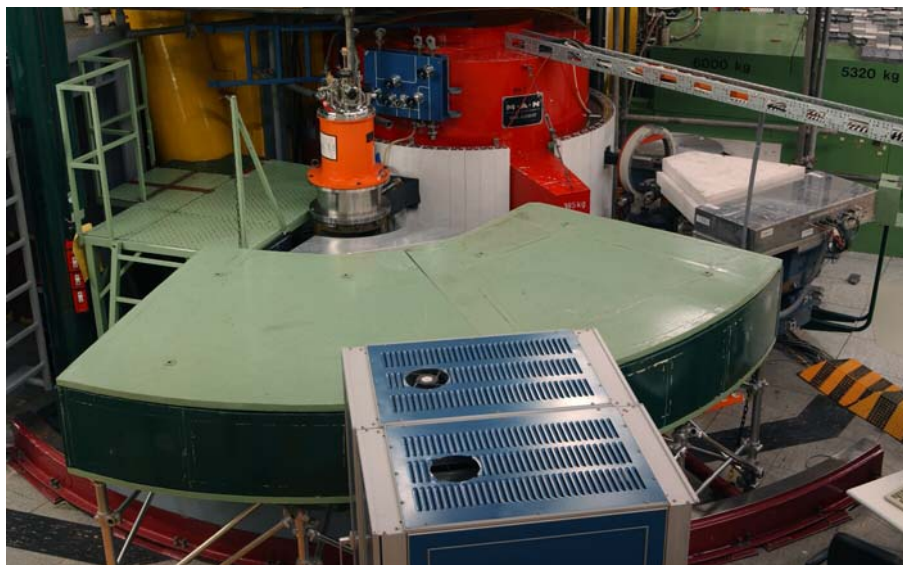


Twin Diffractometer (SV7)



Instrument Parameters

Monochromators - <i>standard:</i> - <i>optional:</i>	Ni (220), PG (002) Ni (200), Cu (111), Pb (311), Ge (111)/(311)
Monochromator angles:	52° and 40°
Wavelengths:	$1.0 \text{ \AA} \leq \lambda \leq 2.3 \text{ \AA}$
Collimators - <i>primary beam:</i> - <i>scattered beam (optional):</i>	Soller: 12 min radially oscillating
Max. beam size:	$25 \times 40 \text{ mm}^2$
Mean sample volume (cylinder):	10 mm Ø, 30 mm high
Scattering region:	$0^\circ \leq 2\Theta \leq 90^\circ$
Detectors:	linear JULIOS units
Max. neutron flux at sample:	$10^6 \text{ n/cm}^2 \text{ s}$
d-spacings:	$0.7 \text{ \AA} \leq d \leq 35 \text{ \AA}$
Mean resolution $\Delta d/d$:	10^{-2}
Sample environment:	Closed cycle He-cryostat: 4 ... 293 K; He-bath cryostat: 1.5 ... 4.2 K; He-3 cryostat: 0.3 ... 293 K; Split coil magnet cryostat: 0 ... 7 T, 4 ... 293 K; cryofurnace 4 ... 400 K; Full circle goniometer with external ω -rotation: 293 K; Flow cryostat containing full circle goniometer: 4 ... 293 K

Instrument Responsible

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Rolf Skowronek

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Email: j.walter@fz-juelich.de
Email: r.skowronek@fz-juelich.de



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-04-001		
Experiment title:	Texture analysis of antique Dutch tin spoons from Amsterdam: 1350-1750 AD		
Dates of experiment:	30.01.05-03.02.05 03.03.05-04.03.05	Date of report:	14.06.2006
Experimental team: Names	Addresses		
Visser, Dirk Hallebeek, Peter Kockelmann, Winfried Veerkamp, Jorgen	Department of Physics, University of Warwick, Gibbet Hill Road, Coventry CV4 7 AL, UK and NWO-Physics, ISIS Facility, RAL, Chilton, Didcot OX11 0QX, UK Netherlands Institut for cultural Heritage (ICN) Gabriel Metsustraat 8, Postbus 76709 1070 KA Amsterdam, NL Mineralogisch-Petrologisches Institut , Universität Bonn, Poppelsdorfer Schloss, D- 53115 Bonn Archeology Amsterdam, Noordermarkt 45, 1015NA Amsterdam, NL.		
Local Contact:	J. M. Walter		

Experimental report text body

Introduction

The Department of Archeology of Amsterdam has a collection of 861 tin spoons of which the earliest examples are dated around 1350 and the last ones around 1750. At this time the production of tin spoons in Amsterdam ceased. In order to make tin (Sn) usefull for appliances, it has to be alloyed with another material to make it harder. Often used alloying metals are lead (Pb) or copper (Cu). At that time it was already known that a too high percentage of Pb caused a public health hazard. A safe percentage should not be higher than 10%. It is known that in the early period one often did not adhere to this rule due to the fact that lead was much cheaper than tin. After 1530, the production of tin spoons in Amsterdam was regulated and overseen by the Guild. Also other alloying metals were used, e.g. Cu or even Cd. The amount of Cu in Sn should be determined by its solubility, in general only a few percent. However, objects have been found with much higher Cu-concentrations, up to 20% (15th century Coventry [1]). The alloying with Pb shows, however, a much larger distribution.

Measurements and Results

Apart from the main phase composition of the spoons, obtained on ROTAX, we obtained more information on the production methods provided by texture analysis. Complementary to measurements on ROTAX, where we could obtain semi-quantitative texture information on some of the spoons, we in these measurements confirm these results and obtained more quantitative information on the crystallographic orientation distributions of more objects. We conducted texture measurements of fourteen spoons, distinguishing between different parts of the spoons as some of the spoons have also an iron core in the handle. Additionally production methods may vary for different parts of the spoons. For all the spoons we related the existing metallurgy data of the objects. Due to the form anisotropy of the spoons, it was not possible to measure complete pole figures of the spoons,

which would in this case also show form texture. Therefore only distinct ϕ -rotations in certain χ -tilts were selected for the measurements. From the resulting cascading diffraction patterns as a function of the orientation, it is possible to obtain qualitatively by the comparison of the different peak heights, whether the spoon was casted in a mould from molten material or worked by mechanical force. Spoon parts that were casted in a mould show only distinct singular peak heights of single crystallites (see Fig. 1), whereas mechanically worked spoon parts show a classical bulk texture distribution, which varies in a wavy distribution depending on the measured angle (see Fig. 2).

