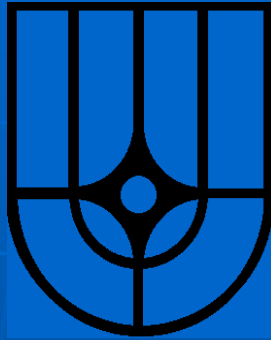
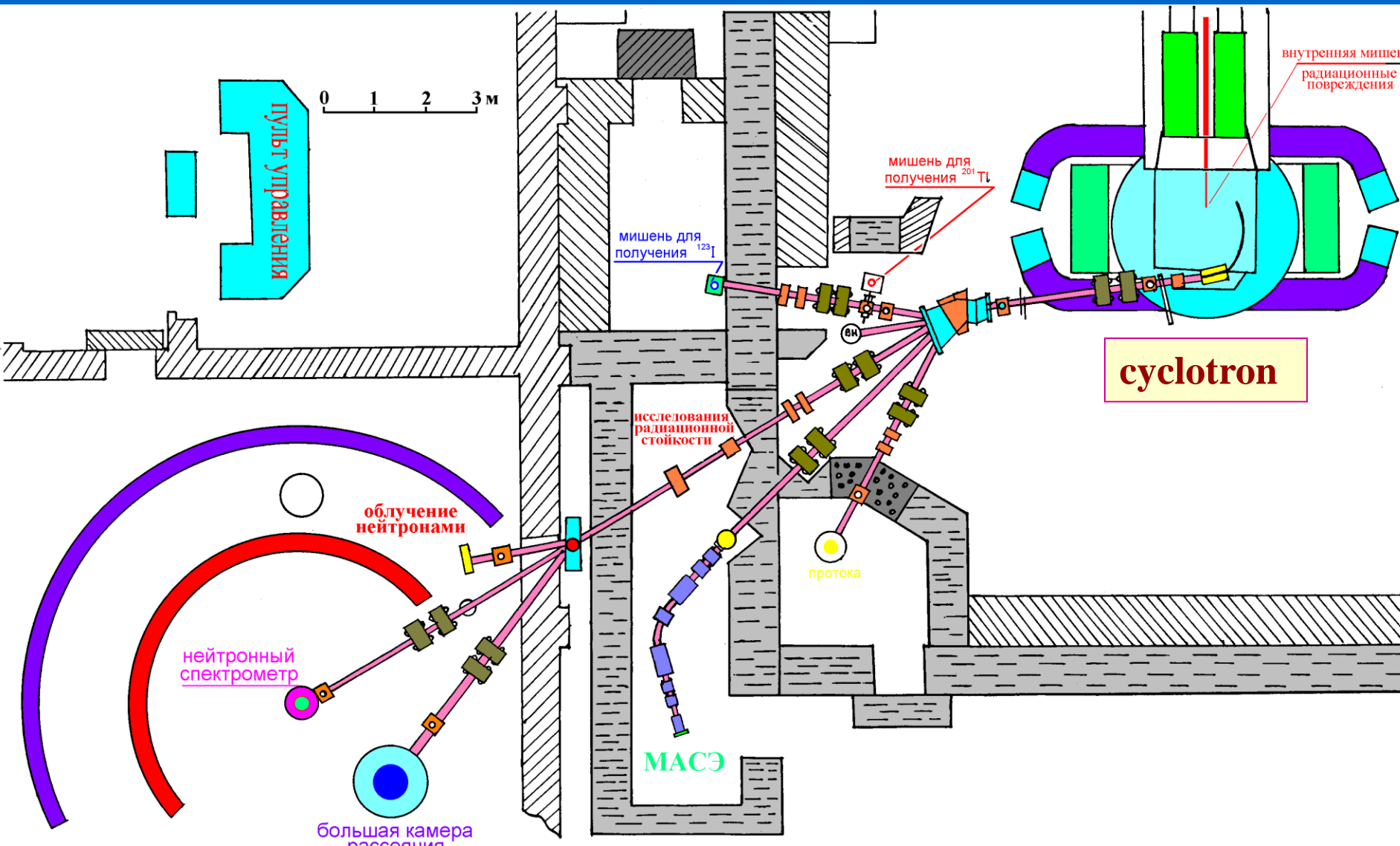


National Research Center” Kurchatov Institute”



Investigations of fast proton irradiation effects on Mo-Diamond collimator materials for LHC at NRC “Kurchatov Institute” and comparison with the results for Co-Diamond materials.

Alexander Ryazanov



System of cyclotron transportation

Accelerators of Charge Particles at National Research Centre “Kurchatov Institute”

Cyclotron of NRC KI:

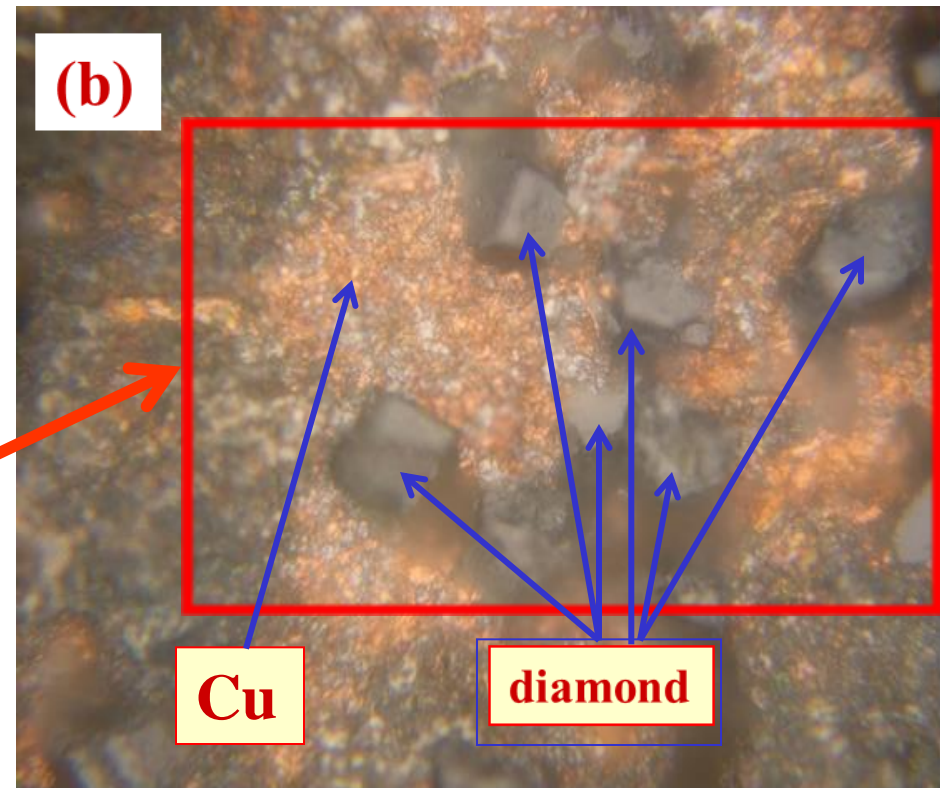
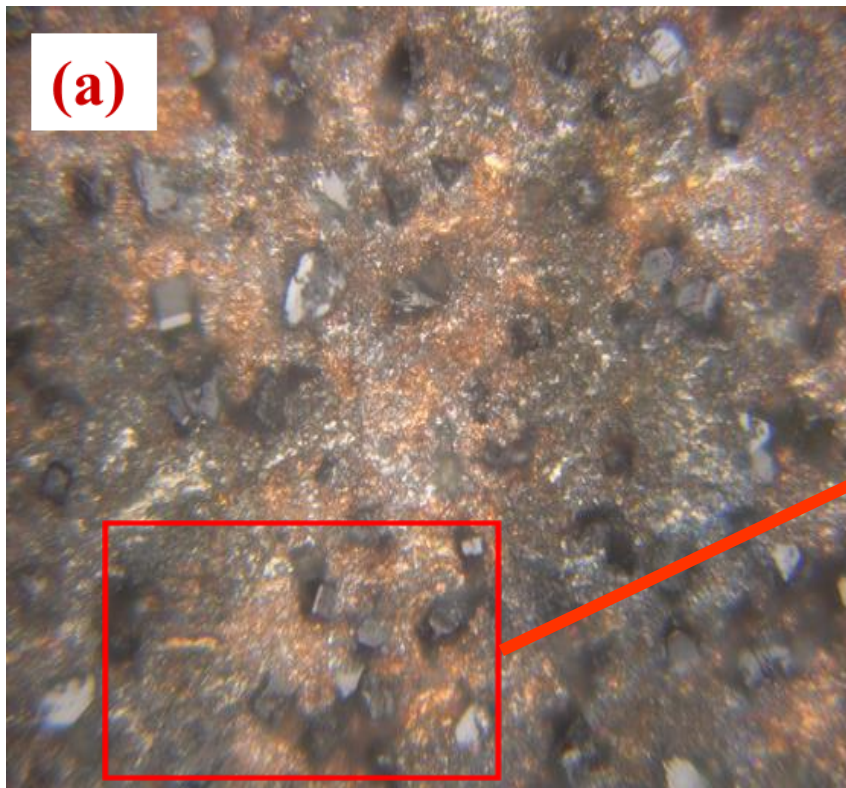
protons with energy < 35 MeV, current $J < 30$ mA

helium ions He^4 with energy < 60 MeV,
current $J < 20$ mA

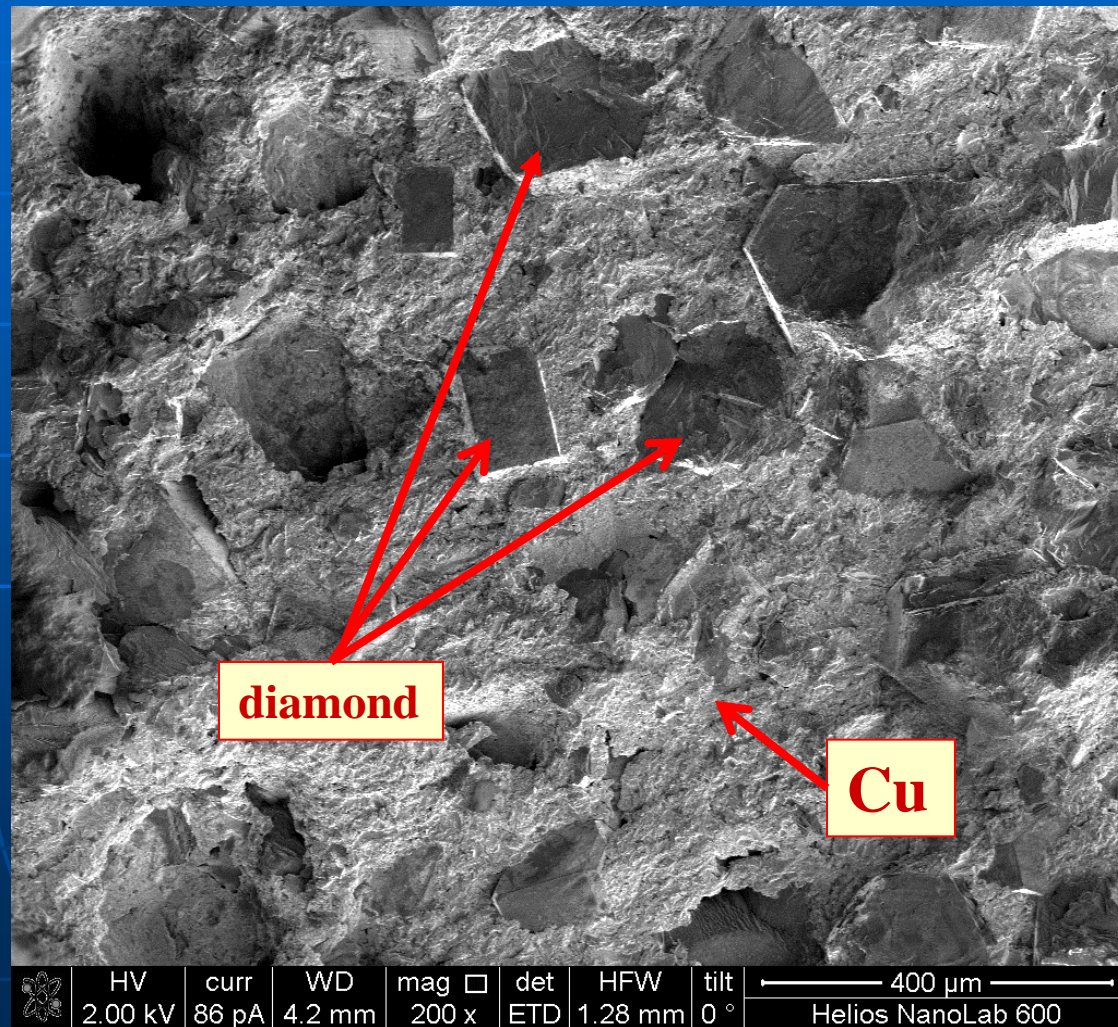
ions O^{16} with energy < 120 MeV, current $J < 5$ mA

ions C^{12} with energy < 80 MeV, current $J < 5$ mA

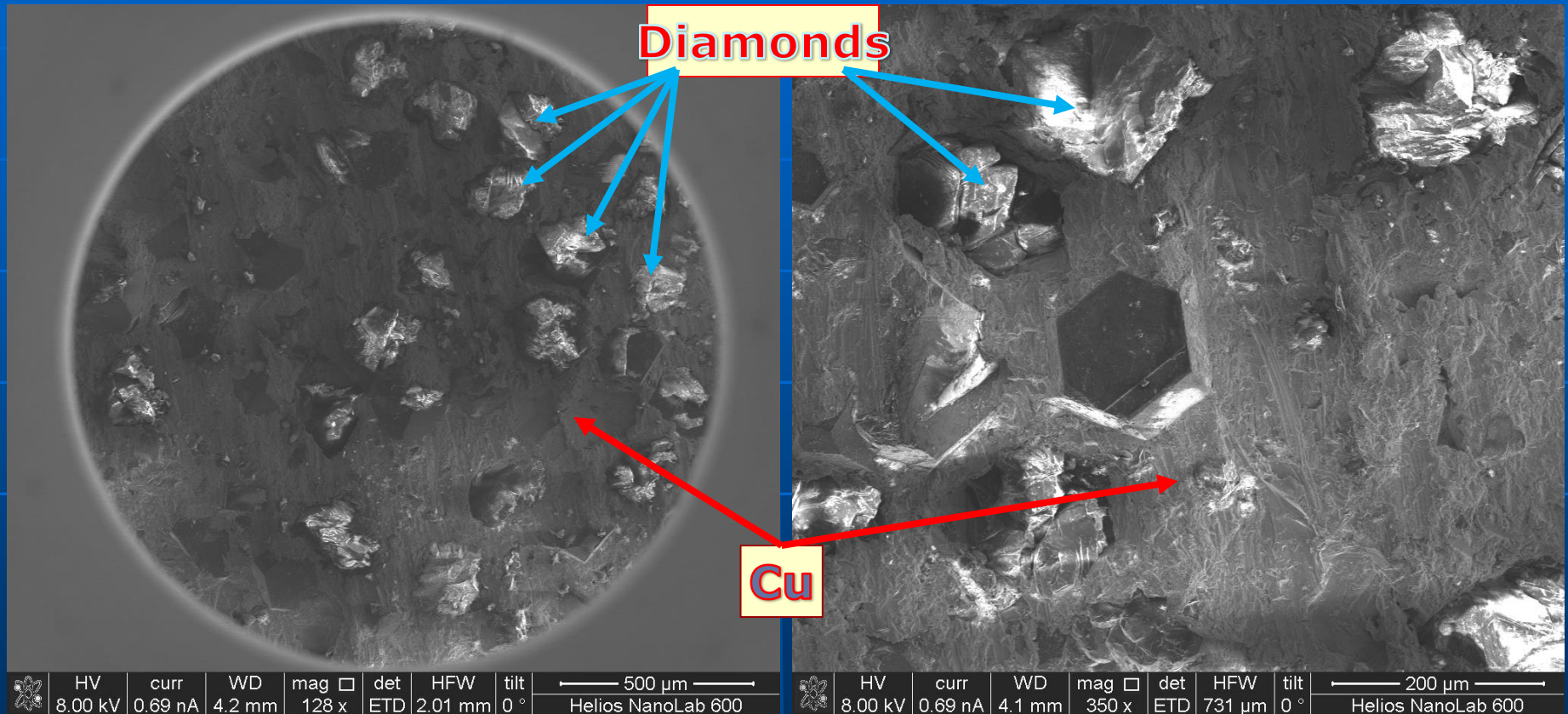
Microstructure of Mo - diamond sample surface under different magnification (-200 (a) and -500 (b)) made on optical microscope Carl Zeiss Axio Observer D1m



STEM Study of Cu-diamond composite



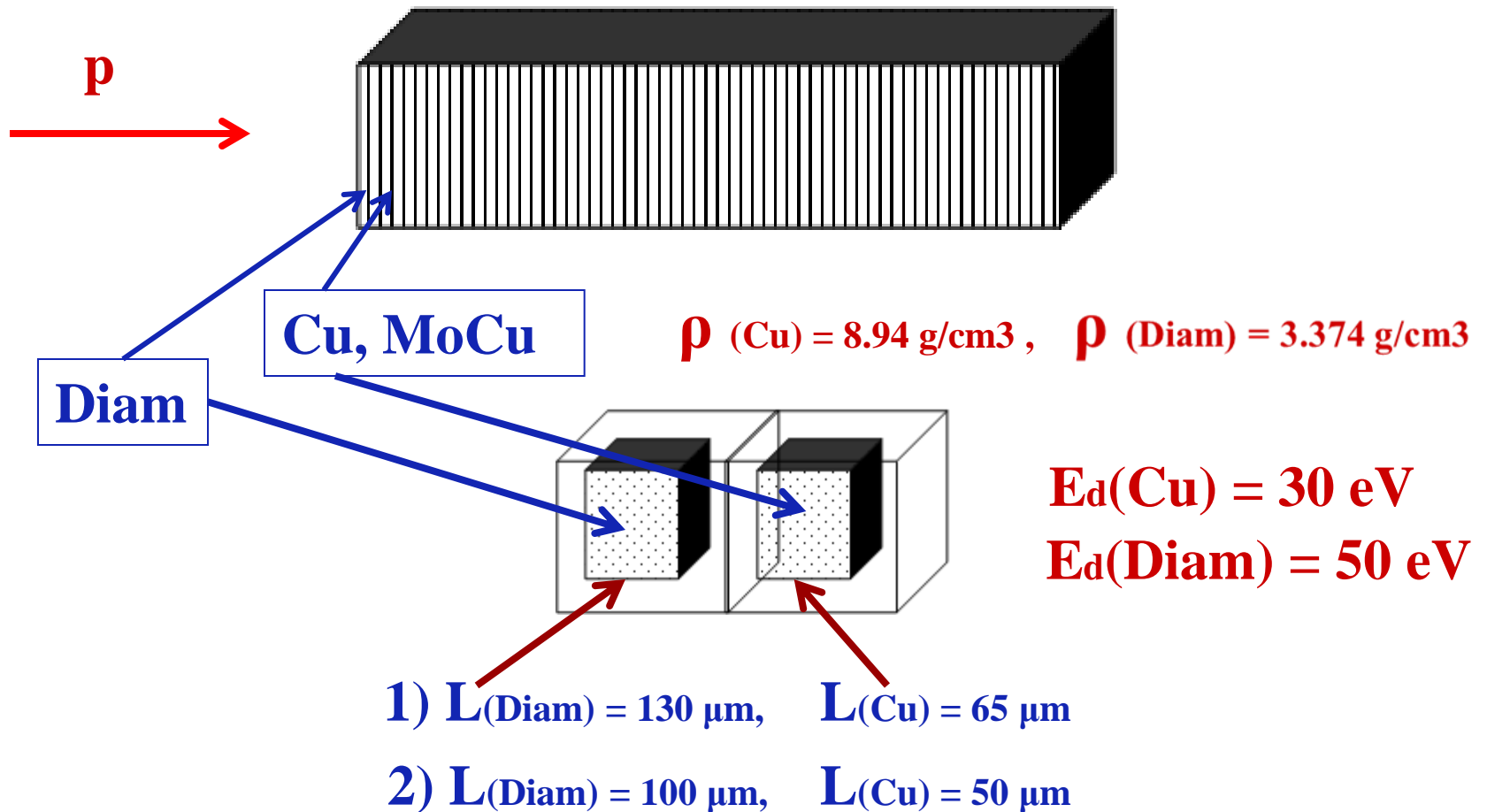
The SEM study and sample preparation



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

Theoretical modelling of radiation damage profiles (dpa) in Cu-Diamond and Mo-Cu-Diamond materials irradiated by fast protons on NRC KI cyclotron with the energy up to 35 MeV

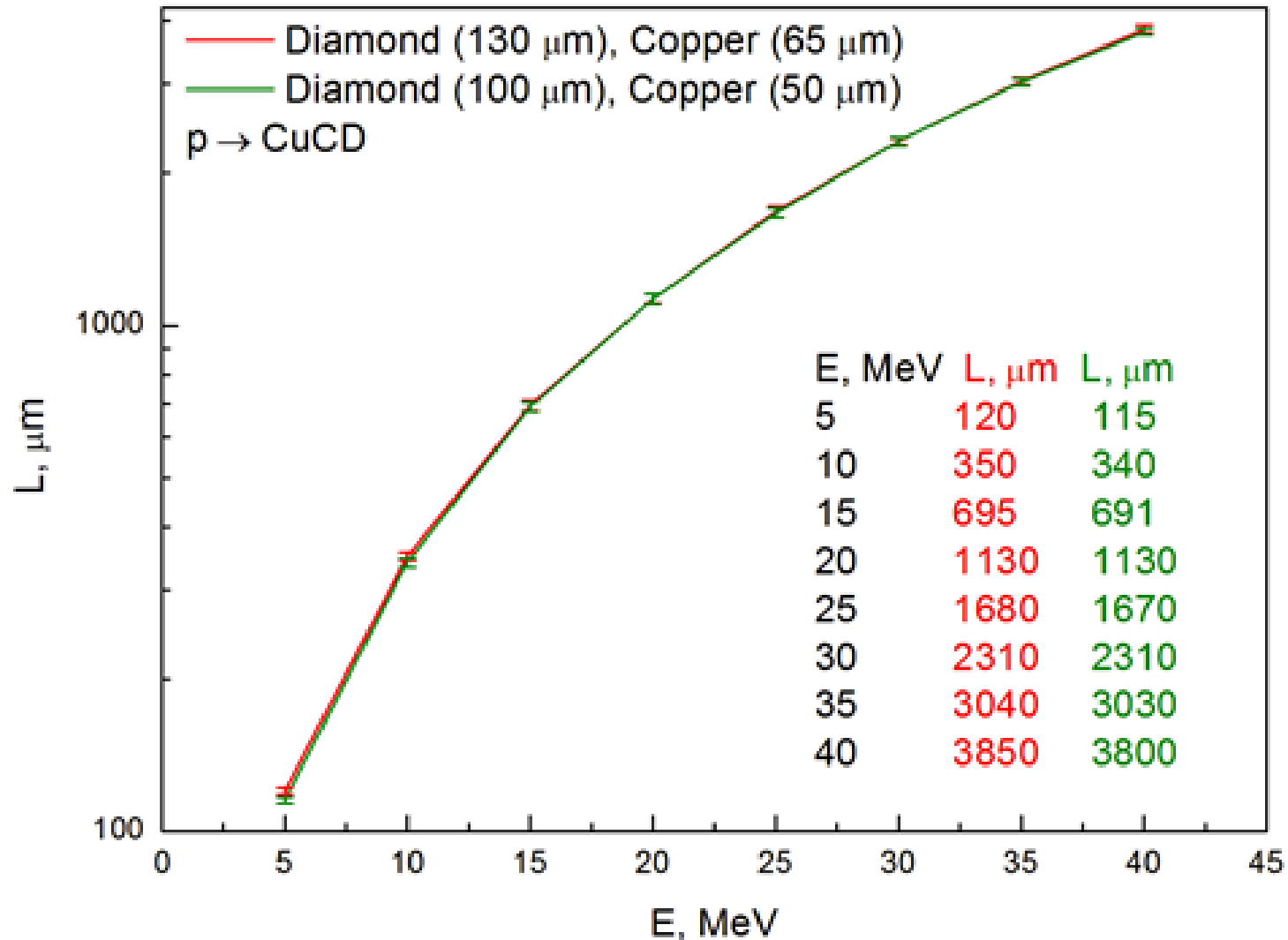
Theoretical modeling of point radiation damage accumulation in Mo-Diamond and Cu-Diamond materials irradiated by 30 MeV protons



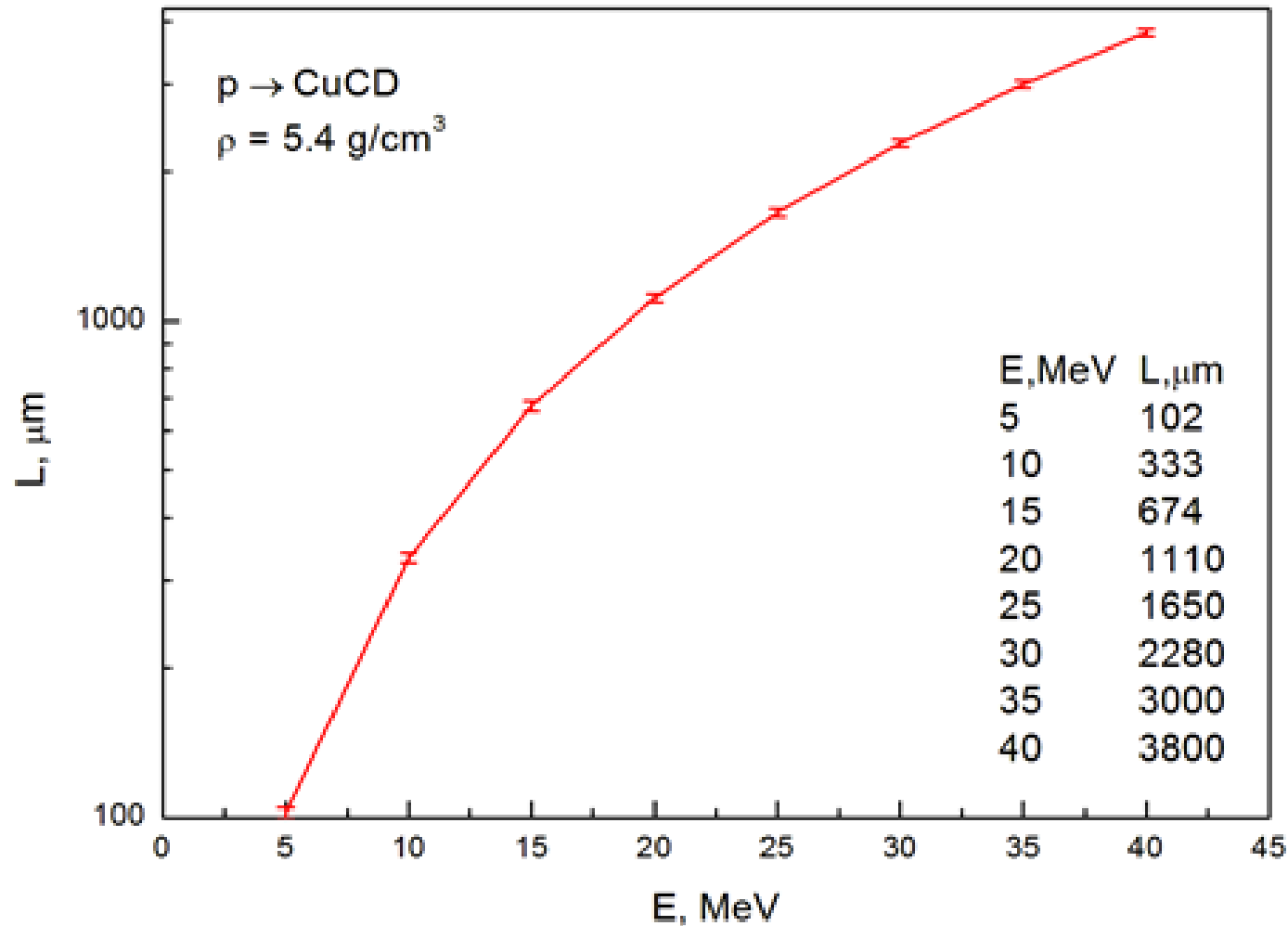
Physical parameters used for numerical calculations in two theoretical models in both materials CuCD and MoCuCD

