ESRF	**Experiment title:** *Investigation of the martensitic transformations in Lithium and Sodium by three-dimensional X-ray diffraction*	**Experiment number**: **HS 1586**
Beamline: **ID11**	**Date of experiment**: from: **31/01/2002** to: **04/02/2002**	**Date of report**: **22/02/2002**
Shifts: **12**	**Local contact(s)**: **Dr. Stephan GRIGULL**	*Received at ESRF:*

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Report:

In the low-temperature phase diagram of lithium 24 crystallographic variants of a close-packed rhombohedral 9R phase coexist with the bcc matrix below 80K. On heating, the 9R phase gradually transforms into a fcc structure. At 120K all three phases coexist [1,2]. The atomic-scale mechanisms of these phase transitions are now quite well understood in the framework of Landau theory [3]. On the other hand, the mesoscopic domain structure and its relation to the atomistic transformation mechanism were almost unexplored up to now. In *in-situ* optical microscopy [4] a highly irregular and complicated martensite-austenite microstructure (structural units of the 9R phase of about 25µm, embedded in the matrix) was observed, in clear contrast to expectations from the geometrical theory of martensite. Therefore it was presumed that the irregular microstructure might be specific to the surface region and not representative of the bulk.

In the present experiment we used the 3DXRD microscope [5] with a micro-focussed beam for three-dimensional structural characterization of the bulk martensite phase in high-purity Li single crystals. Depth resolution was obtained by a conical slit (CS) device [6]. Up to now this technique has been used for the study of the grain structure in polycrystalline, but single-phase material. The CS device available at the beamline ID11 has been designed for fcc structures. It was, however, found that at an X-ray energy of 50.77keV three reflections of the 9R phase, namely (009), (110) and (0,0,18) also fit perfectly to the conical slits (Fig. 1). This allows to distinguish between 9R and fcc, since only 3 reflections of the former but 6 reflections of the latter can be recorded by use of the CS.

A new method for aligning the CS with respect to the monochromatic beam had to be invented. The standard method described in [6] uses a fine powder of the investigated material. In the present case it was, of course, not possible to produce a powder of the low-temperature 9R and fcc phases of Li. As an alternative, it was found that the *c* lattice parameter of hcp Hf coincides almost exactly with that of the non-primitive hexagonal unit cell of 9R Li. Therefore, the alignment could be performed by means of the (002) and (004) reflections of a thin, polycrystalline Hf foil. This alternative method of alignment, designed specifically for the present experiment and performed for the first time in this way, was very time-consuming and in fact

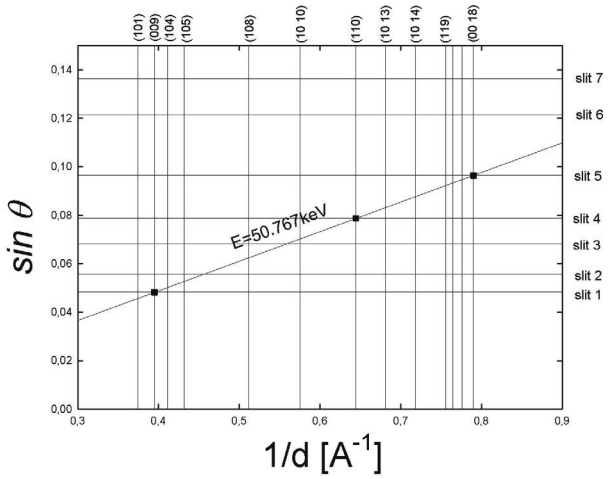

Fig.1. Matching of the conical slit device with reflections of the 9R phase of lithium

required more than 50% of the granted beam time. The experience acquired in the present experiment will, however, enable a considerably faster alignment in future studies of phase transitions by 3DXRD.

The original plan of the experiment was to study the bcc-9R microstructure below 80K on cooling and subsequently the bcc-fcc microstructure on heating. As a consequence of the time-consuming alignment procedure, however, two scans at different temperatures of a same sample volume of reasonable size could not be performed. Therefore it was decided to investigate the microstructure only at 125K, where all three phases coexist. A total sample volume of 100 x 100 x 350µm was scanned in steps of, respectively, 10µm in y and z (normal to the beam) and 50µm parallel to the beam. In order to distinguish between the crystallographic variants of the martensite phase, at each (x, y, z) position the sample was rotated through a total angle of 45° and 9 images in different angular positions were recorded. A typical image is shown in Fig. 2. Apart from the Li reflections, a Debye-Scherrer ring of the beryllium sample container appears in every image. Fortunately it does not impede the data evaluation, since it corresponds to the one conical slit where neither a 9R nor a fcc reflection of lithium may occur.

Fig.2. Diffraction pattern of lithium at 125 K recorded with the conical slit device. The outer Debye-Scherrer ring is due to the beryllium sample container.

As a consequence of the late date of the experiment (Jan.31 - Feb.03, 2002), complete evaluation of the data has not yet been performed. Reconstruction of the microstructure from the recorded images will be performed by the 3DXRD software at the beamline ID11. Some modifications of the software will be required since in the present stage it applies to single-phase materials only. Therefore, an additional stay at ESRF for data evaluation is planned during March. The present preliminary report is submitted in order to serve as a necessary prerequisite for the application for additional beamtime.

References

[1] W. Schwarz and O. Blaschko, *Phys.Rev.Lett.* **65** (1990), 3144
[2] W. Schwarz, O. Blaschko and I. Gorgas, *Phys.Rev.* **B44** (1991), 6785
[3] O. Blaschko, V. Dmitriev, G. Krexner and P. Tolédano, *Phys.Rev.* **B59** (1999), 9095
[4] M. Krystian and W. Pichl, *Phys.Rev.* **B62** (2000), 13956
[5] U. Lienert, H. F. Poulsen and A. Kvick, Proc. 40th AIAA Conf. *Structures, Structural Dynamics and Materials,* St. Louis, USA, 1999, p. 2067
[6] S. F. Nielsen, A. Wolf, H. F. Poulsen, M. Ohler, U. Lienert and R. A. Owen, *J. Synchrotron Rad.* **7** (2000), 103