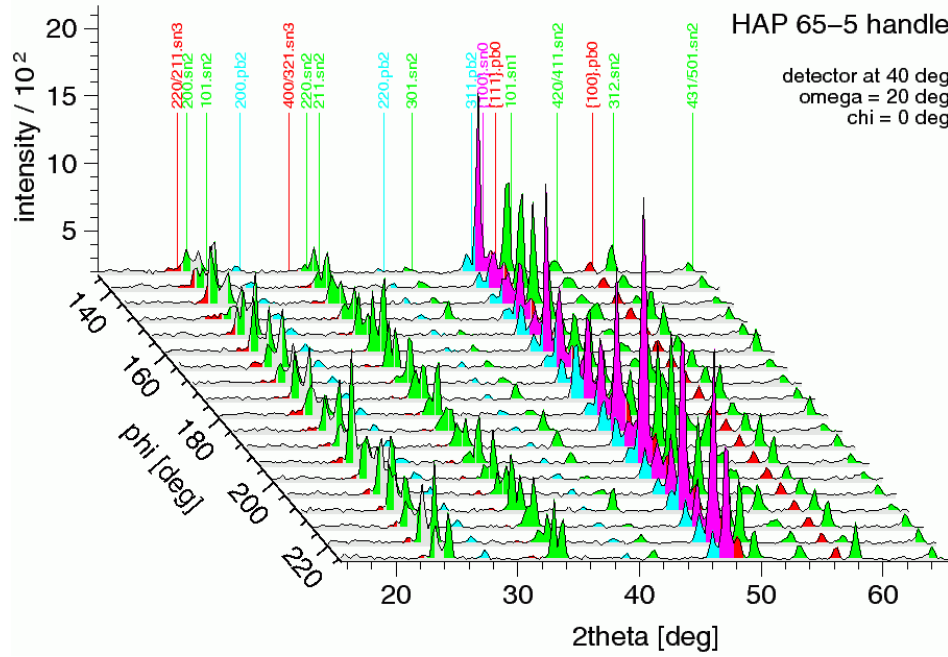


Fig.1: Casted spoon handle shows single crystals not in preferred orientation

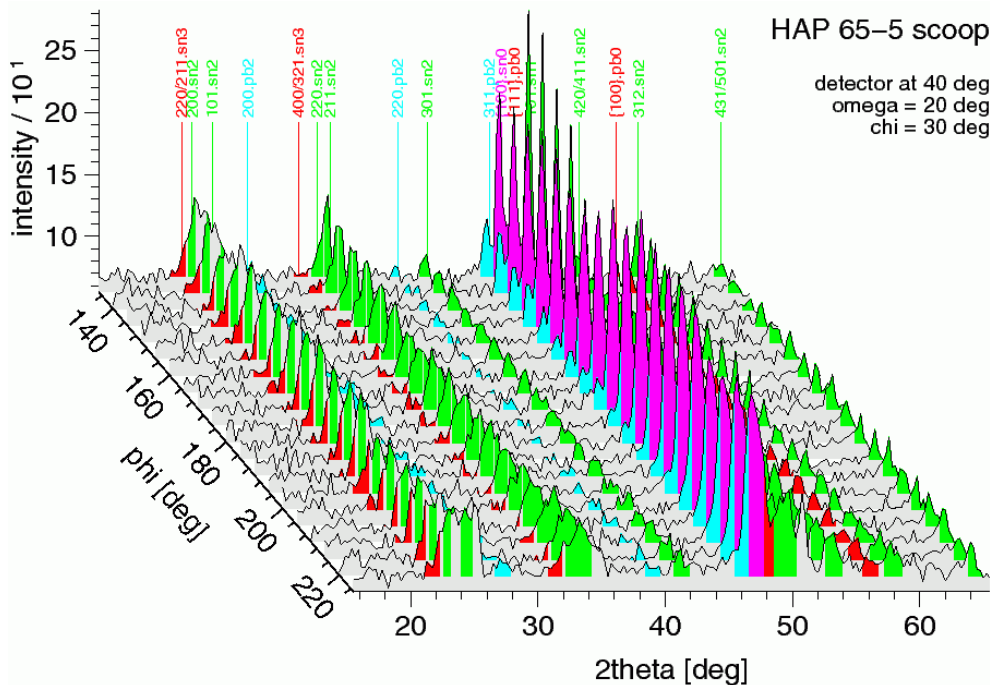


Fig. 2: Mechanically worked spoon scoop shows crystallographic preferred orientation



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-04-007		
Experiment title:	Neutron diffraction studies of magnetic transitions in TmNi₄Al - continuation		
Dates of experiment:	2005	Date of report:	23.03.2006
Experimental team: Names	Addresses		
T. Toliński, A. Kowalczyk	Institute of Molecular Physics Polish Academy of Sciences Smoluchowskiego 17 60-179 Poznań, Poland		
A. Hoser R. Skowronek	Institut fuer Kristallography, Aachen MIN/ZFR (Univ. of Bonn)		
Local Contact:	Dr. A. Hoser		

Experimental report text body

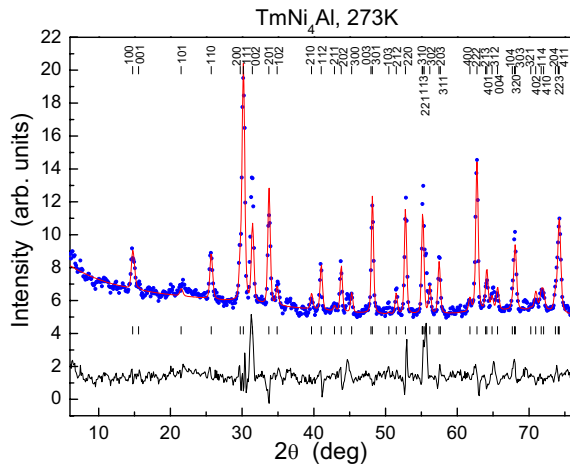
The RN₄Al compounds (R = rare earth or Y) crystallize in the hexagonal CaCu₅-type of structure (Fig. 1), point group *P6/mmm*. Ce occupies the 1a site (0,0,0) and Ni(1) the 2c site (1/3,2/3,0). Ni(2) and Al are statistically distributed over the 3g site (1/2,0,1/2) [1].

In the year 2005 we performed the second part of the measurements on the TmNi₄Al compound using the SV7 instrument. The polycrystalline TmNi₄Al compounds were synthesized by induction melting under an argon atmosphere. As the ingot a stoichiometric amounts of the constituent elements were used. The neutron wavelength was equal to 1.096 Å. The sample was contained in a cylindrical vanadium can mounted (1) in a refrigerator cryostat and (2) in a cryomagnet with external magnetic fields up to 5 T perpendicular to the horizontal diffraction plane. The magnetometric measurements were performed using a MagLab 2000 System (Oxford Instruments Ltd.).