Material	Layer thickness, microns	E_d , eV	Density, g/cm ³
CuCD (layered)	130 (CD)	50	3.373
	65 (Cu)	30	8.94
MoCuCD (layered)	45 (CD)	50	3.373
	45 (MoCu)	50	9.581
CuCD (average)	-	30/50 (Cu/CD)	5.4
MoCuCD (average)	-	50	6.7

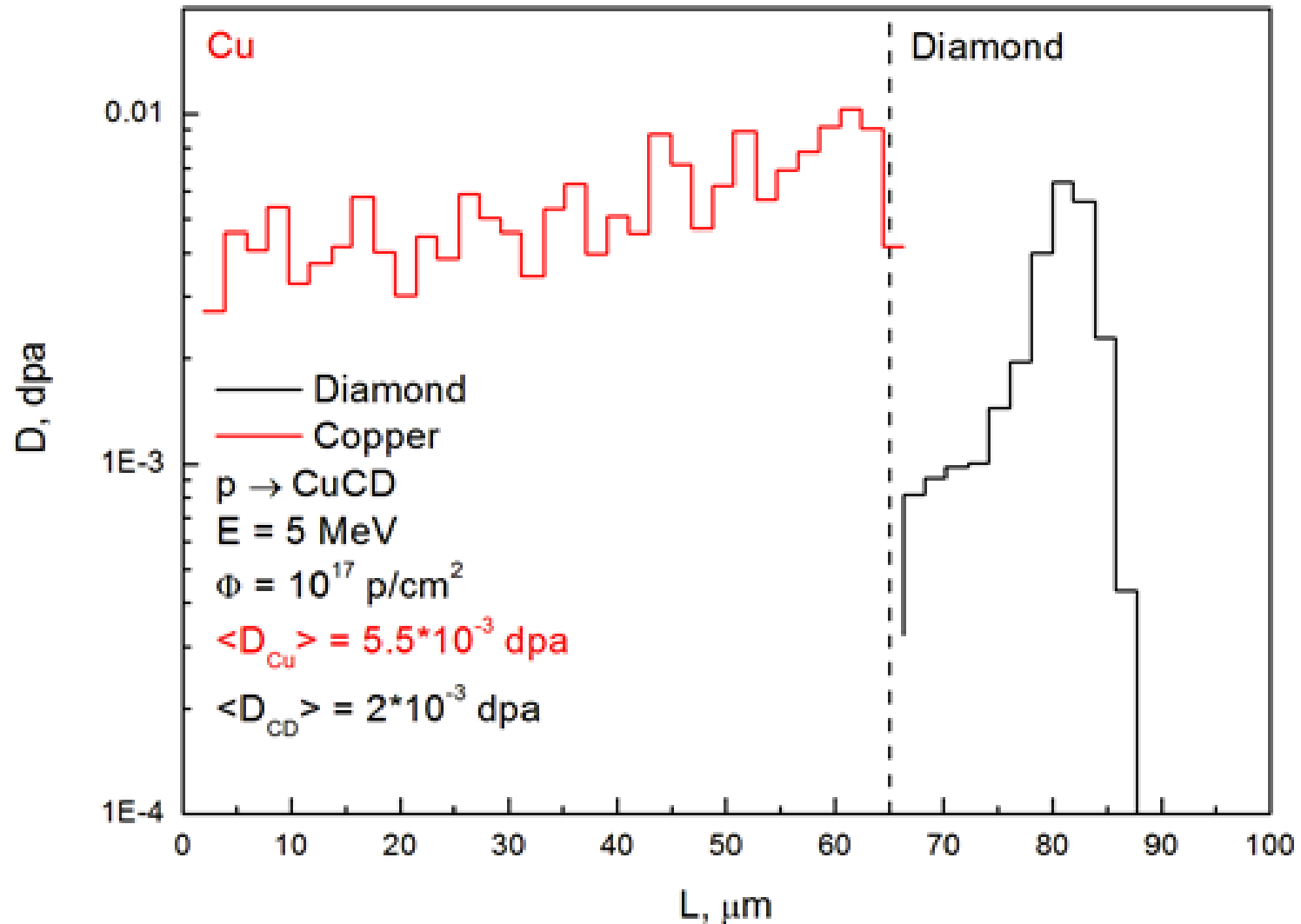
Ranges of protons in CuCD layered structure of two different layer configurations versus proton energy



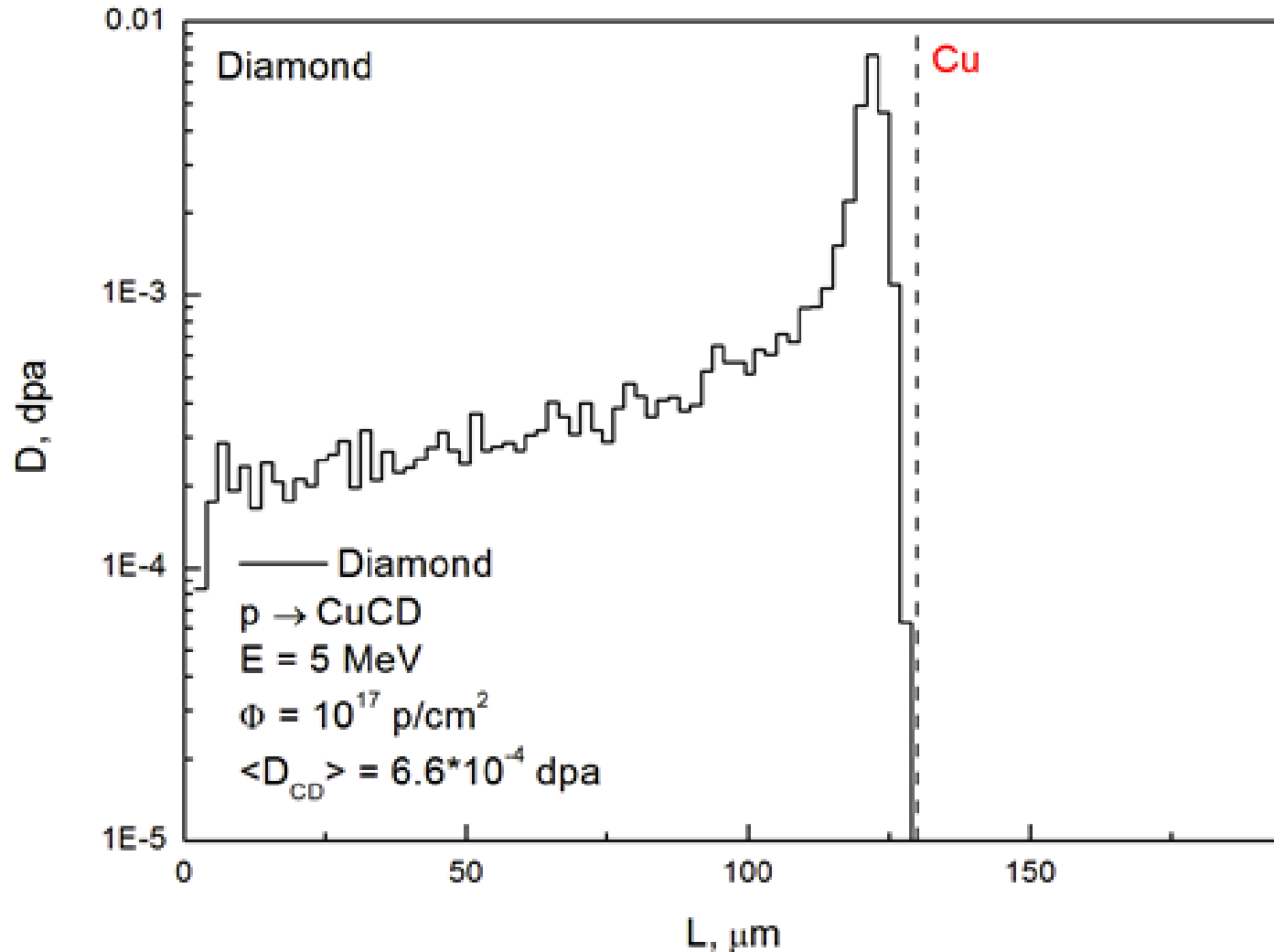
Ranges of protons in CuCD material with average density as a function of proton energy



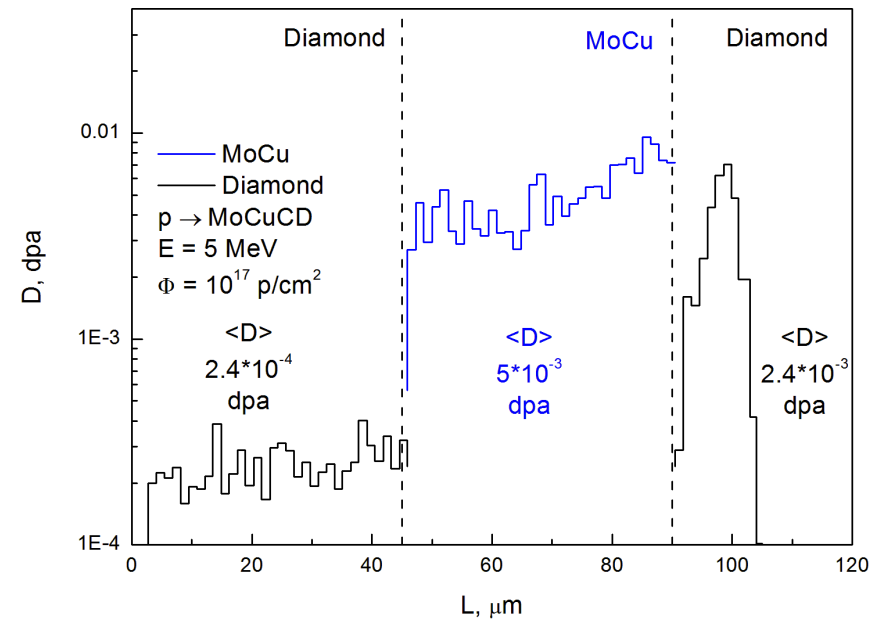
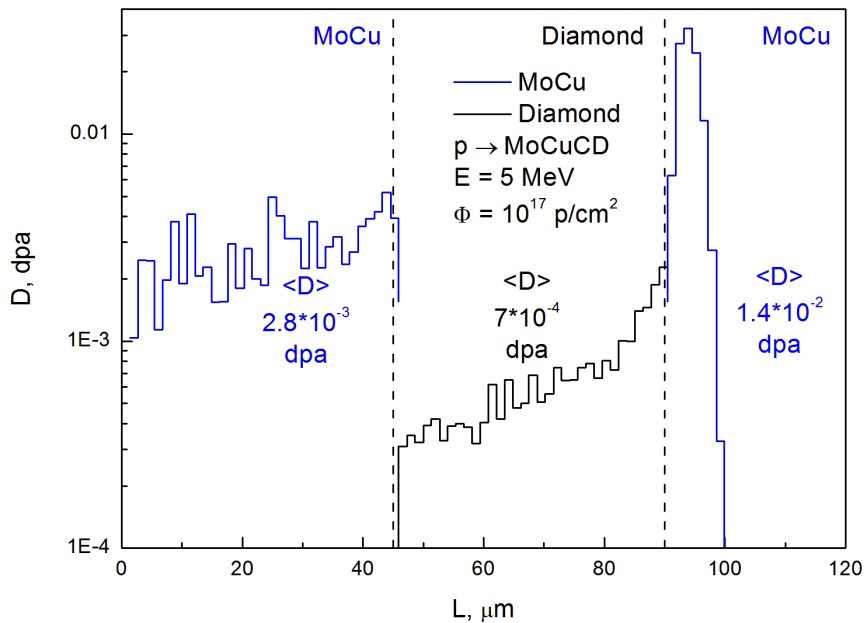
Radiation damage profiles from protons with energy 5 MeV in the layered structure of CuCD material for two different configurations of layers



Radiation damage profiles from protons with energy 5 MeV in the layered structure of CuCD material for two different configurations of layers

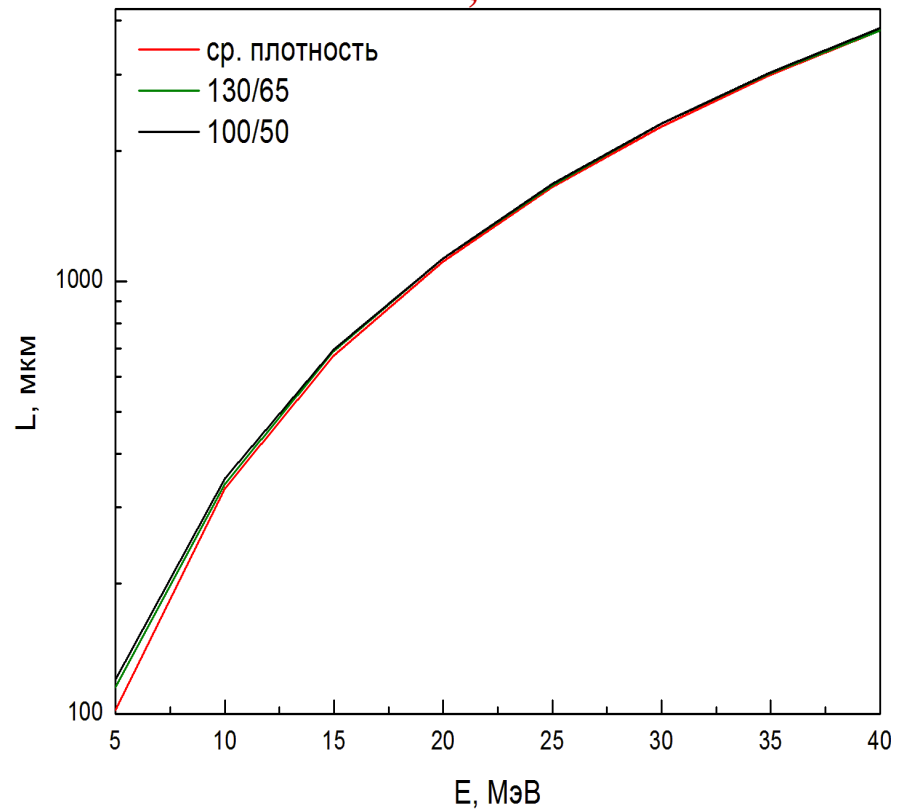
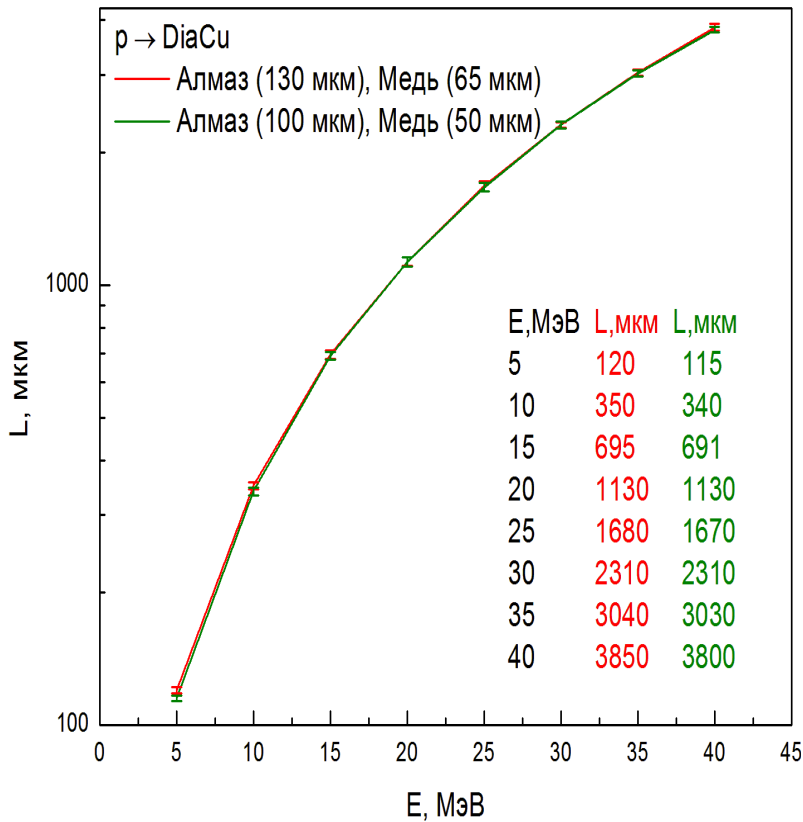


Distribution of radiation damage profiles (dpa) in Mo-Cu-Diamond irradiated by protons with the energy 5 MeV at fluence $\Phi = 10^{17}$ p/cm²

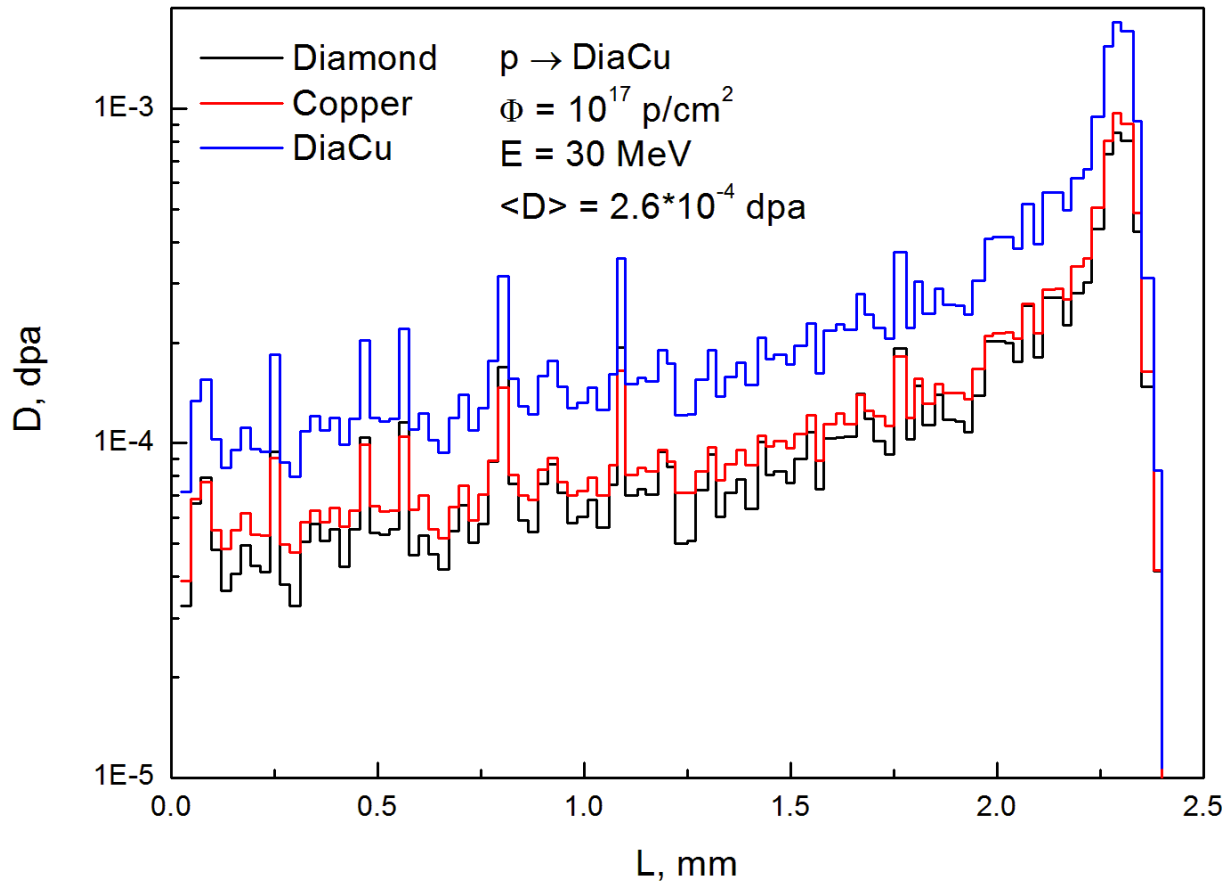


Dependence of penetration depth of fast protons in Copper-Diamond Samples on proton energy

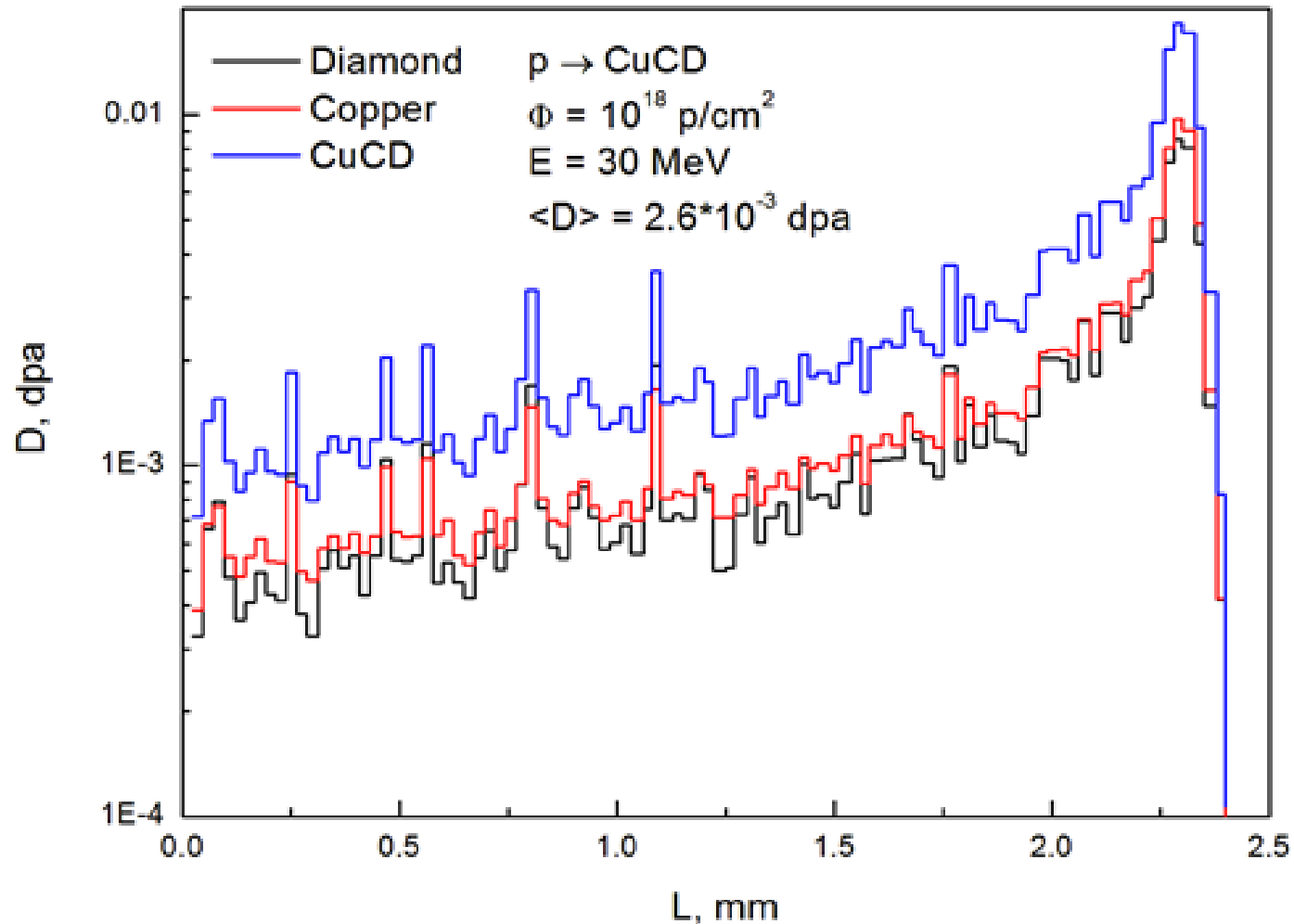
$$\langle \rho(\text{Cu-D}) \rangle = 5.4 \text{ g/cm}^3$$



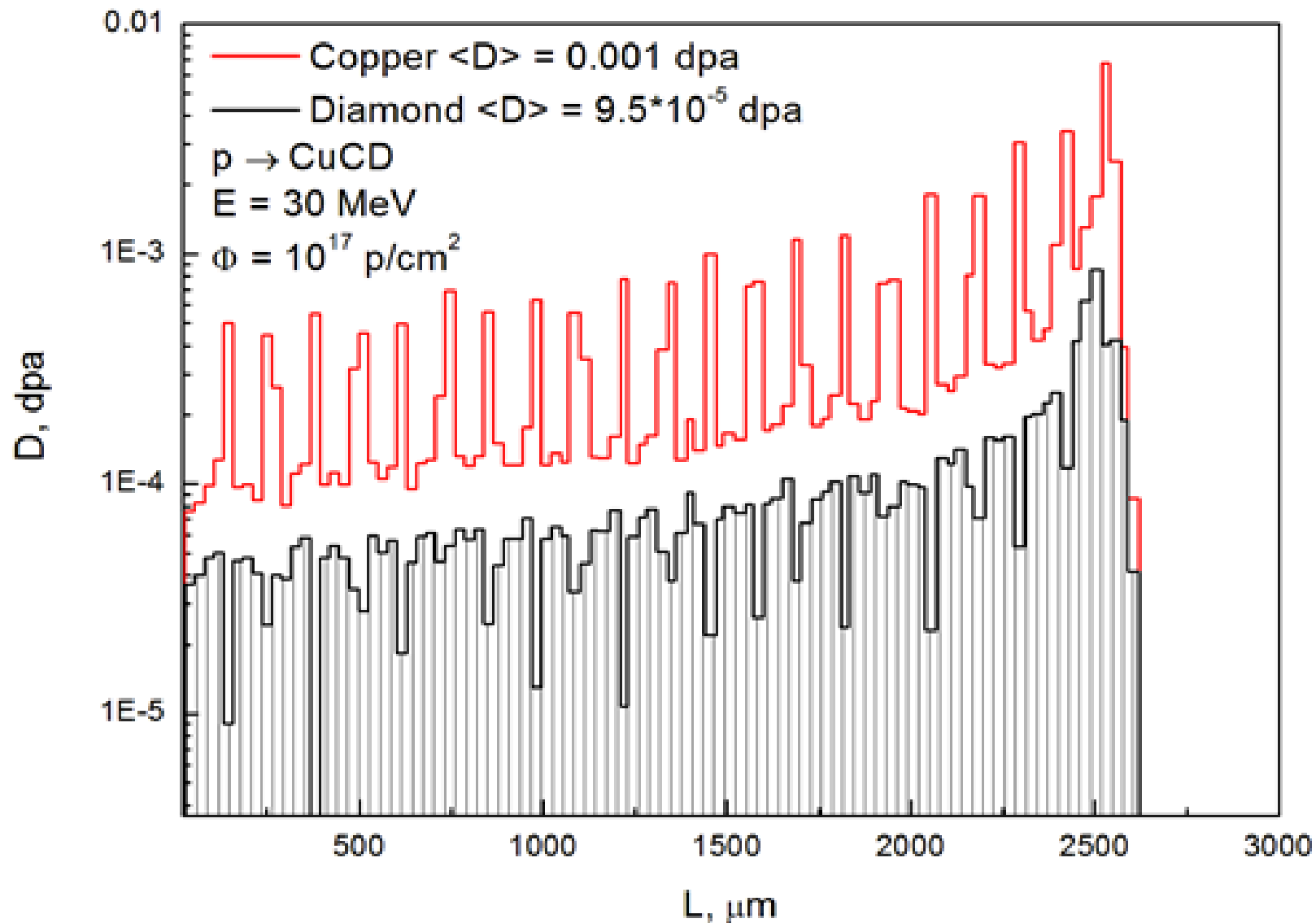
Displacement damage profiles in Copper-Diamond irradiated by 30 MeV protons up to dose $\Phi = 1 \times 10^{17}$ p/cm²



