ESRF	**Experiment title:** Study of the mesoscopic phase separation in the $Mg_{1-x}Al_xB_2$ system in the proximity of a Lifshitz electronic topological transition (ETT)	**Experiment number**: HE-2403
Beamline: ID13	**Date of experiment**: from: 16 April 2007 to: 21 April 2007	**Date of report**: 28-August-2007
Shifts: 12	**Local contact(s)**: Dr. Dmitry Popov	*Received at ESRF:*

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Report:

The aim of this experiment was to identify the mesoscopic phase separation in the proximity of a Lifshitz electronic topological transition (ETT) in the $Mg_{1-x}Al_xB_2$ ternary systems, using micro X-ray diffraction. High resolution X-ray power diffraction measurements on this system showed that this critical point happens at the Al content x=0.28, where the Al-poor phase and the Al-rich phase has the same relative weight.

During the allocated beam-time we have tried to select the single micro-crystals to check if this phase separation is intrinsic or estrinsic. Our local contact Dmitry Popov helped us to individualize the single micro-crystals through the optical microscope and the SEM apparatus, and to select them through the nano-manipulator. At low Al contents we could not find any good micro-crystal as they where smaller of 200 nm. At higher Al content we found bigger microcrystals. Our local contact selected some crystals of about one micron size from the sample with Al content x=0.28, where the phase separation was expected.

We mounted these crystals on a glass capillary and we characterized them through the synchrotron X-ray diffraction using the one-micron size beam with $\lambda = 1$ Å. To select a single hexagonal crystal we tried with a micro-mesh on the tip of the glass in order to find a single bragg peak. Then we found the better orienation of the sample and we made an angular scan to solve the structure.

In figure 1 we show a CCD image of one micro-crysal.

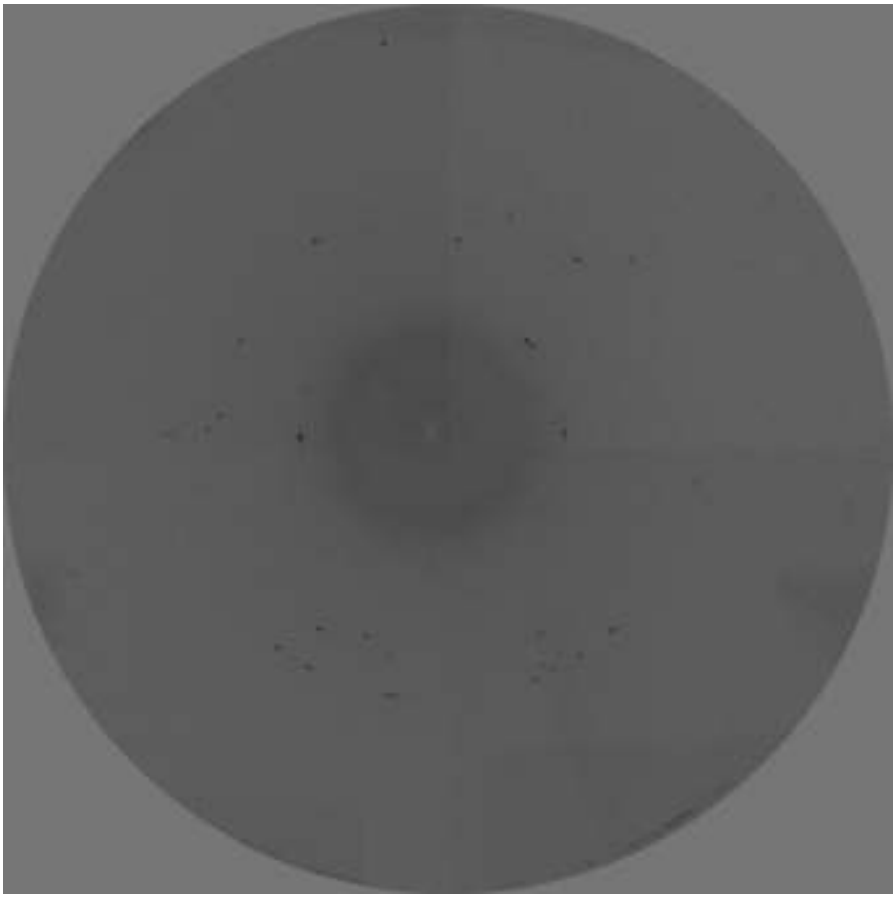

Fig 1: CCD image of a micro-crystal of the sample $Mg_{0.72}Al_{0.28}B_2$

In the table 1 we report the values of the crystallographic axes compared with the results obtained through high resolution x-ray powder diffraction. This experiment shows that it is possible to separate single crystal at 0.28 Al-content, showing that the micro-crystal we have found corresponds to the phase with lowest Al content.

Experiment	a axis (Å)	c-axis (Å)	Relative weight
High resolution x-ray powder diffraction: phase1	3.062	3.397	0.55
High resolution x-ray powder deffraction: phase2	3.069	3.432	0.45
X-ray micro-diffraction on a single crystalline	3.07	3.43	1

Tab. 1: crystal axis measured through micro-crystal diffraction compared with the ones obtained through high resolution X-ray powder diffraction.