Magnetometric measurements for TmNi₄Al have shown a double peak in the ac magnetic susceptibility at 5.8 K and 2.8 K (Fig. 2), contrary to the other RN₄Al members (R = rare earth) showing a single phase transition. The first part of the neutron diffraction experiments revealed only an increase of the low angle peaks' intensities between 4.2 K and 1.8 K. Therefore, the two peaks in the ac susceptibility may be due to a switch between two different slightly non-collinear orders of the magnetic moments. Tm and its alloys are known to create non-collinear magnetic phases, therefore this same can be expected in the case of TmNi₄Al.

The second part of the neutron diffraction experiment have shown that, alternatively, the two peaks in the ac magnetic susceptibility may be due to a small distortion of the crystallographic structure. The latter possibility results from the observed broadening of the diffraction peaks (Fig. 3). Besides, the effect of the magnetic field has been studied in more details. It appears that numerous reflections disappear after application of the magnetic field - this behavior is still not fully explained.

The analysis of the magnetic structure shows the magnetic moments ordering in the c – direction, whereas in the case of RNi_4Al for $R = Nd, Tb$, and Dy the magnetic moments have ordered within the hexagonal plane [1-3]. It can be explained by the crystalline electric field (CEF) effect and the change of the sign of the related second order Stevens factor [4].





Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-04-013		
Experiment title:	Study of the stress on 16NiCrMo quenched steel		
Dates of experiment:	28.01.2005-1.02.2005	Date of report:	15.02.2006
Experimental team: Names	Addresses		
Salvatore Magazù	Dipartimento di Fisica Universita' di Messina C.da Papardo Sperone S. Agata 98166 Messina Italy phone: 0039-090-6765025 fax: 0039-090-395004		
Local Contact:	Dr. Jens M. Walter		

Experimental report text body

Turning is one of the most employed processing technologies for mechanical components production. Continuous efforts of the manufacturers have always been oriented on the study and research of cutting tools production to allow a higher costs-saving preserving product quality. In these last years the research has been involved on the development of stronger tool materials, able to resist to the thermo-mechanical stresses that are present during machining, with the aim to increase the tool-life. The new design of the tool corner radius has allowed preserving the same product quality due to the “wiper effect” generated during the cut.

In recent times different tools that can exploit the wiper effect have been produced. They allow substantial productivity improvements or, which is the same, to reduce the time of cut and then to reduce production costs. In particular, if the feed can be increased to double, the cutting time is halved and almost twice as many components are made in the same time maintaining the same surface finish. Otherwise by employing the same feed rate it is possible to achieve excellent surface finish and eliminate, in such cases, the grinding operation.

With the aim to verify the possibility to realize good finishing on hardened materials with the above mentioned new tool-manufacturing technology we used SV7 to perform a set of measurements on 16NiCrMo quenched steel. The main aim is to compare the quality of the manufactured product with the one of the same workpiece obtained by turning with traditional tools and grinding and to completely determine the mechanical performances of such products.

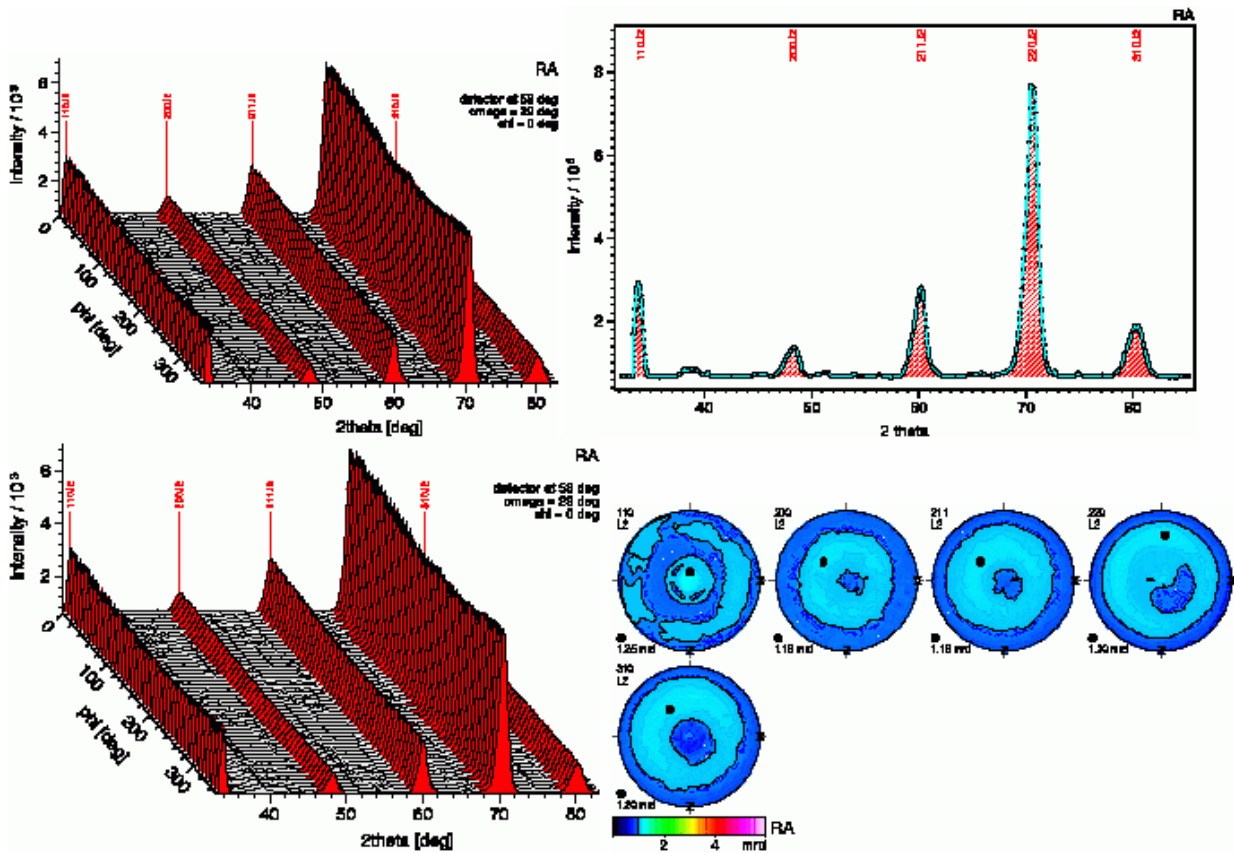
The samples were cementation steels 16NiCr4 which have been submitted to carbo-cementation treatment and therefore they present an external layer rich of carbon which has been transformed in martensite after hardening treatment.

