HS3274

STUDIES OF THE VERWEY PHASE TRANSITION IN MAGNETITE

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This experiment was the continuation of the experiment HS2806 [1], and was aimed to observe the temporal changes of magnetite Fe_3O_4 lattice symmetry occurring at the Verwey transition and to study the fluctuations of the lattice close and at the transition. No such studies were conducted on this material till now.

At the Verwey transition at T_V=125K large latent heat manifests the abrupt change of major physical characteristics, e.g. the crystal symmetry, that turns from monoclinic (space group *Cc*) below T_V to cubic *Fd-3m*. Despite 60 years of interest the transition is still not entirely understood. In view of that, we have set up the project aimed to simultaneously observe how magnetic susceptibility χ_{AC}, electrical resistivity ρ and the specific heat change exactly at the transition [2]. Due to large latent heat of transition, the time of this observation may be largely extended. In the present experiment we added yet another characteristics, crystal lattice symmetry, that can be observed simultaneously with others mentioned above, i.e. while the transition develops.

Our experiment was conducted in ID10A on Troika 1, with the *E*=7.1 keV radiation. Two samples of stoichiometric magnetite, each ca. 0.5 g, were measured. One was cut parallel to (110) plane (hereafter referred to as "110" sample), the other parallel to (553) plane ("553" sample). For each measurement the miniature Pt1000 thermometer (ca. 1X1X0.5 mm^3) was glued on top of the sample, adjacent to the beam, to precisely monitor samples' temperature. Additionally, the special setup (Fig.1) for the observation of AC susceptibility was constructed (two pairs of coils, wound together, one of each supplied AC magnetic field of f=125Hz, the other were pick up coils; the subtracted signal was measured by the lockin amplifier hooked up to SPEC) and

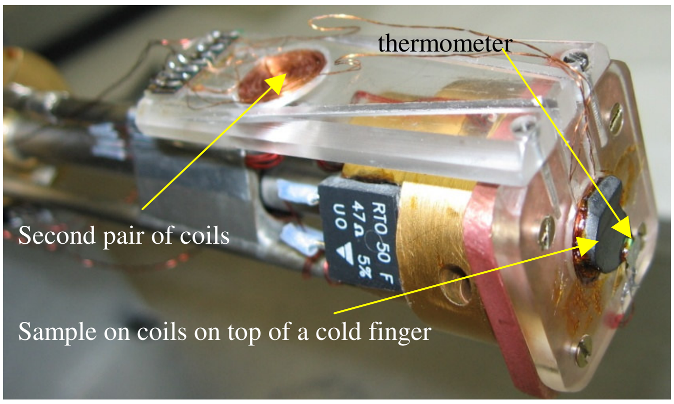

Fig. 1. The sample on top of AC susceptibility setup (attached to the cryostat cold finger) and with Pt1000 therometer

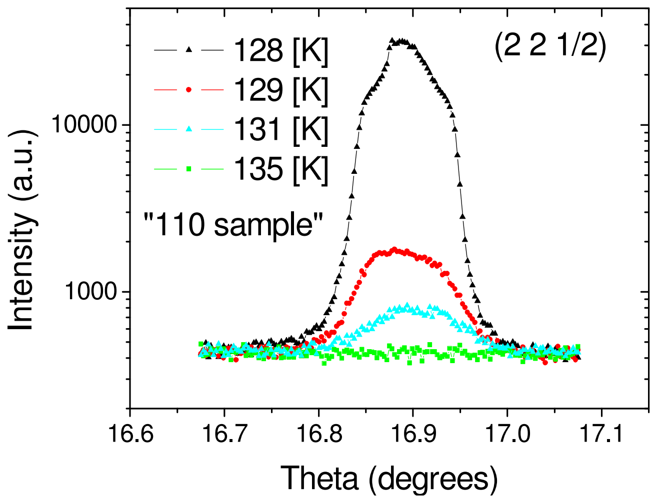

Fig. 2. Temperature dependence of part of superlattice peak Q-scan of just above the transition for "110 sample"

was used for 553 sample measurements. This setup was also treated as the heat barrier between the sample and the cold finger that should allow for the long time of transition observation. For each sample, the three primary high-T phase peaks were found (to calculate the orientation matrix), the superlattice peaks ((2 2 ½) in case of "110" sample and (1 1½ 2) in case of "553" sample) were located and traced through the Verwey transition, first by the point detector and then by CCD camera. In this last case, we wanted to observe crystal lattice dynamics, making use of the coherent beam (we had to diminish the before-the-sample slit openings that critically lessened the number of diffracted photons). The most important results (as judged from the still rudimentary analysis) are:

1. Characteristic diffuse scattering was found for “110” sample just above the transition (see Fig. 2), despite the fact that the transition is discontinuous. This resembles the neutron measurements results in [3] and will be the subject of the next proposal for the beamtime in ESRF.