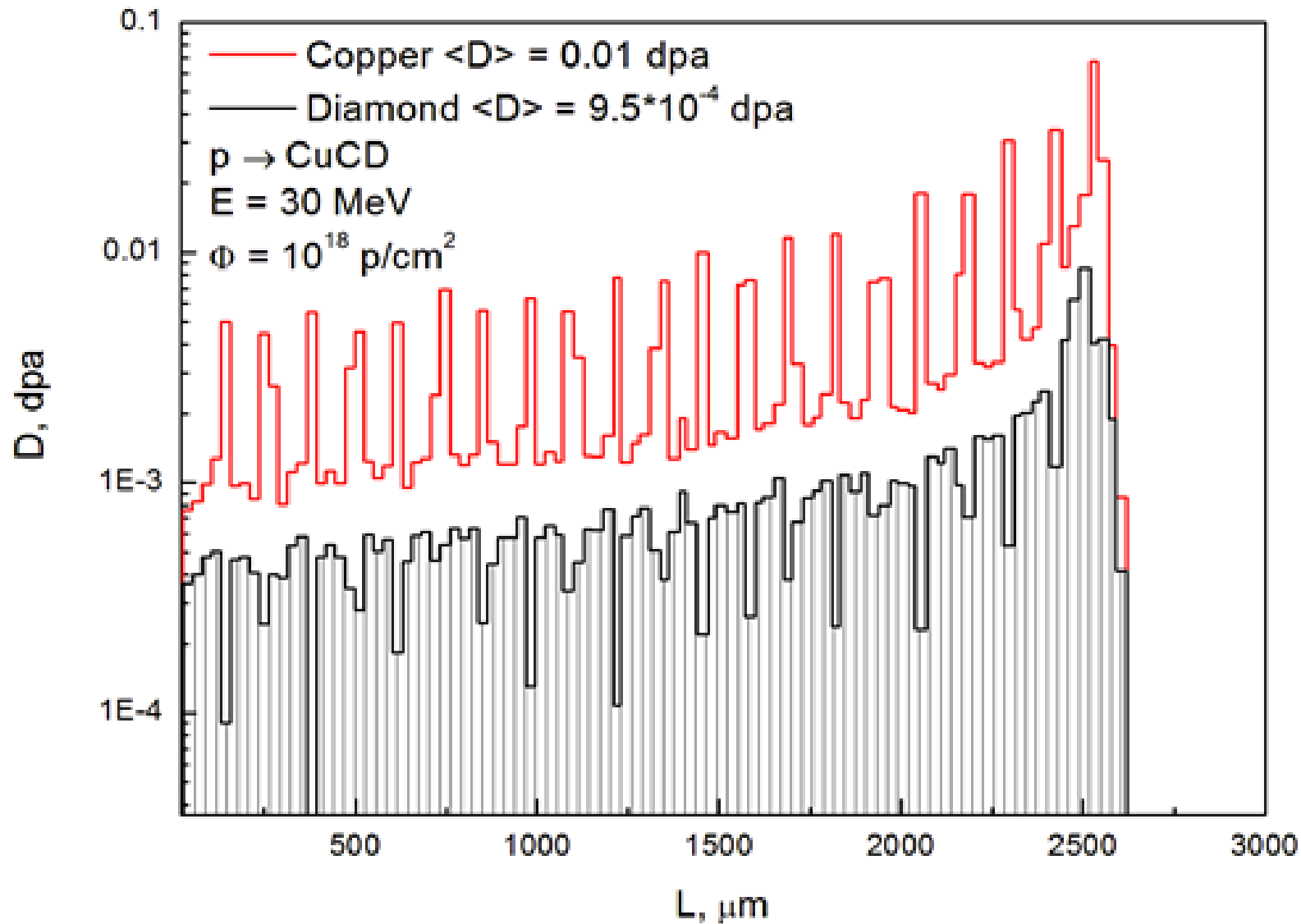
Displacement damage profiles in Copper-Diamond irradiated by 30 MeV protons up to dose $\Phi = 1 \times 10^{18}$ p/cm²



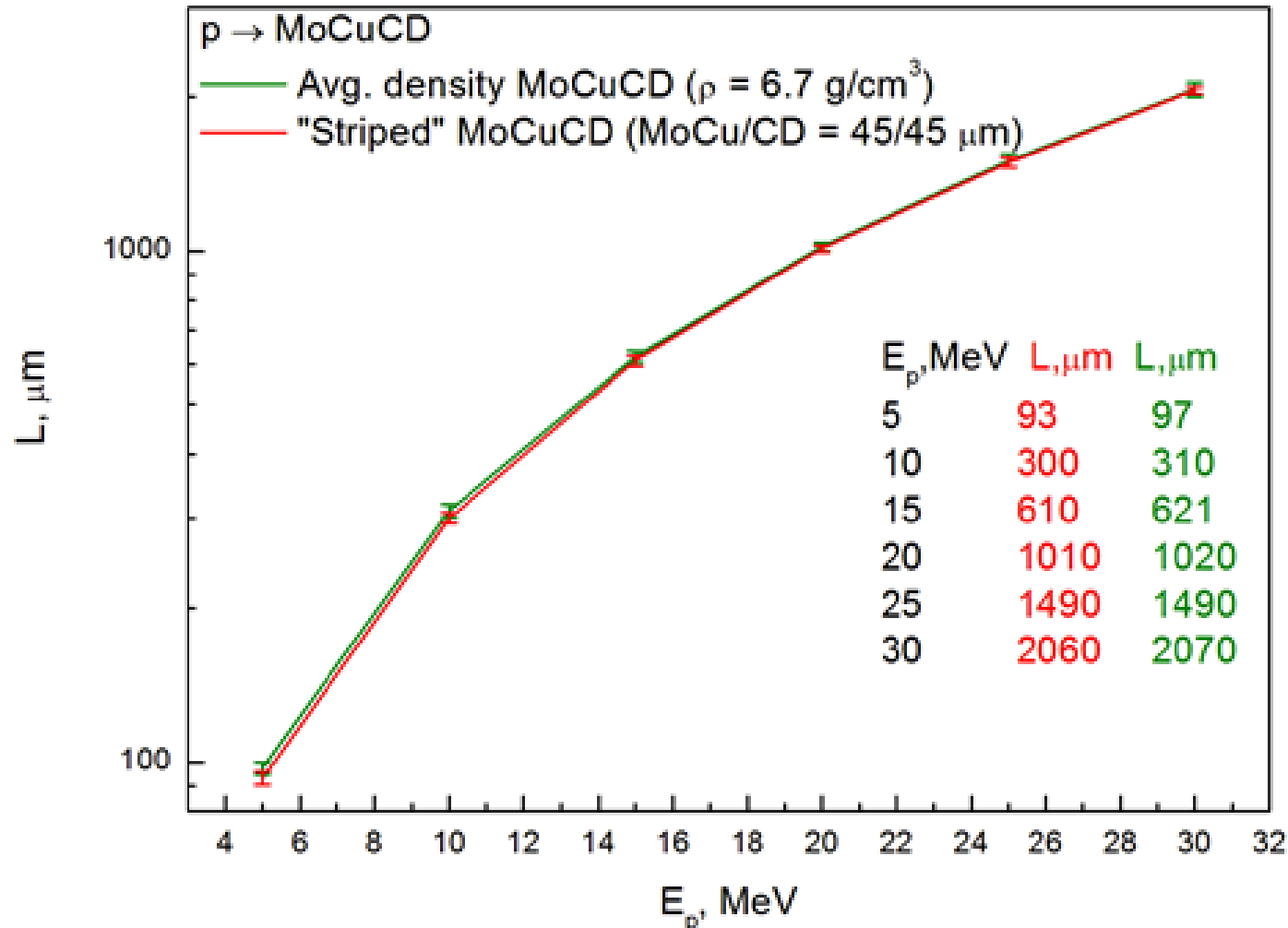
Displacement damage profiles in Copper-Diamond irradiated by 30 MeV protons up to dose $\Phi = 1 \times 10^{17}$ p/cm²



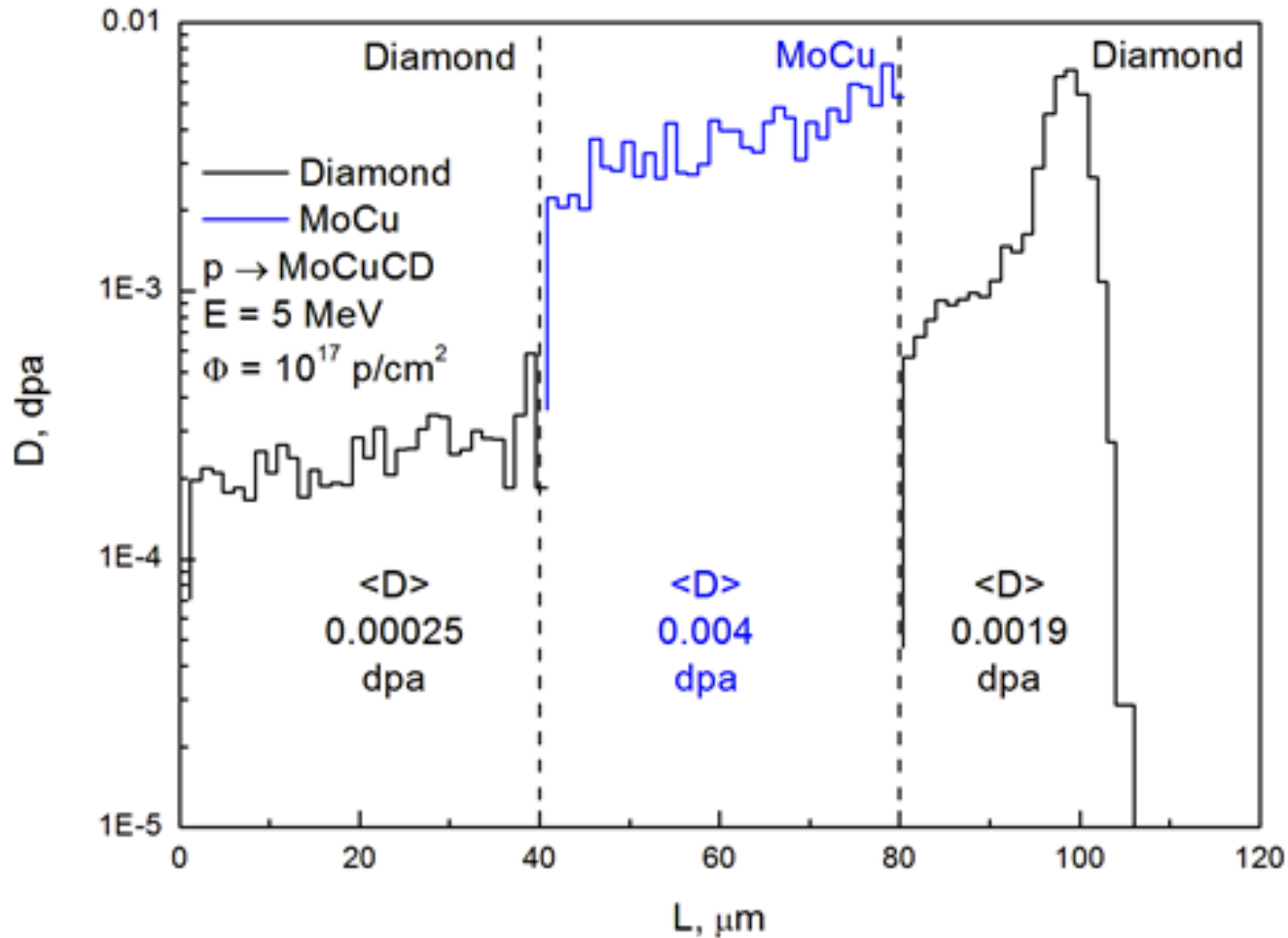
Displacement damage profiles in Copper-Diamond irradiated by 30 MeV protons up to dose $\Phi = 1 \times 10^{18}$ p/cm²



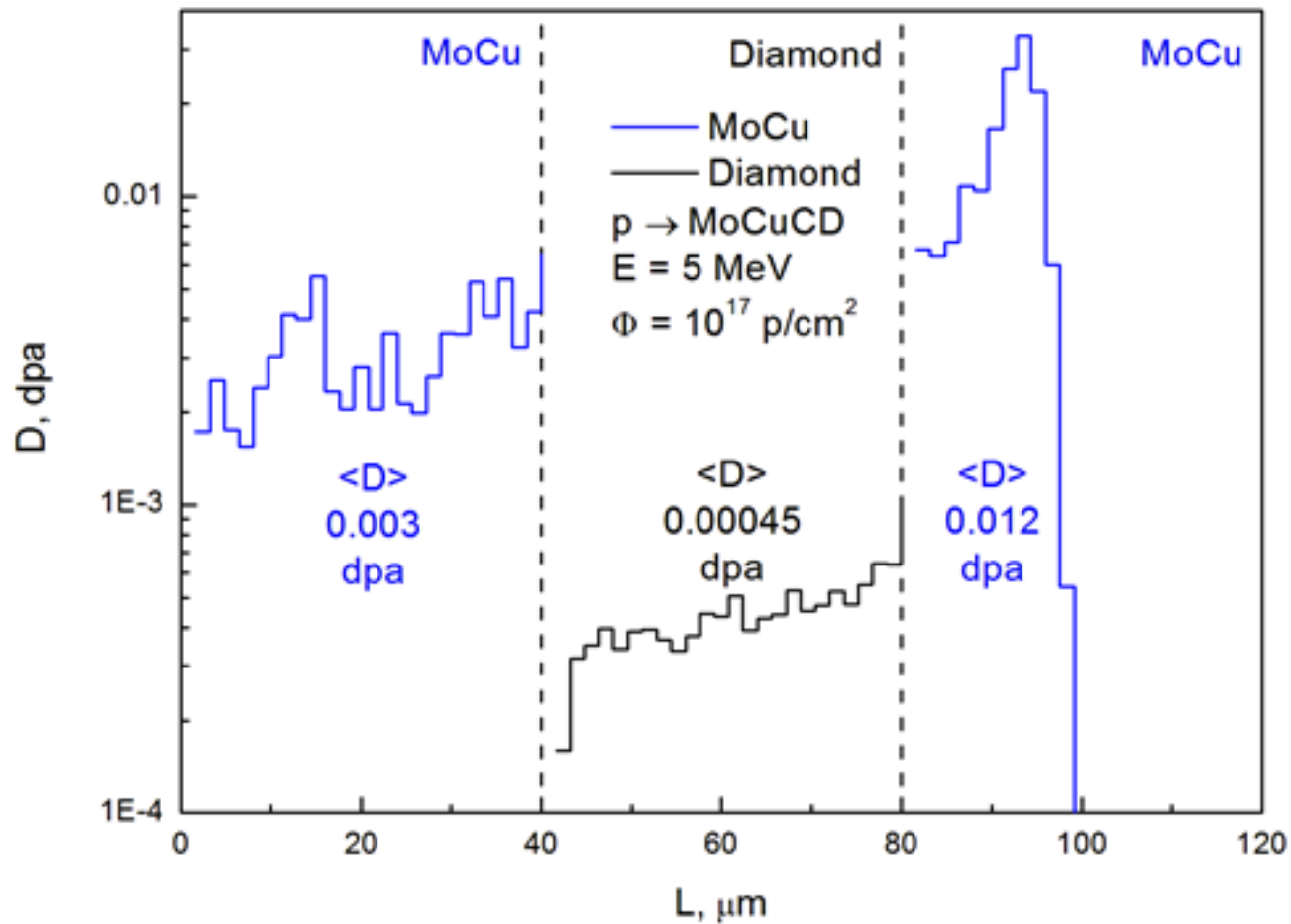
Ranges of protons in MoCuCD layered structure of two different layer configurations versus proton energy



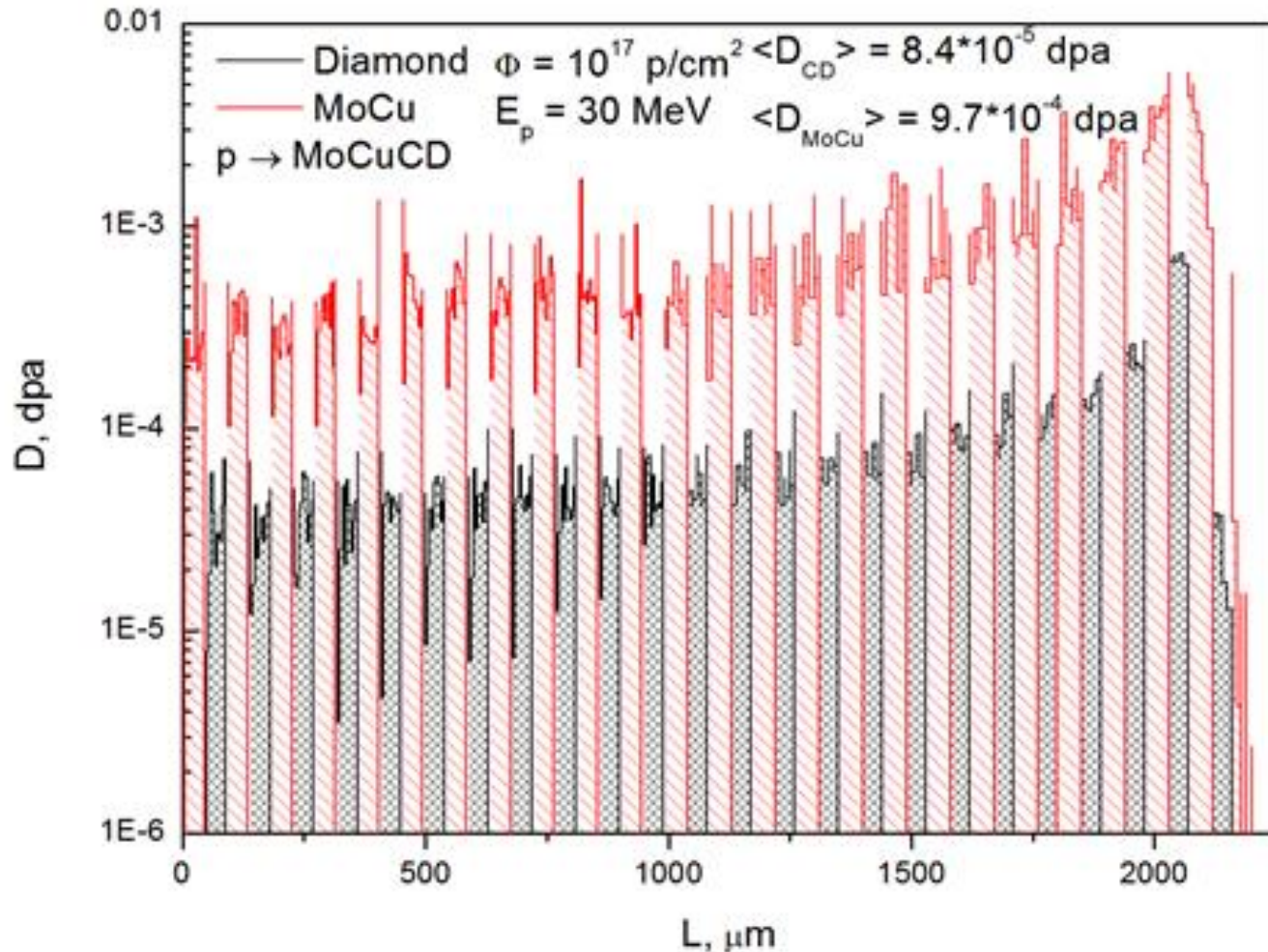
Radiation damage profile from protons with energy 5 MeV in the layered structure of MoCuCD material for two different configurations of layers



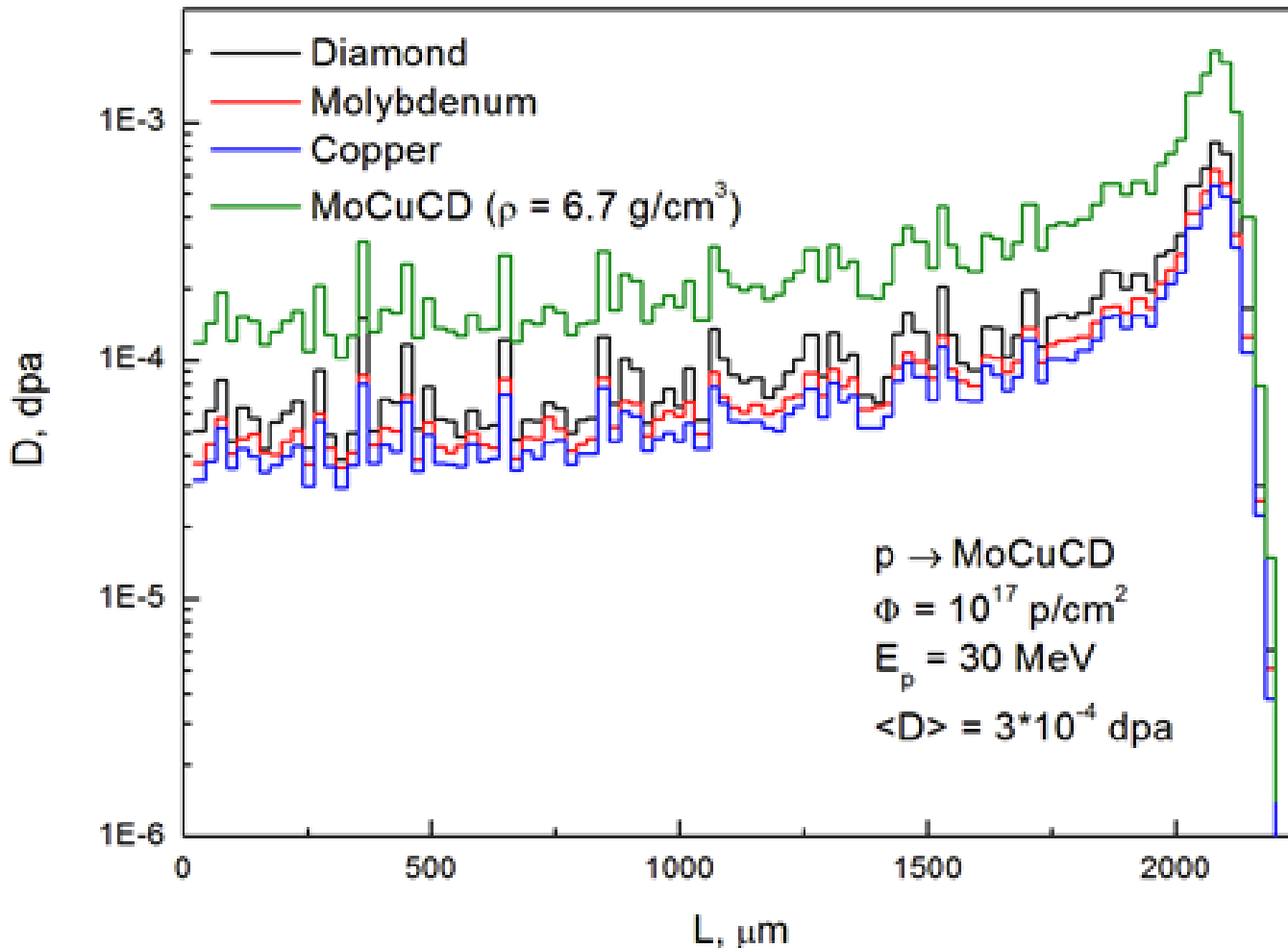
Radiation damage profile from protons with energy 5 MeV in the layered structure of MoCuCD material for two different configurations of layers



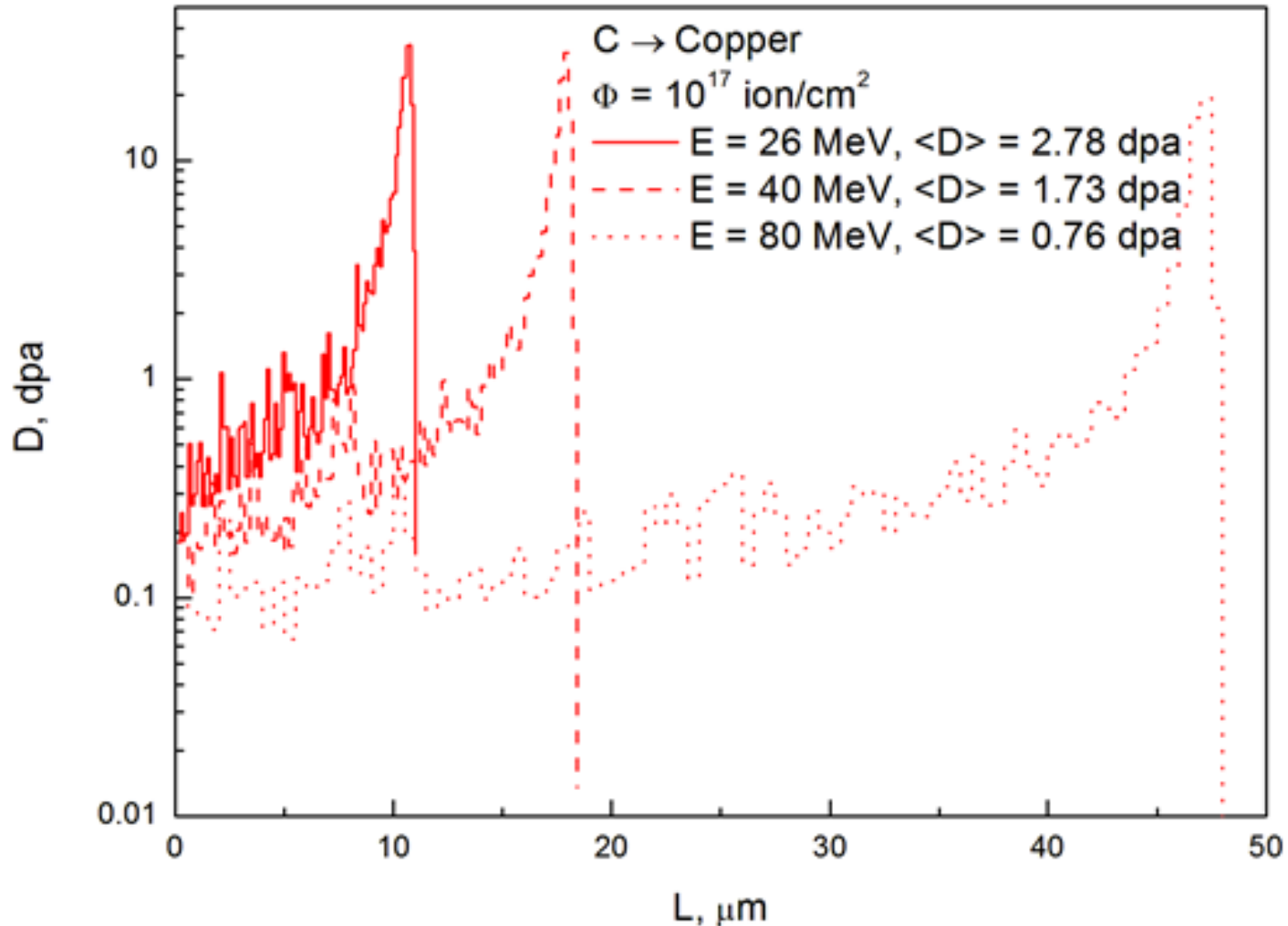
Radiation damage profile from protons with energy 30 MeV in the layered structure of MoCuCD material for a dose up to 10^{17} p/cm²