Before the neutron measurements, the samples have been treated by machine-tools with wiper tools, by varying the treatment parameters. One of the samples has been ground for having the opportunity to do the comparison. We measured the hardness of the samples, before other treatments, obtaining for the cemented external layer the value of 58 HRC or 1050HV, for the intermediate un-cemented layer the value of 38 HRC or 750 HV and for the core the value of 20 HRC or 500 HV.

The samples are of three different kinds:

- Sample 1: it has been turned with wiper tools (tool covered by CBN; Radius=0,8mm; V=150 m/1'; a=0,1mm/rev; final ruggedness Ra=0,4micron);
- Sample 2: it has been turned with wiper tools (ceramic tool 6050; Radius=0,8mm; V=250 m/1'; a=0,1mm/rev; final ruggedness Ra=0,6micron);
- Sample RA: it has been ground (Ra = 1micron).

In Figure the results concerning Sample RA are shown. For iron we calculated the pole figures from the diffraction data. The orientation of the pole figures is the direction of view into the pole figure is parallel to the long axis of the cylindrical core samples.



The comparison among the three samples has been performed. At the present we are performing other measurements by using complementary techniques. The production of other samples obtained by using the two methods is also in progress.



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-04-015		
Experiment title:	Correlation between structural and magnetic properties of $\text{La}_{7/8}\text{Sr}_{1/8}\text{Mn}_{1-\gamma}\text{O}_{3+\delta}$ with controlled nonstoichiometry		
Dates of experiment:	09.03.05-11.03.05 14.06.05-26.06.05	Date of report:	28.03.06
Experimental team: Names	Addresses		
Mr. H. F. Li Dr. Y. Su Mr. J. Persson Dr. P. Meuffels Prof. Dr. Th. Brueckel	Institute of Solid State Research, Research Center Jülich, D-52425 Jülich, Germany		
Local Contact:	Mr. R. Skowronek, Dr. J. Walter		

Experimental report text body

Introduction

The defect chemistry such as cation nonstoichiometry and excess oxygen is a crucial issue for the understanding of manganites. It determines e.g. the orthorhombic-rhombohedral phase transition and the value of the Curie temperature T_c . Moreover, nonstoichiometric manganites are of extremely interest in application to the solid oxide fuel cells. Structural as well as physical properties are very sensitive to the synthesis conditions that can control the relative amount of Mn^{3+} and Mn^{4+} ions. In this report, We performed simultaneous Rietveld refinement combining both neutron and X-ray powder diffraction data of air sintered, Ar and O_2 annealed (1 atm pressure) $\text{La}_{0.875}\text{Sr}_{0.125}\text{Mn}_{1-\gamma}\text{O}_{3+\delta}$ samples.

Results

In order to explore the effect of Ar and O_2 annealings on the magnetic properties, the zero-field-cooling (ZFC) and field-cooling (FC) magnetizations as a function of temperature at an applied field of 20 Oe for the three samples were measured (Fig. 1(a)). Structural parameters were determined by the Rietveld method using the FULLPROF program. For each sample, scale factors, zero shifts, lattice constants, atomic positions, isotropic thermal parameters and the occupancies of Mn and oxygen were refined. From Figs. 2(A) and 2(B), one may observe three Bragg reflections, i.e. [400], [224] and [040] in NPD pattern [Fig. 2(a)] and one [111] in XPD pattern [Fig. 2(a')] of the Ar annealed sample. For the air sintered sample, the three reflections degenerate into two reflections [220] and [208] [Fig. 2(b)] and the [111] reflection disappears [Fig. 2(b')]. This shows that the Ar annealed sample has an orthorhombic structure ($Pbnm$, $Z = 4$; O), while the air and O_2 annealed samples belong to the rhombohedral space group ($R\bar{3}c$, $Z = 2$; R). The refined formulas of Ar annealed, air sintered and O_2 annealed samples are $\text{La}_{0.875}\text{Sr}_{0.125}\text{Mn}_{0.960(8)}\text{O}_{3.024(4)}$ (Ar), $\text{La}_{0.875}\text{Sr}_{0.125}\text{Mn}_{0.986(8)}\text{O}_{3.090(2)}$ (Air) and $\text{La}_{0.875}\text{Sr}_{0.125}\text{Mn}_{0.985(8)}\text{O}_{3.144(8)}$ (O_2), and they can be expressed as $\text{La}_{0.868(1)}\text{Sr}_{0.124(1)}\text{Mn}_{0.952(8)}\text{O}_3$ (Ar),

$\text{La}_{0.850(1)}\text{Sr}_{0.121(1)}\text{Mn}_{0.957(8)}\text{O}_3$ (Air) and $\text{La}_{0.835(2)}\text{Sr}_{0.119(1)}\text{Mn}_{0.940(8)}\text{O}_3$ (O_2), respectively. At the O - R structural transition, the averaged Mn-O-Mn bond angle increases from $161.08(4)^\circ$ (Ar) to $163.38(1)^\circ$ (O_2) and $163.64(1)^\circ$ (Air), respectively. Moreover, the tolerance factor t calculated from the averaged La(Sr)-O and Mn-O bond lengths increases from 0.990(1) (Ar) to 0.992(1) (O_2) and 0.993(1) (Air), respectively. Ar and O_2 annealings cause a decrease of the Curie temperature T_c from 240(1) K (Air) via 237(1) K (O_2) to 192(1) K (Ar). The Curie temperature is reduced by $\sim 20\%$ for the sample with orthorhombic structure (Ar) as compared to the samples with rhombohedral structure (Air and O_2). This is explained by a decrease of the total strength of magnetic interactions, due to the changed Mn-O-Mn bond angle, the decrease of the numbers of nearest magnetic neighbors (Fig. 1(b)).