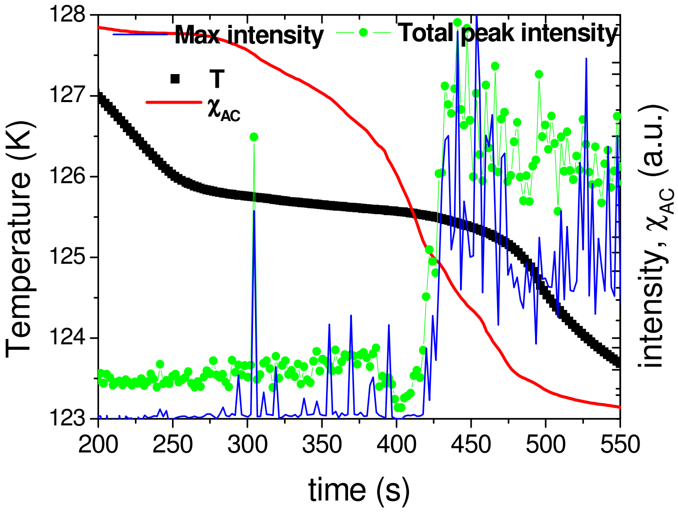

Fig. 3. Time profile of temperature, AC susceptibility and the intensity of (1 1½ 2) superlattice peak of “553” sample at the Verwey transition

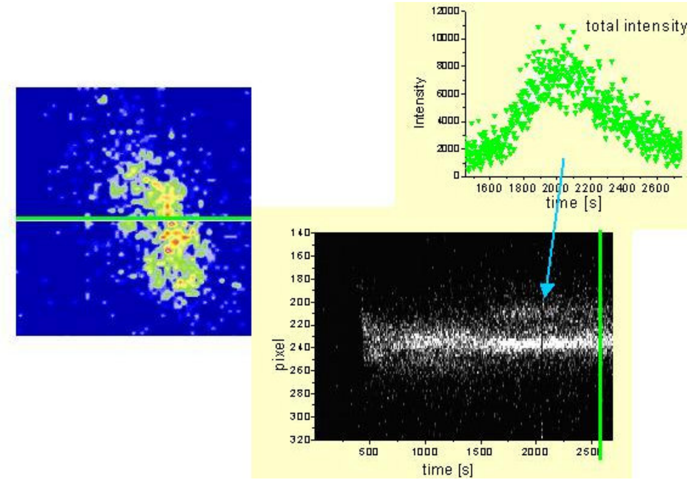

Fig. 4. The evidence of a long time dynamics in (1 1½ 2) superlattice peak of “553” sample at and slightly below the Verwey transition. The left panel is a typical slide from CCD with the peak profile. Time dependence of the green cut through the peak is shown in a lower panel. The details of the long-lasting fluctuation (pointed by the arrow) are shown on the upper panel

2. The superstructure peak fully appears at the end of the transition, as observed on cooling (fig. 3), i.e. where both T profile-plateau and a χ_{AC} step already signal the proximity of a new high T phase. The same result is valid for cooling: the superlattice peak always appears close to low-T side of a transition. This is an important point and may suggest that the Verwey transition is caused by the structural changes that trigger the transformation of other subsystems.
3. Although we did not find the typical speckles temporal changes as observed e.g. in [4], it may mean that time scale of lattice fluctuations at the Verwey transition is beyond the time window probed by XPCS technique. In fact, we have observed traces of both very fast and very slow dynamics, the last one lasting hundreds of seconds, as shown on Fig. 4.
4. The changes of the (1 1½ 2) peak center of gravity (observed on CCD camera, but not shown here) most probably are due to structural twins (visible also in the emerging splitting of (2 2 ½) reflection on Fig.2) dynamics. This problem is the subject of analysis which si still in its introductory form.

In the our opinion, the HS3274 experiment was successful: almost all from the planned issues were addressed. We have not have, however, enough beamtime to further study the Q scans for “553” sample as well as we could not use the AC susceptibility setup for “110” sample due to problems with heat transfer between the sample and the cold finger. Finally, the problem of diffuse scattering at $T>T_V$, found for “110” was not even attempted for “553” ((1 1½ 2) dynamics was observed the last night, just before the beam time was over): this should be thoroughly studied in a separate experiment.

We highly appreciate the cooperation with ID10A team. Especially, the help offered by Peter van der Linden can not be overestimated.

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