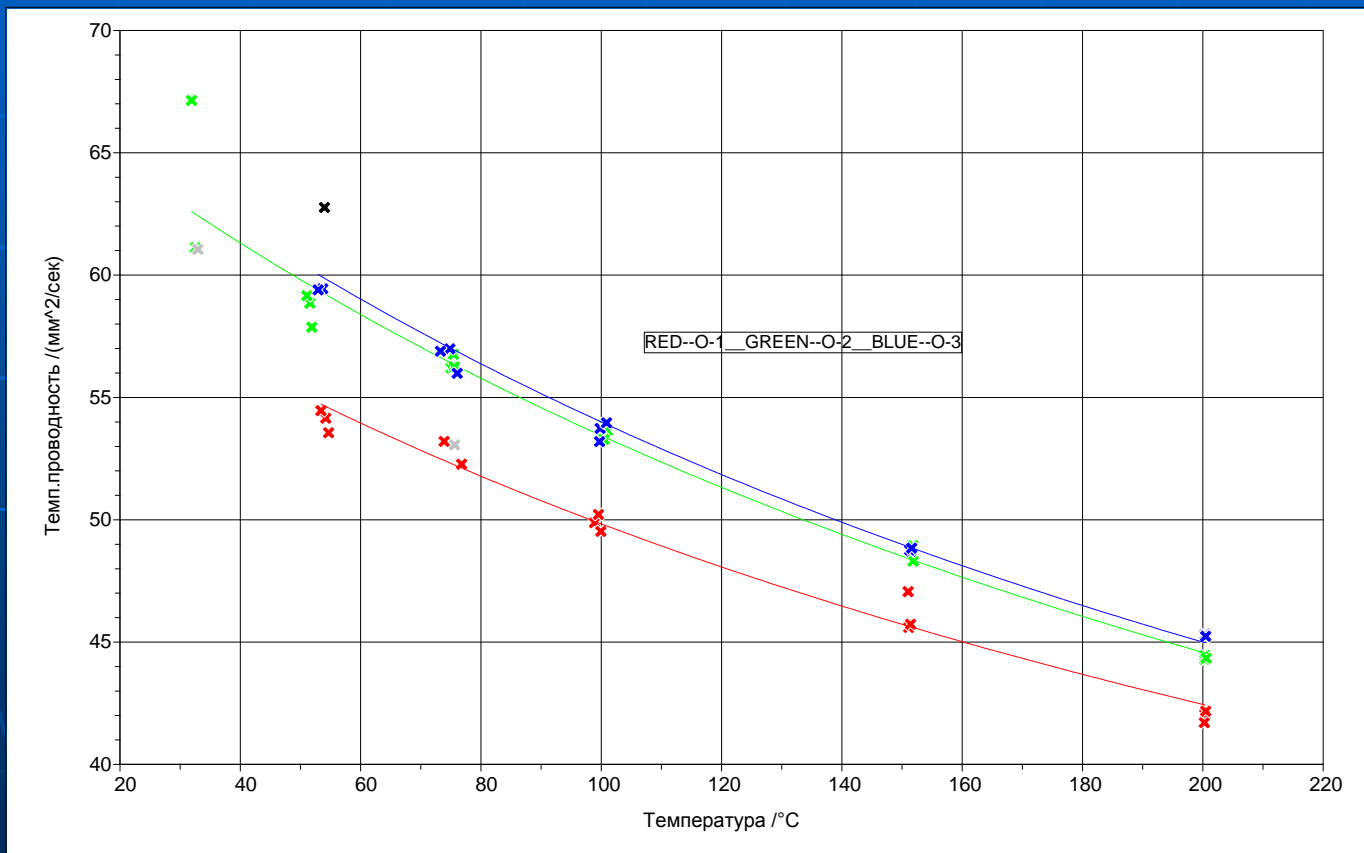
[#]	Приб...	Файл	Дата	Код образца	Образец	Дл...	С..	Диапазон	Атм...	Корр.
[1]	DIL 40...	Рязан-Р3-Мол-медь-алм_20-500_5_He-50.dle	2013-03...	Рязан-Р3-Мол-медь-алмаз...	Рязан-Р3-Мол-медь-алмаз...	16...	1...	20.0/5.0(К/мин)/5...	He/5...	dL:813, Аль...
[2]	DIL 40...	Ряз_мол-медь-алм_Р-3_2-обл_20-1К-215-40К-20_Н...	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

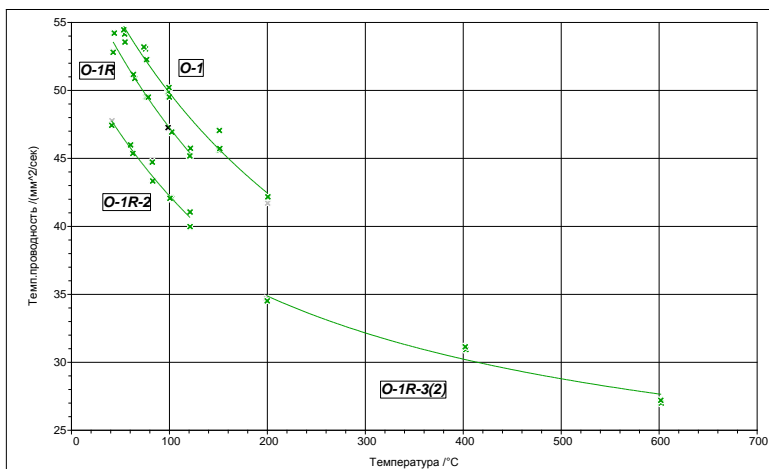
* - values are given in the nominator before irradiation and in the denominator after irradiation.