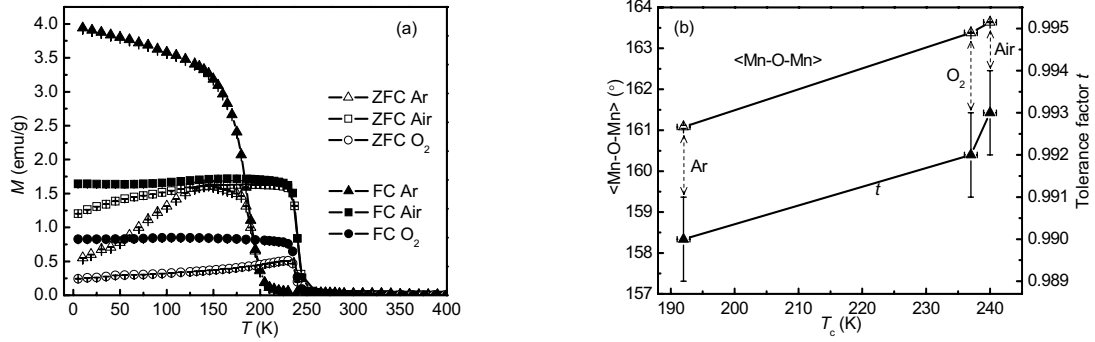


Fig. 1. ZFC and FC magnetization M vs. temperature measured at $H = 20$ Oe (a), and mean $\langle \text{Mn-O-Mn} \rangle$ bond angle and tolerance factor t vs. the Curie temperature T_c (b) for the three samples.

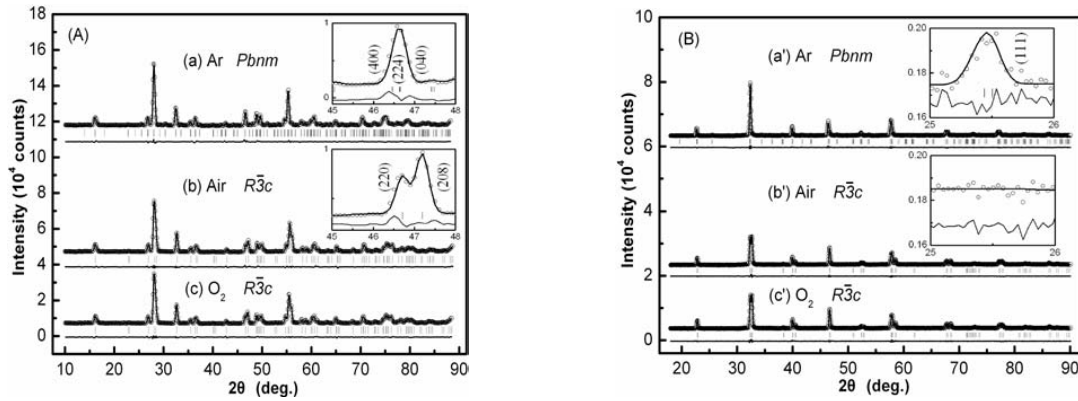


Fig. 2. The observed (circles) and calculated (solid lines) patterns of neutron [(A)] and X-ray [(B)] powder diffraction diagrams for Ar annealed [(a) and (a')], air sintered [(b) and (b')] and oxygen annealed [(c) and (c')] $\text{La}_{0.875}\text{Sr}_{0.125}\text{Mn}_{1-\gamma}\text{O}_{3+\delta}$ samples at ambient conditions. The bars mark the positions of Bragg reflections. The lower curve in each panel represents the difference between the observed and calculated patterns. The inset shows magnified patterns in certain 2θ regions.

Conclusions

We have systematically studied the effect of Ar and O_2 annealings on $\text{La}_{0.875}\text{Sr}_{0.125}\text{Mn}_{1-\gamma}\text{O}_{3+\delta}$ manganites with a fixed Sr doping. Our results clearly demonstrate that how extremely small change in the stoichiometry can have huge effects on structural and magnetic properties in these compounds based on the perovskite structure. Only $\sim 2.2\%$ relative decrease of oxygen content can lead to a structural transition from rhombohedral to orthorhombic and to a decrease of the transition temperature by $\sim 20\%$. Such a huge response on small changes of parameters is characteristic of this complex system. To our knowledge, only a combined refinement of X-ray and neutron diffraction data allows one to determine the stoichiometry with the required precision to be able to compare results from different samples.



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-04-020		
Experiment title:	Magnetic ordering and superstructure in bornite Cu_5FeS_4 polymorphs		
Dates of experiment:	11.10.04-20.10.04 08.11.04-10.11.04 25.11.04-28.11.04	Date of report:	13.02.2006
Experimental team: Names	Addresses		
M. Romanelli A. Caneschi	University of Florence, Dept. of Chemistry, via della Lastruccia 3 – I50019 Sesto Fiorentino, Italy		
M. Borgheresi	University of Florence, Dept. Earth Sciences, via La Pira 4 - I50121 Firenze, Italy		
F. Di Benedetto G. Pratesi C. Cipriani	Museo di Storia Naturale, Università di Firenze, via La Pira, 4, I50121, Firenze (Italy)		
Local Contact:	J. M. Walter		

Experimental report text body

Introduction

Bornite has three temperature-dependent polymorphs, termed high, intermediate, and low. The intermediate (I-bornite) and low (L-bornite) forms can be described as superstructures of the high-temperature cubic form (H-bornite), the various superstructures arising from an increase in the ordering of vacancies and metal atoms with reducing temperature (Koto and Morimoto 1975; Putnis and Grace 1976; Pierce and Buseck 1978). The L-bornite crystallises in the orthorhombic (pseudo-tetragonal) space group *Pbca* where the metals ions hosted in the tetrahedral site of two different sub cells: sphalerite- and antiferite-type (Kanazawa et al., 1978). The metals locations proposed by Koto & Morimoto (1975) are, according to Collins *et al.* (1981), conceivable but not certain, because the scattering of Cu and Fe is too similar in the X-ray measurements. Moreover, the neutron diffraction data obtained from Collins *et al.* (1981) can be interpreted on the basis of different metals distributions. The problem of discerning between Cu and Fe positions and distributions become very important for the magnetic data interpretation which are directly related to the short-range ions interactions.

Measurements and Results

The measurements were planed at the SV-7 powder diffractometer in order to study the lattice constants variation in the high temperature but also to investigate the magnetic ordering phenomena (long- and short-range ordered magnetic structures) that appear at 70 K.

The bornite sample, from the Natural History Museum, was previously studied using micro analytical

(XRD, EMPA) and advanced spectroscopic and magnetic techniques (EPR, HF^2EPR , ESE, SQUID magnetometry). The measurements were taken on 1 g of powdered sample placed in a cylindrical vanadium can of 7.6 mm diameter and 30 mm height by using the closed cycle He-cryostat (4-293K) and the furnace (293-1500K) for the temperature excursions. The neutron diffraction measurements were obtained, in fact, in the high temperature range (at 200°C, 300°C and from 220°C to 250°C every 5°C), at room temperature and finally in the low temperature range (at 4 K and from 10 to 115 every 5 K) in order to assess and describe the magnetic polymorphs.

