

PECULIARITIES OF PHASE TRANSFORMATION IN CRYSTALLINE SILICON UNDER LOCAL LOADING

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The investigation of phase transformation induced by nano- and microindentation of Si (100) under different loading conditions has been carried out in this work. The influence of different loading regimes was considered by using instrumented nano/microindentation technique with continuous registration of load-displacement ($P-h$) curve, as well as by using quasistatic microindentation technique. The effect of different types of indenters was also taken into consideration: Berkovich trihedral diamond pyramid with 150 nm tip rounding for the former technique and Vickers tetrahedral diamond pyramid with 4,5 μm tip rounding for the latter one. The investigation of phase transformation under local loading was studied by means of micro-Raman spectroscopy.

A consecutive transformations from the initial diamond cubic phase (Si-I) to high pressure β -Sn conductive phase (Si-II) during loading and then, body-centered cubic (Si-III), rhombohedral (Si-XII) and amorphous conductive phases (a-Si) during unloading are known to take place during nano- and microindentation process. The formation of amorphous phase is accompanied by a so-called “pop-out” effect on the unloading part of $P-h$ curve. In this work it was investigated the development of this effect under various loading conditions: increase of the holding time under the constant load from standard 5 s to 15 s, 1 hour and 14 hours and different loading-unloading rates (1; 2; 10; 50 mN/s). The “pop-out” step became larger with load increase and loading rate increase (Fig. a, b) that is probably due to the extending of the volume of material which undergone the amorphization. But for the loading regime with holding under peak load during 900 s the “pop-out” effect became very weak (Fig. c).

At the same time the Raman peaks of $488,8\text{ cm}^{-1}$ corresponding to amorphous phase was detected for 300 mN Vickers indentations made under holding time of 1 hour and 14 hours by using quasistatic microindentation technique. This discrepancy may be connected with stronger deformation and hence more dense and complicated dislocation structure created by sharper Berkovich indenter as a result of its geometry, comparatively with Vickers one [1]. This fact can lead to the stabilization of the defect structure during holding under the load using Berkovich indenter resulting in more difficult relaxation of material at the unloading and, respectively, less pronounced amorphization process.

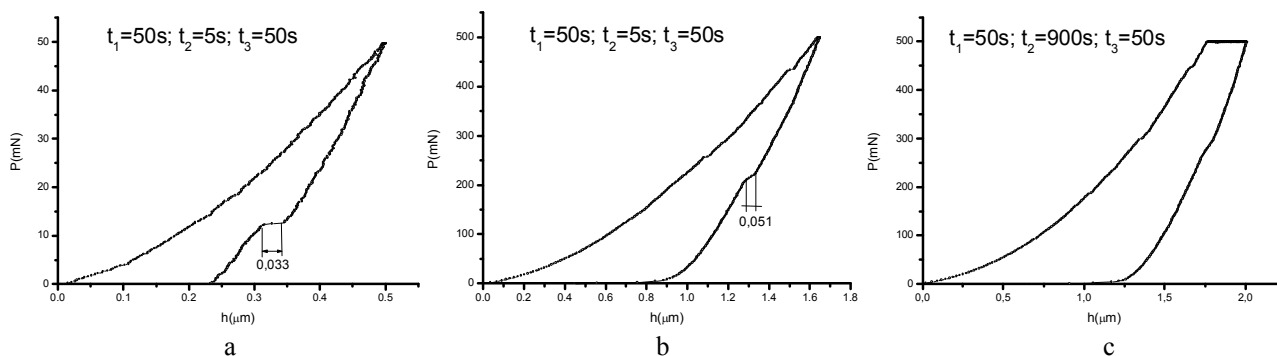


Figure. Indentation $P-h$ curves made on Si (100) by using Berkovich indenter with loads $P=50$ mN (a) and 500 mN (b, c); indicated on the figure t_1 is the loading time, t_2 is the holding time under the load, and t_3 is the unloading time.

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[1] O. Shikimaka, D. Grabco. Deformation Created by Berkovich and Vickers Indenters and its Influence on Surface Morphology of Indentations for LiF and CaF₂ Single Crystals. Journ. Phys. D: Applied Physics, 2008, v. 41, 074012, p. 1-6.