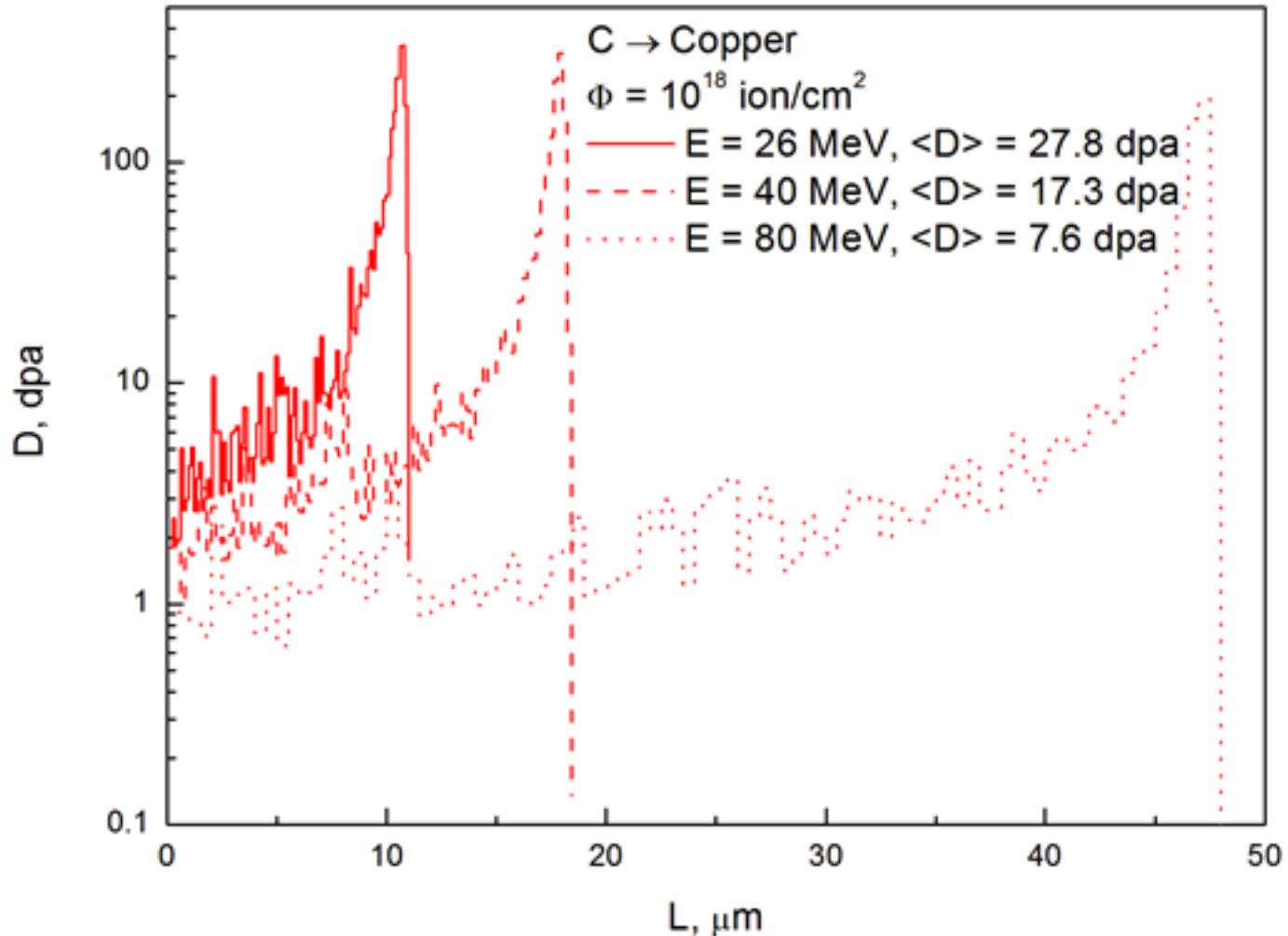
Radiation damage profile from protons with energy 30 MeV in MoCuCD material with average density for a dose up to 10^{17} p/cm²



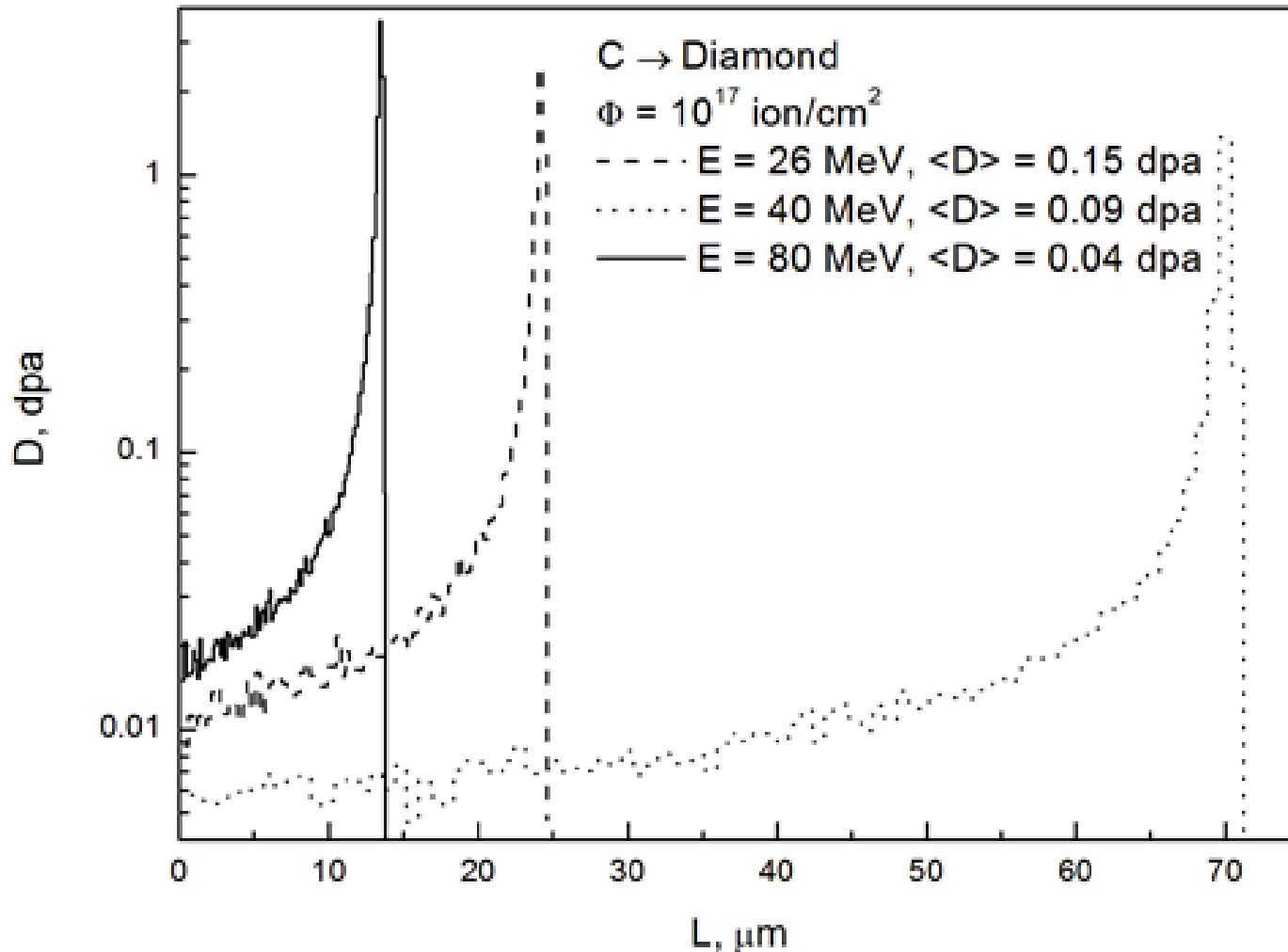
Radiation damage profiles from carbon ions with energy 26-80 MeV in copper for a dose up to $10E17$ ion/cm²



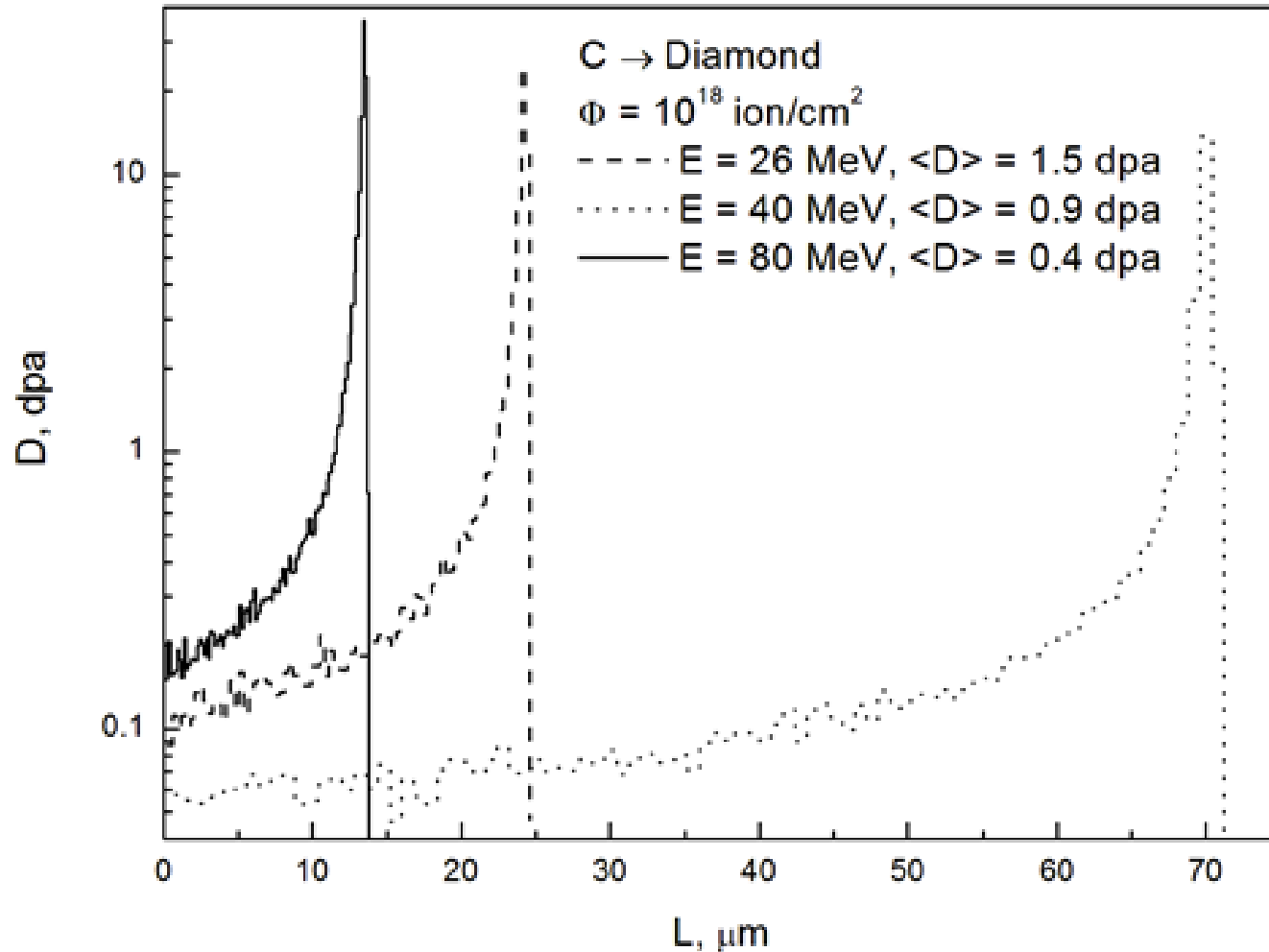
Radiation damage profile from carbon with energy 26-80 MeV in copper for a dose up to $10E18$ ion/cm²



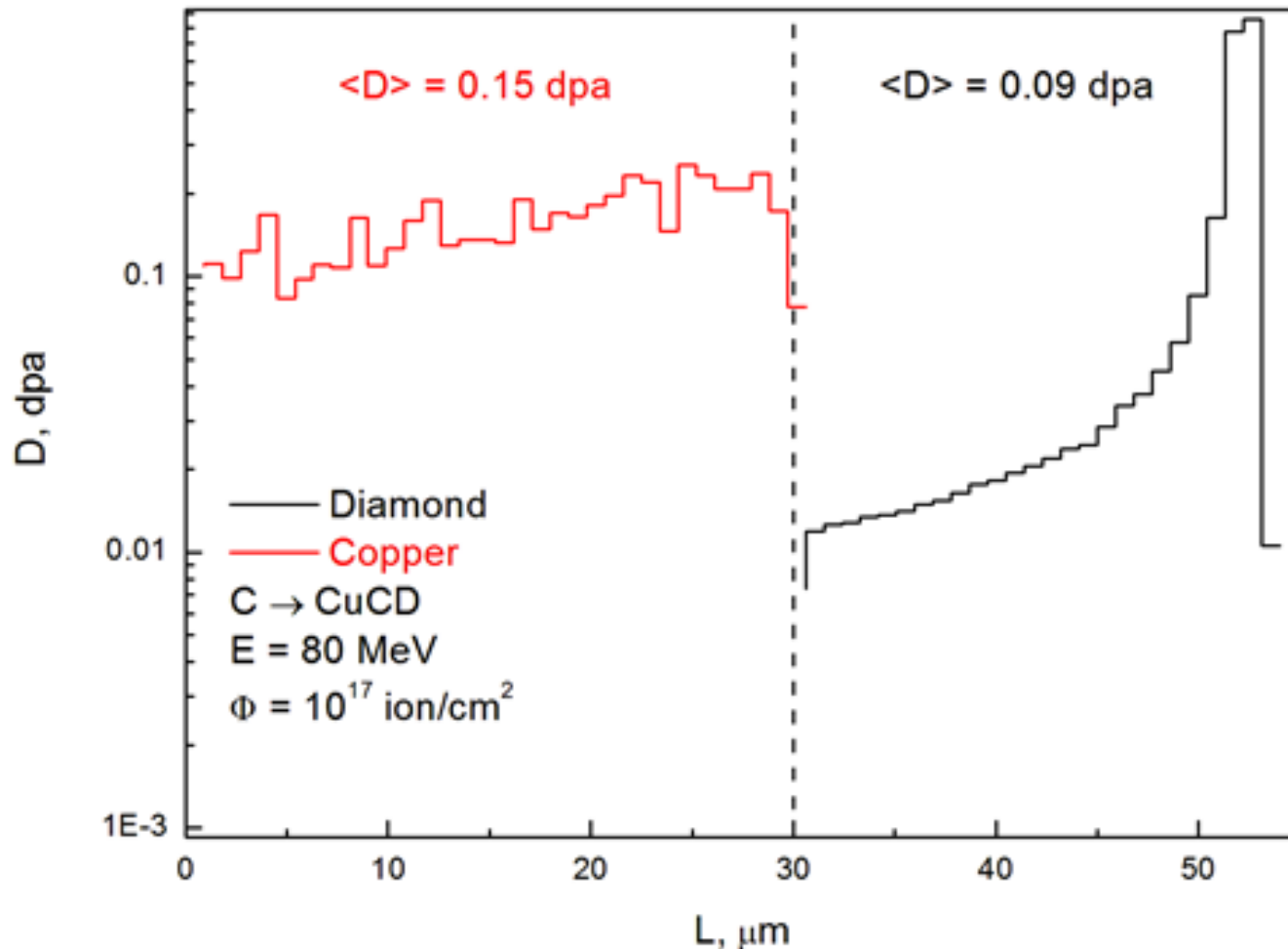
Radiation damage profiles from carbon ions with energy 26-80 MeV in diamond for a dose up to $10E17$ ion/cm²



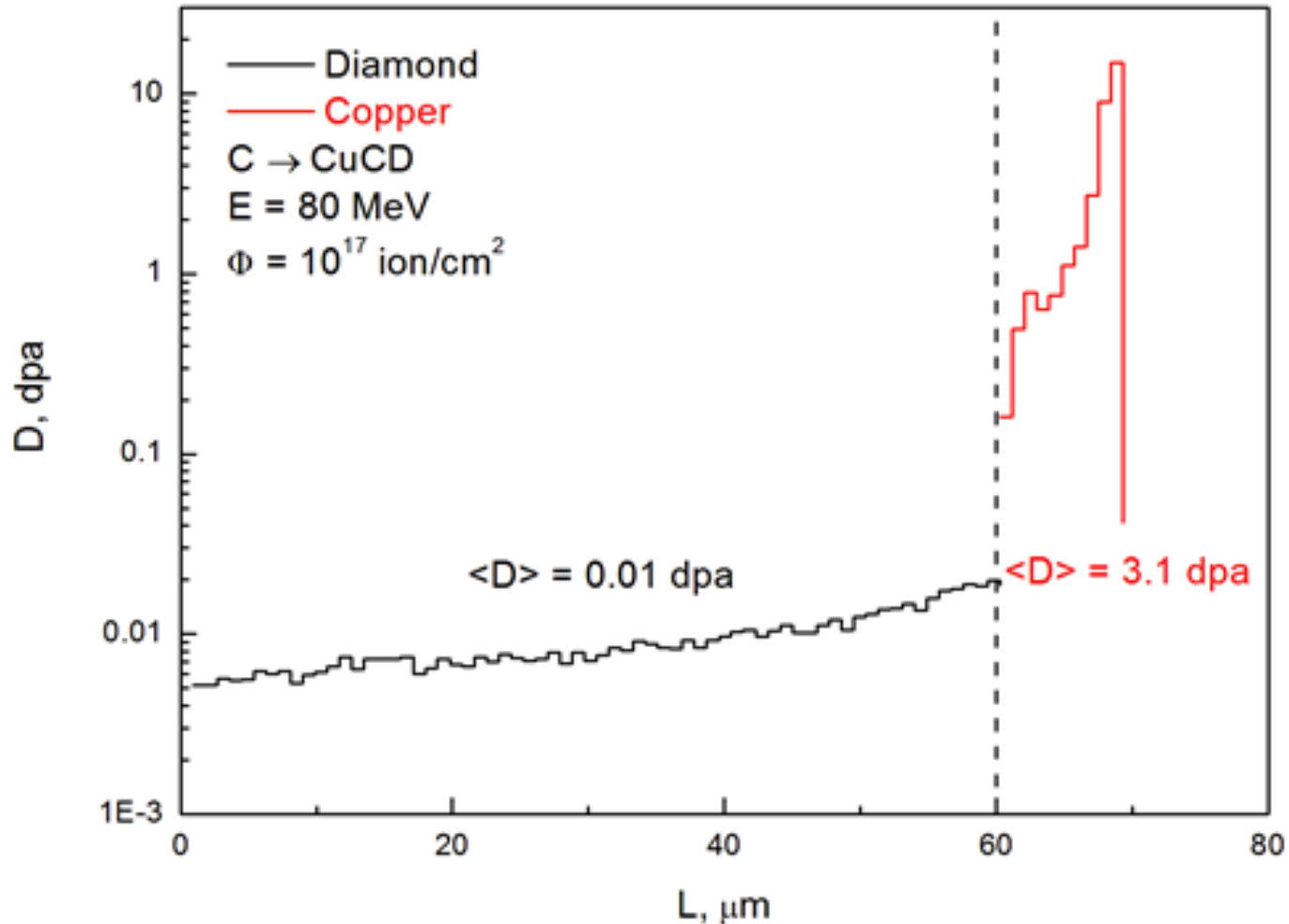
Radiation damage profiles from carbon ions with energy 26-80 MeV in diamond for a dose up to $10E18$ ion/cm²



Radiation damage profiles from carbon ions with energy 80 MeV in the layered structure of CuCD material for two different configurations of layers.



Radiation damage profile from carbon ions with energy 80 MeV in the layered structure of CuCD material for two different configurations of layers.



Conclusion

- On the basis of the proposed theoretical models of the layered structure for the irradiated materials, a number of calculations have been performed to determine radiation damage profiles.
- Calculations show that the damage levels in diamond inclusions approximately an order of magnitude lower than that of copper and molybdenum-copper alloy.
- The average damage for both irradiated materials correspond to 10^{-4} dpa for diamond inclusions and 10^{-3} dpa for copper and copper-molybdenum alloy under irradiation of 30 MeV protons to doses 10^{17} p/cm².
- Calculations carried out with the average density approach correspond to values about $3 \cdot 10^{-4}$ dpa. The ranges of protons for the materials with the layered structure and with the average density coincide within the margin of error for protons with energies greater than 10 MeV.
- Irradiation of samples with carbon ions leads to a higher levels of displacements, but the ranges of 80 MeV carbon ions are limited with 70 microns. The average damage is generally distributed within individual layers of copper or diamond and it may vary significantly for carbon ions passing the interface between diamond inclusions and metal.

Development of experimental methods for investigations of physical-mechanical properties in Mo - Diamond collimator materials before and after fast proton irradiation with the energy 30 MeV

**Determination of the density in Mo - Diamond samples
before and after 30 MeV proton beam irradiation on NRC
KI cyclotron up to dose 10E17p/cm2**

Sample, shape	Weight, g	Sizes, mm	Hydrostatic density, d, g/cm³	Density change, (d_{об.л}-d_{исх})/d_{исх}, %
O-1, cylinder	2,225/2,254*	D=10,115 H=4,225	6,569/6,585	0,2
O-2, cylinder	2,093/2,090	D=10,00 H=4,185	6,285/6,289	0,1
O-3, cylinder	2,515/2,485	D=10,225 H=4,505	6,745/6,681	- 0,9
P-1, Parallepepid	1,918/1,903	L=16,0	6,686/6,737	0,2
P-2, Parallepepid	-/1,982	L=16,0	6,686/6,705	0,3
P-3, Parallepepid	1,970/1,944	L=16,0	6,665/6,668	0,0

*** - before, * - after irradiation**

**Sizes of Mo-Diamond samples for mechanical tests based on
ASTM C1161 02c and ASTM D6272–02 Standards before
and after (*) proton irradiation**

Sample, №	h, mm	b, mm	h*, mm	b*, mm	L, mm¹⁾	S, mm²	S*, mm²	ΔS/S, %
Mo-Cu-D-1	4,40	5,68	4,41	5,70	~61	24,96	25,12	0,6
Mo-Cu-D-2	4,50	5,92	4,18	5,32	~61	26,62	26,82	0,7
Mo-Cu-D-3	4,43	5,71	4,36	5,38	~60	25,27	25,26	0,0

h-height, b-width and L-length

**Results of mechanical tests of Mo-Diamond samples
before and after (*) 30 MeV proton irradiation on
NRC KI cyclotron**

№ Sample	$F_{M(E)}$, N	$F_{M(E)}^*$, N	σ_E, MPa	σ_E^*, MPa	E^B, GPa	E^{B*}, GPa	$((E^{B*} - E^B) / E^B) \cdot 100$, %
Mo-Cu-D-1	100	50	34,2	17,1	158 ± 8	184 ± 9	16
Mo-Cu-D-2	100	50	31,2	15,6	179 ± 8	210 ± 9	17
Mo-Cu-D-3	100	50	33,8	16,9	155 ± 9	180 ± 8	16

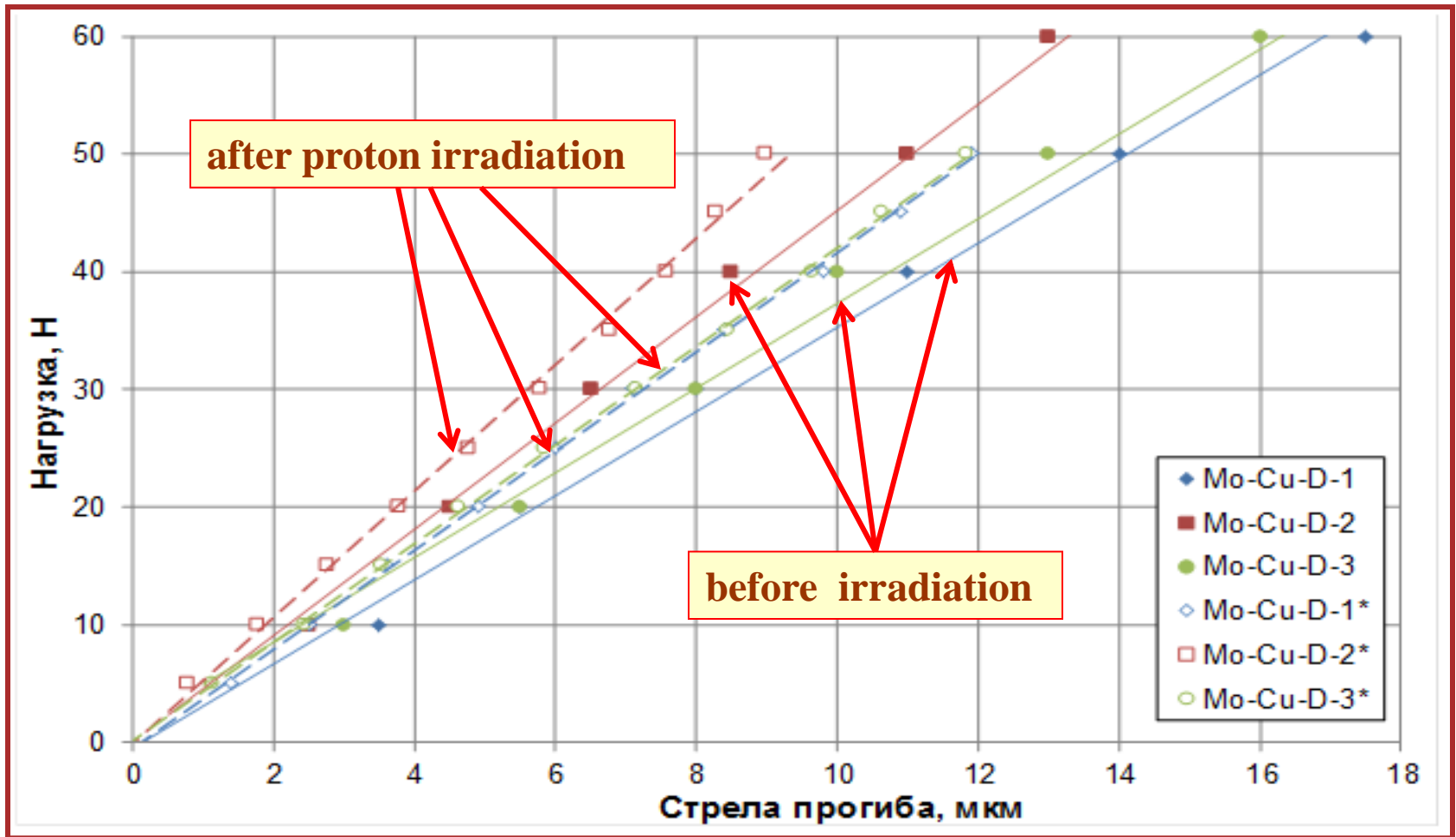
**$F_{M(E)}$, (N)- maximum applied load,
 σ_E , (MPa) – obtained maximum stress before rupture
 E^B , E^{B*} - Young's Modulus of elasticity of pure bending
of non-irradiated and (*) irradiated samples, respectively.**

Young's Modulus changes in Cu-Diamond materials before and after 30 MeV proton irradiation using four points pure deformation (bending) according ASTM C 1161-02c Standard