The neutron diffraction data (Fig. 1,2) collected in the low temperature region, which reveal at least 4 extra peak, confirmed the presence of an antiferromagnetic structure formed below 70 K and associative with an ordered distribution of the iron cations. No evidence of further magnetic discontinuity, as suggested in literature, has been proved from this diffraction data. The refinement of all the obtained data will carry out some other relevant information, in particular the distribution of Fe ions and their reciprocal interaction.

References

- Collins, M. F.; Longworth, G., Townsend, M. G. (1981); *Can. J. Phys.*; **59**(4), 535-9
- Kanazawa, Y., Koto, K., and Morimoto, N. (1978) Bornite (Cu_5FeS_4): stability and crystal structure of the intermediate form. *Canadian Mineralogist*, **16**, 397–404.
- Koto, K. & Morimoto, N. (1975); *Acta Cryst.*, **B31**, 2268-73.
- Pierce, L. & Buseck, P.R. (1978); *Am Mineral.*, **63**, 1-16
- Putnis, A. and Grace, J. (1976) The transformation behaviour of bornite. *Contributions to Mineralogy and Petrology*, **55**, 311–315.

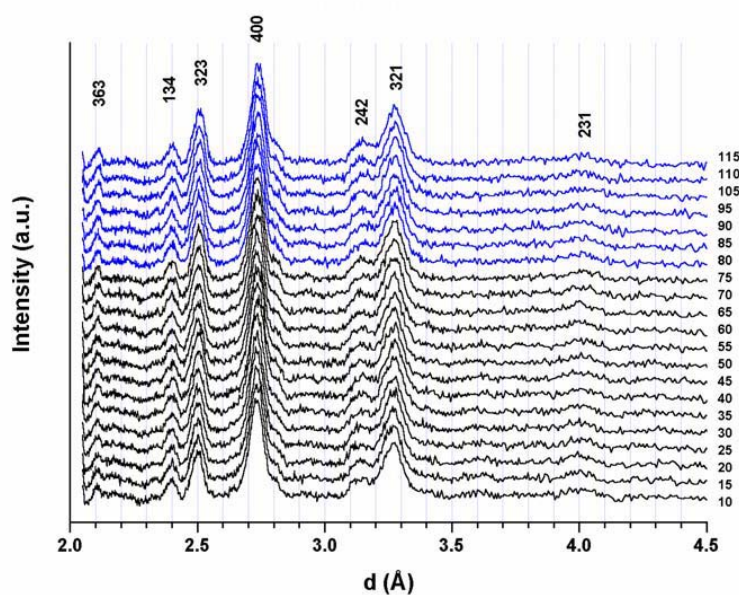


Figure 1

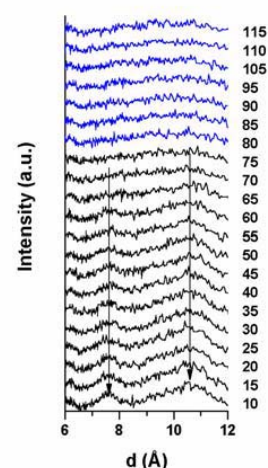


Figure 2



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-04-021		
Experiment title:	Polyphase deformation of quartzitic mylonites of the Monte Rosa nappe (Southern Switzerland/Northern Italy)		
Dates of experiment:	20 days	Date of report:	12.02.2006
Experimental team: Names	Addresses		
Pleuger, Jan Froitzheim, Nikolaus	Geologisches Institut der Universität Bonn, Nußallee 8, 53115 Bonn		
Local Contact:	J.M. Walter, E. Jansen, R. Skowronek		