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

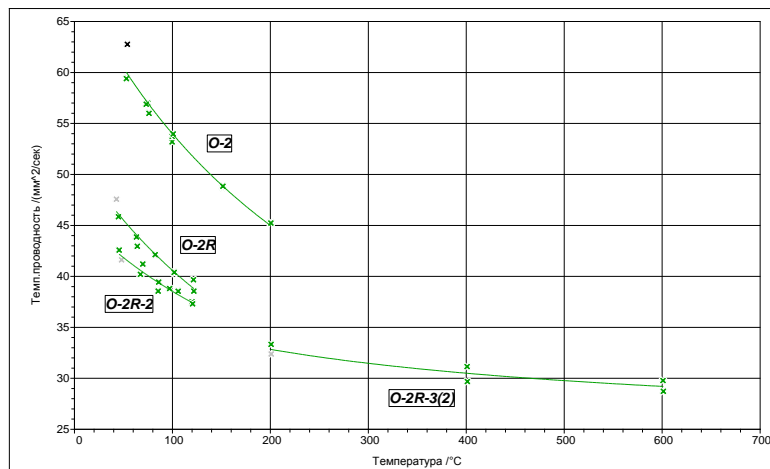


Код образца	Образец	Толщина /мм	Материал	Атмосфера	Дата/Время	Код вычисл.
Mo-C O-3	O-3	4.4900	---	Вакуум	2012-09-14 11:50:26	A+p/l/x-x-0
Mo-C O-2	O-2	4.1850	---	Вакуум	2012-09-10 14:53:05	A+p/l/x-x-0
Mo-C O-1	O-1	4.2250	---	Вакуум	2012-09-07 15:33:50	A+p/l/x-x-0

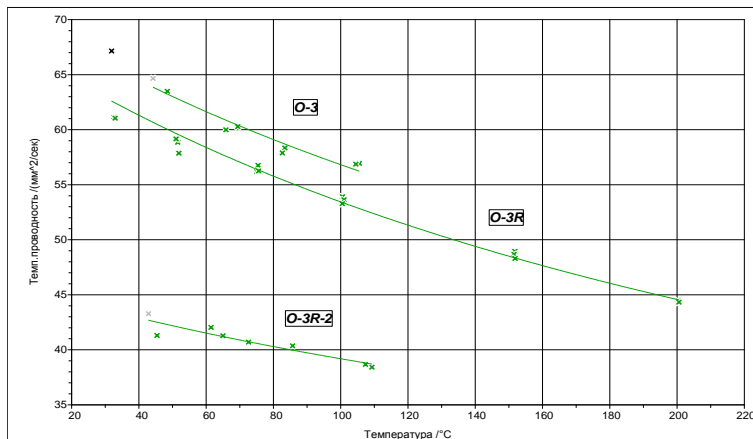
Temperature dependence of thermal conductivity for three Mo-Cu-Diamond samples (O-1, O-2, and O-3) up to 200°C (initial), up to 120°C (after first irradiation (marked as R), after second irradiation (marked as R2)), and up to 600°C (after second irradiation (marked as R3))



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	АнР/Хх-0
Mo-C-1R-2	O-1R-2	4.2250	---	Вакуум	2016-04-15 14:13:01	АнР/Хх-0
Mo-C-O-1R	O-1R	4.2250	---	Вакуум	2014-04-22 14:03:00	АнР/Хх-0
Mo-C-O-1	O-1	4.2250	---	Вакуум	2012-09-07 15:33:50	АнР/Хх-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	АнР/Хх-0
Mo-C-2R-2	O-2R-2	4.1850	---	Вакуум	2016-04-14 14:12:59	АнР/Хх-0
Mo-C-O-2R	O-2R	4.1850	---	Вакуум	2014-04-23 12:33:30	АнР/Хх-0
Mo-C-O-2	O-2	4.1850	---	Вакуум	2012-09-10 14:53:05	АнР/Хх-0