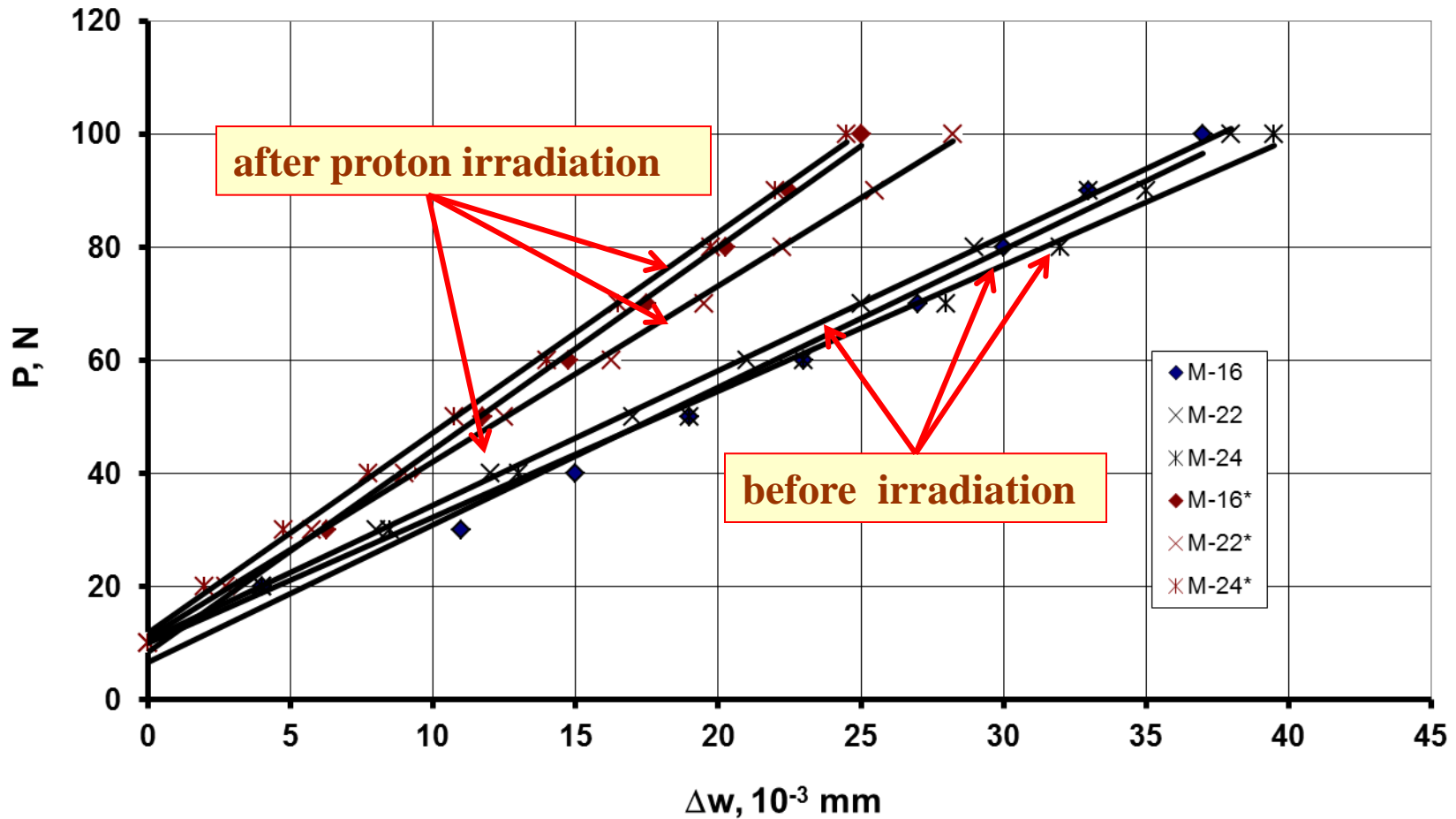
Маркировка/Назначение	F_M , N	σ_{FB} , MPa	E^H , GPA (Unirradiated)	E^H GPA (Irradiated)	% (difference)
M-18 (before)	241,5	90	272±39	-	-
M-18 (before)	413,6	127	-	-	-
M-20/ (before)	241,2	86	224±17	-	-
M-16/ (Before and after Irradiation)	101,7	35	240±52	338±28	41
M-22/ (Before and after Irradiation)	101,4	38	241±22	308±19	28
M-24 (Before and after Irradiation)	101,6	39	230±19	343±31	49

Young's Modulus changes in Cu-Diamond materials after 30 MeV proton irradiation are higher (39%) comparing with Mo-Diamond materials (16%)

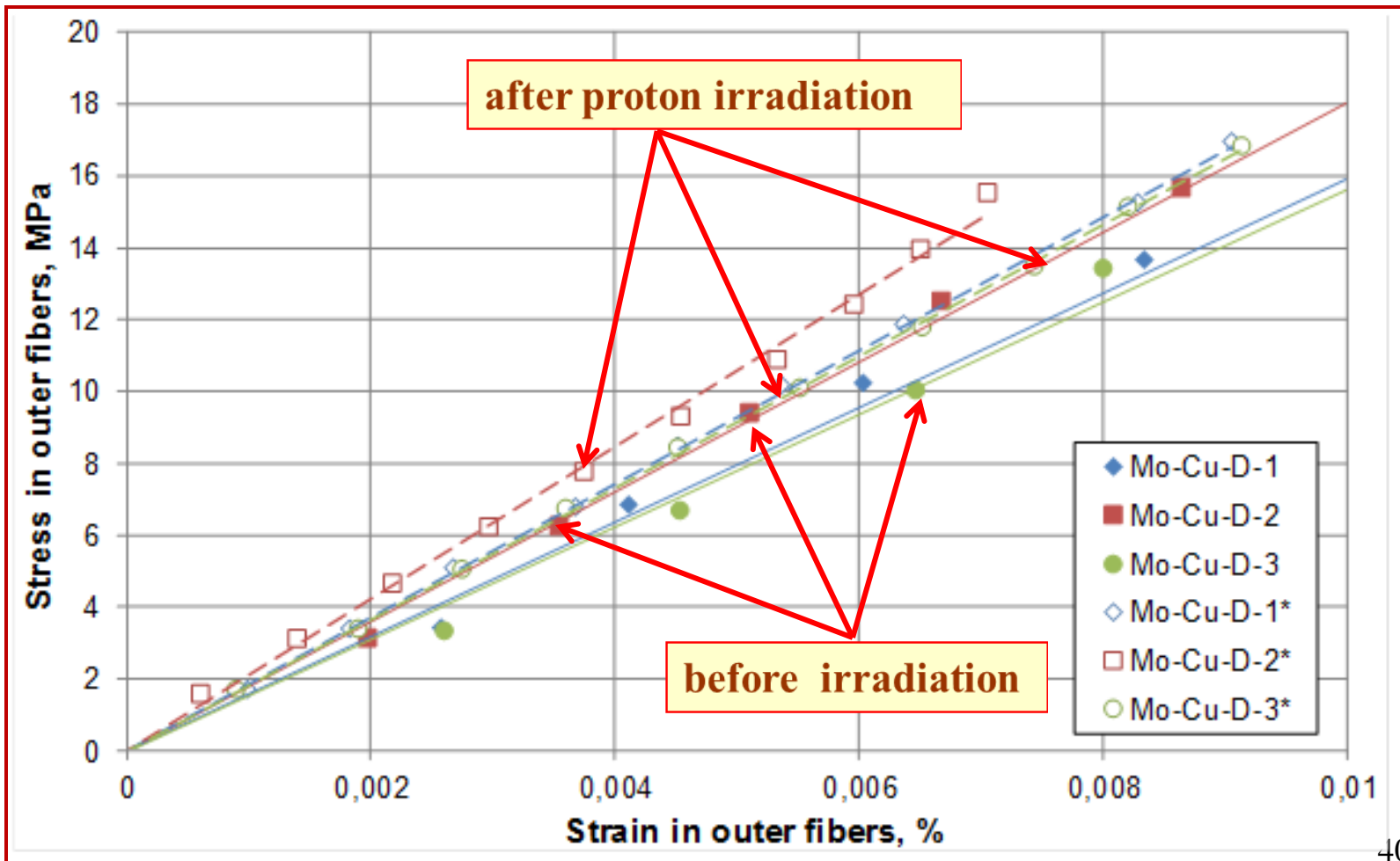
Deformation curves for irradiated (*) by 30 MeV protons and unirradiated Mo-Diamond samples for LHC collimator materials



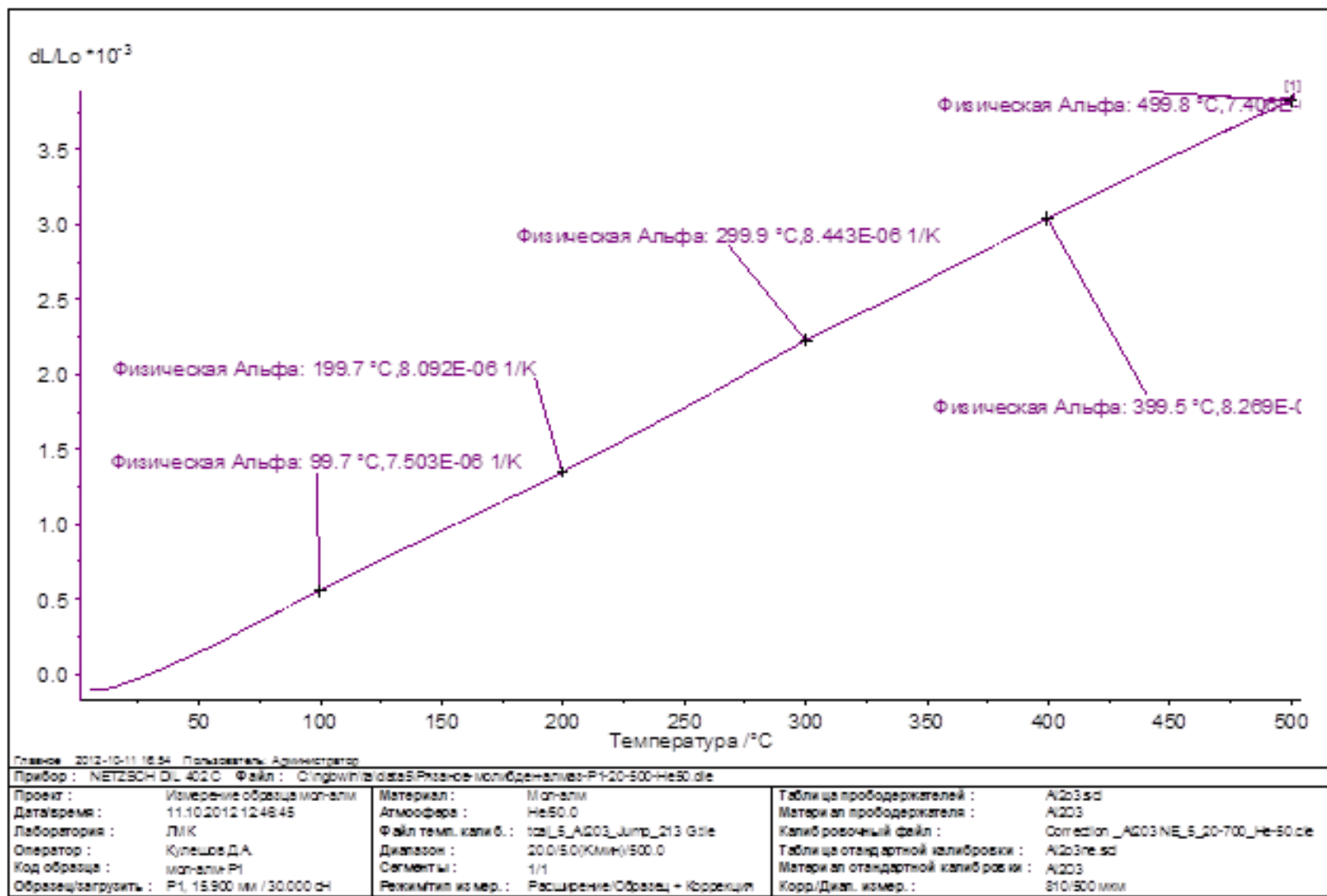
Deformation curves for irradiated by 30 MeV protons and unirradiated Cu-Diamond materials



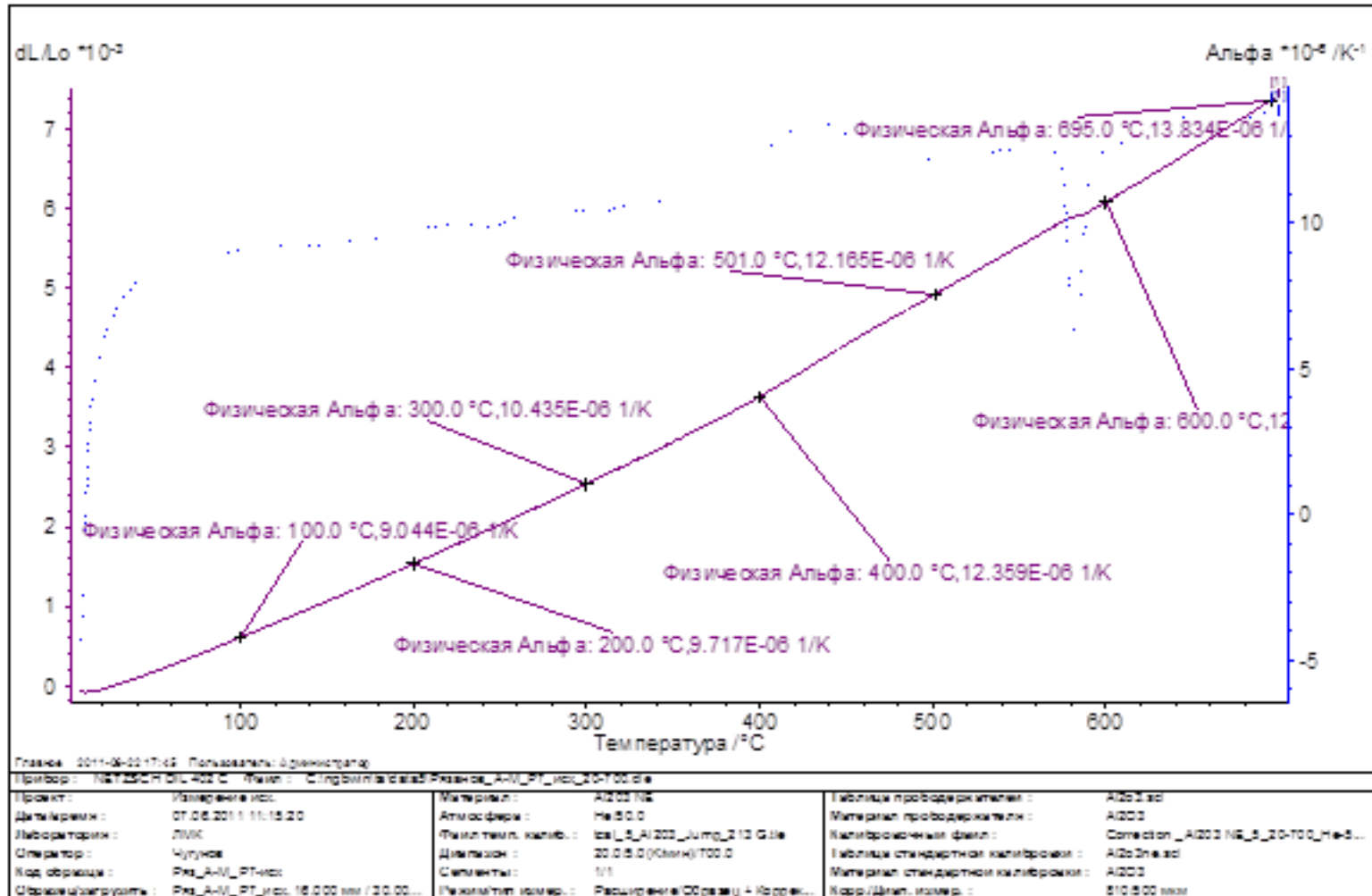
Dependence of deformation curves in outer fibers of MoCuD on applied stress for unirradiated (\blacklozenge , \blacksquare , \bullet) and irradiated by 30 MeV protons (\diamond , \square , \circ) during measurements of Young's Modulus of elasticity



Temperature dependence of thermal expansion coefficient in Mo-Diamond collimator material (Sample P1)

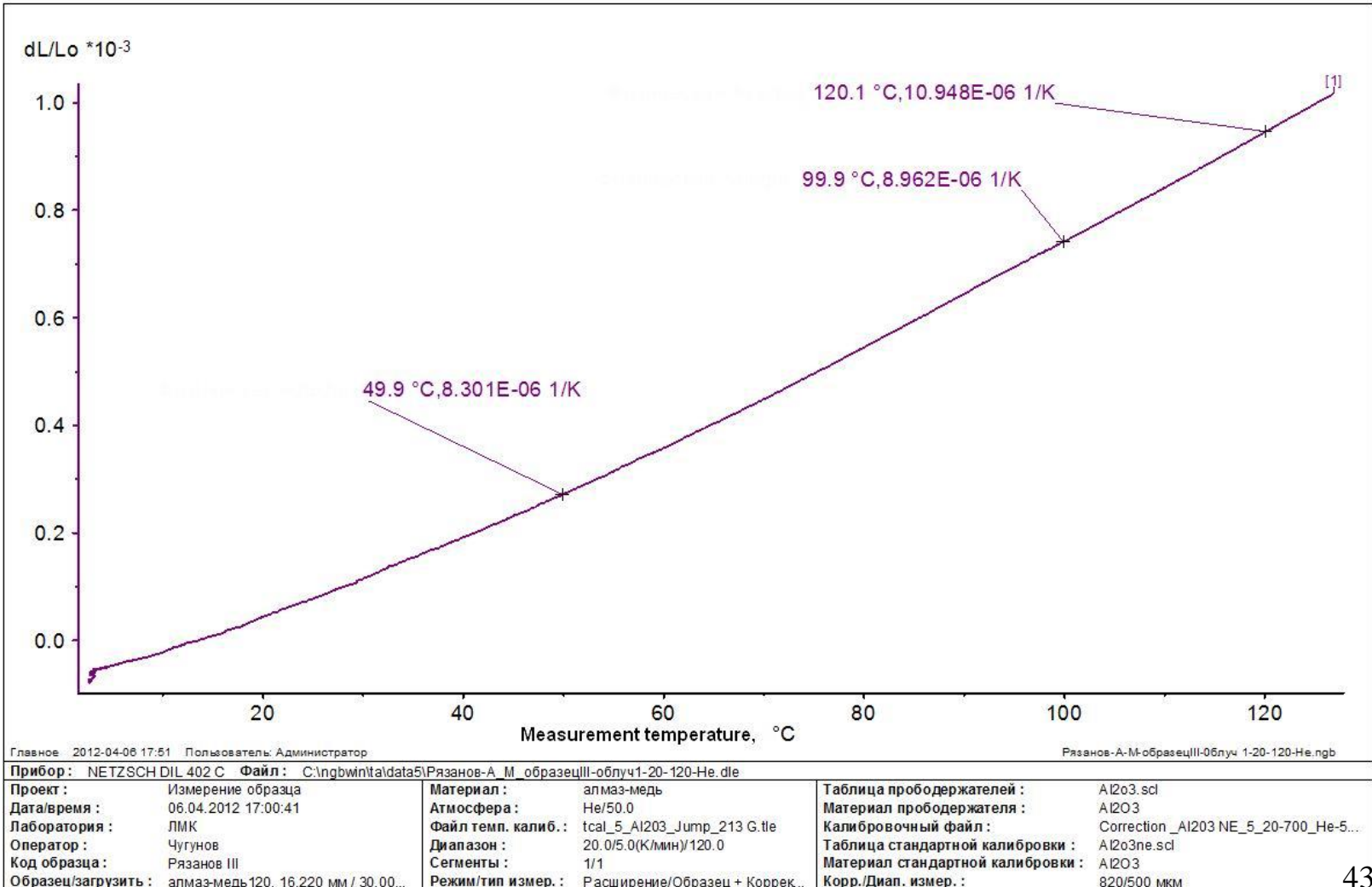


Temperature dependence of thermal expansion coefficient in Cu-Diamond collimator material (Sample P7)



In Cu-D is higher comparing with Mo-D

Measurements of thermal expansion coefficient in Cu-Diamond collimator materials after 30 MeV proton beam irradiation at $T = 100^{\circ}\text{C}$ on NRC KI cyclotron up to dose $10\text{E}17\text{p}/\text{cm}^2$



Comparison of thermal expansion physical coefficient (α) in Cu-Diamond collimator materials before and after 30 MeV proton beam irradiation at T =100°C on NRC KI cyclotron up to dose 10E17p/cm2

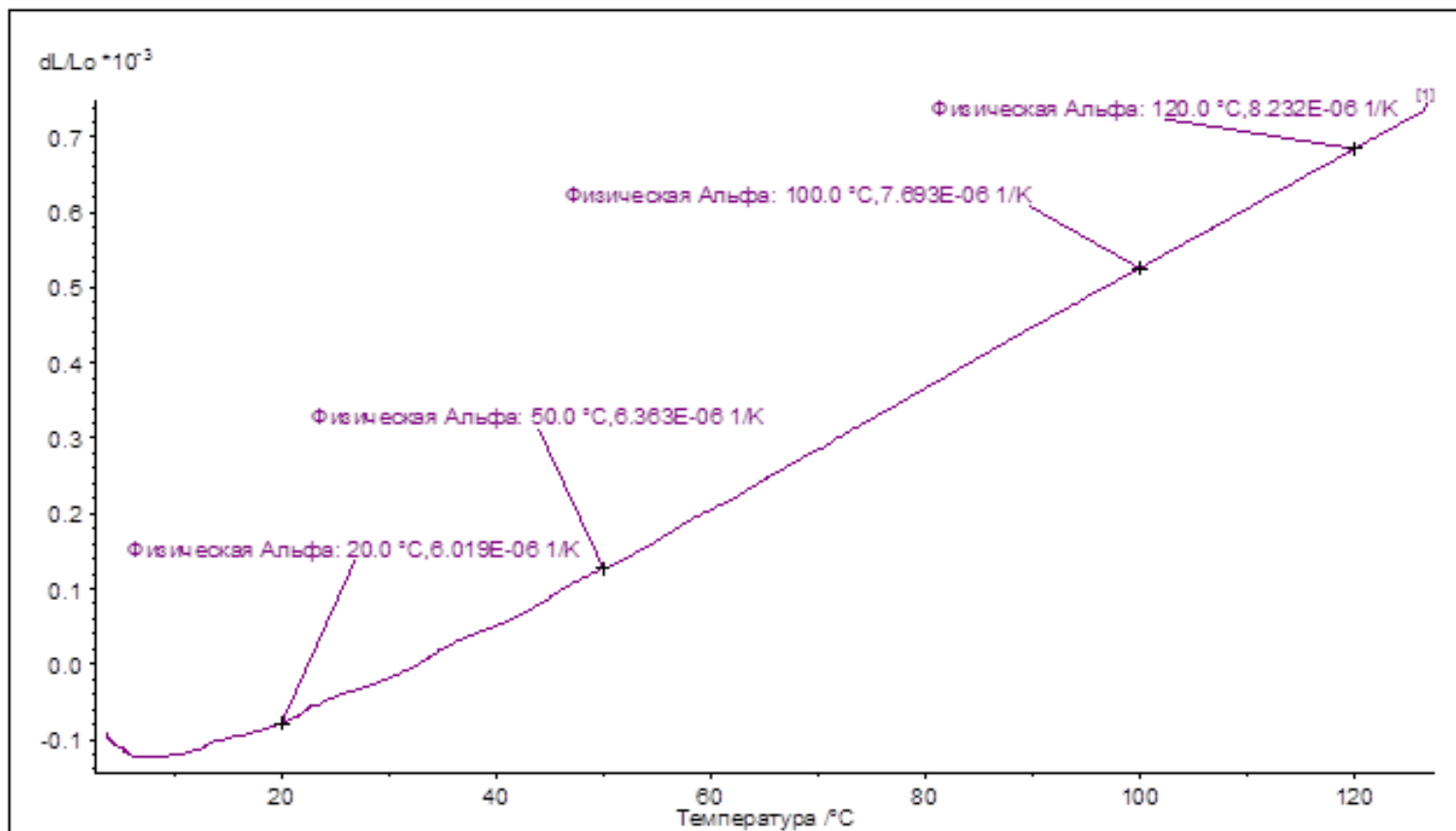
T_{measurement}, °C	50	100	120
α_{ineatial}, 1/K, 10⁻⁶	7,8	8,5	9,0
α_{irradiated}, 1/K, 10⁻⁶	8,3	9,0	10,9

The increasing of TEC (α) is equal 5-7 %

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

T, oC	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
MoCu-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
MoCu-Diamond (Sample P3)	8,3	8,5	9,0	8,7	7,6

Temperature dependence of thermal expansion coefficient in irradiated by 30 MeV protons and dose 10E17 1/cm2 Mo-Diamond collimator material (Sample P1)

