INDENTATION UNLOADING PHASE TRANSFORMATIONS IN SILICON: A NEW PERSPECTIVE

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This work deals with the pressure-induced phase transformation silicon undergoes during nanoindentation. From high-pressure studies, it is known that silicon transforms from diamond-cubic to beta-tin structure when exposed to pressures exceeding 11-13 GPa [1,2]. Upon pressure release, silicon does not directly transform back to the diamond-cubic structure, instead different metastable phases are formed [2,3]. For the small volumes and fast unloading rates, often encountered in nanoindentation, also transformations to amorphous silicon can be observed [4].

It is well established that these phase transformations in silicon affect the indentation unloading curve due to an increase in the volume of the material. The transformation to metastable, crystalline phases happens in a fraction of a second. Accordingly, the sudden decrease in displacement is observable as a pop-out in the unloading curve. Contrary to this, the amorphous transformation happens in a continuous manner. Therefore, the rate of displacement reduction increases if the contact pressure falls below a certain threshold. This is seen as a bend in the unloading curve commonly referred to as an elbow. [5,6]

From the load-displacement signal, only the onset loads of these transformations are known. In the past, concepts to calculate the actual transformation pressures from the load-displacement data have been proposed [7,8]. However, much like continuous stiffness measurement (CSM) allows acquiring the hardness (= contact pressure) during the loading process, basing the calculation of unloading contact pressure on actual contact stiffness data would be beneficial.

Here, a new nanoindentation protocol [9] is used to achieve exactly such a determination of unloading-contact pressure. The protocol uses the CSM signal during unloading. However, a problem arises when calculating a contact pressure from the stiffness data: The calibrated area-function loses its validity when unloading, as the contact area is not only determined by the elastic sink-in and the indenter shape, but also by the residual impression forming [10]. In the new protocol, this problem is bypassed by calculating a contact area from the stiffness data under the assumption of a constant reduced modulus.

Additionally, constant-load-holding segments were added at various loads during unloading. This was done to investigate the time-dependent transformation under constant pressure, and therefore constant driving force. Here, a slow but continuous reduction of displacement and sudden drops in displacement can be observed. Corresponding, Raman spectroscopy investigations show that the slow reduction is linked to the formation of amorphous silicon while the sudden drops correlate to metastable crystalline silicon. This behavior resembles that of pop-out and elbow during continuous unloading.

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