ESRF	**Experiment title:** Examination of molten $BiCl_3$ by anomalous x-ray diffraction	**Experiment number**: CH-600
Beamline: ID15 A	**Date of experiment**: From: 31.03.99 to: 06.04.99	**Date of report**:
Shifts: 21	**Local contact(s)**: Dr. Michael Ohler, Dr. Klaus-Dieter Liss	*Received at ESRF:*

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Report

Compared to other molten salts, bismuthtrichloride $BiCl_3$ has some unusual physical properties, as the low melting point of 224°C and the unusual low critical temperature of 905°C. In the composition KCl*2 $BiCl_3$ it is a glass former and it is able to solve metallic bismuth [1]. Owing to these properties, $BiCl_3$ is an outstanding model system for the thermodynamic states “molten salt”, “molten metal” and “glass” and the corresponding phase transitions. In order to study this very interesting system, we got beamtime in the period of Easter. As the microscopic structure of $BiCl_3$ can be described by three atom pair correlation functions, we intended to determine these three functions by means of the anomalous x-ray diffraction at the K- adsorption edge of Bi at 90.5 keV. The L-adsorption edge of Bi at 13.42 keV cannot be used due to high absorption by container and sample.

Experimental setup

The sample consisted of a quartz tube, which was sealed when it was filled with the sample. The quartz tube was heated at both ends with a heater connected to a temperature controller. The temperature was 260°C with a stability of ± 1°C. The scattered intensities were measured at the instrument ID15A in the angular range of 1° to 20° with an energy dispersive multichannel analyser in order to see if any fluorescence radiation is generated when the incident wavelength is near the adsorption edge. Since at the present stage nobody has performed anomalous x-ray diffraction on amorphous systems at high energy, we had to modify the experimental setup. In order to record the complete energy spectrum of the scattered beam, we removed the analyser crystall. Since the background scattering was rather large after the removal of the analyser we tried to install some slit systems. After several attempts we succeeded to reduce the background finally.

Results

After optimization of the thickness of the sample we measured the scattering intensity of the empty container and the container, filled with the molten $BiCl_3$, at the energy of 90.15 keV i.e. 380 eV below the K-edge of bismuth. Then the change of the atomic formfactor f'of Bi has to be determined because this value could not be found in the literature. For this purpose, the absorption of the molten salt was measured as function of the energy of the incident radiation. These spectra were corrected for coherent and incoherent scattering, delivering the true photoabsorption coefficient, which is proportional to the imaginary part of the dispersion correction. The tabulated values of the photoelectric absorption coefficient of the anomalous scatterer were replaced by the experimental data in the measured range.
Then the real part f'was determined by Kramers-Kronig transformation. The resulting change of the atomic formfactor amounts to 6 electron units at the K-edge (Fig.1). After having determined the dispersion correction of bismuth at the K-edge we were able to select the wavelength appropriate for a significant change of the atomic form factor of Bi. The

following measurements were performed with an energy of the incident beam of 90.15, 88.65 and 79.65 keV. Due to several problems with the instrument itself and the computer control system of the instrument as well as some temporal beam dumps we were unable to complete this experiment. Therefore we could not evaluate our data, with respect to anomalous x-ray diffraction. Nevertheless, we corrected our first measurement for container-scattering and self-shielding and normalized to the sum of atom-, compton-, and multiple scattering. The Fourier-transform of the distinct part of the coherent scattering, namely the total pair correlation function, is given in fig.(2) . The interpretation of the data is in progress, but a first comparison with neutron diffraction results [2,3] on the identical system shows a good agreement, and indicates that our measurements and data evaluation are correct.

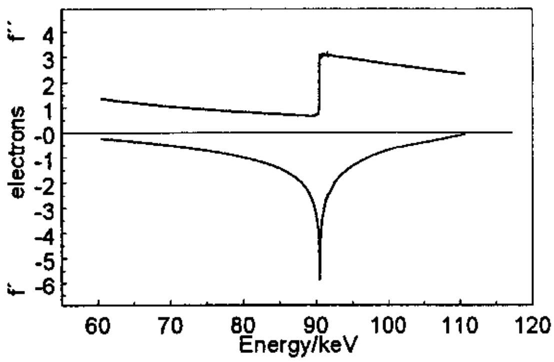

Fig.1: Change of the atomic form - factor f' with the incident energy

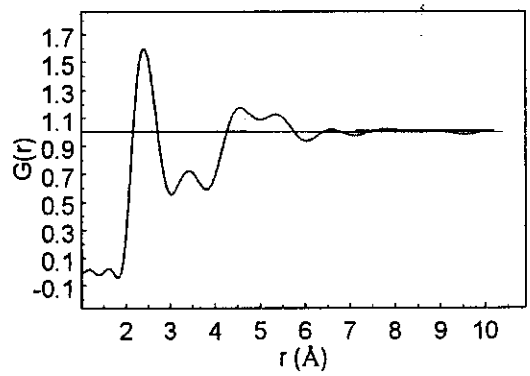

Fig. 2: Total atom pair correlation function G(r).
Temperature: T=260°C

References

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