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# Indentation behavior of superelastic hard carbon

Olga Chernogorova  
*IMET*, tchern@imet.ac.ru

Ekaterina Drozdova  
*IMET*

Iraida Ushakova  
*IMET*

Evgenii Ekimov  
*Vereshchagin Institute for High Pressure Physics RAS*

Vicente Benavides  
*Lulea University of Technology*

*See next page for additional authors*

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[1] O. Chernogorova, I. Potapova, E. Drozdova, et al. Applied Physics Letters 104, 043110-1 - 043110-4 (2014); doi: 10.1063/1.4863470.

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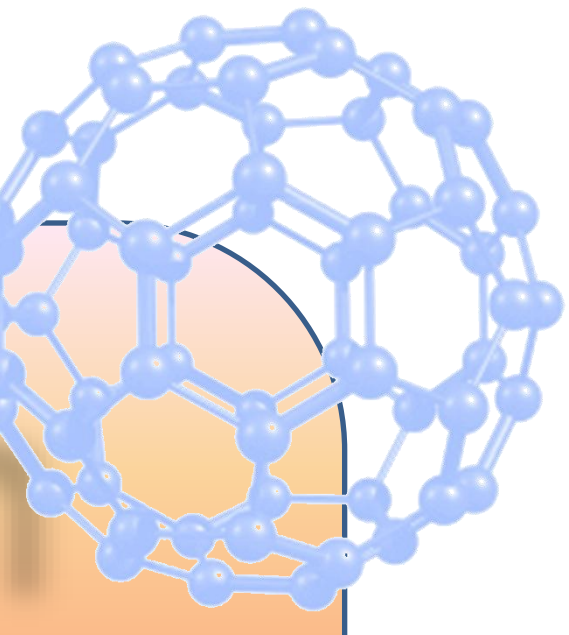
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**Authors**

Olga Chernogorova, Ekaterina Drozdova, Iraida Ushakova, Evgenii Ekimov, Vicente Benavides, and Aleksander Soldatov



# Indentation behavior of superelastic hard carbon



**Olga Chernogorova, Ekaterina Drozdova, Iraida Ushakova**, Baikov Institute of Metallurgy and Materials Science (IMET), 119991 Moscow, Russia, [tchern@imet.ac.ru](mailto:tchern@imet.ac.ru),

**Evgenii Ekimov**, Vereshchagin Institute for High Pressure Physics RAS, Troitsk, Moscow Region, Russia,

**Vicente Benavides, Alexander Soldatov**, Lulea University of Technology, SE - 97187, Lulea, Sweden and Department of Physics, Harvard University, Cambridge, MA 02138, USA

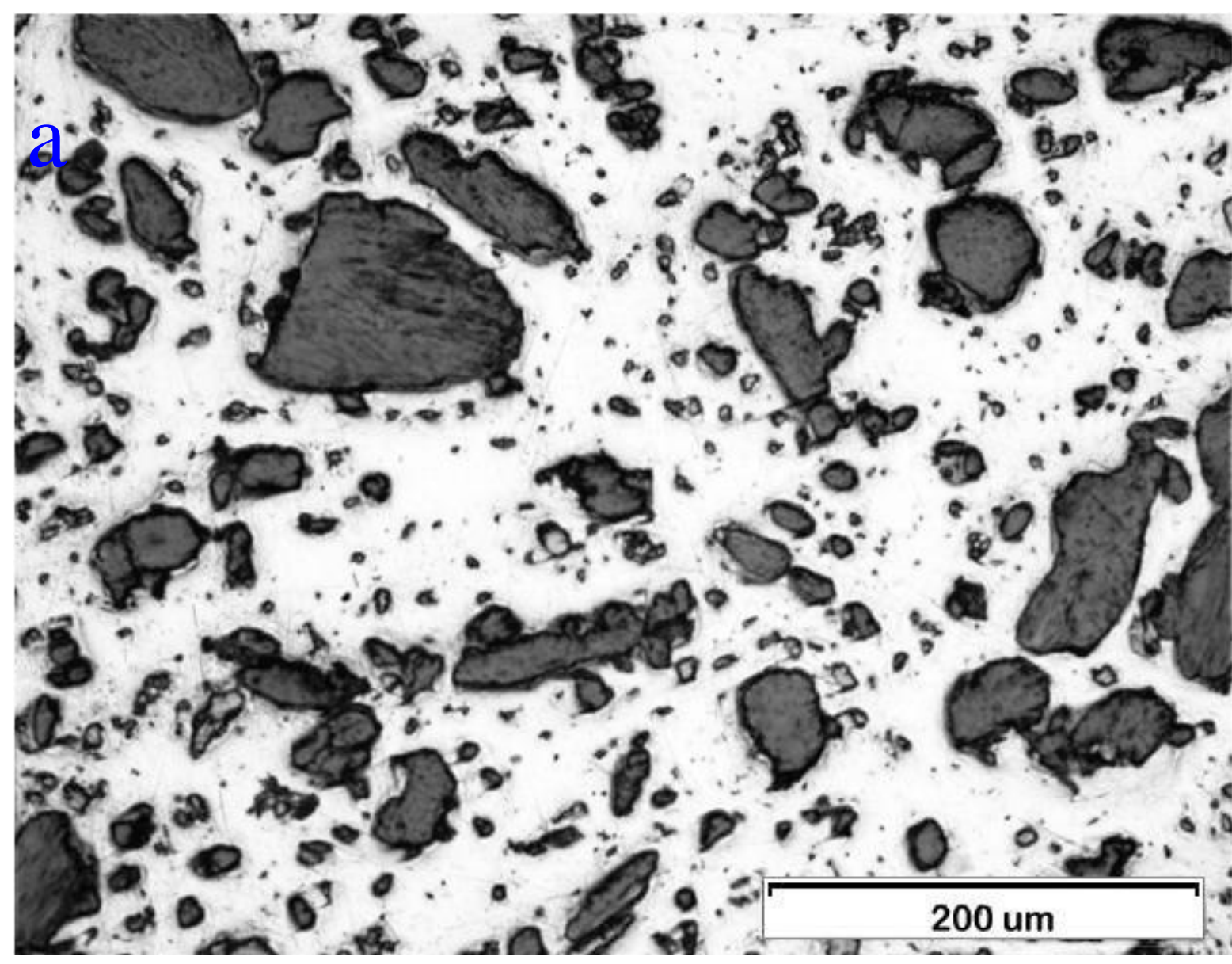


Fig. 1. Microstructure of (a) the cobalt-based composite material reinforced by carbon particles obtained from fullerite C<sub>60</sub> at a pressure of 8 GPa at 1000 C and (b,c) the particles from (b) C<sub>60</sub> and (c) SE in polarized light.