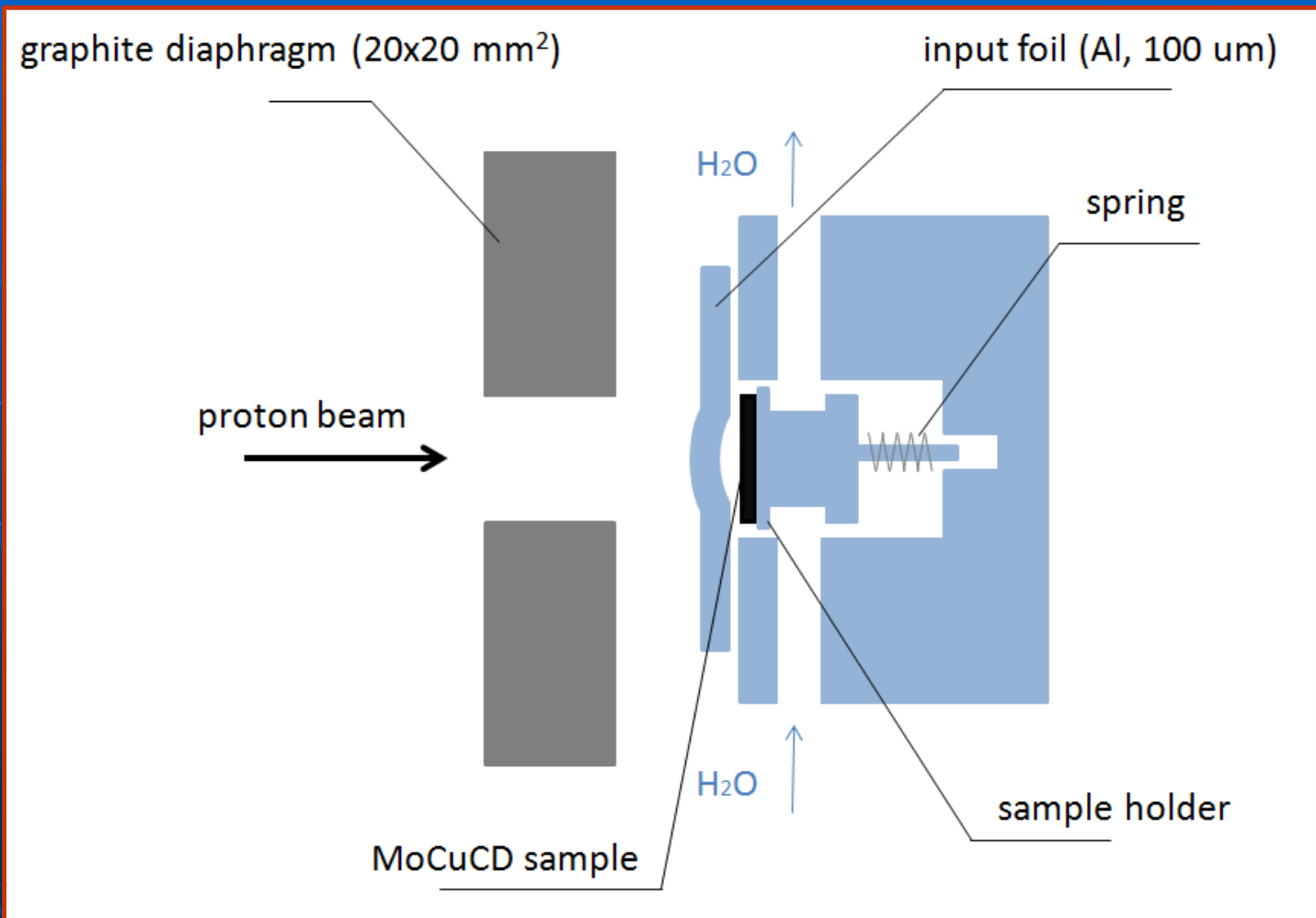


Код образца	Образец	Толщина /мм	Материал	Атмосфера	Дата/Время	Код вычисл.
Mo-3R-2	O-3R-2	4.5000	---	Вакуум	2016-04-13 14:21:53	АнР/Хх-0
Mo-C-3R	O-3R	4.5000	---	Вакуум	2014-04-23 16:29:54	АнР/Хх-0
Mo-C-O-3	O-3	4.4900	---	Вакуум	2012-09-14 11:50:26	АнР/Хх-0

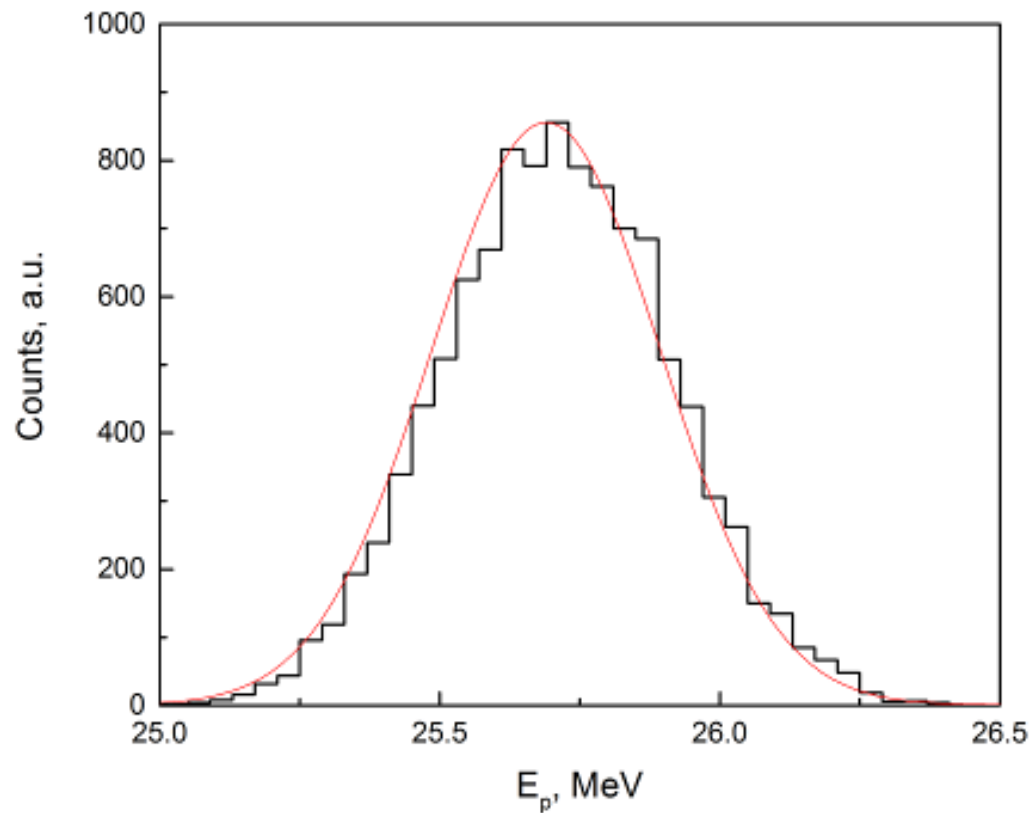
Measurement results of specific resistivity for Mo-Cu-CD collimator materials before and after two irradiations by fast protons with the energy 30 MeV at two doses: $\Phi_1=10e17$ p/cm² and $\Phi_2=10e18$ p/cm²

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
1 st 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
2 nd 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