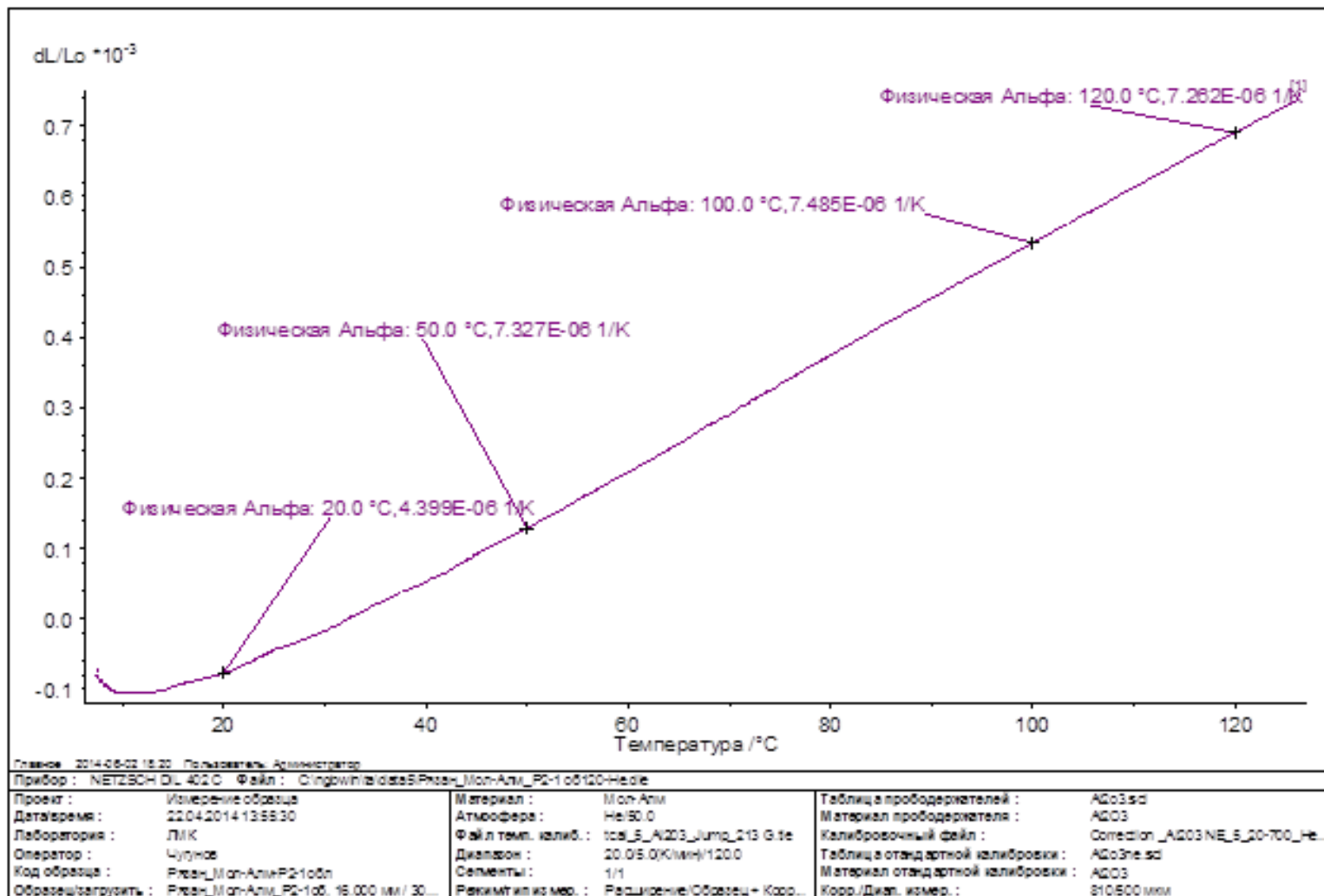


Главное: 2014-08-02 15:20 Пользователь: Администратор

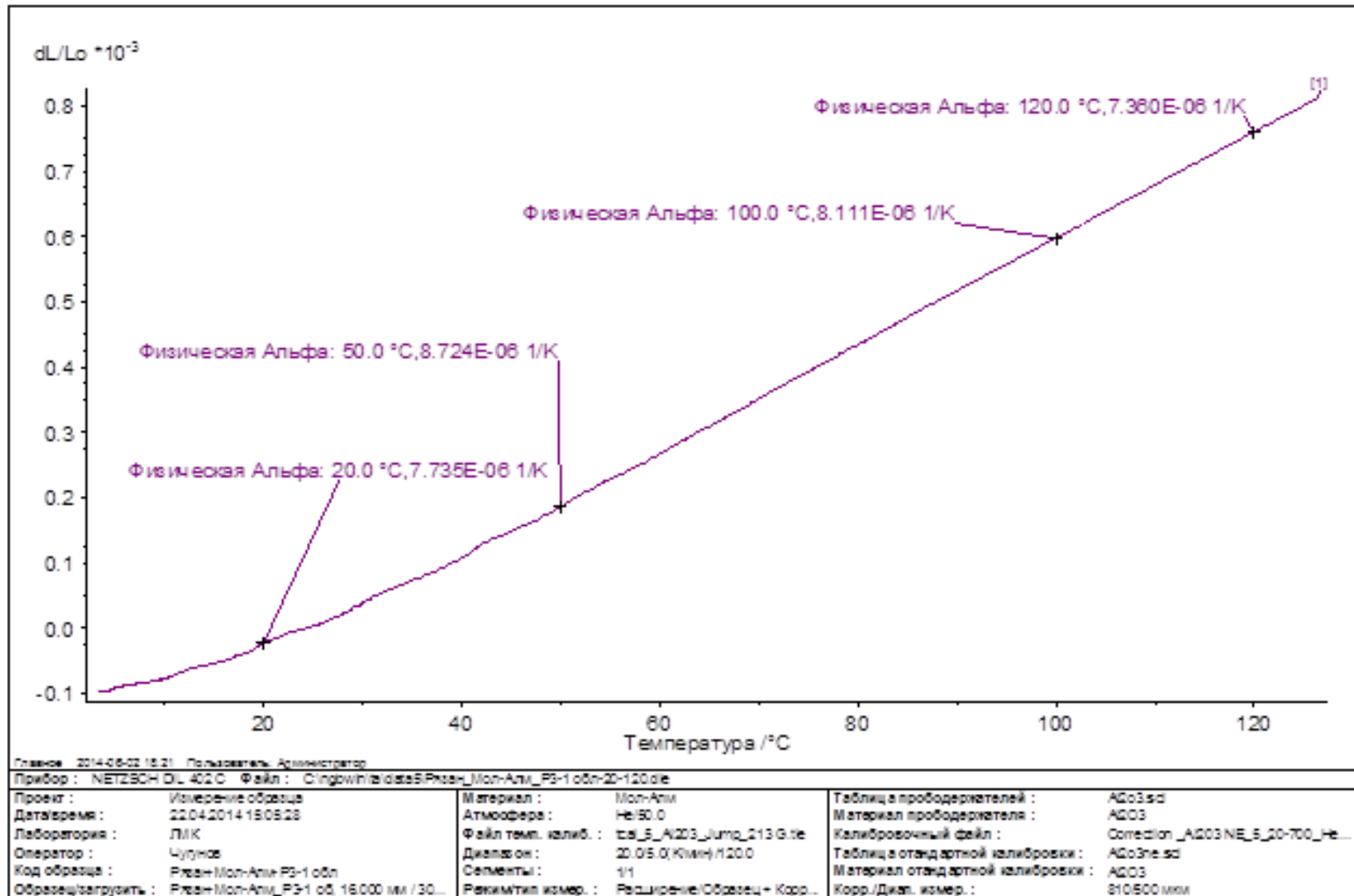
Прибор: NETZSCH DL 402C Файл: C:\ngwin\data5\Резул_MoD_Am_P1-1 обл-120-He.de

Проект: Измерение образца	Материал: Моn-Аm	Таблица прободержателей: A203.scf
Дата/время: 22.04.2014 12:58:01	Атмосфера: He/50.0	Материал прободержателя: A203
Лаборатория: ЛМК	Файл тем. калиб.: tsdLS_A203_Jump_213 G.te	Калибровочный файл: Correction_A203_NE_S_20-700_He...
Оператор: Чулюков	Диаметр: 20.050(КМин)/1200	Таблица стандартной калибровки: A203nescl
Код образца: P1e_MoD_Am_P1_1 обл	Семплеты: 1/1	Материал стандартной калибровки: A203
Образец/загрузчик: P1e_MoD_Am_P1-1 обл, 16.000 мм/ 30...	Режим тип из мер.: Расширение/Образец + Корр...	Корр.Диал. из мер.: 810/500 мм

Temperature dependence of thermal expansion coefficient in irradiated by 30 MeV protons and dose 10E17 1/cm2 Mo-Diamond collimator material (Sample P2)



Temperature dependence of thermal expansion coefficient in irradiated by 30 MeV protons and dose 10E17 1/cm2 Mo-Diamond collimator material (Sample P3)



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

T, °C	20	50	100	120
Unirradiated	$\alpha, 10^{-6} \text{ 1/K}$			
Sample 1			7,5	
Sample 2			8,1	
Sample 3			8,3	
Irradiated	$\alpha, 10^{-6} \text{ 1/K (growth)}$			
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

Thermal expansion coefficient in Mo-Diamond materials after 30 MeV
proton irradiation is less comparing with unirradiated material

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
$\rho, 10^{-6}, \text{Om}\cdot\text{m}$	110	142	85	113	91	137
$(\rho_{\text{irr}} - \rho_{\text{in}}) / \rho_{\text{in}}, \%$	29		33		51	

The increasing of ER in Mo-D after proton irradiation is larger (37,7%)

Measurements of electrical resistivity in Cu-Diamond collimator materials before and after 30 MeV proton beam irradiation on NRC KI cyclotron up to dose $10E17p/cm^2$

Sample	I		II		III	
Condition	Initial	Irradiated	Initial	Irradiated	Initial	Irradiated
$\rho, 10^{-6},$ $Om \cdot m$	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

TEM study of Mo - Diamond composite

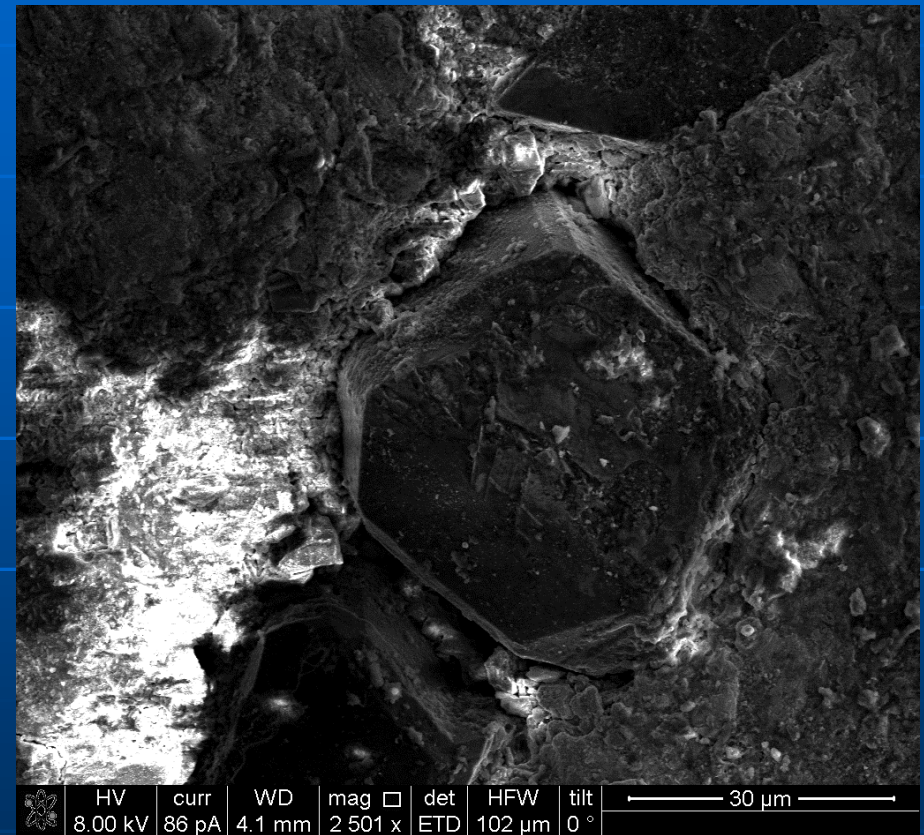
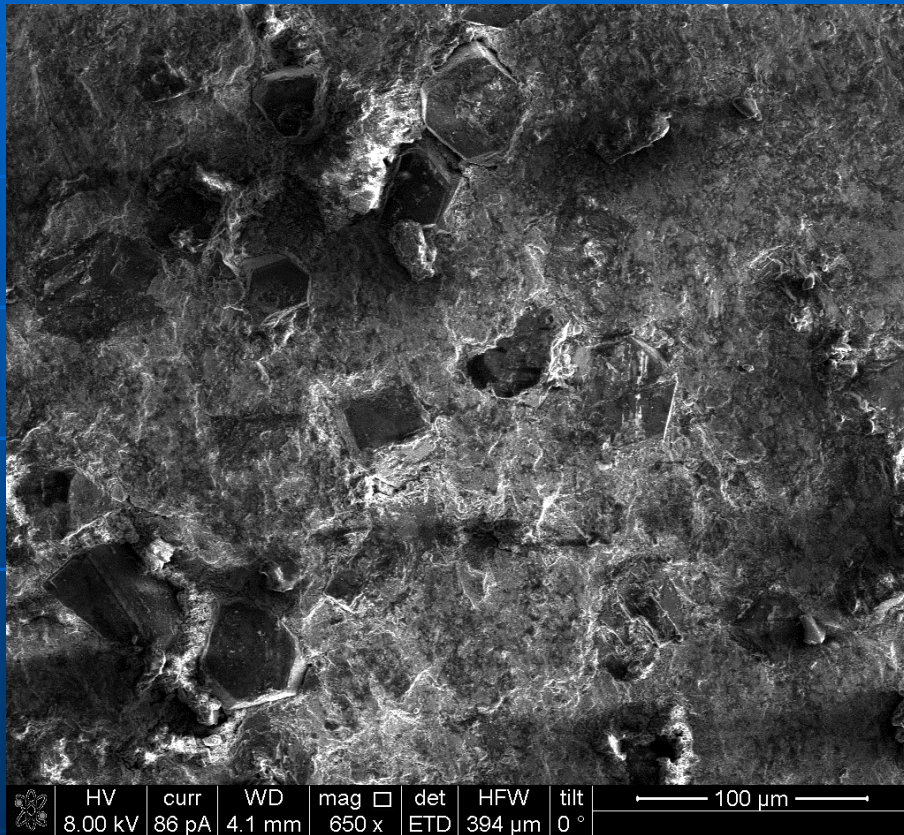
sample preparation

Cross-sectional samples for TEM were prepared by focus ion beam (FIB) in the Helios (FEI, US) dual-beam electron-ion microscope. The Ga⁺ ions energy during sample prep was 30 keV in the beginning and 2 keV at the end of the procedure.

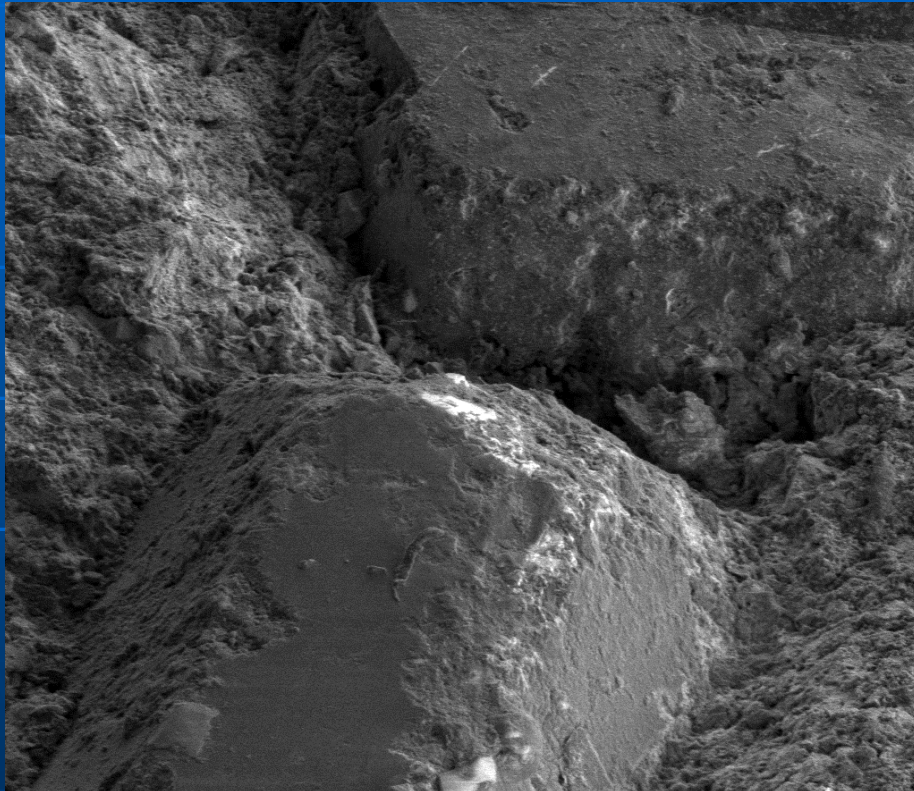
Transmission electron microscopy/Scanning transmission electron microscopy (TEM/STEM)

TEM/STEM study were performed in TITAN 80-300 TEM/STEM (FEI, US) operated at 300 kV and equipped with Cs probe corrector system, energy dispersive X-ray spectrometer (EDXS) (EDAX, US), electron energy loss spectrometer (EELS), (Gatan, US) and high angle annular dark field (HAADF) detector (Fischione, US)

The Scanning Electron Microscopy (SEM) study of Mo –Diamond composite




The Scanning Electron Microscopy (SEM) study of Mo –Diamond composite



	HV	curr	WD	mag	det	HFW	tilt
2.00 kV	86 pA	4.3 mm	5 008 x	ETD	51.1 μm	52 °	

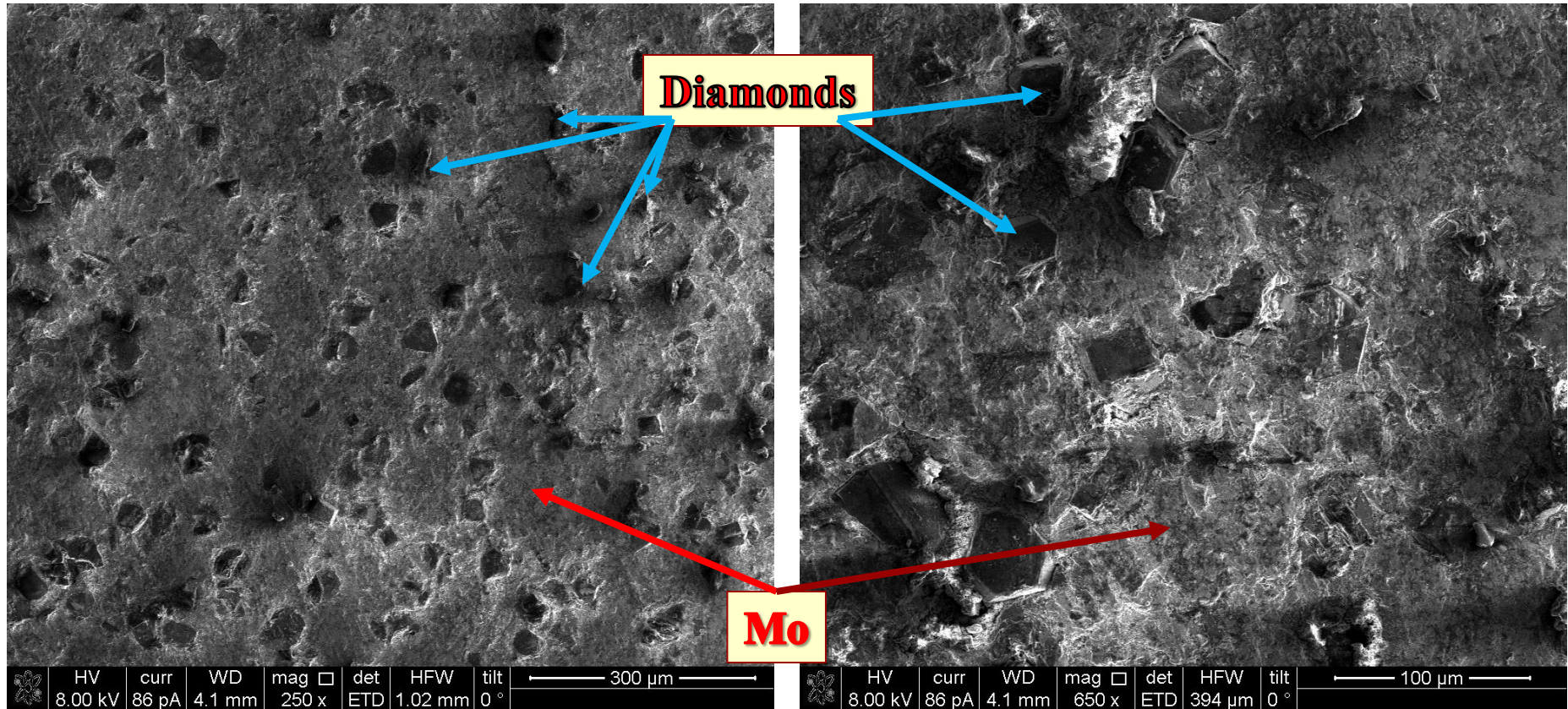
10 μm



	HV	curr	WD	mag	det	HFW	tilt
2.00 kV	86 pA	4.3 mm	1 966 x	TLD	130 μm	52 °	

40 μm

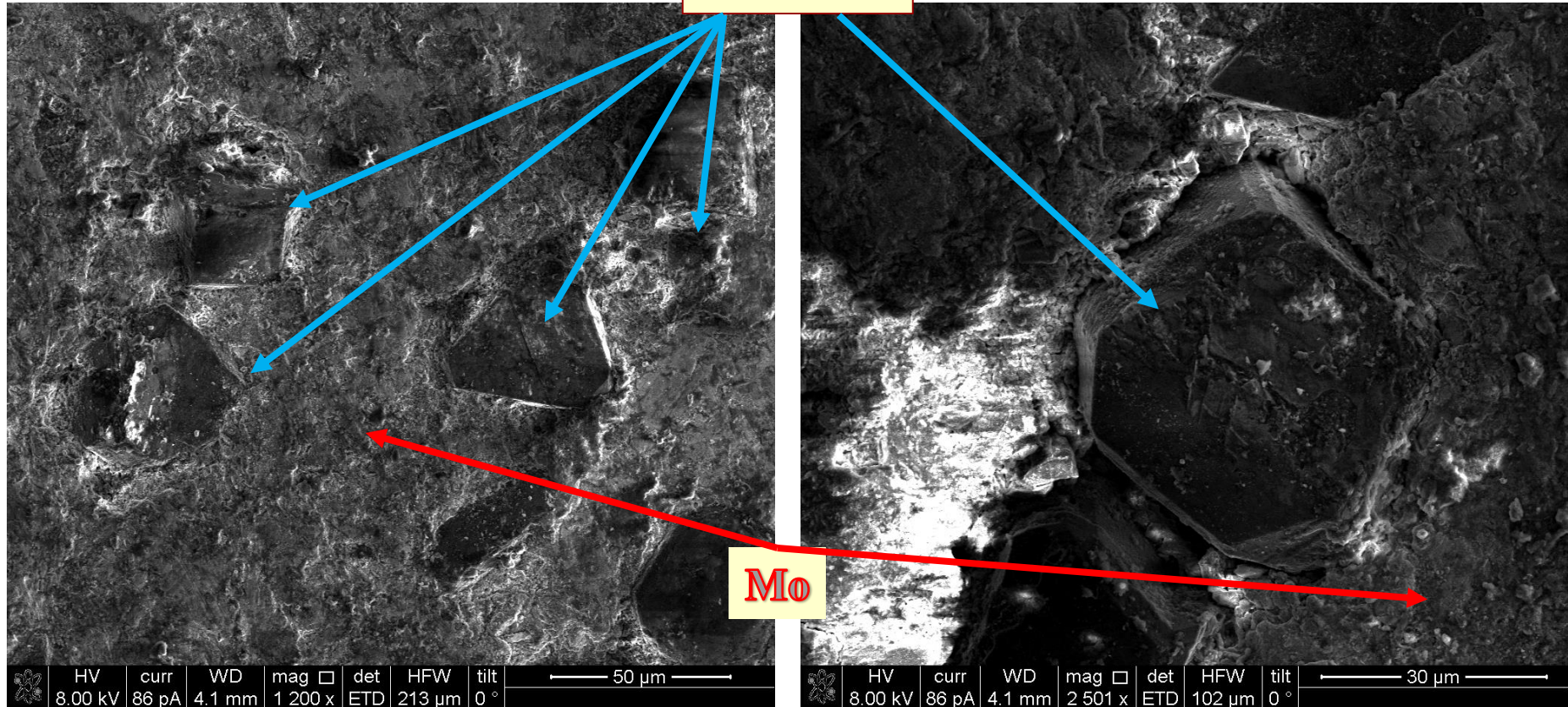
The Scanning Electron Microscopy (SEM) study of Mo-Diamond composite



SEM (secondary electrons -SE) images of the Mo-diamond composite

The SEM study

Diamonds

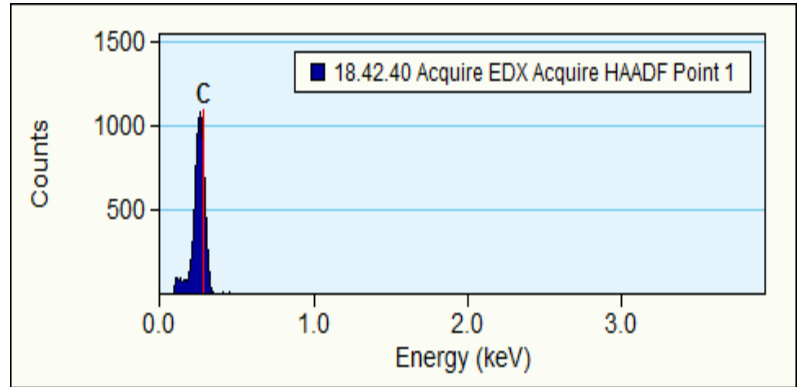


Mo

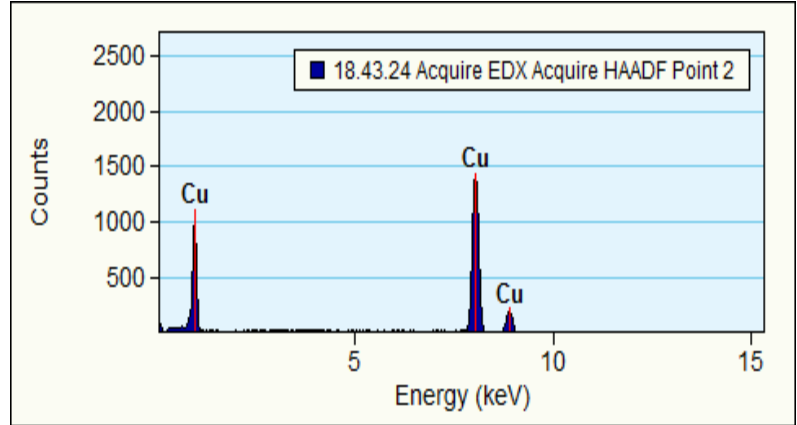
SEM (secondary electrons -SE) images of the Mo - diamond composite

The STEM and microanalysis (EDXS) study

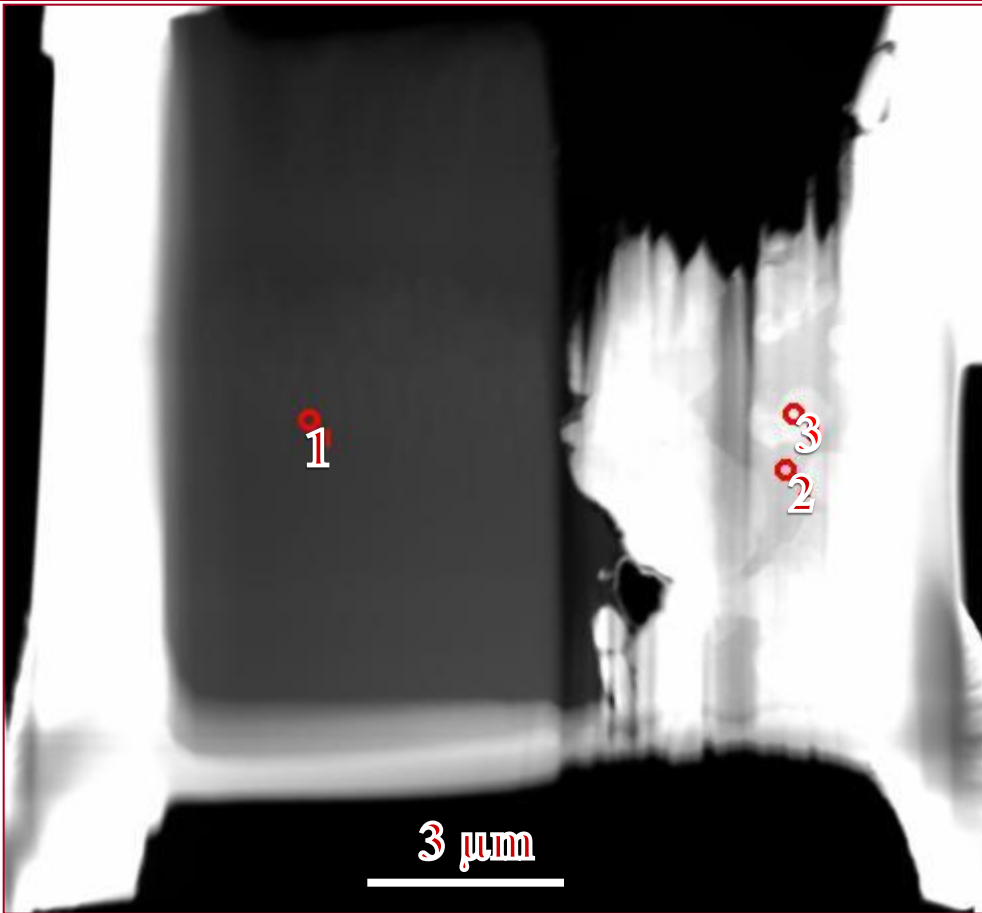
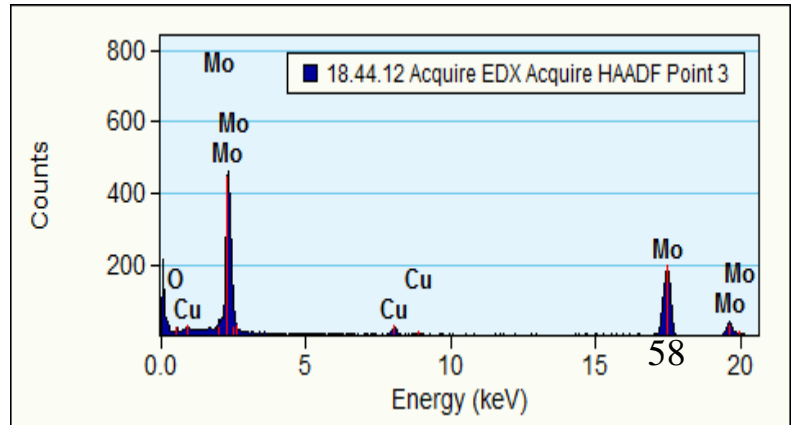
1