HREM images and Fourier transform for atomic carbon phase obtained at 8 GPa (800°C) of (1) crystalline and (2) amorphous (mechanically activated) fullerite C<sub>60</sub>

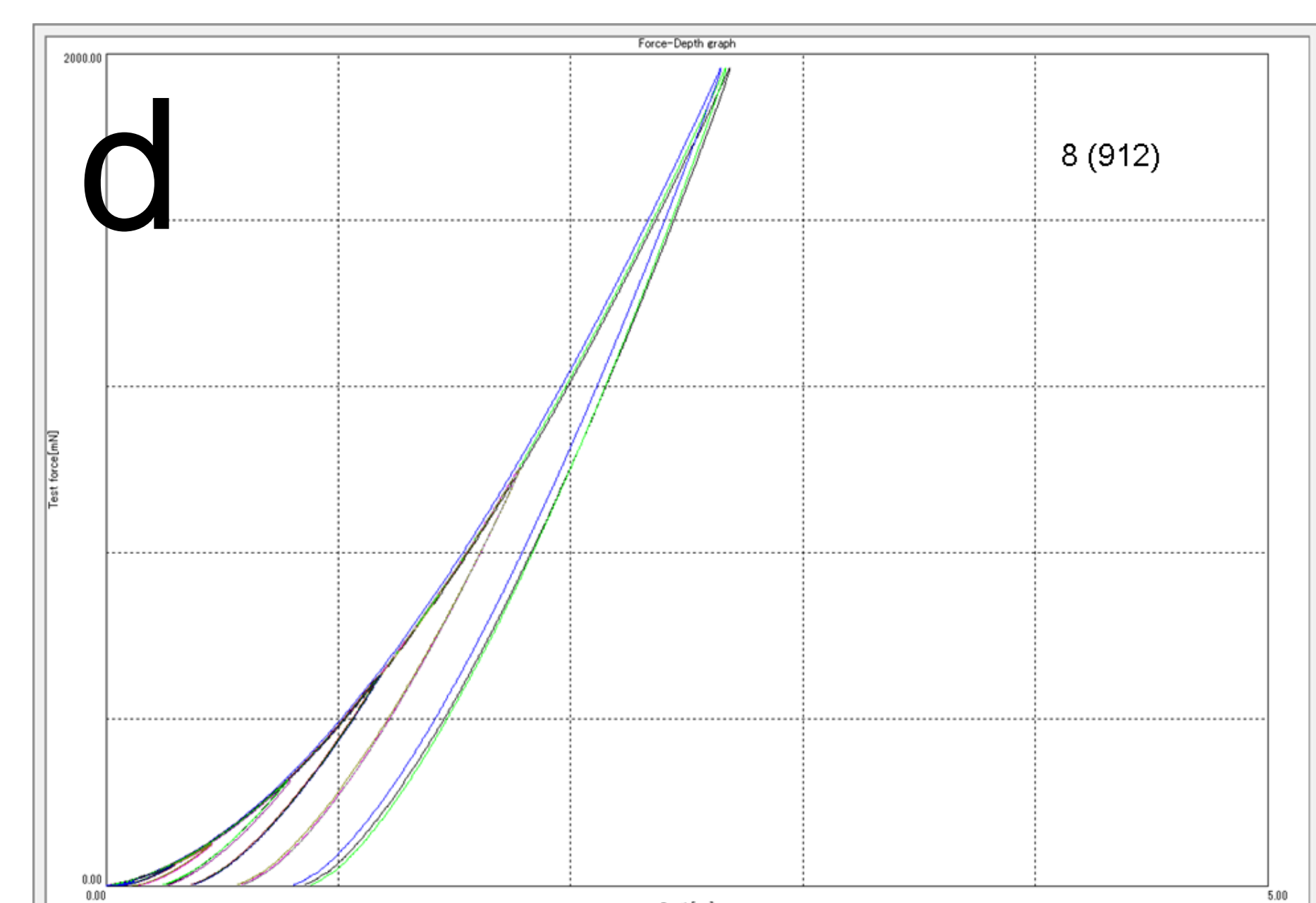