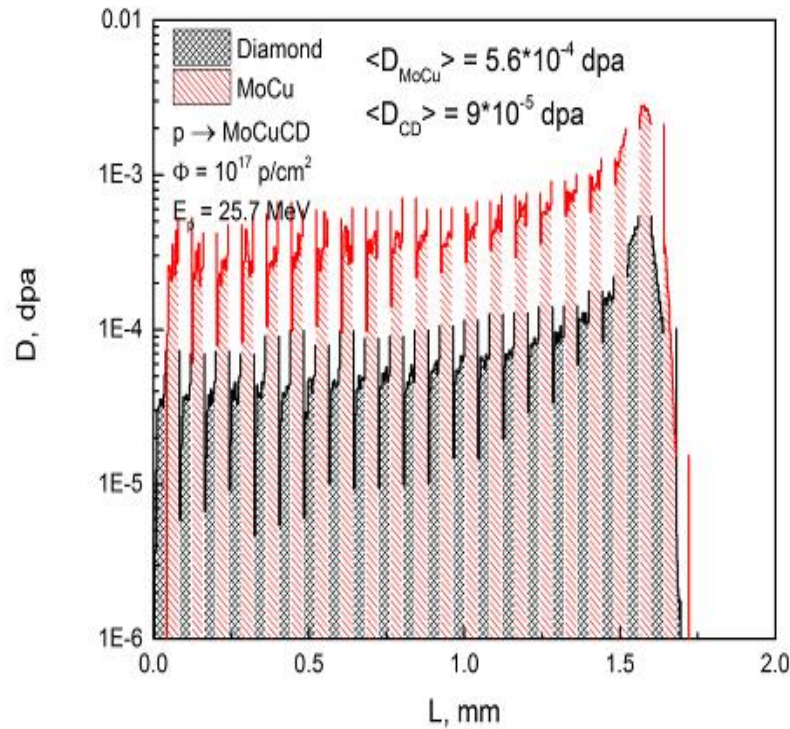
Simulation of radiation damage in Mo-Cu-CD materials taking into account water-cooling circuit.



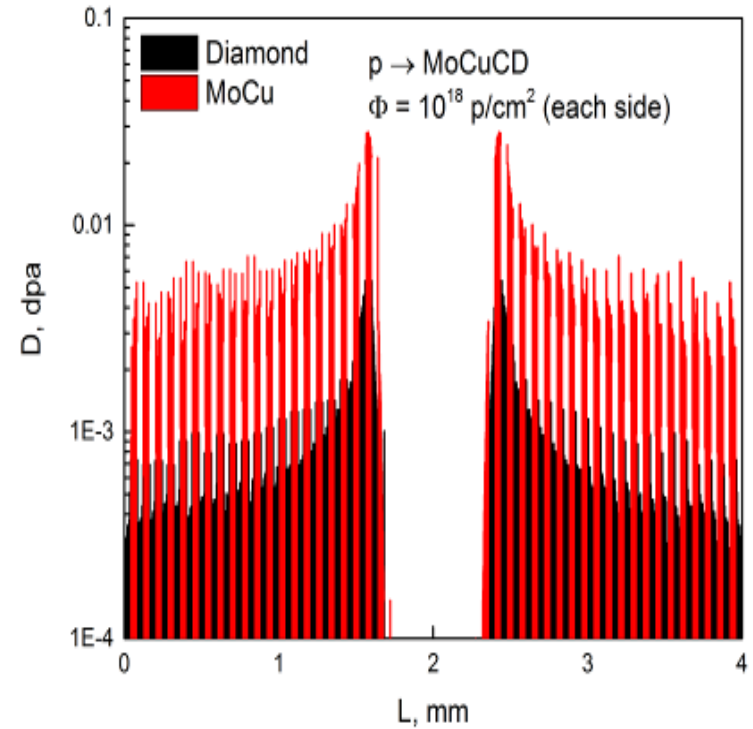
Energy spectrum of 32 MeV protons after passing through 100 μm of aluminium and 3 mm of water.



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/cm² (a) and for a dose up to 10^{18} p/cm² for both sides of the sample irradiation (b).

