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# Experiment Report Form

**The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF**.

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*http://193.49.43.2:8080/smis/servlet/UserUtils?start*

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## Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

## Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

| ESRF | **Experiment title: Interplay of magnetic ordering and lattice modulation in GdB6 and other heavy rare earth hexaborides** | **Experiment number**: HE-897 |
|---|---|---|
| **Beamline**: BM16 | **Date of experiment**: from: 14/09/2000 to: 18/09/2000 | **Date of report**: 27/ 08/ 2001 |
| **Shifts:** 12 | **Local contact(s)**: Andrew Fitch | *Received at ESRF:* |


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## Report:

The $RB_6$ series crystallise in a simple cubic, $CaB_6$ type structure. Despite the simplicity of the crystallographic structure, these compounds present very unusual electronic and magnetic properties.

In order to determine the symetry lowering associated to the magnetic ordered phase, in the heavy rare earth hexaborides, we performed the diffraction experiment on a $GdB_6$ and $DyB_6$ powder.

In the case of **$GdB_6$**, a first diffraction experiment on the single cristal [1], showed the presence of the superlatice reflections of the *[h/2 k l]* form in the high temperature ordered phase, and of *[h/2 k l]* and *[h/2*

*k/2 l]* forms in the low temperature phase; these reflections are $10^2$ greater than a reflection expected for magnetic scattering. In order to confirm this resultats and for a comprehensive search of the satellites wave vectors, we performed diffraction experiment on a $GdB_6$ powder, for temperatures: 4 K, 10 K, 11.5 K, 30 K and 295 K. No reflections of the *[h/2 k l]* form in the high temperature ordered phase, and *[h/2 k l]* and *[h/2 k/2 l]* forms in the low temperature phase, with an incident wave length $\lambda$= 0.35433 Å, could be observed (fig.1), which indicates that their intensities are less than $10^{-4}$ a lattice reflections. Neither splitting of the lattice peaks was observed, which indicates that even with a high incident X ray flux, we can't see such phenomena on a polycrystalline sample. It appears that the weakness of both the charges sattelites reflections and the magnetoelastic phenomena in $GdB_6$ requires X-ray investigations on single crystal only.

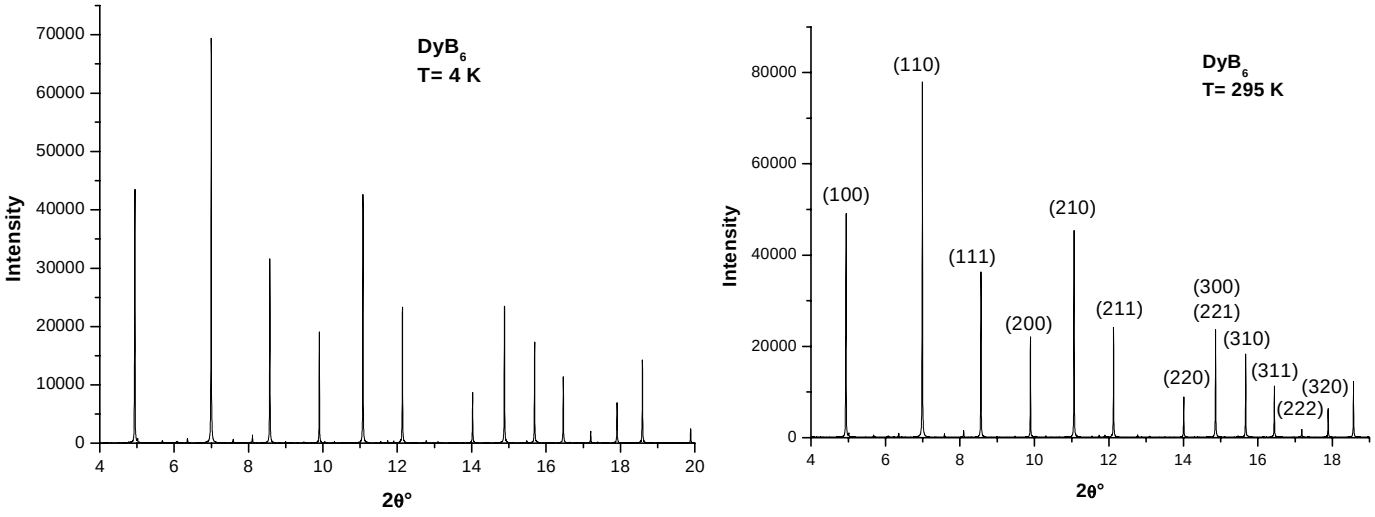


*Fig 1. Diffraction patterns for $GdB_6$ in the low temperature phase and at the room temperature*

Unlike $Gd^{+3}$ ion, $Dy^{+3}$ ion in **$DyB_6$** has an orbital momentum, so we can expect huge lattice effects in relation with a magnetic or orbital transition. $DyB_6$ is reported to present only one antiferromagnetic phase, below $T_N$= 26 K and an anomaly at $T_{JT}$= 35 K, which has been ascribed to a Jahn Teller (JT) effect [2]. In order to determine the supposed JT deformation in the ordered phases, we performed X-ray powder diffraction experiment for a series of temperatures, ranging from 5 K to 40 K. Due to the incongruent melting of Dy and B, the used sample contained extra phases of $DyB_4$ and $DyB_{12}$, which reflections can be easily distinguished from the ones of $DyB_6$.