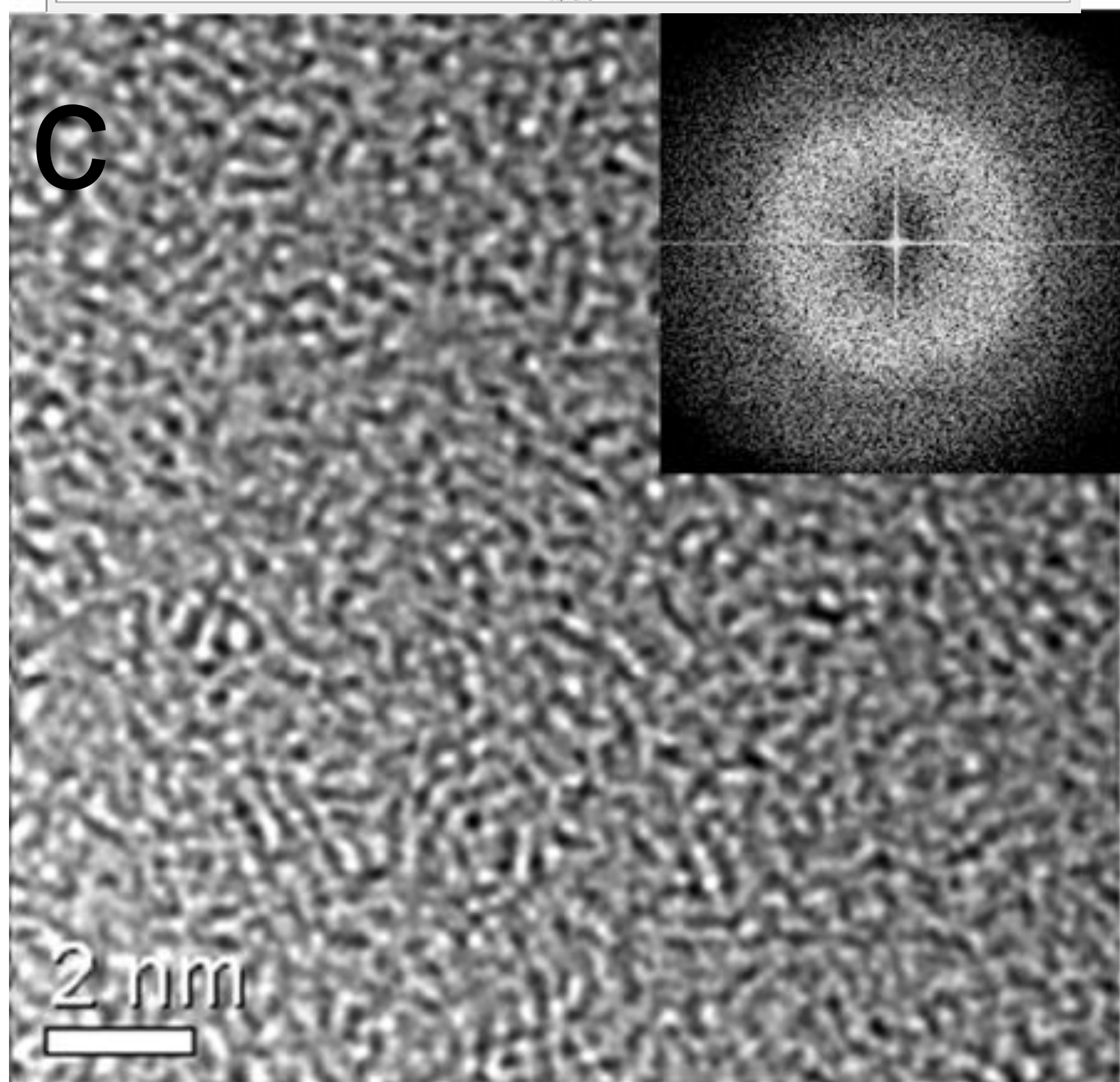
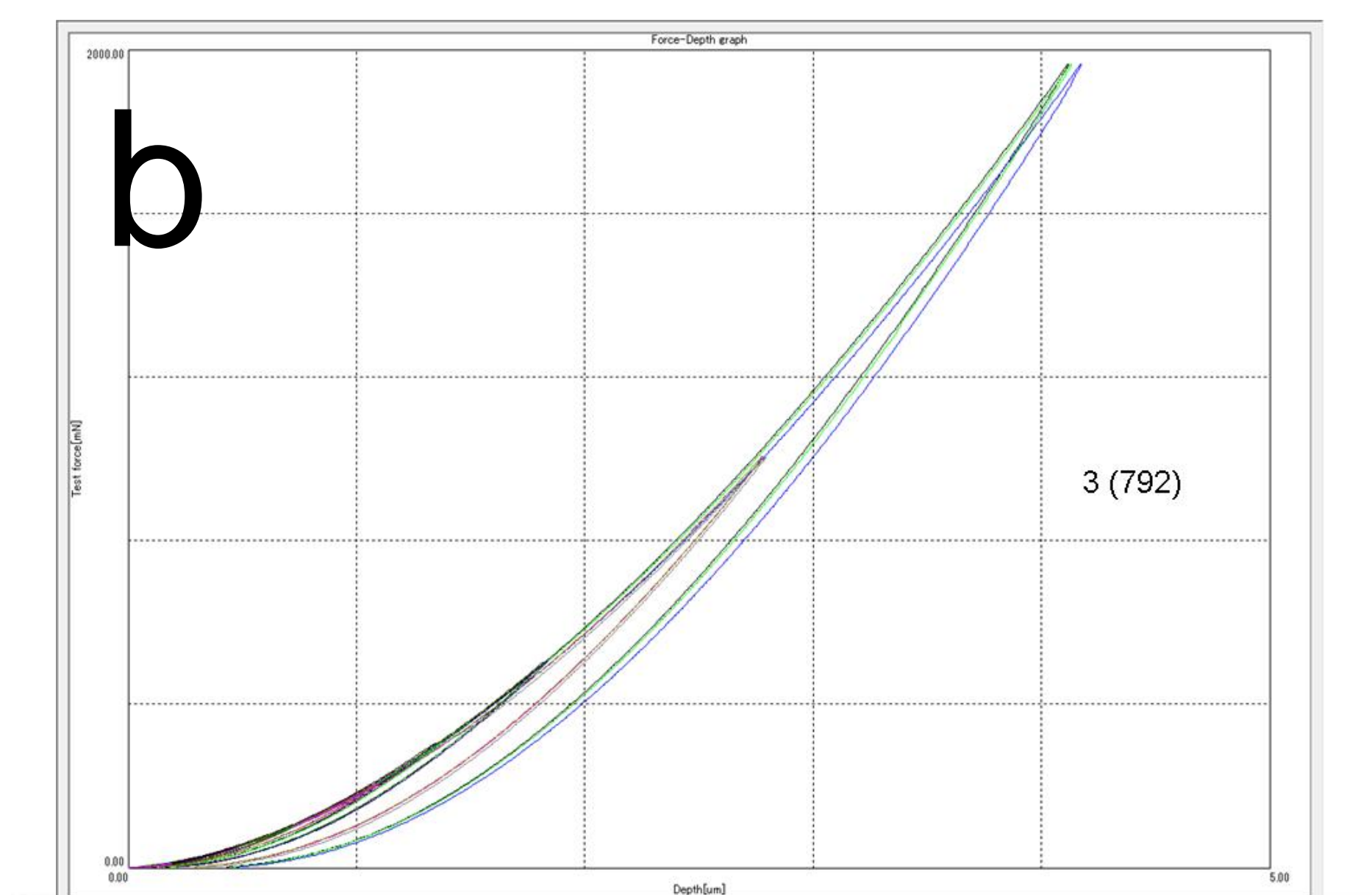
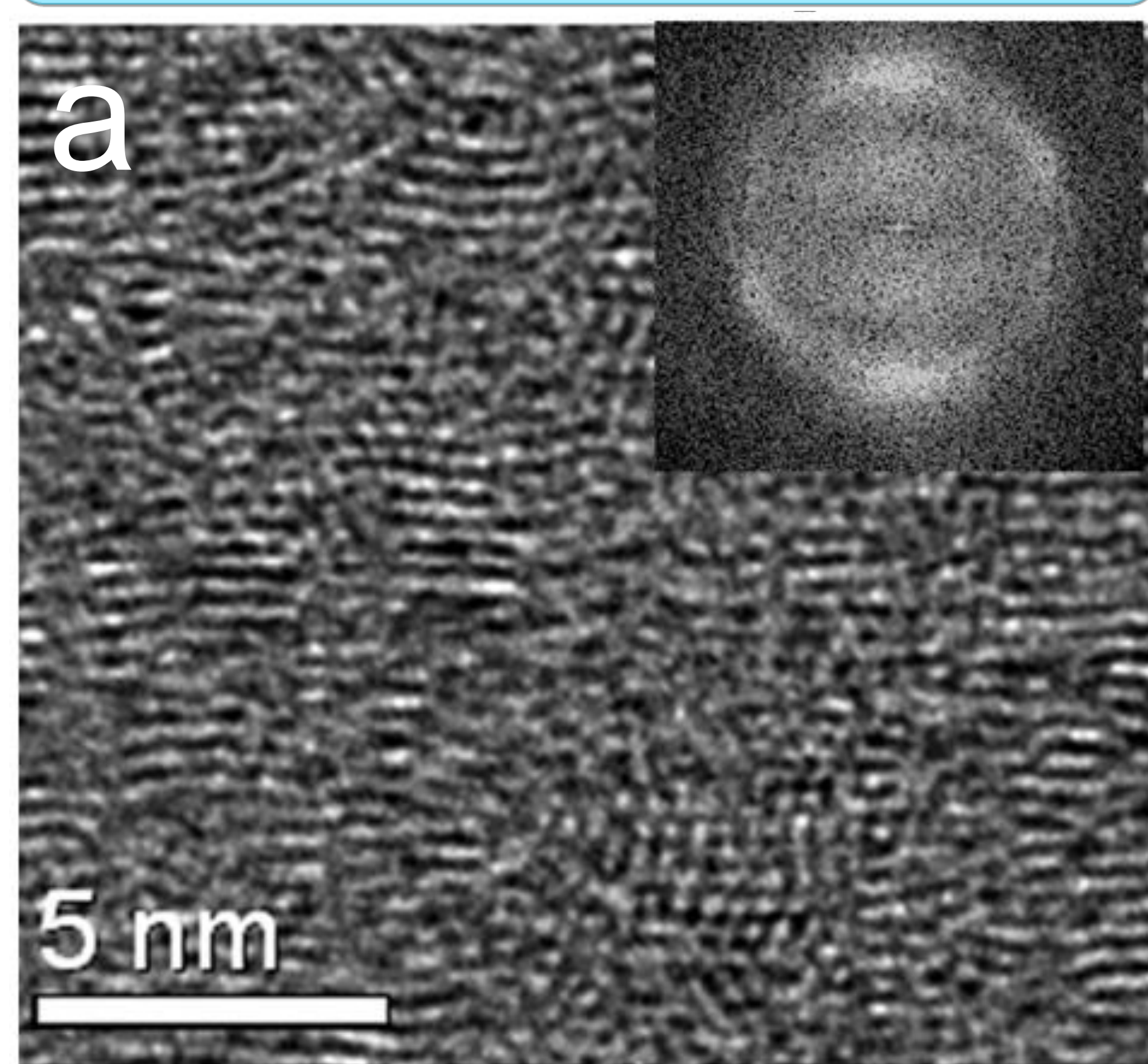


