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Report: **First observation of X-ray quadrupolar diffraction peaks**

NdMg is a cubic compound (CsCl-type) which displays a multiaxial magnetic structure below T_R = 35 K[1]. In the plane defined by the moments' directions, this structure corresponds to an antiferroquadrupolar arrangement with a [l/2 l/2 0] wave-vector. This magnetic structure is then suitable for the observation of X-ray antiferroquadrupolar diffraction peaks, at reciproqual space nodes Q = [h k l]+[l/2 l/2 0], where [h k l] is a reciprocal lattice vector.
The experiment for checking the existence of the quadrupolar Bragg reflections has been performed using the 7-circles goniometer of the D2AM beamline and a closed-cycle helium refrigator. As very weak signals had to be measured, the signal to background ratio had to be drastically optimised. In view of this, a short X-ray wavelength λ = 0.8943 Å, well below the Neodymium L absorption edges, has been used. To further decrease the background level, a Ge(111) analyser was mounted in front of the detector.

The enlightened face of the crystal, perpendicular with the [1 0 0] direction, was tilted in order to have the φ axis confused with the [1 1 0] direction. All measurements were performed in reflection conditions.

The sample was then cooled down to 19 K. Scanning the lattice reflections, it appeared that the tetragonal striction was large enough to separate in Q-space the peaks associated with the three magnetic domains. As only one domain could result in quadrupolar diffraction peaks in the accessible (h k 0) plane, we had to focuse on the corresponding reciprocal lattice.

We first focused on the [5/2 5/2 0] quadrupolar reflection which is expected to be the most intense. First, the background counting level was estimated about the [5/2 5/2 0] node, its value being a little less than 2 counts/s. Considering the order of magnitude of the expected quadrupolar reflection (some tenth of count per second), counting times of at least ten minutes per point were necessary for the peak to emerge from the background. Due to the ω extension of the Bragg reflections, a diagonal scan in the (h k 0) plane appeared to be the most appropriate process. Another advantage of this h,k scan was to measure the equivalent [5/2 5/2 0] nodes of the two other domains, which allows one to check the systematic emergence of peaks at such positions, in relation, for instance, with the h/2 harmonic of the monochromator. As result of these scans, with total counting time of 20 minutes per point (10^8 monitor counts), only one peak was clearly defined and located at the expected position for the quadrupolar scattering , whereas no h/2 peaks were identifiable. The maximum of the peak reached some more than 200 counts above the background, that is about 0.2 count per second, which is typically the expected order of magnitude.

Equivalent measurements were performed about the [3/2 5/2 0] reflection. This scan also revealed a peak at the expected position, even better defined than the previous one. In both cases, the peaks' Full-Widh-at-Half-Maximum are comparable to the lattice reflections close to these reciproqual space positions.

A check of these reflections at high temperature shows a background level increased by about 40%, due to the thermal incoherent scattering of the lattice, but peaks could no longer be identified at the [5/2 5/2 0] and [3/2 5/2 0] positions.

In summary, the existence of diffraction peaks at the positions and with the order of magnitude expected for quadrupolar reflections has been established in NdMg. These peaks disappear as the temperature is raised up to the paramagnetic range and cannot be ascribed to a l/2 pollution. They have been observed despite an unfavourable domain partition of the crystal and using a Neodymium based sample, which doesn't correspond to the maximum quadrupolar scattering amplitude among the rare-earths. This experiment demonstrates that the tiny X-ray reflections associated with 4f orbital orderings can be efficiently measured thanks to the high flux available with synchrotron radiation.

[l] M. Deldem, M. Amara, R.M. Galéra, P. Morin, D. Schmitt and B. Ouladdiaf, *Multiaxial magnetic ordering in NdMg*, Journal of Physics : Condensed Matter 10(1) (1998) 165.