2

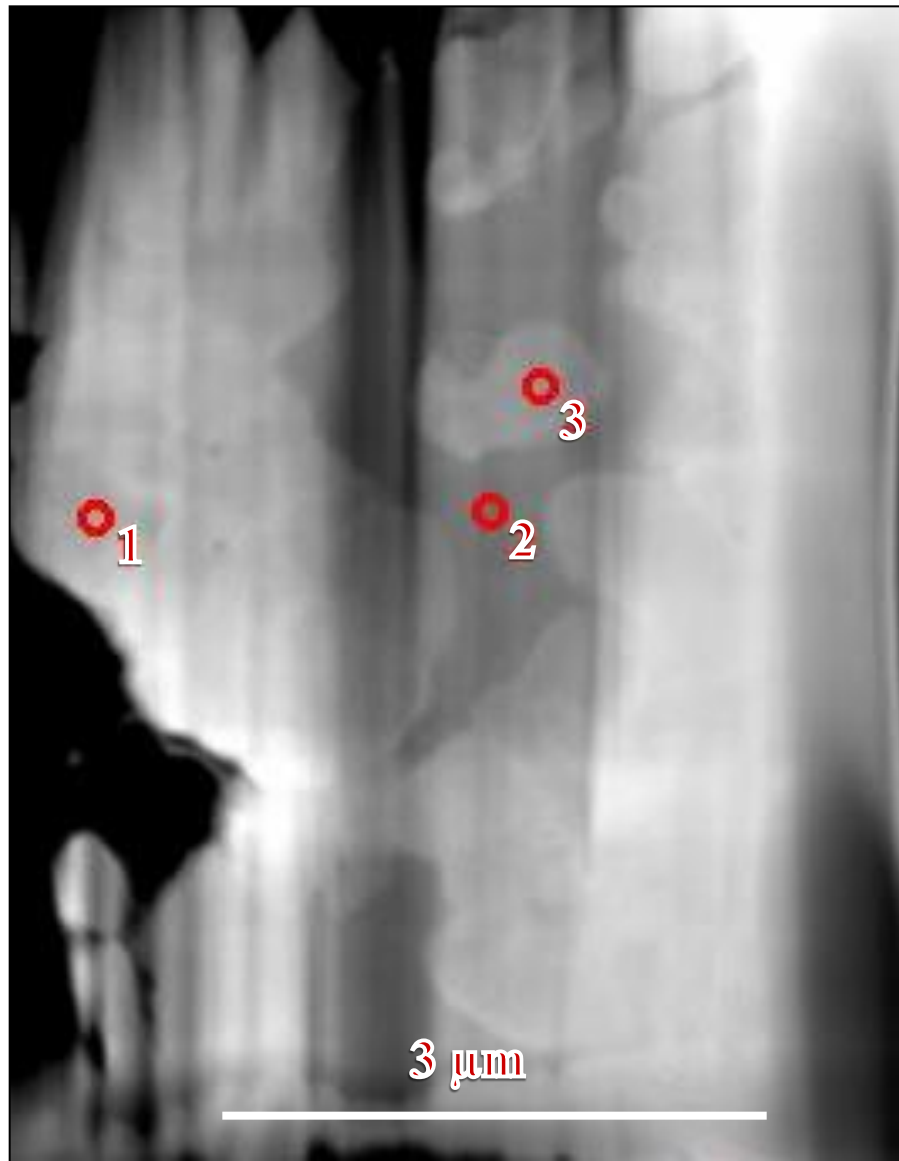


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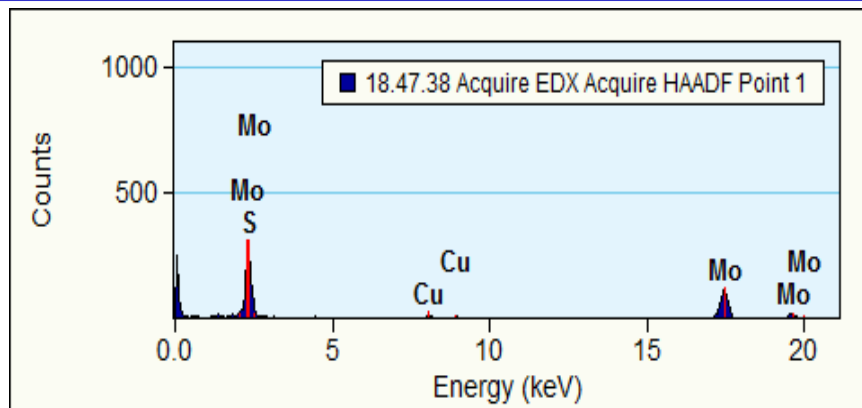


The Dark field (HAADF - High angle annular dark field) STEM image of the sample and results of EDXS analysis The microanalysis results demonstrated that left part of sample was diamond and right part –Mo-Cu alloy. The brighter areas corresponds to Mo and darker - to Cu.

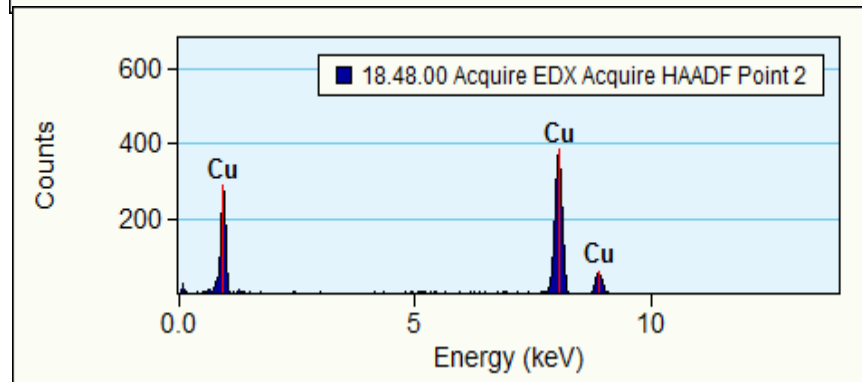
The STEM and microanalysis (EDXS) study



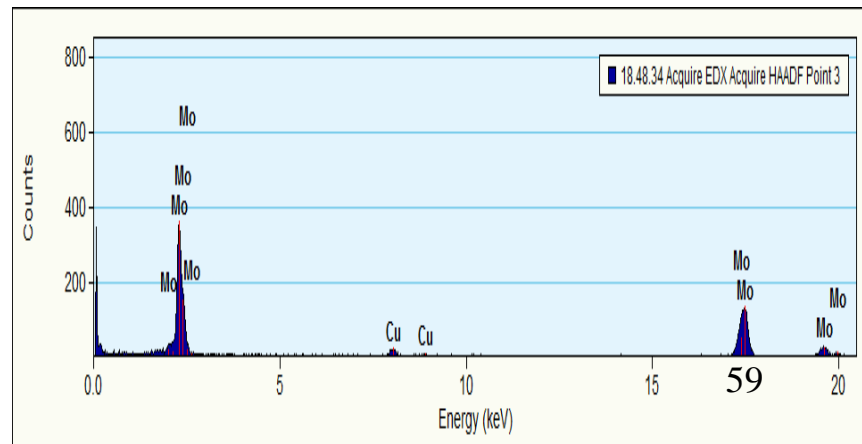
1



2

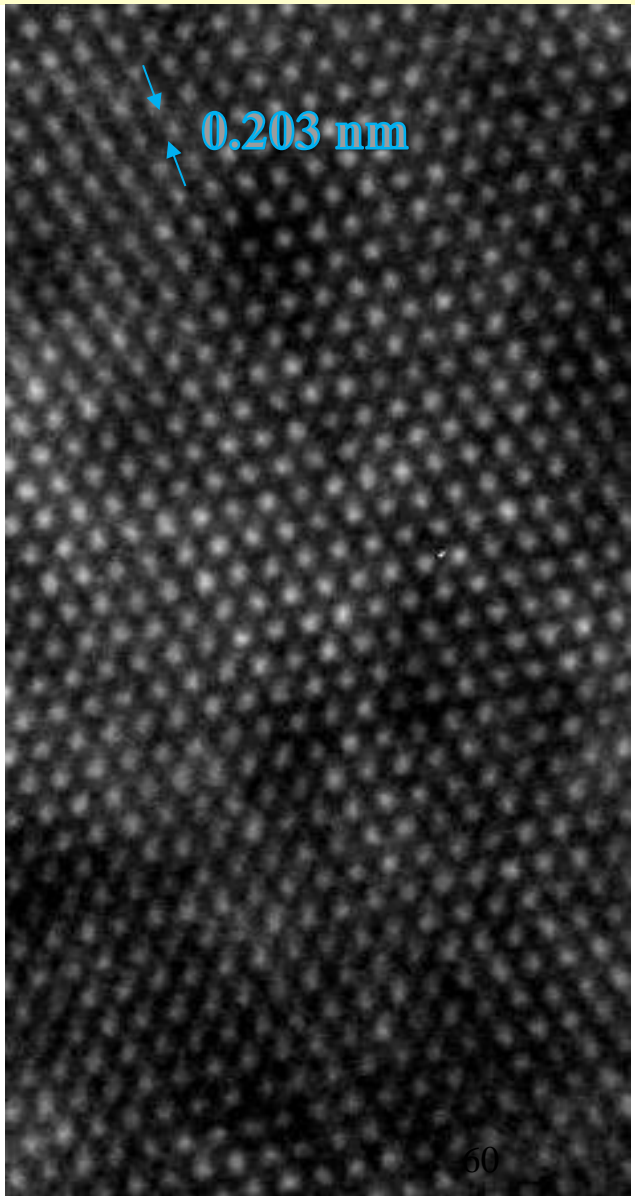
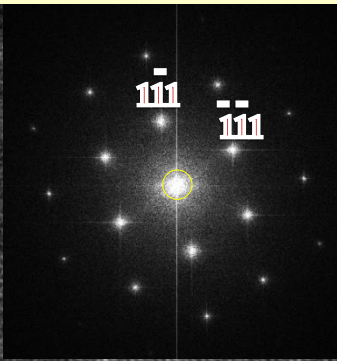
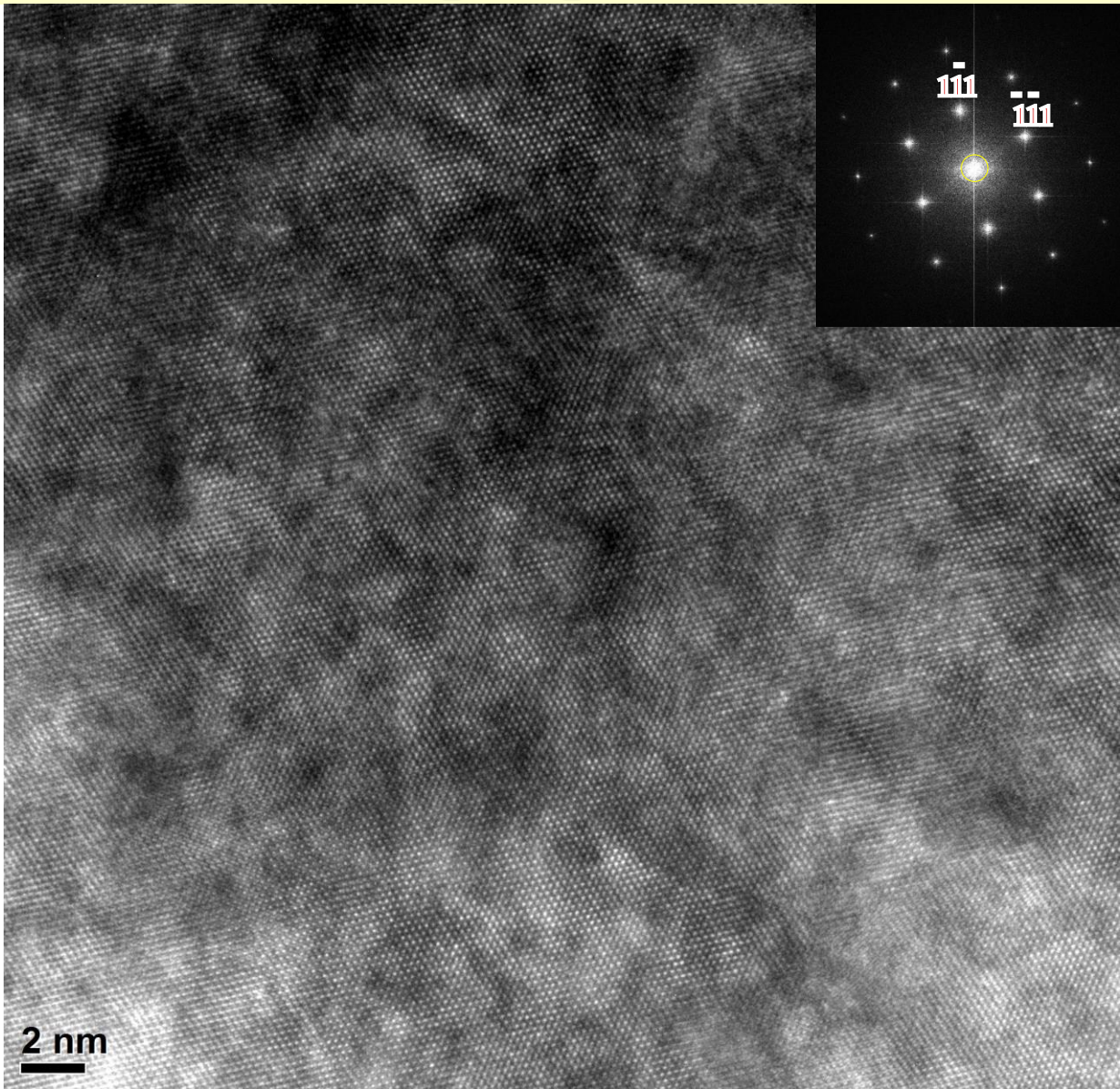


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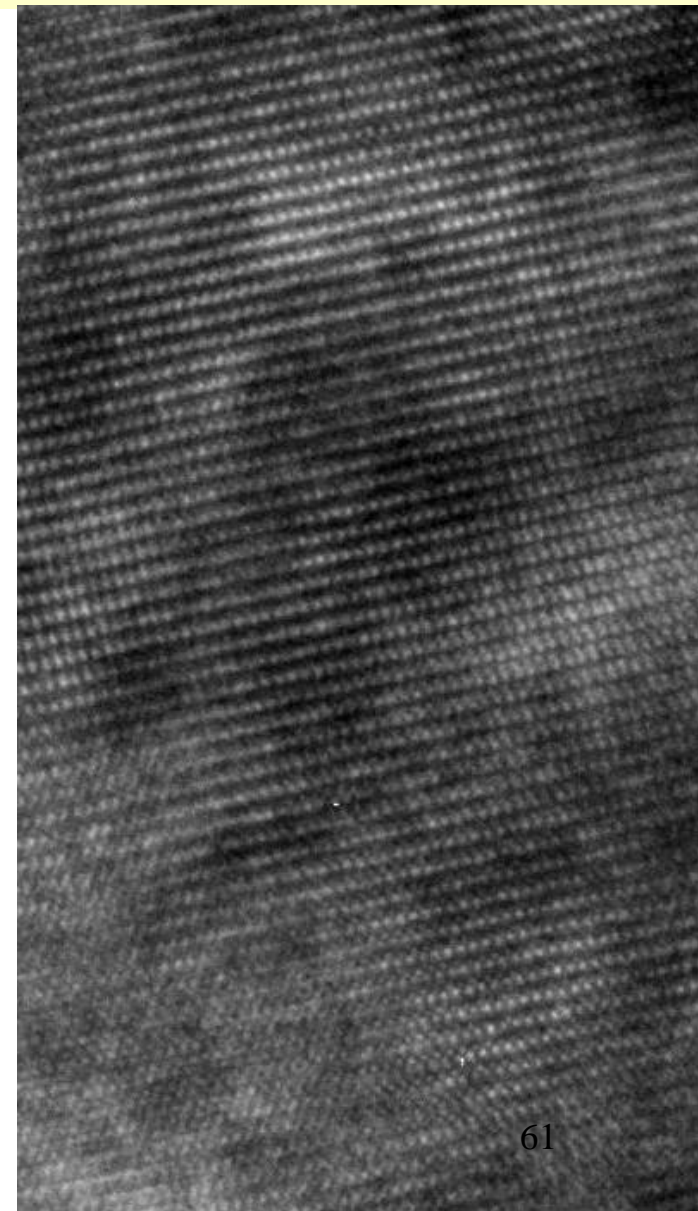
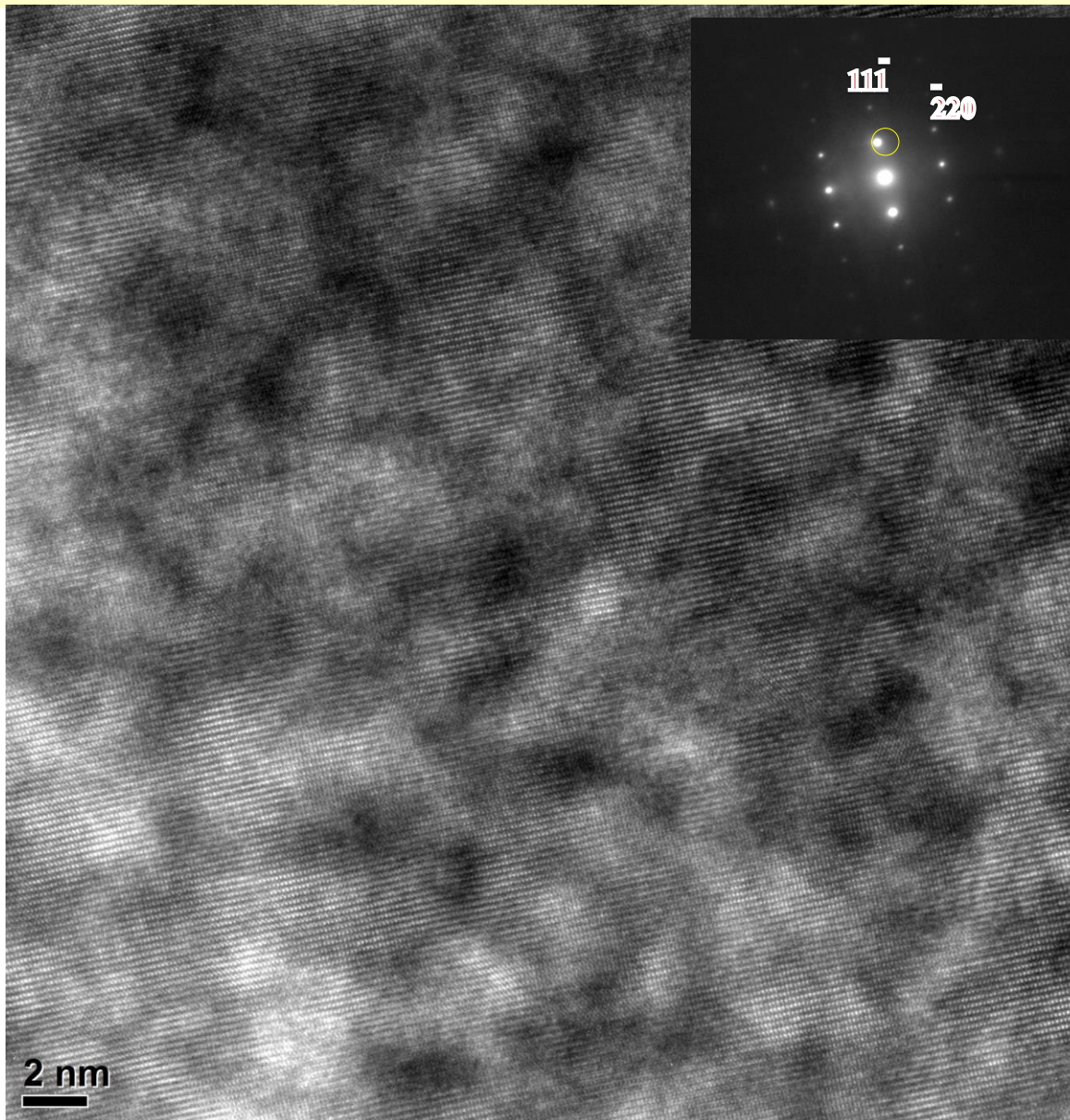


The HAADF STEM mage of the sample and results of EDXS analysis

The bright field (BF) High resolution (HRTEM) image of a diamond in $B=[001]$ zone axis and Fast Fourier Transform



The bright field (BF) High resolution (HRTEM) image of a diamond in $B=[112]$ zone axis and Electron diffraction



Development of experimental methods for investigations of mechanical properties in Molybdenum-Copper - Diamond collimator materials before and after fast proton irradiation with the energy 30 MeV up to dose $10E17$ 1/cm²

Elastic modulus tests

Development of methodology for elastic modulus tests were conducted on high-density graphite samples (MPG-6), by comparing quantities of graphite elastic modulus, measured in a variety of three ways:

- 1) dynamic method,**
- 2) axial compression method with an add-on detector,**
- 3) tests on a four-point bend with measuring deformation on traverse displacement.**

Elastic modulus measurement by pure bending

Elastic modulus calculation by pure bending was conducted in accordance with ASTM D6272–02 standard for the applied facility:

$$E^H = 0.17 \frac{l^3 m}{bh^3}$$

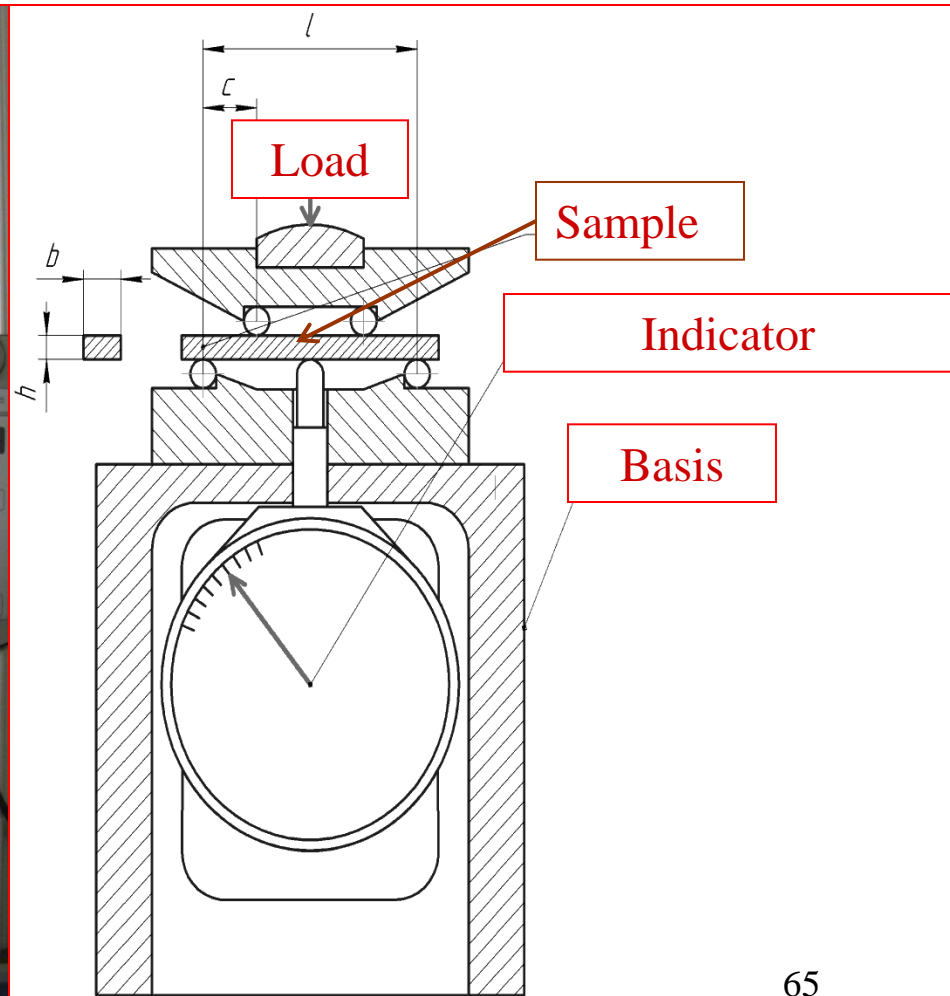
where E^H - elastic modulus, MPa;

m - inclination of the initial straight-line portion of loading curve, N/mm;

l - distance between lower bearings, mm (see pic. 4.1);

b, h - samples' breadth and height, accordingly, mm;

**Equipment for measurements Young's Modulus changes before and after
30 MeV proton irradiation using four points pure deformation (bending)
according ASTM C 1161-02c. standard**



Mo – Cu - Diamond sample sizes

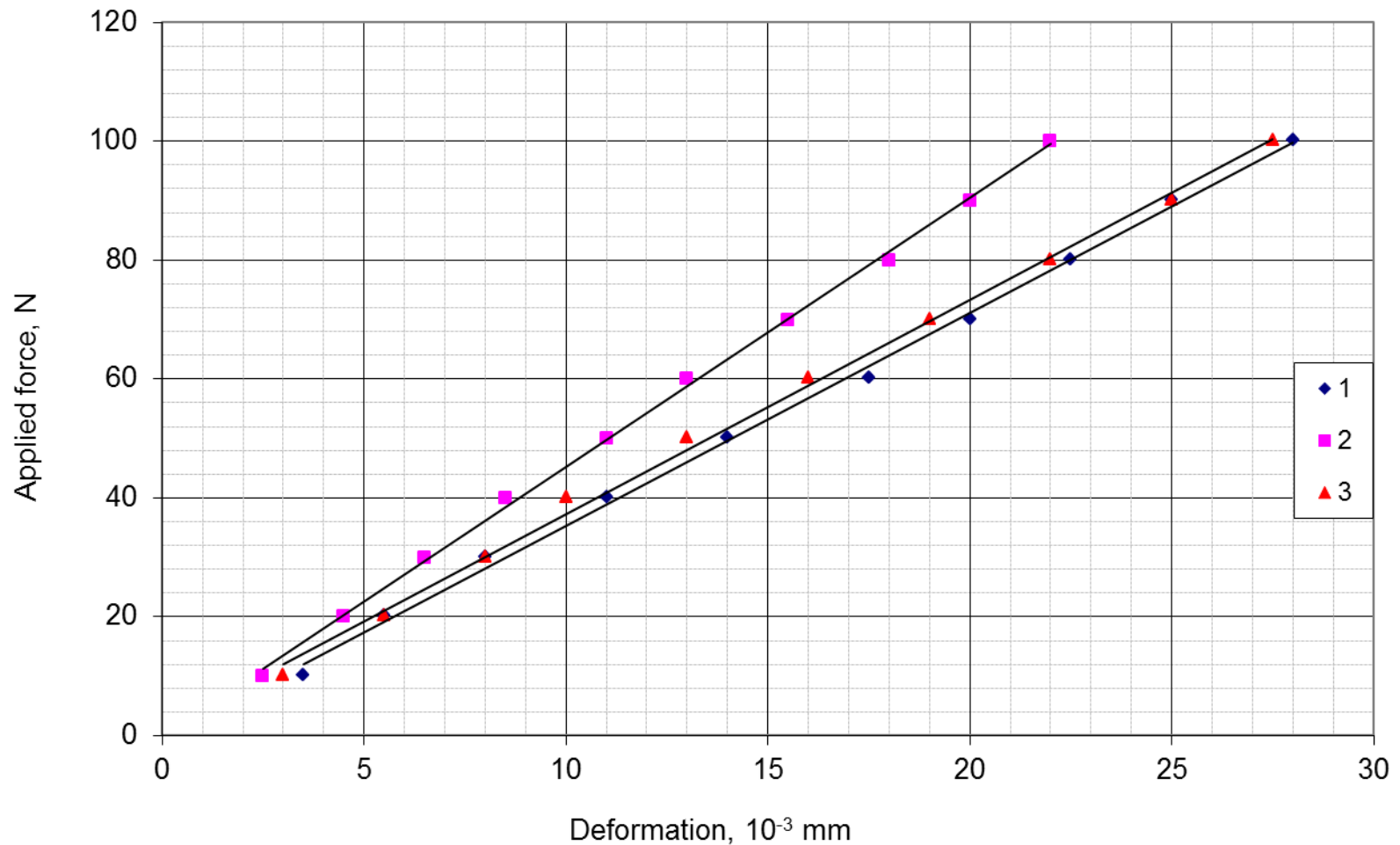
Sample №	h, mm	b, mm	L, mm1)
1	4,40	5,68	~61
2	4,50	5,92	~60
3	4,43	5,71	~60