Fig. 2. HRTEM images of (a) nanoclustered graphene phase (samples 1-3) and (c) highly disordered hard carbon phase and (b, d) their indentation curves, respectively, recorded at loads of 10 – 1970 mN).

Superelastic hard carbon particles up to 1 mm in size were produced by fullerene collapse upon high-pressure high-temperature treatment with simultaneous sintering of metal-matrix composite materials (CM) reinforced by such particles (Fig. 1). The hardness of carbon particles can be varied in a wide range by changing the parameters of their structure, which consists of curved graphene planes or their packets of different sizes (Fig. 2a). Such carbon phase was called “nanoclustered graphene phase” (NGP) [1]. The properties of the carbon particles were controlled by changing treatment pressure (5 and 8 GPa) and temperature (1100-1800 K), composition of parent fullerites (C<sub>60</sub> or C<sub>60</sub>/70), and pre-treatment (ball milling) of parent fullerites. The carbon particles formed from fullerites under pressure are close to diamond-like carbon coatings in mechanical properties (combination of high hardness and high elasticity), but they are sufficiently large for their microhardness testing at rather high loads (Fig. 3). The mechanical characteristics of the carbon particles were tested with a DUH 211S (Shimadzu) tester (Fig. 4) according to ISO14577 with a Vickers indenter at loads of 10-1970 mN in load-unload regime at a strain rate of 70 mN/sec. The Martens hardness HM measured at a load of 500 mN on the carbon particles of 28 CM samples was varied from 2700 to 13600 N/mm<sup>2</sup>; the corresponding indentation hardness H<sub>IT</sub> changes from 8100 to 42400 N/mm<sup>2</sup>, i.e., for the carbon material under consideration, H<sub>IT</sub>/HM = ~3 (see the table). Such great difference between the above hardness parameters is due to a great contribution of the elastic deformation to the total deformation upon indentation. The ratio between the corresponding deformation works  $\eta_{IT} = W_{elast}/W_{total}$  (%) with increasing hardness decreases from 87 to 78%, but still remains very high, exhibiting the superelastic behavior of the NGP carbon particles. All samples are characterized by the indentation size effect (ISE), which manifests itself as decreasing HM, H<sub>IT</sub>, and E<sub>IT</sub> with increasing F<sub>max</sub>. The ISE becomes more pronounced with increasing hardness of the carbon particles, for example, HM of the least and most hard particles with increasing F<sub>max</sub> decreases by a factor of 1.7 (from 3900 to 2300 N/mm<sup>2</sup>) and 5.4 (from 40200 to 7400 N/mm<sup>2</sup>), respectively (Fig. 5). The intensity of hardness reduction in the range of small loads (10-250 mN) is significantly higher than in a range of 250-1970 mN. The elastic recovery upon indentation expressed as  $\eta_{IT}$  at F<sub>max</sub> ranging between 250 and 1970 mN is virtually unchanged while, at lower loads, the dependence of  $\eta_{IT}$  on F<sub>max</sub> is non-monotonic, with a small peak at F<sub>max</sub> = 50 mN in all cases (Fig. 6). Indentation creep C<sub>IT</sub> (%) was measured at F<sub>max</sub> = 500, 1000, and 1970 mN, the holding times at F<sub>max</sub> were 60, 300, and 600 sec. For all samples, C<sub>IT</sub> decreases with increasing F<sub>max</sub> and increases with holding time (Fig. 7). The time dependence curves of C<sub>IT</sub> tend to saturation with increasing F<sub>max</sub>. C<sub>IT</sub> increases with increasing hardness of the superelastic hard carbon particles.

Reference

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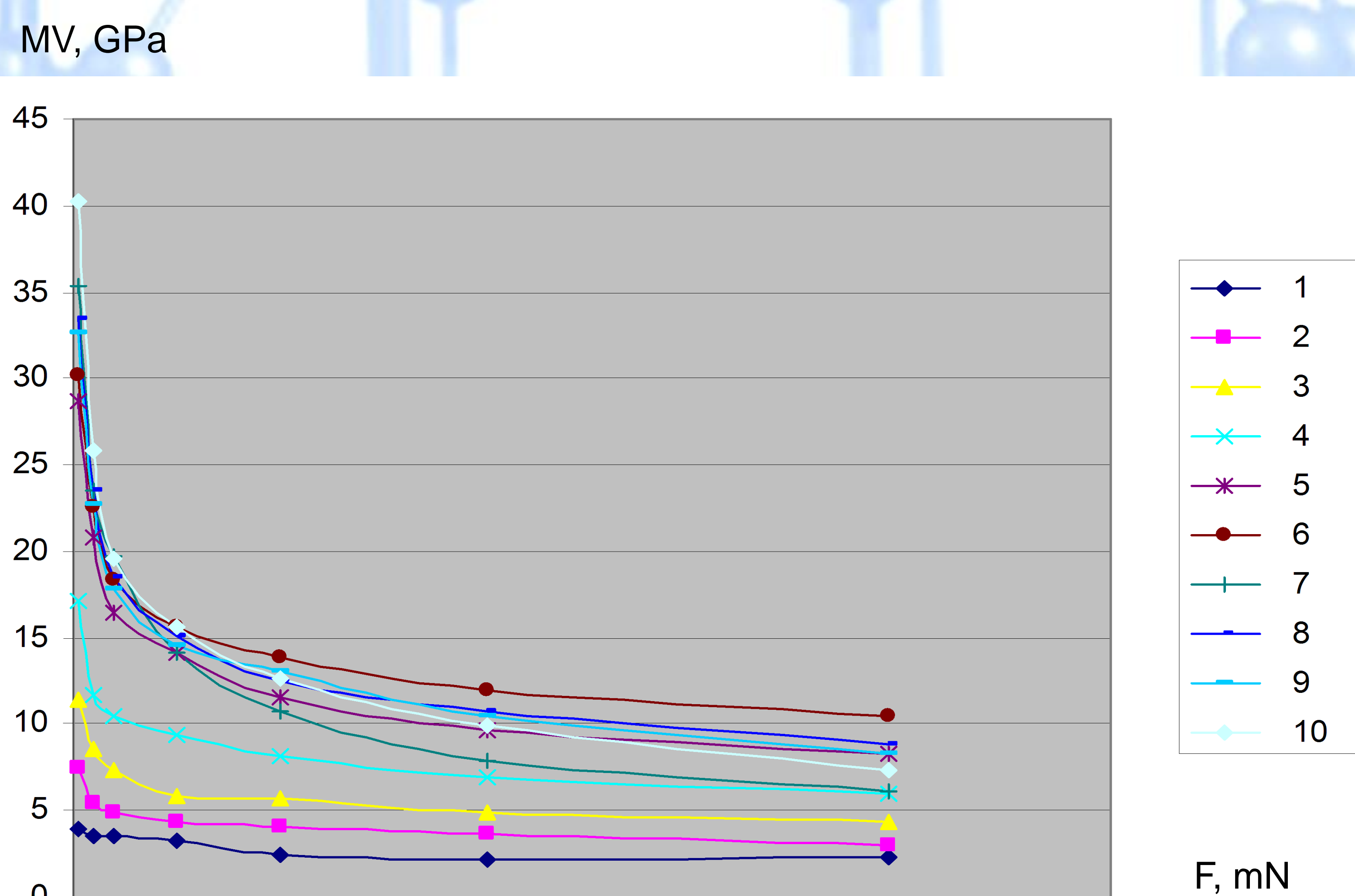


