ESRF	**Experiment title:** Low-temperature charge order in NaV_2O_5	**Experiment number:** HS-1427
Beamline: ID10A	**Date of experiment:** from: 13/06/01 to: 18/06/01	**Date of report:** 21/08/01
Shifts: 17	**Local contact(s):** Dr. Federico Zontone	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):
Prof. Dr. Sander van Smaalen,*
Dr. Peter Daniels,*
Mr. Lukas Palatinus,*

Laboratory of Crystallography,
University of Bayreuth, D-95440 Bayreuth,
Germany

E-mail: smash@uni-bayreuth.de

Report:
The goal of this experiment was to study the low-temperature superstructure of NaV_2O_5. The phase transition in NaV_2O_5 at $T_c = 34$ K is characterized by the development of magnetic order and by the development of superlattice reflections in the x-ray scattering. General agreement exists, that the phase transition is associated with charge order on the vanadium sublattice. In a previous study with synchrotron radiation at the Hasylab synchrotron we have determined the superstructure with space group Fmm2 on an $2a \times 2b \times 4c$ supercell [1,2]. Particularly based on NMR experiments [3,4], the correctness of this structure was questioned, and it was proposed that presumed additional superlattice reflections might have been overlooked, or that the superstructure has monoclinic symmetry.
In the present experiment it was necessary to measure the intensities and shapes of a series of Bragg reflections in various directions at low temperatures (T = 20 K). This required a four-circle diffractometer equiped with a Helium Cryostat. The set-up at beamline ID10A gave the full functionality of a four-circle diffractometer, whereby the long collimator in front of the detector was an important aspect to suppress background radiation and to achieve a great sensitivity towards weak scattering effects. A problem was encountered with the cryostat owned by the beamline. Due to its special design it imposed too many restrictions on the available movements of the diffractometer. This problem was solved by using a borrowed cryostat. In possible future experiments, we intend to bring our own closed-cycle He cryostat, which fits into the HUBER cradle and which allows sufficient freedom for the omega, chi and phi movements.

With the borrowed cryostat we were able to measure about 80 reflections at T = 20 K on the primitive $2a \times 2b \times 4c$ superlattice. Intensities were not observed at any of the measured positions that were forbidden by the F-center (51 reflections), while all first-order and second-order satellites did have non-zero intensities. We thus established that the true superlattice is indeed F-centered on a $2a \times 2b \times 4c$ supercell. The profiles of the Bragg reflections in various directions were found to be slightly broader at 20 K than above the phase transition at 40 K, but splitting of reflections was not observed. Thus we could establish the absence of monoclinic distortions of the lattice.
The results of this experiment at ID10A were used for new refinements using intensity data of the first-order satellites as collected by Bernert *et al.* [5]. It was found that the true structure of NaV_2O_5 might be a stacking of layers with zigzag charge order on all ladders, that shows stacking disorder such that the average structure has space group Fmm2 on the $2a \times 2b \times 4c$ supercell. Variations in the stackings then also provide an explanation for the devil's staircase behavior of $2a \times 2b \times 4c$ supercell, as has recently been observed under pressure [6].

The results were summarized in a manuscript, that was submitted to Phys. Rev. Lett. and to the condensed matter e-print server:
S. van Smaalen, P. Daniels, L. Palatinus, and R.K. Kremer (2001) cond-mat/0108067.

References

[1] J. Lüdecke *et al.* (1999) Phys. Rev. Lett. **82**, 3633.
[2] S. van Smaalen and J. Lüdecke (2000) Europhys. Lett. **49**, 250.
[3] Y. Fagot-Revurat et al. (2000) Phys. Rev. Lett. **84**, 4176.
[4] T. Ohama *et al.* (2000) J. Phys. Soc. Jpn. **69**, 2751.
[5] A. Bernert *et al.* (2001) Eur. Phys. J. B **21**, 535.
[6] K. Ohwada *et al.* (2001) cond-mat/0104286v1.