ESRF	**Experiment title:** Scandium K-edge microXANES in new scandium doped diborides	**Experiment number**: CH-1768
Beamline: ID21	**Date of experiment**: from: 15- september-2004 to: 20-september-2004	**Date of report**: 1-August-2006
Shifts: 15	**Local contact(s)**: Remi TUCOULOU TACHOUERES	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

***Stefano AGRESTINI,** *Matteo FILIPPI, Mauro GIOVANNINI, Adriana SACCONE, Naurang L. SAINI

Unità INFM and Dipartimento di Fisica, Università La Sapienza, Piazzale Aldo Moro 2, 00185, Roma (Italy)

Report:

We have performed measurements of the micro-Fluorescence (μ -XRF) and micro-XANES (μ-XANES) analysis on the phase separated samples belonging to the superconducting section $Mg_{1-x}Sc_xB_2$ for x>0.27, were we have found a phase separation into a Sc-poor and a Sc-rich region. The samples of $Mg_{1-x}Sc_xB_2$ used for the experiments were synthesized by a direct reaction method of elemental magnesium and scandium, boron. High resolution X-ray diffraction have indicated a phase separations for x>0.27. The phase separated samples exhibit a critical temperature slightly higher than the pristine MgB_2.

During the allocated beamtime our focus was to study the actual Sc distribution in the sample and find out if the distribution has any relation with the superconducting properties of the system. We have performed micro-Fluorescence (μ XRF), with a beam spot size below 1 μ m. The results are shown as Fluorescence image in Figure 1, revealing Sc-poor textures of 100 μ m size embedded in a Sc-matrix.

In additions, with μ -XANES we have studied the chemical state of the Sc in the Sc-poor and Sc-rich regions (Figure 2). The Sc K-edge μ -XANES spectra collected in the Sc-rich region

appears very similar to the one of ScB_2, indicating that the Sc coordination in this regions of the sample is the same (hexagonal coordination). The features of the spectra in the Sc-poor region are different from the spectra measured in the Sc-rich region showing typical features of an octahedral coordination, such as in the scandium oxide Sc_2O_3. Although details are to be understood, it is possible to state that the chemical state of Sc in the Sc-poor region is not diborides-like. It appears that the Sc does not enter in the MgB_2 lattice and the enhanced T_c of this system could be due to the strain, which can be induced by the Sc impurities or by the Sc rich matrix.

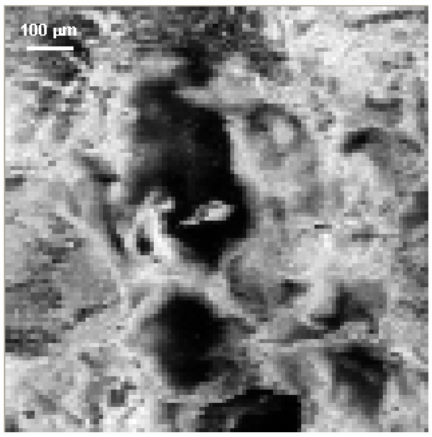

Figure 1: *μ–XRF fluorescence map of Sc Kα intensity on the sample with nominal Sc content x=0.28, with a resolution of 5μm. The Micron marker is shown*

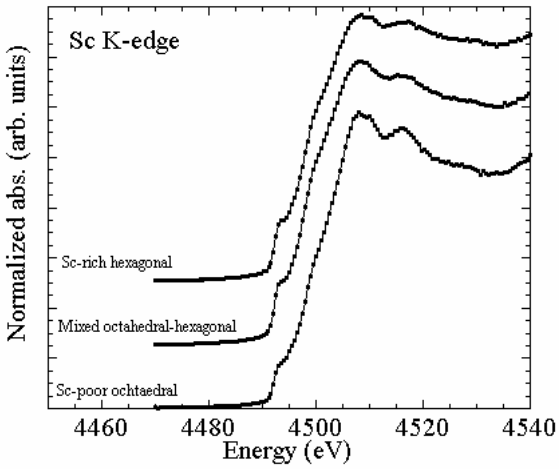

Figure 2: *Comparison between Sc K-edge μ-XANES spectra taken in different phases. The lower spectrum is taken in the Sc-poor while the upper in the Sc-rich phase. The middle spectrum exhibit both coordination*

The results are being published *Spectrochimica Acta B*

("XANES microscopy of phase separation in superconducting $Mg_{1-x}Sc_xB_2$" M. Filippi, S. Agrestini, L. Simonelli, N.L. Saini, A. Bianconi, S. De Negri, M. Giovannini, A. Saccone, *Spectrochimica Acta,* accepted)