Fig. 5. Effect of indentation load on the Martens hardness MV of the samples 1 – 10 (table)

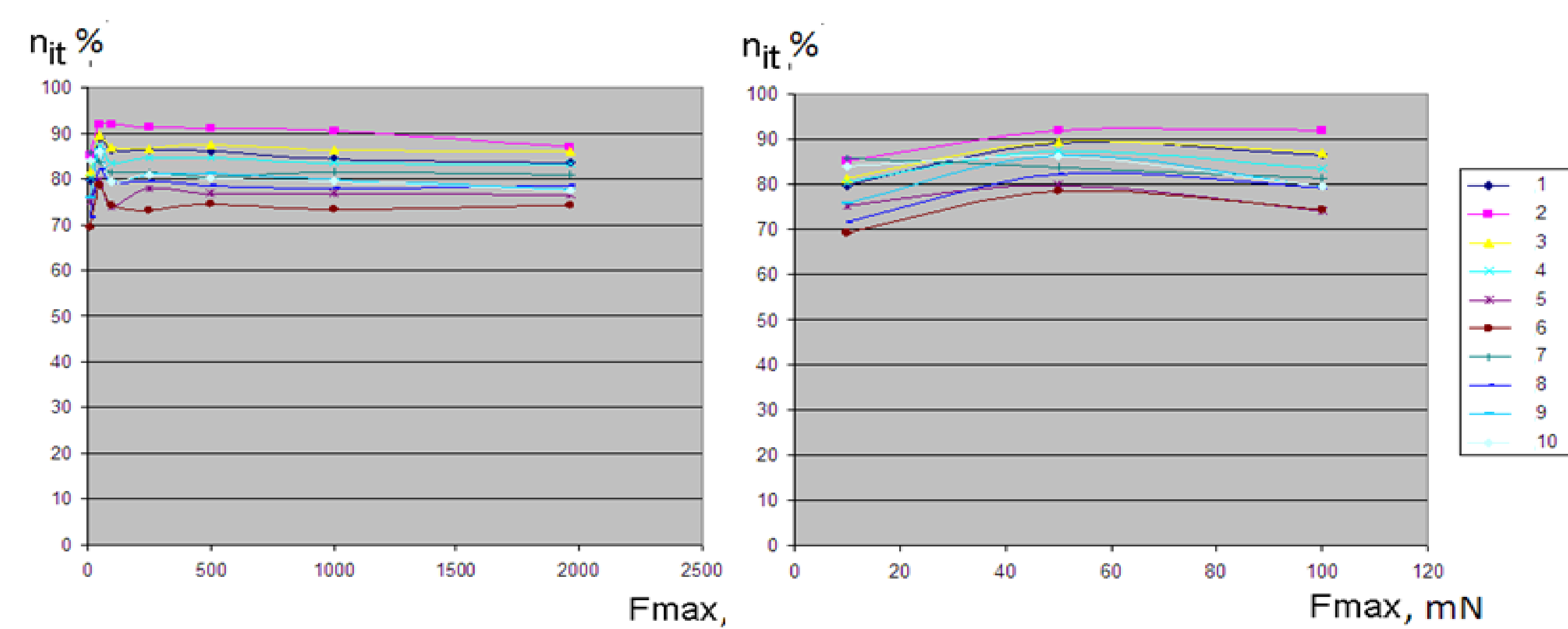


Fig. 6. Effect of indentation load on the elastic recovery of the samples 1 – 10 (table)

Sample No.	Matrix metal, initial fullerite, treatment (H - T, °C)	Properties at 500 mN					HV*	F	Wear mg
		HMV	HMs	H <sub>IT</sub>	E <sub>IT</sub>	nit			
1 (790)	Co+10%C60, 5-1000	2.8	2.4	7.1	43.9	85.09	674		40.9
2 (900)	Co+10%C60/70+BM 8h, 5-1000	4	3.4	12.4	62.1	90.7	1169	0.18	19.7
3 (792)	Co+10%C60/70 5-950	4.9	3.8	14.8	76.1	87.4	1402	0.24	3.3
4 (861)	Co+10%C60+BM 48h, 8-900	8.2	6.1	24.8	133.4	84.1	2340	0.18	1.2
5 (893)	Co+10%C60+BM 8h, 8-800	11.6	7.9	35.5	197.2	78.2	3355	0.14	0.53
6 (920)	Co+10%C60/70 +BM 8h, 8-800	11.6	8.3	34.2	201.1	77.4	3230	0.15	0.15
7 (896)	Co+10%C60/70+BM 8h, 8-800	12.2	8.6	37.7	209.9	80.1	3564	0.16	0.38
8 (912)	Co+10%C60/70+BM 1h 8-800	12.8	9.4	35.6	229.2	75.1	3360	0.16	0.4
9 (908)	Cu+10%C60/70+BM 4h, 8-830	13.3	9.1	41.1	231.3	78.3	3891	0.28	0.37
10 (909)	Cu+10%C60/70+BM 4h, 8-830 + annealing 100°C 10 h	13.6	9.4	42.4	238.9	78	4004	0.11	0.3