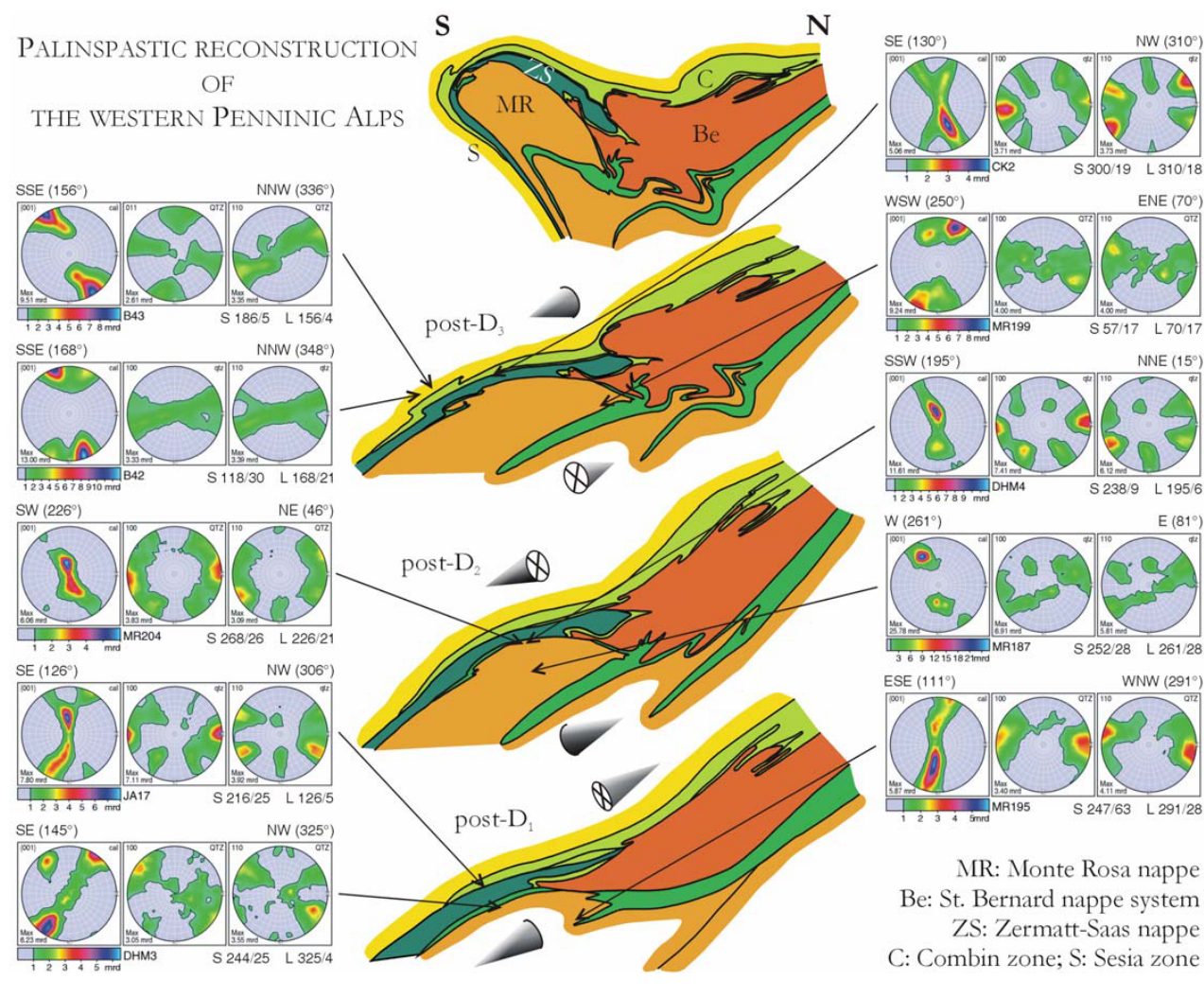
Experimental report text body

The present textures of quartzitic mylonites from the Monte Rosa nappe and adjacent units supplement those obtained earlier in the course of the project and shown in the experimental reports for 2003 (SV7-03-004 & SV7-03-009) and 2004 (SV7-04-005 & SV7-04-006). They provided most useful information about strain during certain stages of deformation while the high-pressure Zermatt-Saas and Monte Rosa nappes were tectonically exhumed. The first deformation phase D_1 led to nappe stacking. The nappes can be viewed as large-scale isoclinal recumbent folds that accommodated intense rotational strain (thrusting) within their strongly thinned overturned limbs. The {c} pole figure of JA17 is an incomplete type I crossed girdle. In terms of flow type, this can be interpreted to result from a weak rotational shear component. The thin section of JA17 shows that strain is strongly partitioned into almost equally large sets of domains of NW-vergent and opposite shear. Other quartz textures also indicate that D_1 strain between the main thrusts had only a small rotational component. It is most probable that larger displacements occurred only along the main thrusts while the nappe stack was otherwise shortened perpendicular to the axial planes of the then developing large nappe folds. Contrarily, the {c} pole figures of samples from the Monte Rosa nappe (DHM3 & MR195) are almost flawless single girdles, the {a} pole figures show one dominant maximum perpendicular to the {c} girdle. These samples with D_1 textures from the Monte Rosa nappe thus yield a stronger rotational component. They originate from levels of intense thrusting, i.e. from the very roof (DHM3) and the base (MR195) of the Monte Rosa nappe.

D_2 is characterized by SW-vergent shearing and intense large-scale folding. D_2 textures from the Monte Rosa nappe (DHM4 & MR204) yield incomplete single girdles of {c} which carry absolute maxima close to y indicating dominant prism <a> slip. Generally, prism <a> slip becomes dominant over basal <a> and rhomb <a> slip at temperatures roughly of upper greenschist- to amphibolite-facies conditions. Comparison of the D_2 textures and those developed in the Monte Rosa nappe during D_1 adumbrates that temperatures may have increased between D_1 and D_2 . Other D_2 textures, however, yield different geometries, some of them triclinic and

therefore difficult to interpret in a straightforward manner, such as MR187. The fact that D_2 textures are ambiguous is best accounted for by the strong heterogeneity of D_2 strain.

D_3 effected the SE-vergent shearing and again large-scale folding. Sample CK2 from the base of the Combin zone yields a D_3 texture with a $\{c\}$ pattern intermediate between type I and II crossed girdles. This and the tendency of $\{a\}$ to array around the stretching lineation testifies constriction. Two samples (B42 & B43) stem from the base of the Sesia zone. B42 reveals two $\{c\}$ maxima close to z . The stronger and the weaker one are rotated by about 20° towards x in a counter-clockwise and clockwise sense, respectively. $\{m\}$ and $\{a\}$ are almost evenly disposed each on two girdles perpendicular to the $\{c\}$ maxima. This texture corresponds well with thin section observations that ribbon domains of quartz display opposite but internally consistent senses of shear. By total volume, ribbons with geographically SE-vergent shear sense, corresponding to the stronger $\{c\}$ maximum, dominate over those with NW-vergent shear sense. The bulk strain of the conjugate ribbon sets was therefore flattening and had a weak rotational component. B43 is dominated by strong single maxima of $\{c\}$ and $\{a\}$ at the periphery of the respective pole figures indicative of basal $\langle a \rangle$ slip. Appendices of the $\{c\}$ maximum extend towards the interior of the pole figure and document some additional influence of rhomb $\langle a \rangle$ slip which acted into the opposite direction than basal $\langle a \rangle$ slip as a conjugate slip system. Consequently, for B43 the same interpretation in regard of strain applies as for B42. A D_3 texture from the Monte Rosa nappe (MR199) has a similar $\{c\}$ pattern as B42 but $\{a\}$ and $\{m\}$ are not that properly arranged on girdles. Variable strain geometries for D_3 must be concluded from D_3 textures from different levels of the nappe stack. CK2 testifies that the rocks along the base of the Combin zone accommodated substantial SE-vergent faulting while elsewhere the strain was rather flattening and only slightly rotational.





Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-04-022		
Experiment title:	Characterisation of ancient pottery of different periods from the excavation of Neos Scopos, Serres in North Greece		
Dates of experiment:	04.04.2005 – 10.04. 2005	Date of report:	12.12.2005
Experimental team: Names	Addresses		
I. M. Siouris V. Mourtzi	SimLab, Department of Informatics and Communication, TEI of Serres Terma Magnisias 62000 Serres, Greece		
Local Contact:	J. M. Walter, R. Skowronek		

Experimental report text body

Introduction

The present study was the first part of a proposed systematic investigation, using non-destructive neutron diffraction for the characterization of ancient pottery from different periods ranging from 8th to 1st century BC, found at the excavation site of Neos Scopos Serres in North Greece. This is the first comprehensive attempt to study ancient Greek pottery using non-destructive neutron diffraction. The aim of this project is to provide a ceramic/pottery data base of the constituent minerals, firing conditions, weathering and trading patterns, available to both archaeologist and scientists in the field.

