ESRF	**Experiment title:** The low-temperature structure of bornite - Cu_5FeS_4	**Experiment number**: MA 3409
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Report:

Introduction

The aim of the present experiment was the study of the eventual occurrence of a polymorph transition of the mineral bornite, Cu_5FeS_4, one the most interesting natural sulphides for its peculiar magnetic properties, whose origin is far to be understood.

Previous studies on this minerals, carried out by many authors, including our research group, during the last 5 decades, have pointed out the occurrence of a magnetic transition well below the liquid Nitrogen temperature. Some of them have also suggested that at 35 K some anomalies in the magnetic behaviour could trace further eventual structural rearrangements. Original (unpublished) Neutron Diffraction data, carried out by us at ILL, confirned the main transition towards an antiferromagnetic (AF) state at 67.5 K, and pointed out a complex magnetic structure, for the solution of which a high resolution synchrotron X-ray diffraction resulted necessary.

Experimental details and procedures

Two bornite samples were brought to the beamline: a natural specimen, coming from the Montecatini Val di Cecina (Tuscany, Italy) mine, and belonging to the collection of the Natural Hystory Museum of the Unversity of Florence, and a synthetic analogue, obtained by laboratory runs carried out under the conventional dry synthesis technique.

The two samples were characterised in previous publications by our group. We want to emphasize that the natural bornite from Montecatini Val di Cecina belong to a very pure Cu sulfide mineralisation, and it can be considered as a sort of standard for this mineral species.

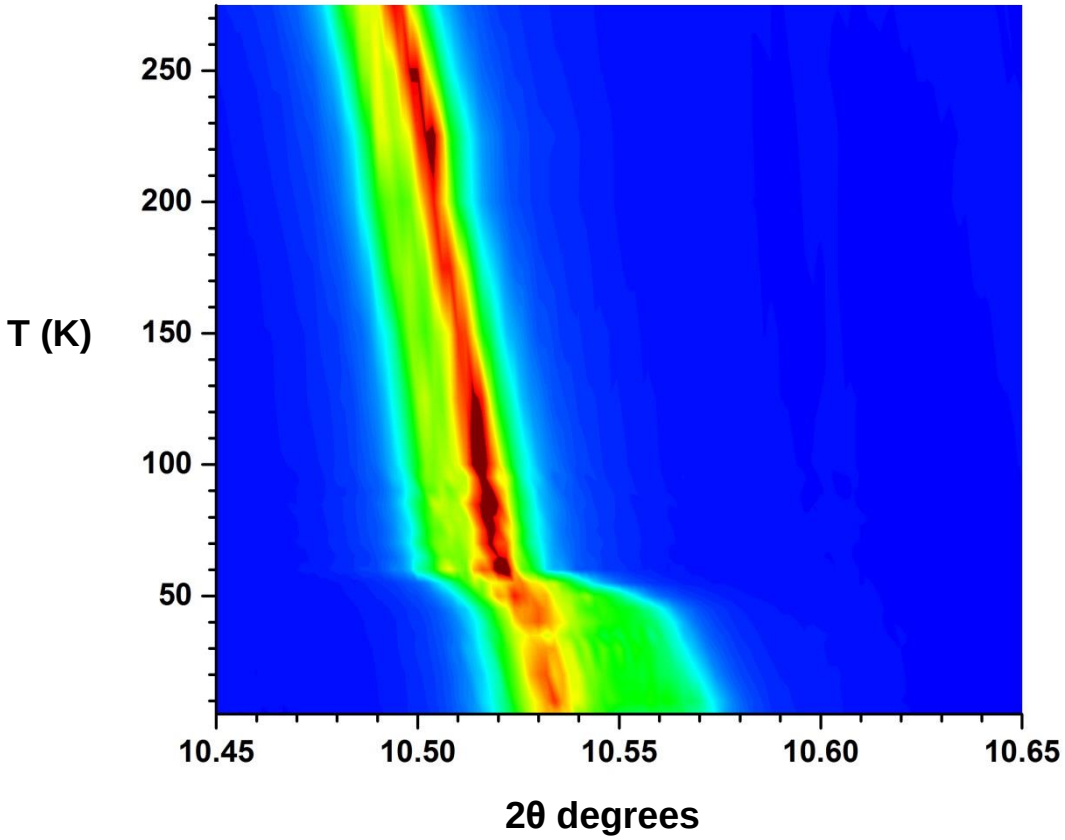
Although resulting almost pure at the preliminary investigation carried out by laboratory powder XRD (the sum of the impurities being in the order of max 1 wt%), the preliminary pattern at the ID22 diffractometer revealed the presence of traces of chalcocite (Cu_2S) as impurity.

Both samples were investigated after having put a small amunt of very fine particles into quartz capillary, left open to exchange the air during the cooling procedure. The tubes were mounted on a rotating sample holder

in the cryostat, and investigated between 0.512 and 52.912 2θ degrees, step size being 0.002 2 θ degrees. The operating wavelength was 0.35417 Å (corresponding to an energy of 35.007 KeV). The two samples were investigated every 5 or 10 K degrees, in the range 5 – 275 K, after having equilibrated the sample to the desired temperature value. The smaller temperature step was realised in the T range were the supposed transitions were hypothesized.
No peculiar problems occurred during the experimental beamtime at ID22, apart from 4 hours of beamtime lost due to a failure in the storage ring.

Preliminary results

The preliminary results obtained on the two samples are well depicted by the Figure reported here below.



In this graph, the behaviour of the main reflection of bornite versus temperature is highlighted (horizontal axis, 2θ degrees; vertical axis, temperature in K; false color convention). Apparently, a monotonic change of unit cell parameters and volume is occurring in the whole T range between 275 and 60 K. Below this temperature a net shift of the peak, coupled to change of its width and asymmetry is apparent.
From this consideration (studied also in relation to the temperature behaviour of the chalcocite impurity, for which the transition is not observed), we can preliminarly conclude that a low temperature polymorh of bornite exists, and further analysis of the data will reveal it structural features, in relation to the AF transition occurring in the same T range.