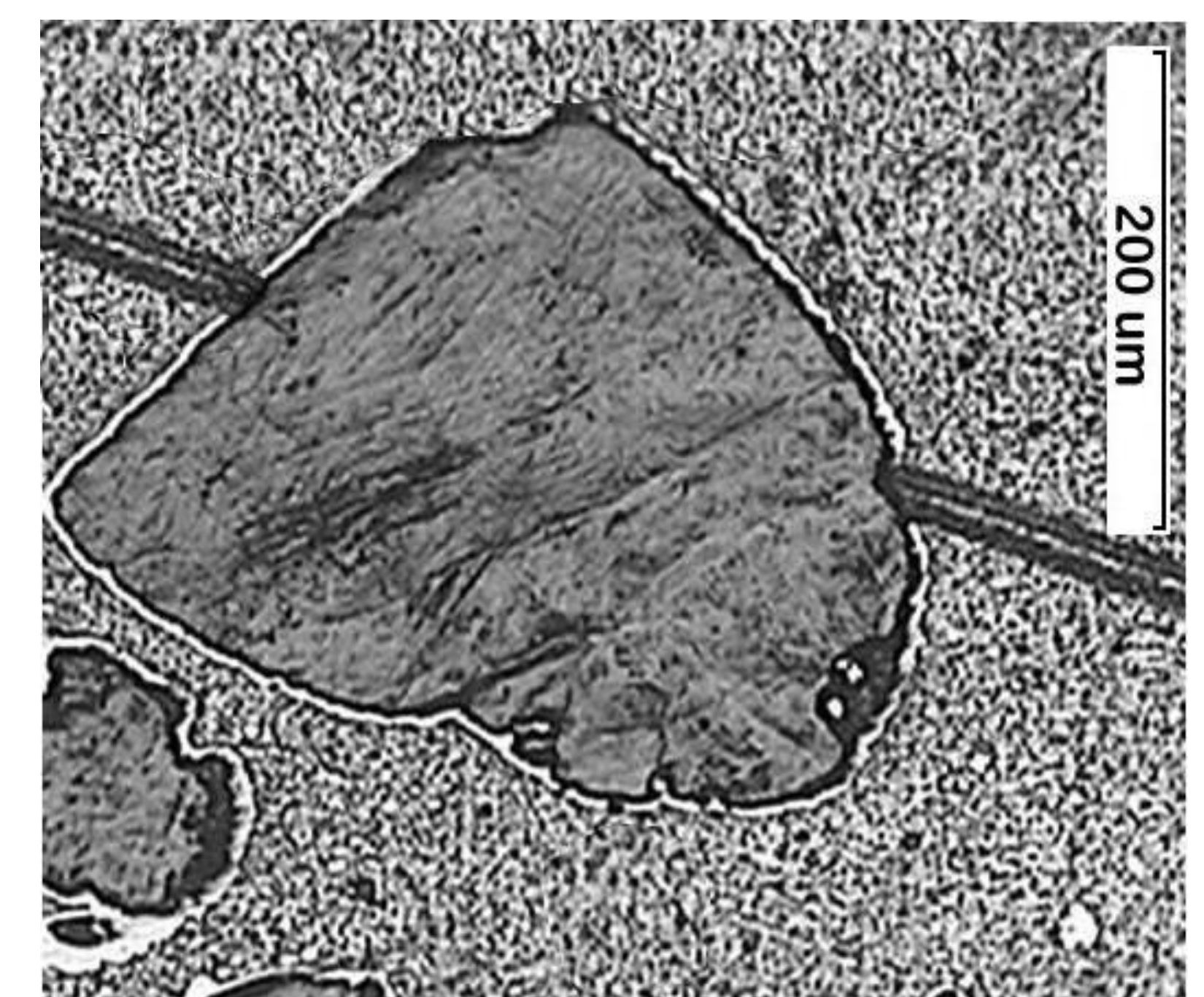


Fig. 3. Reinforcing carbon particle from C<sub>60</sub> in Fe matrix after scratching by diamond pyramid indenter at a load of 0.5 N.



Fig. 4. DUH 211S (Shimadzu) tester and a participant of our work Iraida Ushakova (Potapova) on the cover page of Russian newspaper Poisk.