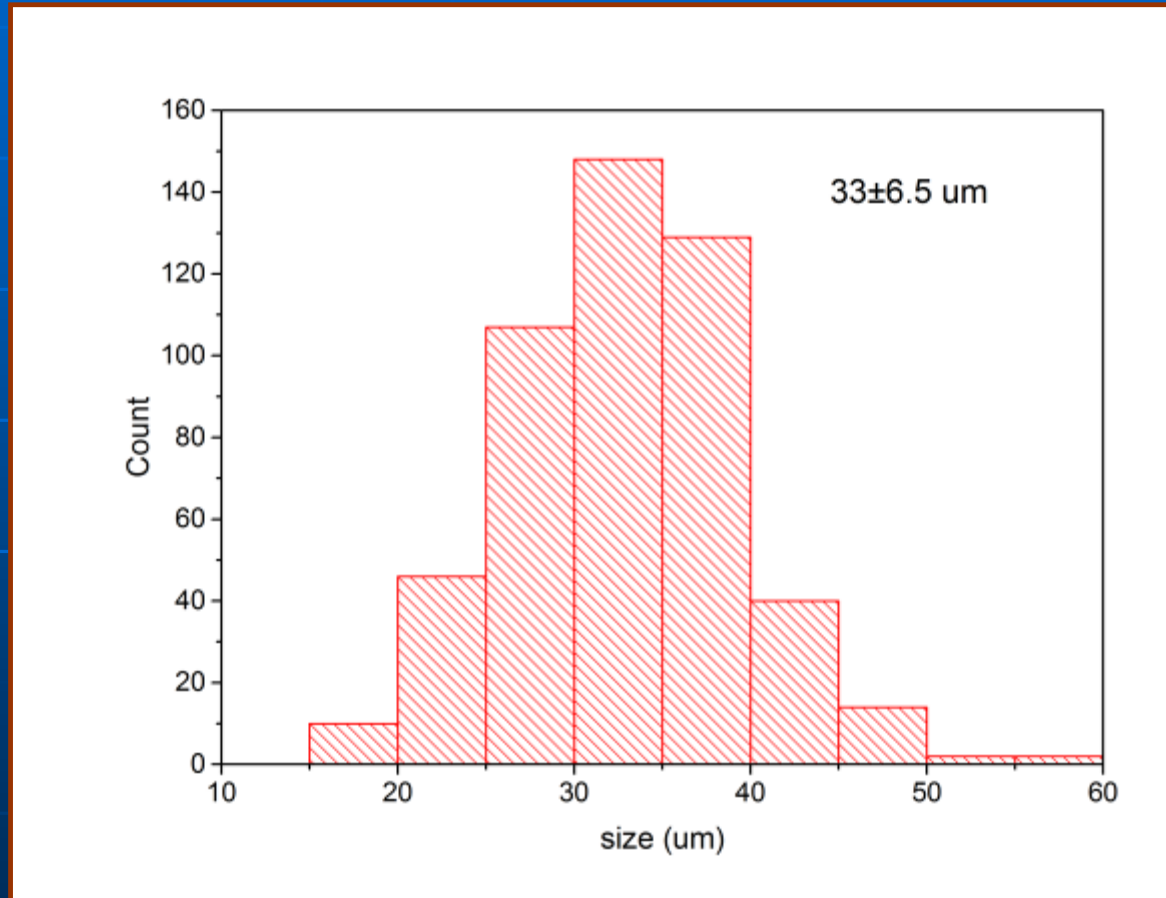


(a)

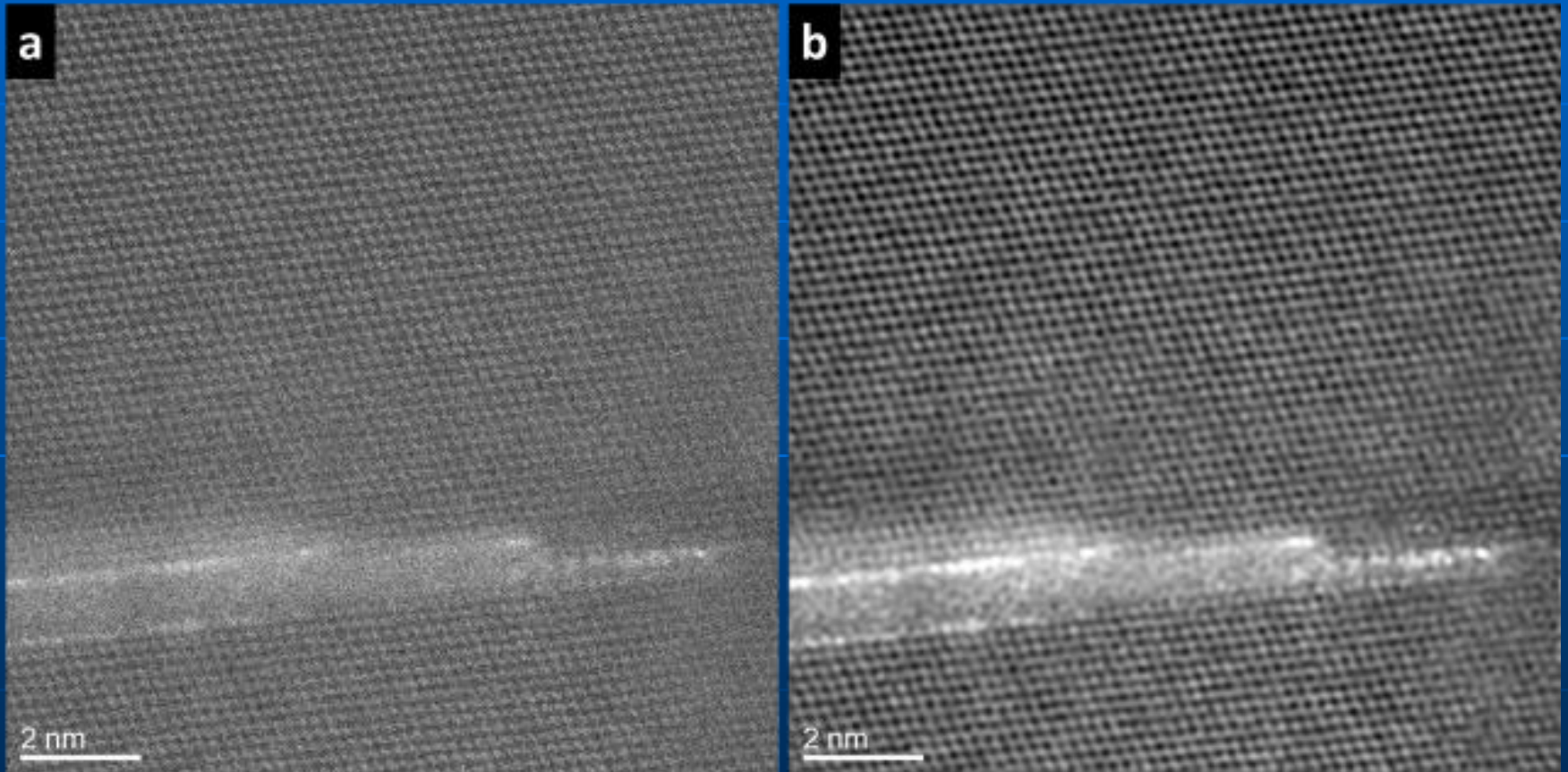


(b)