Samples were not polished and had a surface due to production technique;

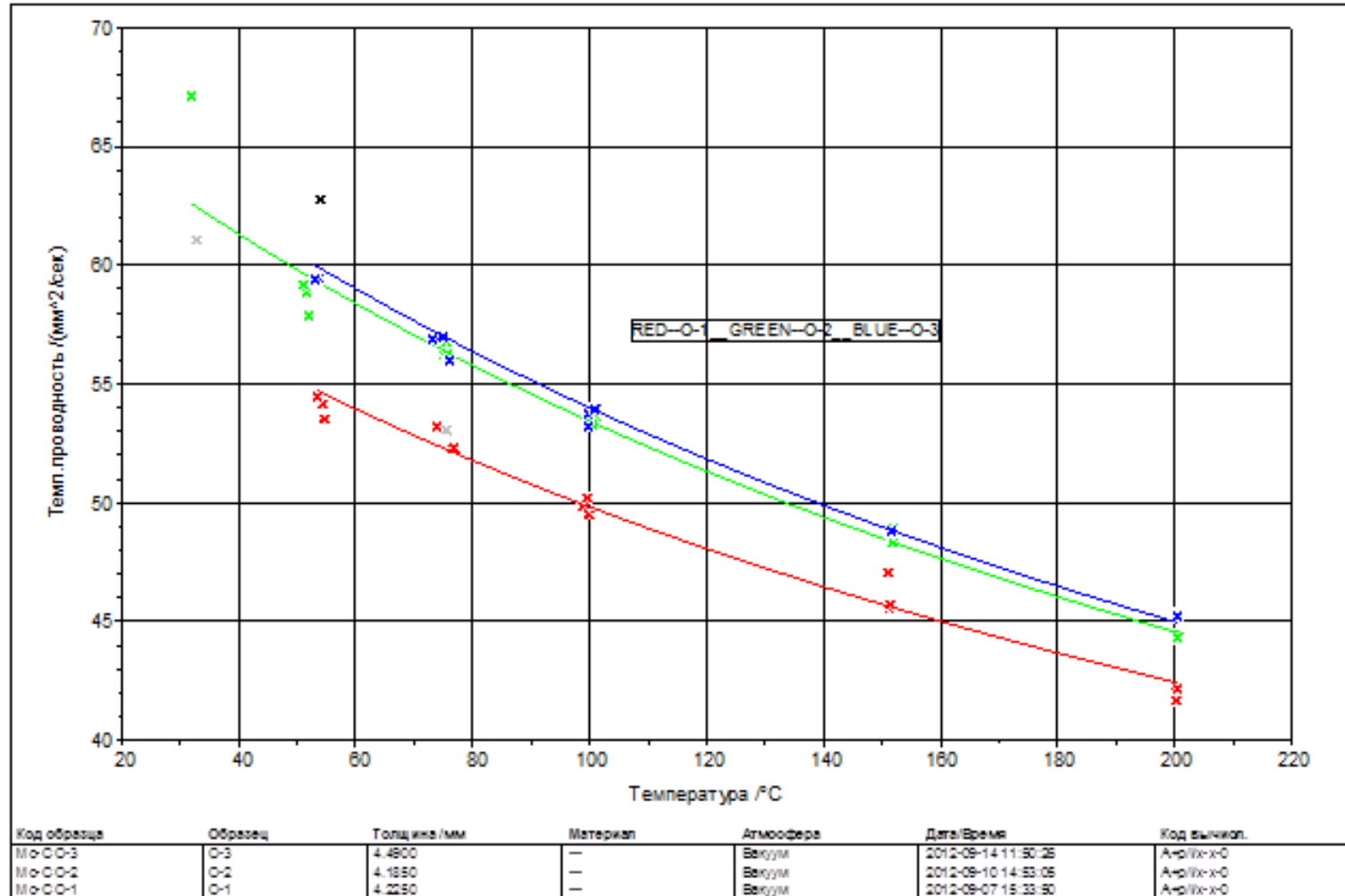
Sample №	$F_{M(E)}$, N	σ_f , MPa	E^H , GPa
1	101,7	34,7	157,9±8,4
2	101,7	31,9	178,7±8,4
3	101,7	34,1	155,1±8,5

$F_{M(E)}$ - maximum load applied to the sample in the process of testing,
 σ_f – achieved tensions in the process of testing for measuring E^H ;
 E^H – **Flexural (elastic)** modulus measured by pure bending.

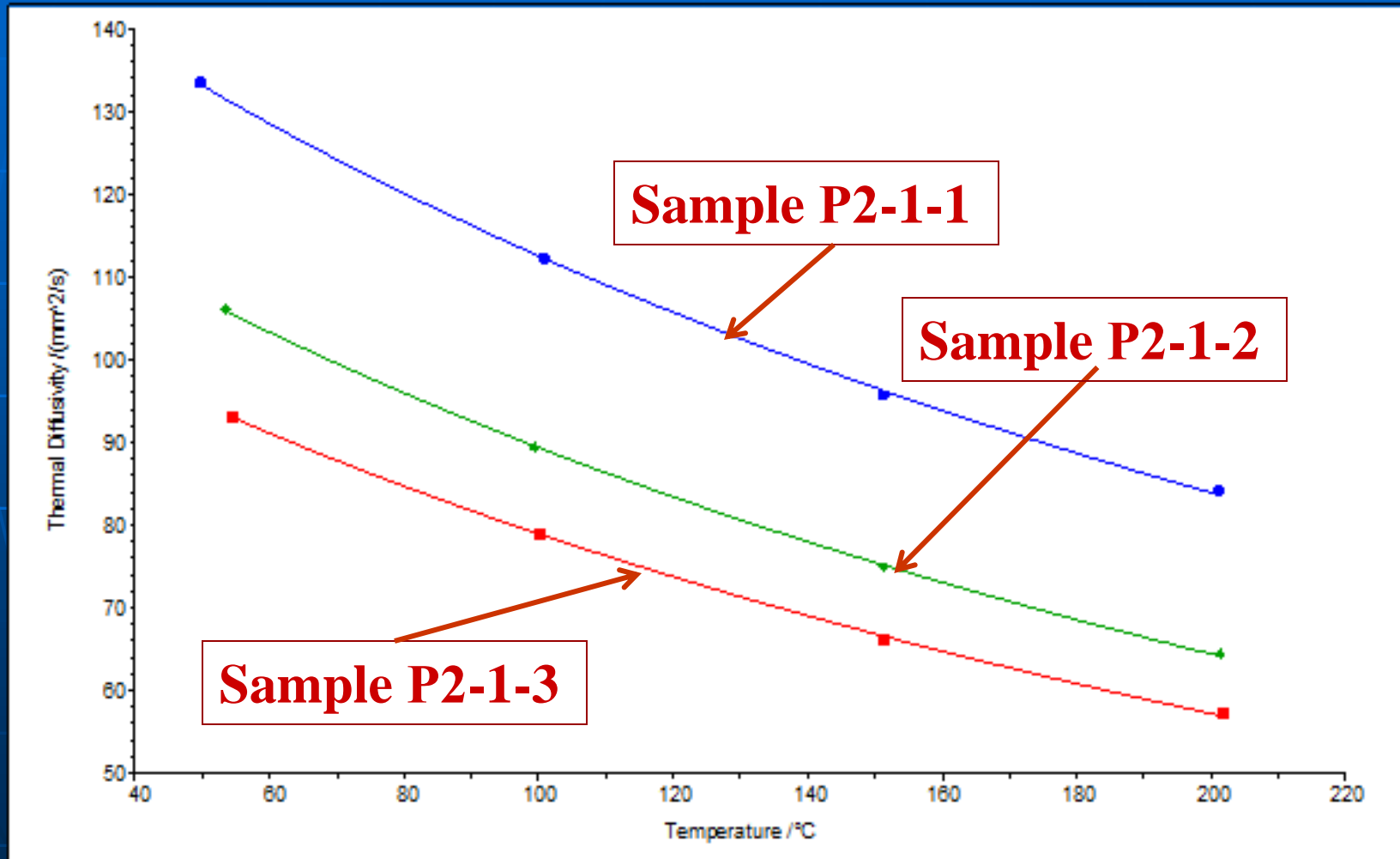
Curves «load – deflection» of «Mo - Cu-Diamond» material samples by measuring elastic modulus



Measurements of temperature dependence of thermal diffusivity in Mo-Diamond collimator materials before proton irradiation on NRC KI cyclotron

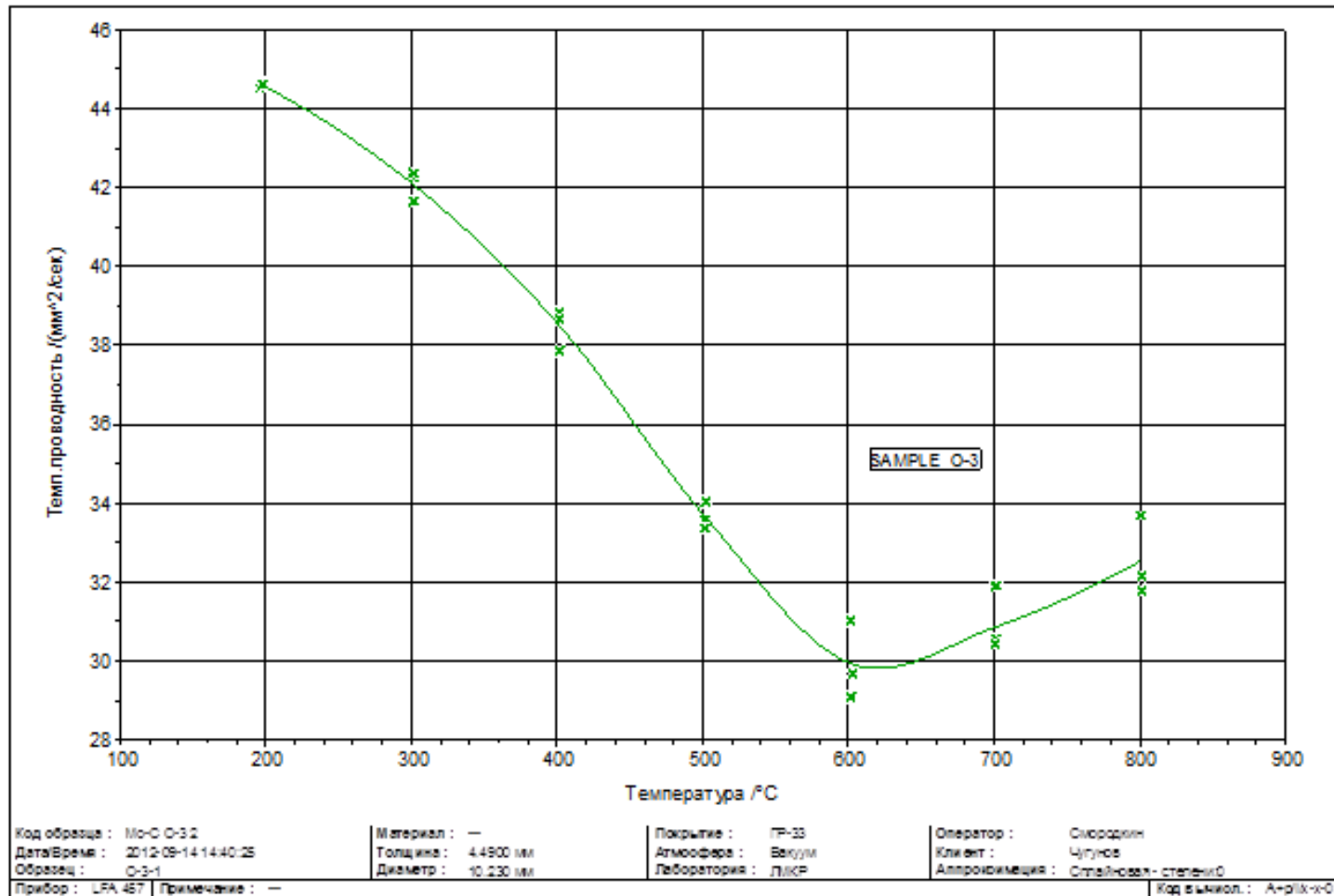


Measurements of temperature dependence of thermal diffusivity in Cu-Diamond collimator materials before proton irradiation on NRC KI cyclotron

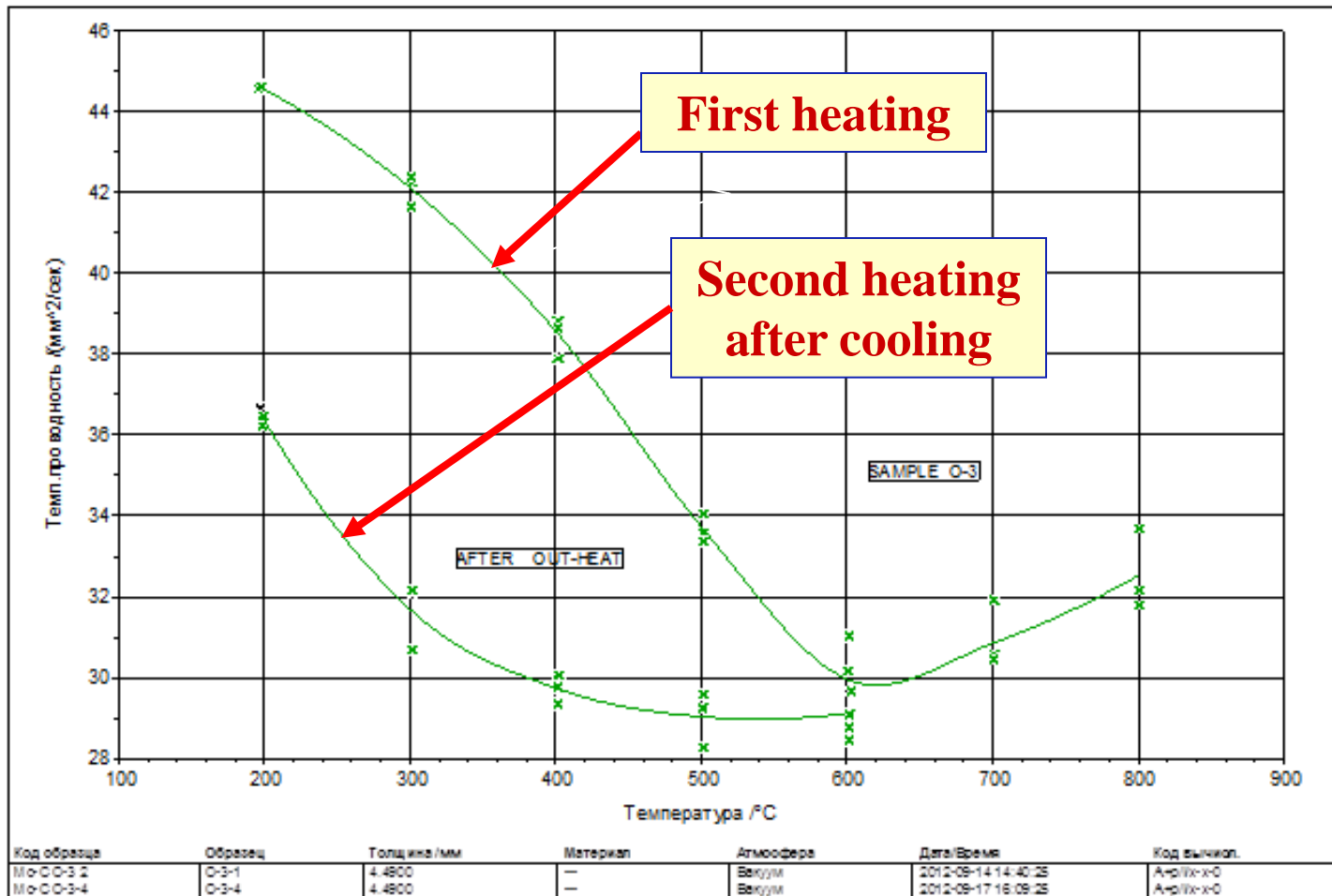


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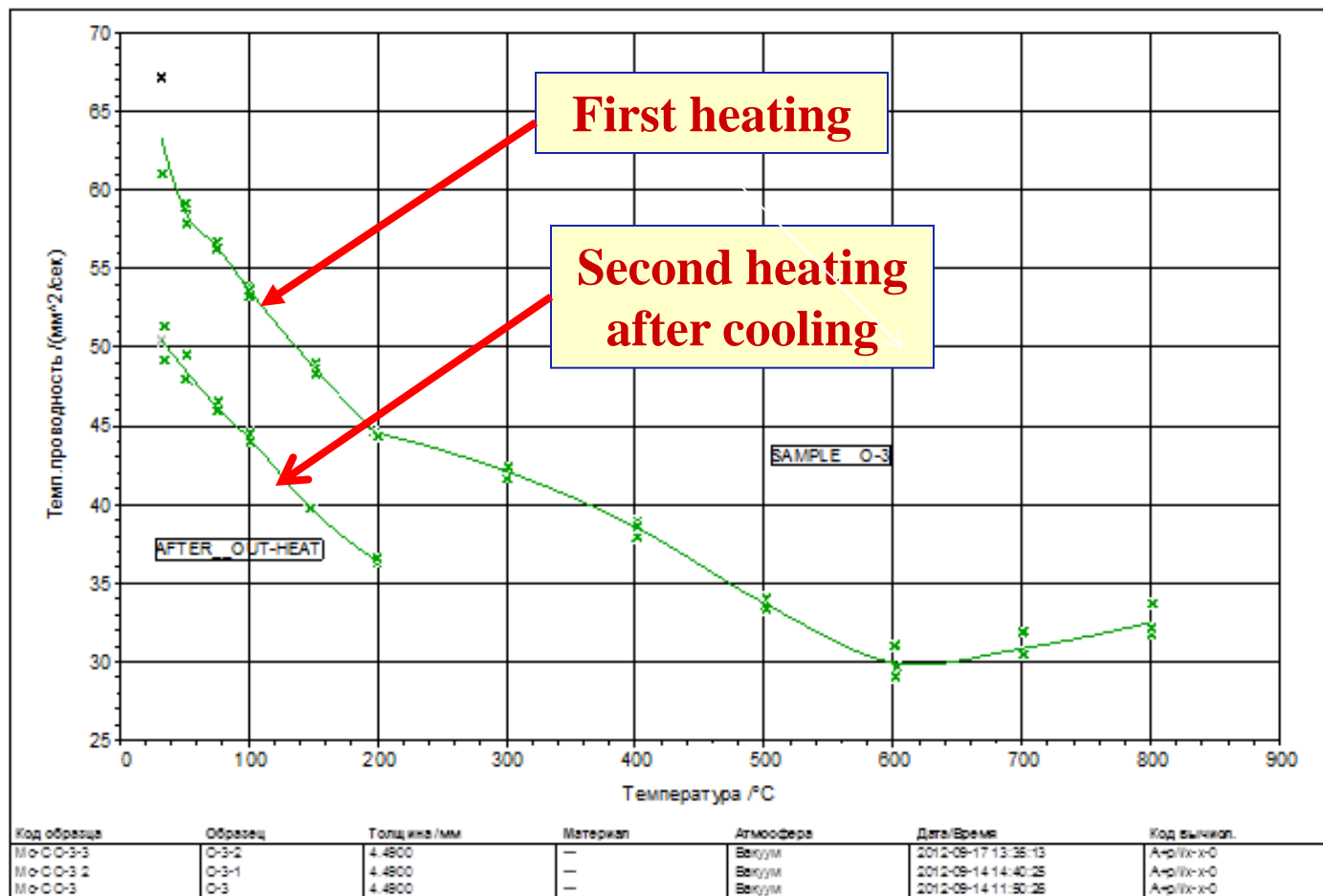
Measurements of temperature dependence of thermal diffusivity in Mo-Diamond collimator materials before proton irradiation during heating from 200°C up to 800°C



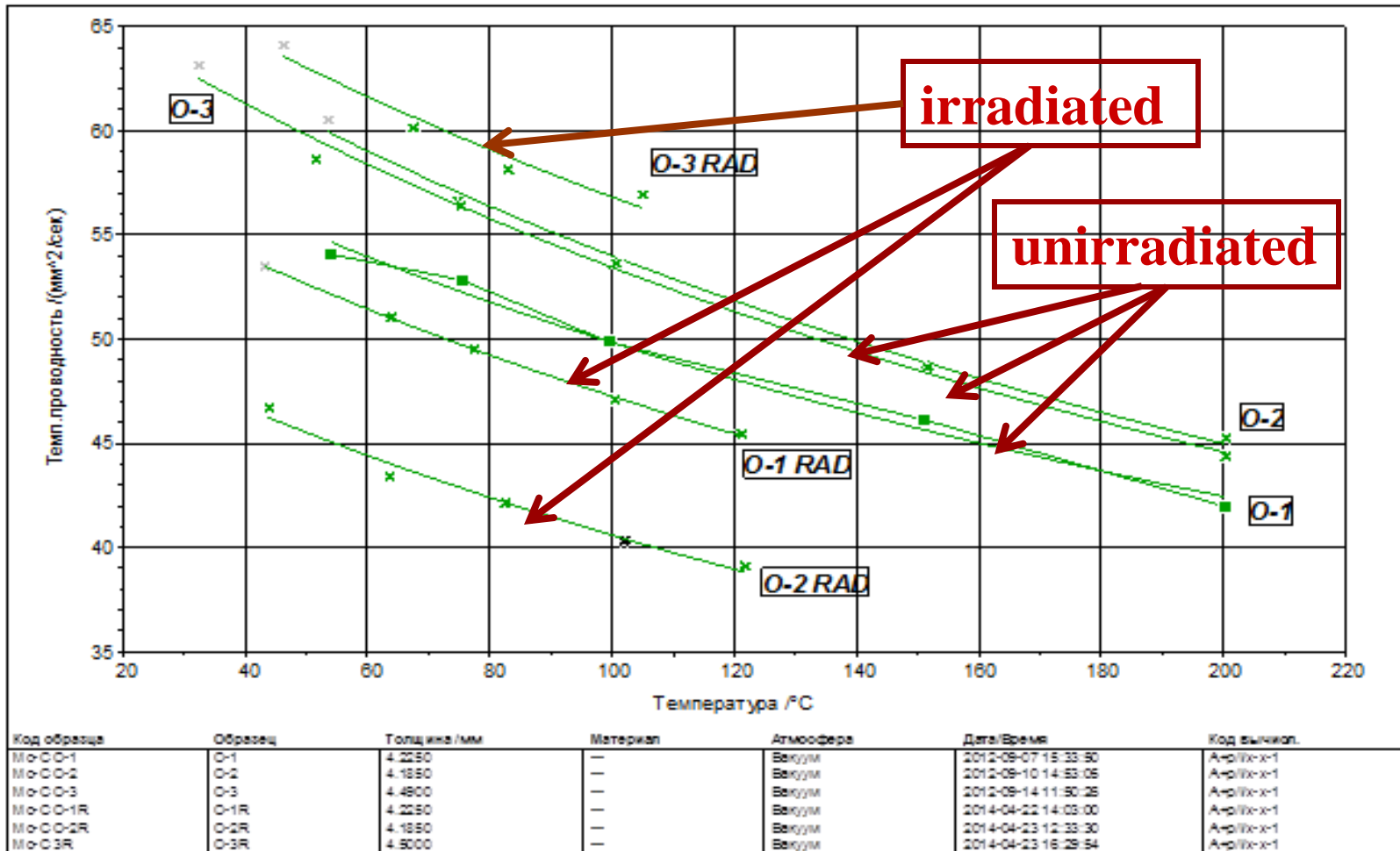
Measurements of temperature dependence of thermal diffusivity in Mo-Diamond collimator materials before proton irradiation during heating from 200°C up to 800°C



Measurements of temperature dependence of thermal diffusivity in Mo-Diamond collimator materials before proton irradiation during heating from 30°C up to 800°C



Measurements of temperature dependence of thermal diffusivity in Mo-Diamond collimator materials before and after 30 MeV proton irradiation on NRC KI cyclotron with dose $10E17$ 1/cm²

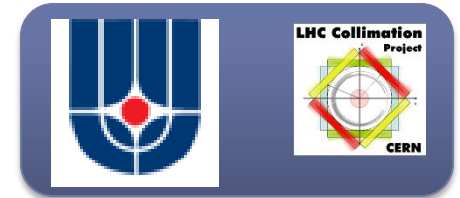


EuCARD-2



ColMat-HDED collaboration and beyond

- ColMat-HDED partners



- Partnership agreement with CERN (KN2045)

BREVETTI BIZZ

- Collaboration CERN with US-LARP



- EuCARD² WP11 ColMat-HDED focusses on further **material developments for collimators and targets**:
 - Producing novel material samples (Brevetti-Bizz, RHP, CERN)
 - Performing irradiation tests in M-branch (from 2011 in GSI) and HiRadMat (from 2012 at CERN), together with well-established irradiation facilities (NRC-KI and BNL) to measure radiation resistance and hardness. (CERN, GSI, UM, KUG, IFIC)
 - Characterising mechanical properties (POLITO and CERN).
 - Simulating mechanical properties (POLITO, NRC-KI, GSI, UM and CERN) and beam induced damage (CERN).
 - **Simulating radiation induced damage (NRC-KI, GSI and CERN).**
 - Integrating collimators into beam environment to give specifications and validate (CERN, HUD, UNIMAN, RHUL, IFIC).

- Heavy ion irradiation tests at GSI (C, Xe, Au, Bi, U, Pb-ions up to 1.15 GeV)
- Proton and C irradiation tests at NRC-KI (up to 35 MeV and 80 MeV).
- Proton irradiation tests (up to 200 MeV) at BNL.
- Planned proton irradiation tests at HiRadMat (CERN)(up to 450 GeV)
- Thermo-mechanical tests at POLITO.
- Material sample measurements at CERN.

Suggestions in the frame of EuCARD 2:

Development of theoretical models and performing of numerical calculations of effect of fast particles (protons, heavy ion) on behavior of LHC collimator materials.

- **Calculations of radiation damage profiles in collimator materials: CuCD, MoCD, MoGr for protons with energy up to 200 MeV (BNL, NRC KI), up to 450 GeV (CERN) and heavy ions up to 1 GeV (GSI).**
- **Calculations of temperature distribution profiles in irradiated collimator materials CuCD, MoCD under fast protons with energy up to 200 MeV (BNL, NRC KI), up to 450 GeV (CERN) and heavy ions up to 1 GeV (GSI).**
- **Calculations of shock waves in collimator materials under proton irradiation with 450 GeV and 7 TeV taking into account “Thermal Spike” and “Coulomb Explosion” models.**

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