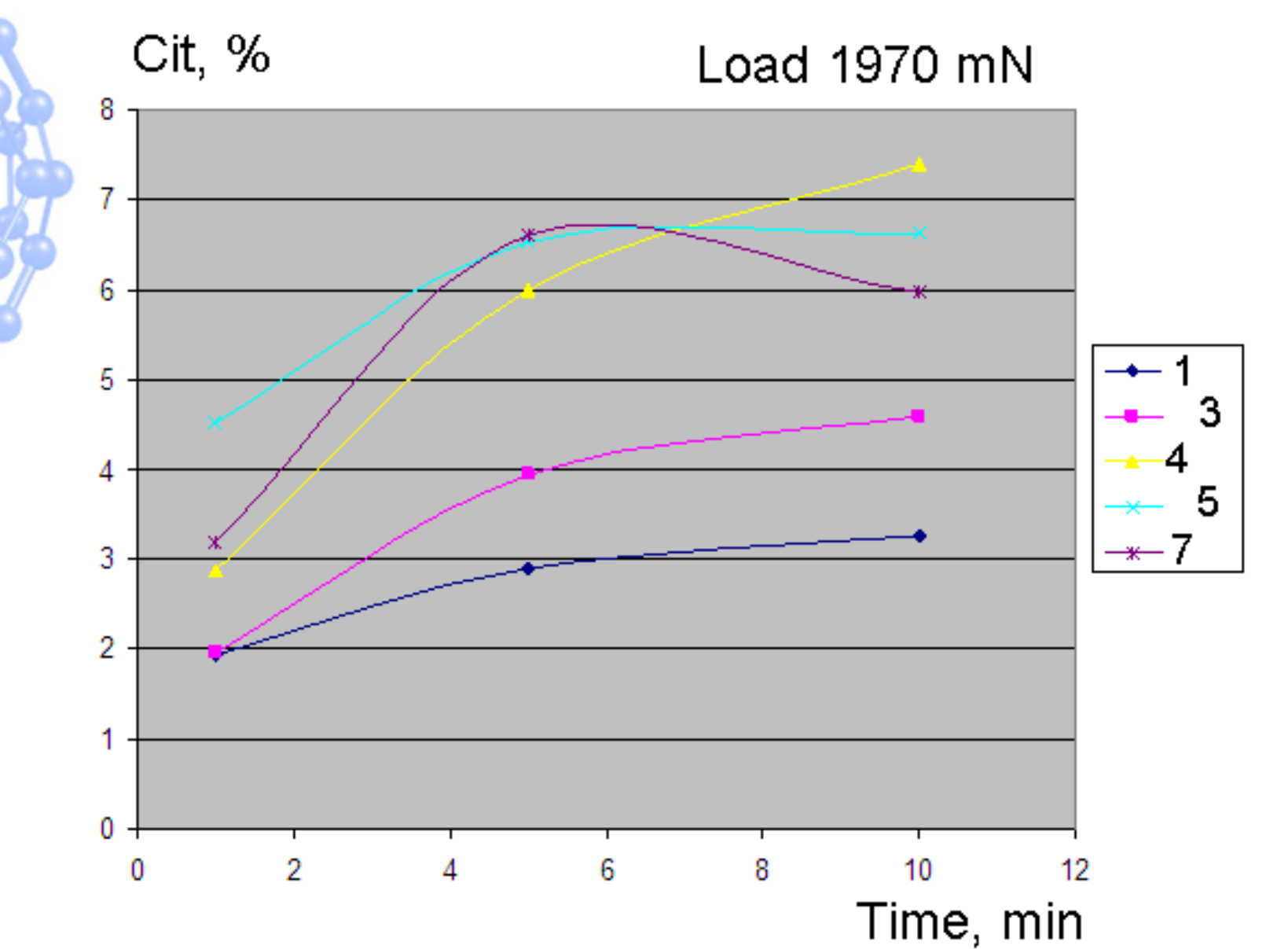
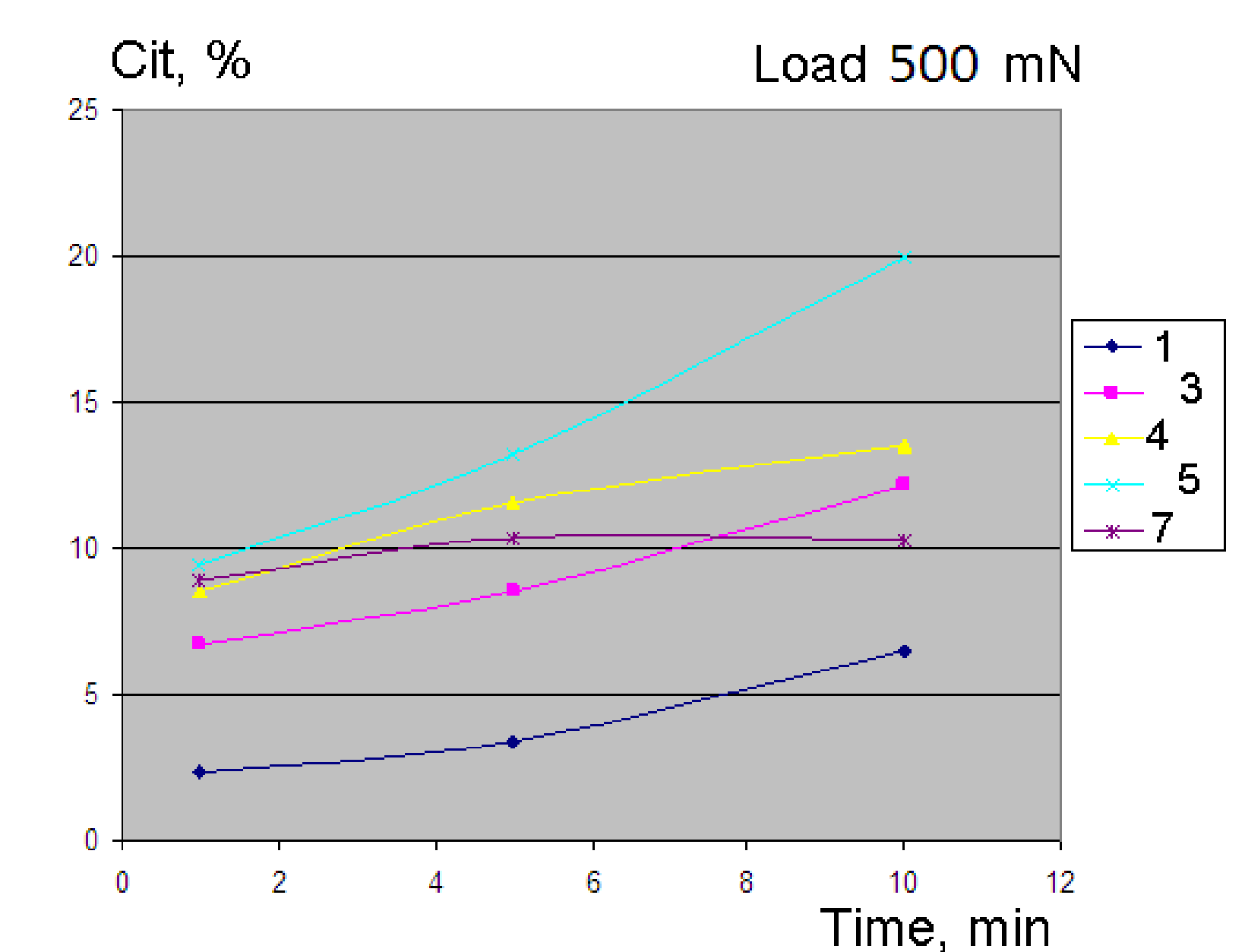


Fig. 7. Indentation creep at different loads as a function of holding time and sample hardness ( see the table).