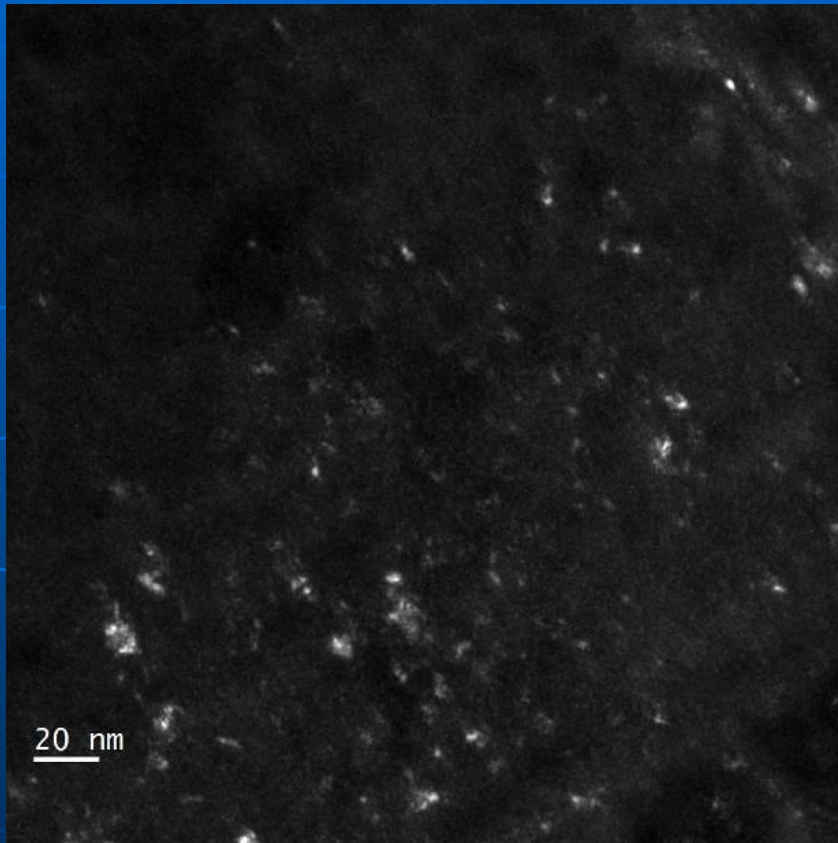
Diamond size distribution in Mo-Cu-CD material



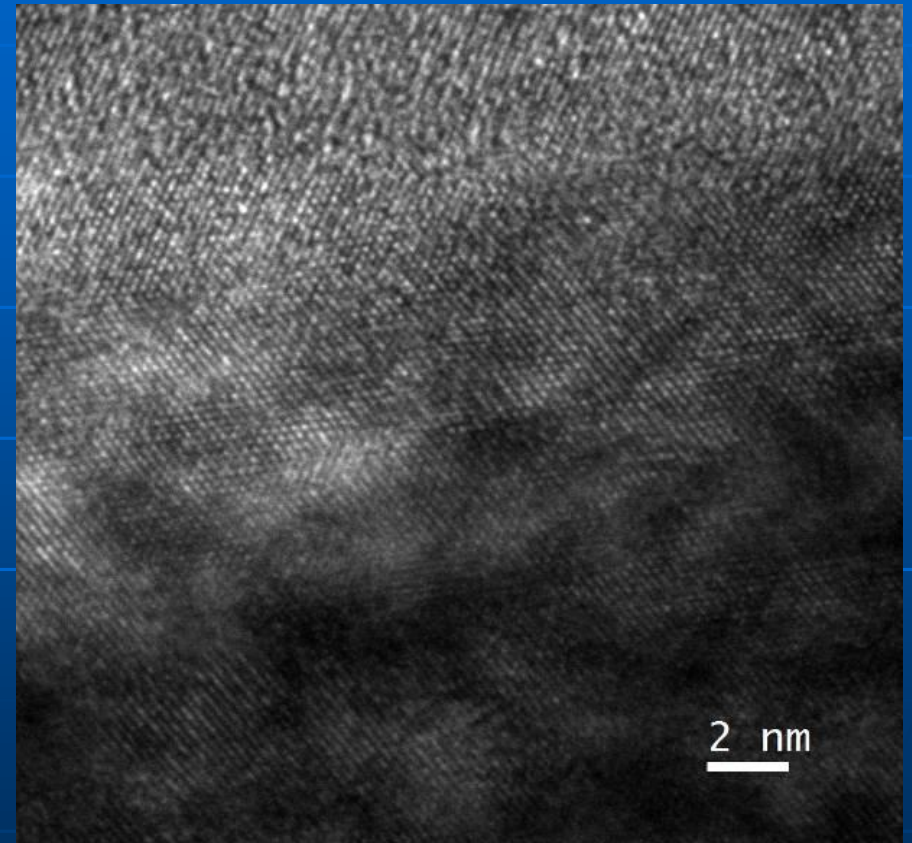
- a) HR HAADFSTEM image of the crack in Mo.**
b) The same image after the filtering.



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 ($T=100^{\circ}\text{C}$) and at the proton dose $\Phi_1 = 10^{17}$ p/cm² and at the radiation damage level ($D=3*10^{-3}$ dpa).

