Influence of Loading Holding Time under Quasistatic Indentation on Electrical Properties and Phase Transformations of Silicon¹

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Abstract—The quasistatic Vickers indentation of Si (100) were applied to investigate the influence of loading holding time on the changes of electrical resistance and phase transformation in the indentation zone. For all used loading regimes with different holding times (2 s, 10 s, 1 h and 10 h) in combination with constant loading—unloading rate (250 mN/s) the electrical resistance in the region of residual indentations was found to be lower than before indentation. It was shown that this is connected with the formation of semimetallic Si-III phase and amorphous Si of higher pressure induced by creep process developed under long lasting pressure. The longer the holding time, the greater lowering of electrical resistance in the indentation region was observed, with the exception of the holding time above 1 h, this being explained by a decelerating creep rate of Si for this interval of time leading to a halt of further extending of amorphous and Si-III regions of lower electrical resistance.

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INTRODUCTION

Being one of the most used materials for microelectronics, optoelectronics, micro- and nano-electromechanical systems, silicon is widely investigated from the point of view of its mechanical behavior for the purpose of deeper understanding of processes taking place under mechanical impact during fabrication and exploitation of devices on its base, as well as with the view of the possibility of using its mechanical peculiarities for the creation of special functional structures and surfaces. Recently, considerable attention has been paid to the structural modification of the silicon single crystal under the influence of the concentrated load action (nanoindentation and microindentation). It is known that Si undergoes a series of phase transformations under high local stresses created during micro- and nanoindentation: the initial diamond cubic phase (Si-I) transforms into β -Sn phase (Si-II) during loading at the contact pressure of about 8-12 GPa, and during unloading, under the pressure release, R8 (Si-XII) and BC8 (Si-III) phases at 5-8 GPa and amorphous (a-Si) phases at about 4 GPa were found to be formed [1-3].

So far under investigation were the influence of different factors and loading conditions such as temperature of deformation [4–6], unloading rate [7, 8], load value and type of indenter [2, 3], cyclic loading– unloading [3, 9, 10] on the peculiarities of phase transformations. Theoretical predictions of silicon behavior under high pressure and experimental investigations under hydrostatic pressure in a diamond anvil cell demonstrated that Si-II is a highly conductive metallic phase [11], Si-XII and Si-III are semimetals [12, 13], and amorphous Si is a narrow-bandgap semiconductor at ambient pressure, exhibiting metallic properties under enhanced pressure [14]. Therefore along with Raman spectroscopy [1, 3-8] and transmission electron microscopy investigations [2, 3, 9], for studying indentation-induced phase transformations in Si, the in-situ measurements of electrical resistance changing in the indentation zone are successfully applied [3, 4, 9, 15] and demonstrate a good correlation with the structural phase formation sequence during the loading-unloading cycle.

Another factor, besides the mentioned above, modeling an eventual condition that could be created during manufacturing and functioning of Si based devices, is the prolonged holding under the local loading that could induce some specific changes in structural transformation of Si. This factor can be investigated by increasing the holding time under the peak load during indentation, which is known to cause the creep (continued deformation under the constant load) for some materials. However the present work does not involve the investigation of the indentation creep directly, although it is an apart interesting problem. The aim of this work is the investigation of electrical resistance and phase transformation changing induced by long lasting constant loading, these

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