ESRF	**Experiment title:** Microfokus powder diffraction investigation of phase transformations in Ge and other semiconductors induced by mechanical contact	**Experiment number:** HS-638
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Report:

As a model case for plastic deformation, we study the effects of hardness indentations on silicon, germanium, and other semiconductors in order to gain new insights into the behaviour of semiconducting materials during machining and further tribological effects. Especially the contact induced phase transformations of Si and Ge to metallic high-pressure phases and their subsequent transformation of crystalline metastable phases at ambient conditions, which we had observed by micro-Raman spectroscopy [1,2], were of interest.

The **aim of the experiment** was to identify metastable phases, which we observed within the residual impressions of hardness contacts by micro-Raman-spectroscopy, and -if possible- to quantify the relative amounts of different phases present.

The **experiment** was conducted using hardness impressions (indentation load 2-5 N, impression size ≈ 20-30 µm) in different single crystalline samples of Si and Ge. The impressions were investigated either in reflection or in transmission. The beam size was 50 to 100 µm (diameter) at an energy of 25 keV (λ = 0.496 Å). The most critical part of the experiment was the centering of the hardness indentations in the beam, which in fact was a very difficult with the technical means available at this beamline. In consequence, time-consuming line-scans were needed.

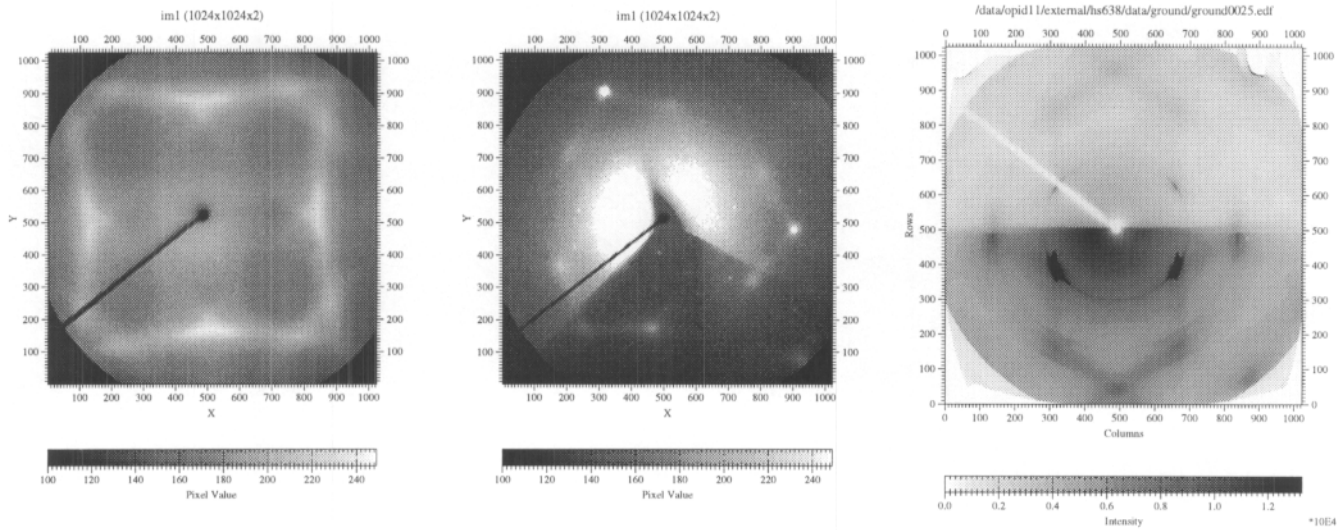

Fig. 1: Diffraction patterns (*Frelon* area detector) of hardness impressions in (100)-Si (a), (111)-Ge (b), and a ground Si (c) surface. No polycrystalline material was found except some debris of Si-I structure on the ground surface.

Unfortunately, our experiment was not successful. Except the diffuse scattering, which resembles the fourfold Si-(100) and threefold Ge-(111) symmetry of the single-crystalline matrix, the expected powder diffraction rings of the phase transformed material were not detected. Even in reflection the phase-transformations were not observed.

As reasons for these results may be discussed:

1. Severe technical problems of finding the small hardness impressions and of positioning them within the beam.
2. The phase-transformed material is of very small crystal size and thus not detectable by X-ray scattering.
3. The amount of phase-transformed material is too low.
4. The background of the single crystalline matrix is too high due to strong diffuse scattering effects.
5. We probably did not hit the impressions exactly.

In contrast, we were able to prove the tetragonal-to-monoclinic Phase-transition in an Y-PSZ-ZrO_2 ceramic, by investigating a large hardness impression ($\approx$ 150 µm) in reflection. This at least shows that despite the negative results of small indentations in Si and Ge there actually exists the possibility of studying phase transformations in materials induced by mechanical contacts, if the experimental procedures and the sample preparation can be refined.