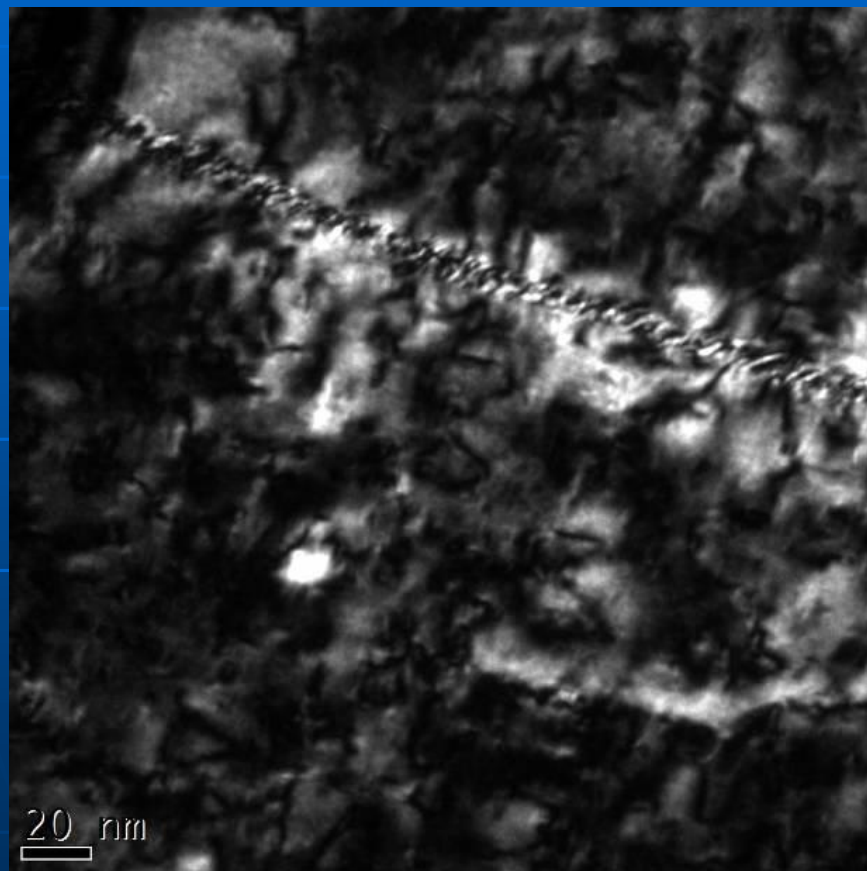


Weak Beam Dark Field image of dislocation loops on Mo metal matrix.

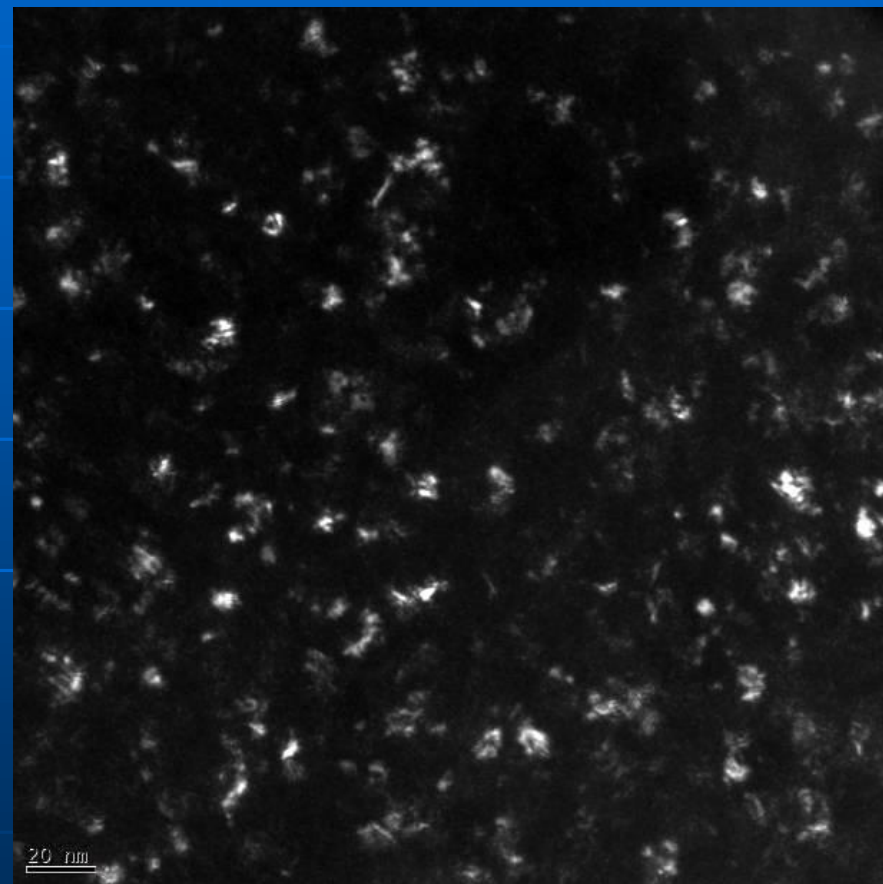


HR TEM bright field image of the Diamond grain after fast proton irradiation.

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 ($T=100^{\circ}\text{C}$) and at the proton dose $\Phi_1 = 10^{18} \text{ p/cm}^2$ and at the radiation damage level ($D=3*10^{-2} \text{ dpa}$).

