ESRF	**Experiment title:** Isotope effect in the negative thermal expansion in diborides	**Experiment number**: HS-2969
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Report:

The aim of this experiment was to resolve the ambiguity occured during our last measurements through a systematic study of the isotopic effect on low-temperature thermal expansion in diborides. Infact in our first experiment (hs2729) we have performed the study of the low-temperature thermal expansion on $Al_xMg_{1-x}B_2$ system with x=0, 0.16, 0.50, synthesized using natural boron. We earlier found a large negative thermal expansion (NTE) in MgB_2 and we obtained that NTE is more than twice for x=0.16 and disappears for x=0.50 [1]. On the contrary in the last experiment (hs2729) we measured the thermal expansion on a sample of MgB_2 syntesized using pure B^{10} but a very small NTE was found.This result led us to ask for some beam-time to measure possible isotopic effect on low temperature thermal expansion.

During the allocated beam-time we performed our measurements on pairs of samples with B^{10} and B^{11} isotopes having the same Al content x and very similar room temperature diffraction profiles, i.e. the same values of *a* and *c*-axis and of peaks broadening and hence the same phase composition. We performed our experiments on four samples with x=0.00, 0.16, 0.28, and 0.37. The measurements were made at about 20 different temperatures in the range between 4 K to 295 K for all samples, while cooling and heating. Silicon powder was used as a standard for the experiments made using wavelength of λ=0.43 Å. Analysing the

diffraction profiles using Rietveld method we found that silicon shows a NTE along the a-axis of (1.5 ± 0.3)x10^{-5}Å·K^{-1} while cooling and of (1.9 ± 0.5) x10^{-5}Å·K^{-1} while heating between 5 and 100K. As for the samples under study we were able to identify two different phases within the pure MgB_2 compound and three phases in the Al doped compounds, i.e. a microscopic phase separation showing different thermal expansion coefficent.

In figure 1 we have plotted the variation of the volume of MgB_2 (B11) as a function of temperature showing a NTE.

As for the samples with x=0.16 and x=0.37 the results suggest that presence of phase separation with several phases with different thermal expansion properties hindering resolution of the NTE in the diborides. A detailed analysis of NTE in different phases is underway.

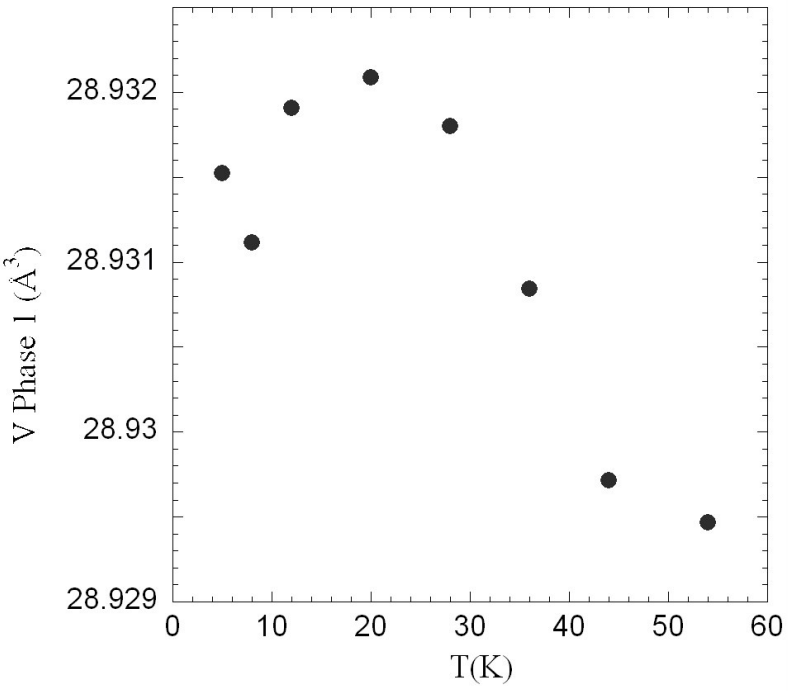

Fig.1 Thermal expansion of the *volume* of the dominant phases in MgB_2 (B11)

[1] V. Palmisano, S. Agrestini, G. Campi, M. Filippi, L. Simonelli, M. Fratini, A. Bianconi, S. De Negri, M. Giovannini, A. Saccone, A. N. Fitch, M. Brunelli and I. Margiolaki, *Journal of Superconductivity: Incorporating Novel Magnetism* **18**, 737 (2005).