We studied the temperature variation and the splitting of peaks *(110), (111), (200), (210)* and *(220)* (fig.2a) of $DyB_6$, in the two phases, in order to determine the symmetry lowering of the ordered structure. By checking the fashion of splitting of such peak, for different symmetry lowering, we determined the new symmetry of the sample, at this temperature. For example, the peaks corresponding to *(110)* must be double in the tetragonal and orthorhombic symmetry, and triple in the case of rhombic symmetry. So we could determine that the symmetry lowering on the ordered phase is of the trigonal mode with the new parameters are *a*=

*2.062 Å* and $\alpha$*= 90.403°*, at 10 K. We also determined the variation of the new lattice parameters with the temperature (fig. 2b).

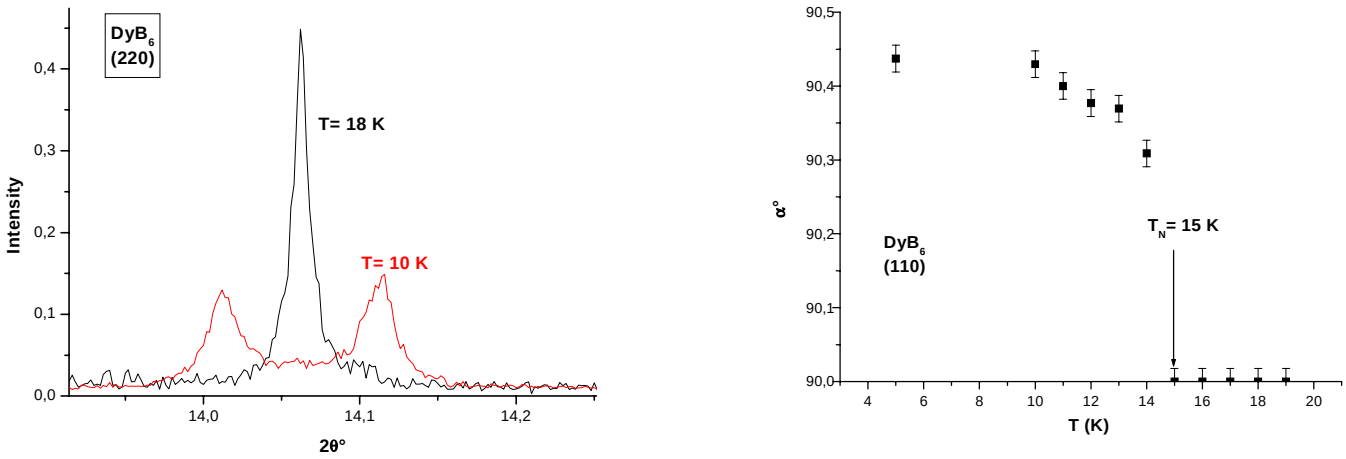


*Fig. 2a) Diffraction patterns for the (220) reflection, for two temperatures T= 10 K and T= 18 K, for $DyB_6$; 2b) Thermal variation of the parameter* $\alpha$ *in the ordered phase*

The scans made in variable temperature, from 10 K to 19 K allowed us to determine the transition temperature, $T_N$= 15 K, which is in agreement with the magnetic measurements made on this sample. For temperatures higher than 15 K, we didn't find any other structural change, so the second, Jahn Teller presumed, transition doesn't exist on this sample. Also the magnetic transition temperature doesn't correspond to the values from the literature. Different transition temperatures have also been found for different samples of $TbB_6$, which shows that the properties of these compounds depend drastically on the quality, crystallization and history of the sample. So the magnetic properties of the compound of this series are still an opened question. We propose an X-ray study on the single crystals, in order to determine the magnetoelastic phenomenon in these compounds.

**References:**

[1] R.M. Galera, D.P. Osterman, J.D. Axe, S. Kunii, T. Kasuya, *J. Appl. Phys*. **63** (1998) 3580- 3582
[2] T. Kasuya, *J.M.M.M*. **174** (1997) L28-L32
[3] K. Takahashi, H. Nojiri, K. Ohoyama, M. Ohashio, Y. Yamaguchi, M. Motokava, S. Kunii, *J.M.M.M*. **177-181** (1998) 1097-1098