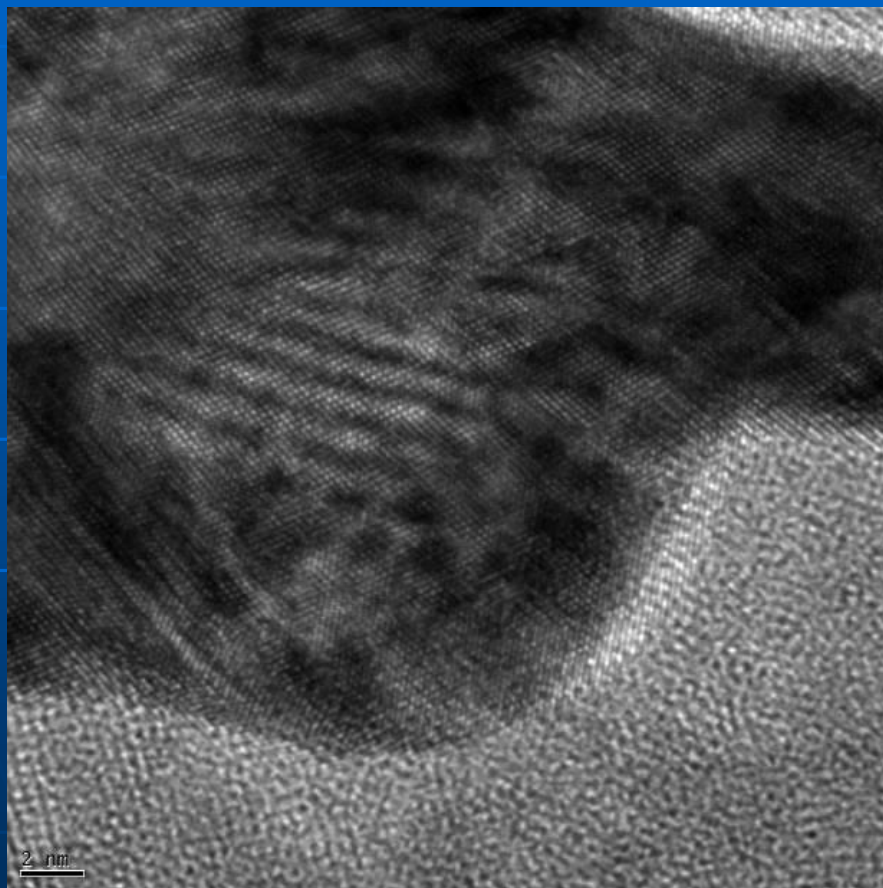


Dark Field image of dislocation loops on Mo metal matrix at 1.5 mm from the surface.

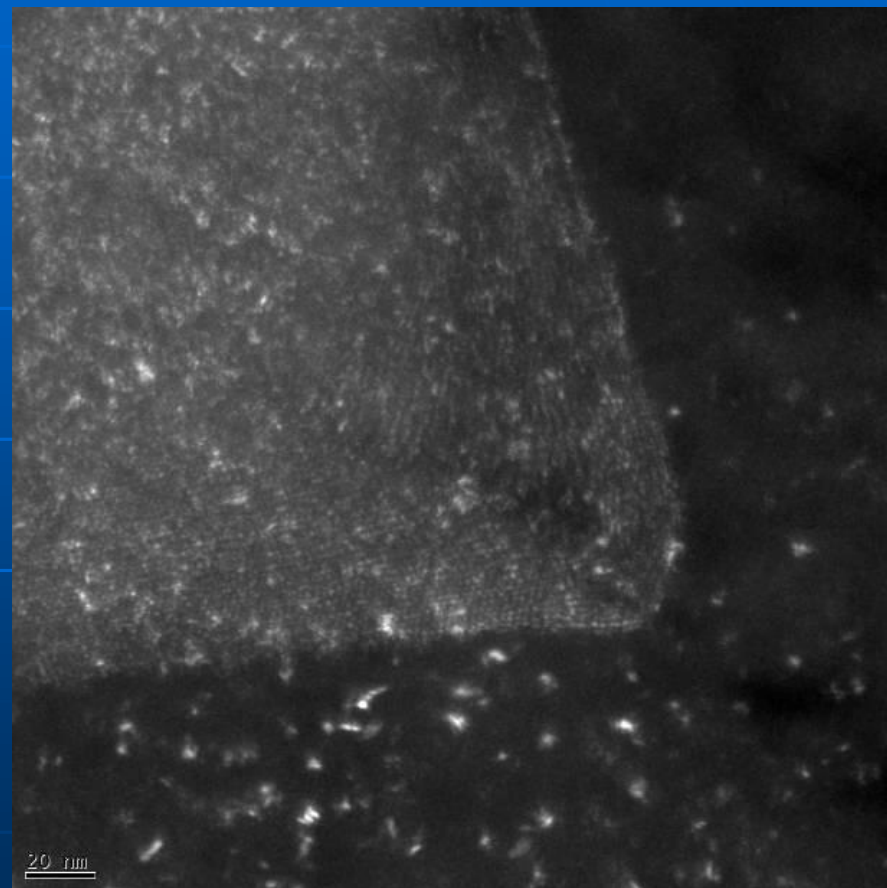


Weak Beam Dark Field image of dislocation loops in Mo metal matrix.

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 ($T=100^{\circ}\text{C}$) and at the proton dose $\Phi_1 = 10^{18}$ p/cm² 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.

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

T, °C	100	200	300	400	500
	$\alpha_{\text{исх}}, 10^{-6} \text{ 1/K}$				
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, °C	20	50	100	120
Unirradiated	$\alpha, 10^{-6} 1/K$			
Sample 1			7,5	
Sample 2			8,1	
Sample 3			8,3	
Irradiated	$\alpha, 10^{-6} 1/K$			
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
$(\rho_{irr} - \rho_{in})$ / ρ_{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	
	Initial	Irradiated	Initial	Irradiated	Initial	Irradiated
$\rho, 10^{-6}, \text{Om}\cdot\text{m}$	9,0	8,8	10,2	10,2	10,1	10,5
$(\rho_{\text{irr}} - \rho_{\text{in}}) / \rho_{\text{in}}, \%$	-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, $\alpha_{\text{исх}}, 1/\text{K},$ $10^{-6} 1/\text{K}$	9,0	9,7	10,4	12,4	12,2
Mo-Diamond, $\alpha_{\text{исх}}, 1/\text{K},$ $10^{-6} 1/\text{K}$	7,5	8,1	8,4	8,3	7,4