	Experiment title: Chemical State and Local Geometry of Pb in Agx(PbCO3)y Derivatives	**Experiment number**: CH1195
Beamline: BM29	**Date of experiment**: from: 29 September 2001 to: 02 October 2001	**Date of report**: 30.08.02
Shifts: 9	**Local contact(s)**: Michael BOROWSKI	*Received at ESRF:*

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Report:

The proposal was motivated by the work reporting signature of superconductivity in a derivative of $Ag_xPb_6CO_9$ at a temperature as high as 340 K. Even though the superconductivity in this derivative was never confirmed, the system shows some anomalous electronic properties. The prposed work was aimed to explore the chemical state and local atomic displacements in this derivative $Ag_xPb_6CO_9$ with respect to the parent compounds $PbCO_3$ (cerussite, existing orthorhombic structure) PbO, PbO_2, Pb_2O_3 and Pb_3O_4, using high resolution Pb L-edge absorption measurements to distinguish the specialities.

During the assigned beamtime, we could measure high resolution Pb L-edge XANES to investigate the instantaneous local structure and the local density of states in powder samples of $PbCO_3$ (cerussite, existing orthorhombic structure) PbO, PbO_2, Pb_2O_3 and Pb_3O_4 and $Ag_xPb_6CO_9$ system. The measurements were made at low temperature using 13 element fluorescence detector. As a usual routine, the emphasis was to obtain the absorption data with high signal to noise ratio and for that several scans were collected to limit the noise level to the order of 10^{-4}.

A preliminary analysis to the XANES data show that the electronic states near the Fermi level in the title system appears quite similar to the one for the $PbCO_3$ (cerussite), however, the local environment is slightly

different, as evidenced by multiple scattering analysis of the Pb L-edge XANES. Detailed analysis of the experimental spectra and interpretaion of different spectral features by calculations of the Pb L-edge XANES using the full multiple scattering theory is underway.

A part of the beamtime was used for a study of local structure of Nb_3Ge intermetallic superconductor by the Ge K-edge absorption spectroscopy. The temperature dependent measurements were made using fluerescence detection mode. Standard procedure was used to extract the extended x-ray absorption fine structure (EXAFS) signal and corrected for the x-ray fluorescence self-absorption before the analysis.

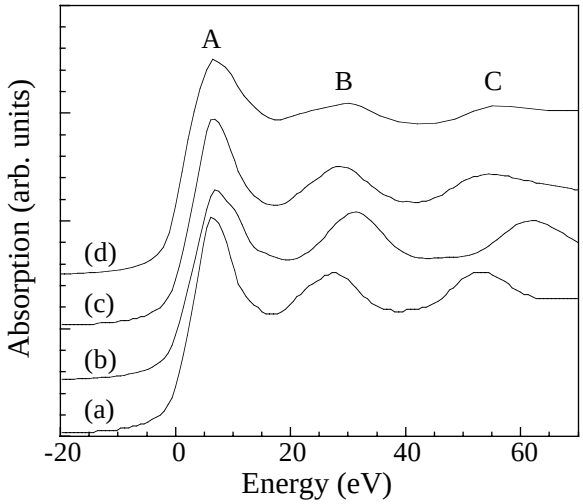

Fig. 1. Multiple scattering calculations for different Nb_3Ge structures; (a) with normal crystallographic Ge-Nb distance of 2.88 ; (b) with shorter Ge-Nb distance of 2.66 and (c) linear combination of the two spectra with probabilities of 0.67 and 0.33 respectively, compared with the experimental spectrum (d).

The EXAFS analysis shows two Ge-Nb distances, i.e., in addition to the crystallographic distance of ~2.88 , there exists a second Ge-Nb distance, shorter by ~0.2 from the average one, assigned to a phase with short range symmetry related to local displacements in the Nb-Nb chains. The X-ray absorption near-edge structure (XANES) spectrum has been simulated by full multiple-scattering calculations considering the local displacements determined by the EXAFS analysis. The XANES spectrum could be well reproduced by considering a cluster of 99 atoms within a radius of about 7 from the central Ge atom, and introducing the determined local displacements (see Fig. 1). The XANES results are found to be consistent with the EXAFS findings confirming a coexisting phase without any long-range crystallographic symmetry due to local displacements in the Nb-Nb chains, in addition to the normal crystallographic structure. The results are being communicated for the publication.

Due to insufficient time, the measurements on the Nb_3Ge were performed only at low temperature. The temperature dependent measurements are due to explore the interplay of inhomogeneous state and superconductivity of this system.