Measurements

At the present experiment 10 articles of pottery fragments with approximate dimensions 10 cm², 0.2-0.4 cm thick were investigated. The measured samples consisted of various types of potshards from three different ancient periods: Archaic 8th BC (4 samples), Classical (2 samples) and Hellenistic 2nd BC (4 samples). The work is focused on determining the mineral phase compositions (fingerprinting), the thermal profiles develop during manufacturing, and the element distribution, of carefully selected ceramic items discovered in a single localized pit. Neutron data were collected for about 5 hours/sample. The neutron diffraction patterns were analyzed and structure refinements on the mineral phases were carried out using the program AMPhOrAe (Archeometric Multi Phase Ornament & Artifacts analyses). This program is a new extended windows version of the IC-POWLS, developed to serve the multiphase modeling requirements for the analysis of the data collected from neutron diffraction measurements. The measured diffraction patterns of three samples at 273 K are depicted in (Fig.1), along with the corresponding fitted profile calculated intensities from the theoretical

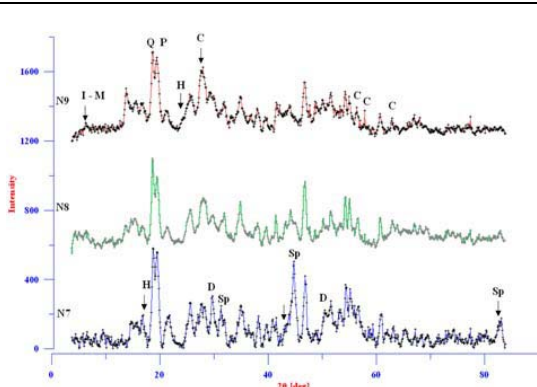


Fig.1: Observed diffraction patterns collected at 273 K of three 2nd century BC samples.

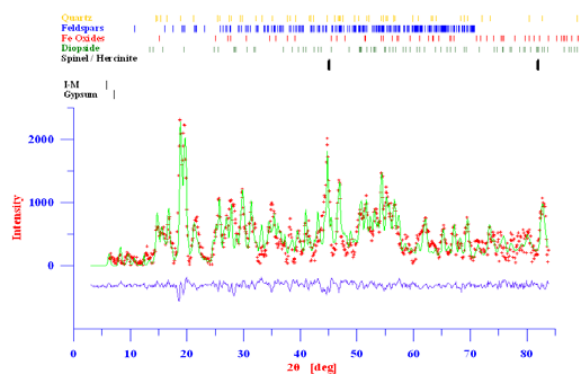


Fig.2: Observed, calculated and difference diagram of sample N7

to final model mineral compositions. Initial modeling of the observed data during the refinement process adopted a quartz, feldspar, plagioclase, hematite and diopside mineral synthesis (see Fig. 3), according to earlier neutron diffraction studies on comparable Greek pottery from Athenian Agora, Krania and Karabournaki.

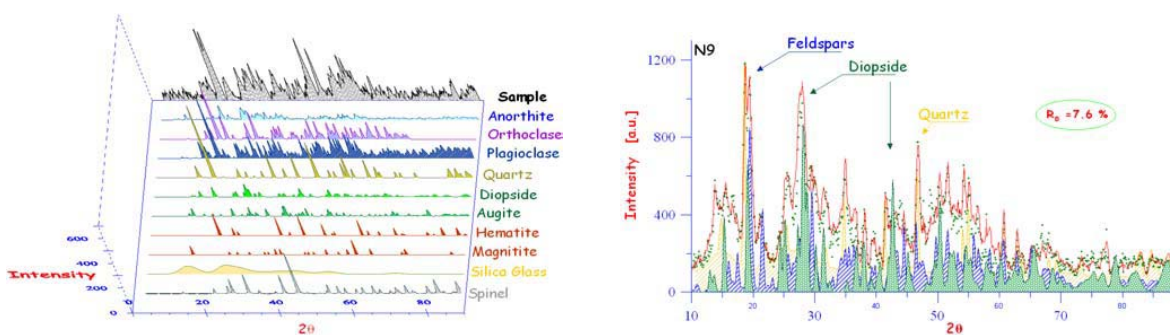


Fig.3: Initial modelling of the observed data at 273 K (left). Final model mineral compositions of N9 (right)

Results and Conclusions

Inspection of the analyzed diffraction patterns reveals the following similarities. All the patterns are dominated by pronounced quartz and feldspar peaks. Additionally, intense Bragg reflections corresponding to diopside (D) and spinel (Sp)/hercynite (Hr) minerals are clearly observed in sample N7 and with lower intensity in samples N8 and N9. Further more, the weak Bragg peaks observed at lower angles may be attributed to the (001) reflection of an illite/muscovite (I-M) phase. The absence of extensive broad humps in the diffraction spectra shows no significant vitreous silica phase, indicating that the firing of the clay could not have exceeded the 1000 °C. The firing temperatures were estimated to be within 900 and 1000 °C. In the table below the relative weight fractions *w* (in percent) of Quartz (Q), Feldspar (F), Diopside (D), K-Spinel/ Hercynite (Sp/Hr) and Hematite (H) are given. Strength of reflection intensities at $2\theta = 13.8^\circ$ for (I-M: illite/muscovite) are indicated by (0), (+), (++), (+++) in this table. R_{wp} are the weight percentages for the different samples.

A/A	w(Q)	w(F)	w(D)	w(Sp/Hr)	w(H)	Q/F	I-M	R_{wp}
N7	18.6	67	11.8	11.5	1.6	0.28	+	6.8
N8	26	58	11	++	5	0.45	++	7.8
N9	42	25	28	+	5	1.68	++	7.6

Full paper: I.M.Siouris, J.Walter to appear in Physica B; **Results presented** at ICNS 2005, in Sydney.



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-04-024		
Experiment title:	Neutron diffraction on PrNi₄Cu, NdNi₄Cu and DyNi₄Cu		
Dates of experiment:	36 days	Date of report:	18.05.2006
Experimental team: Names	Addresses		
Dr. T Toliński, Dr. A. Kowalczyk Dr. A. Hoser	Institute of Molecular Physics, PAS, Smoluchowskiego 17, 60-179 Poznań, Poland		
	Institute of Molecular Physics, PAS, Smoluchowskiego 17, 60-179 Poznań, Poland		
	Institut für Kristallographie, RWTH-Aachen, Germany		
Local Contact:	Dr. J. M. Walter, R. Skowronek		

Experimental report text body

Recently, we have intensively studied the RNi₄M (M = B, Al, Si, Ga, Cu) [1-4] compounds, which originate from the RNi₅ type of compounds (R= rare earth) [5]. The RNi₄Cu series differs from the remaining M substitutions by the reason of the Cu(3d) states being close both to the Fermi level (0eV) and the Ni(3d) (-0.7eV) states. Hence, one can suppose that the Cu(3d) peak, which appears at about -3eV in the valence band can hybridize with the Ni(3d) one, influencing the magnetism of nickel by the electron transfer. However, we know, based also on our previous neutron diffraction studies for M = Al [1], that the magnetism of nickel is already cancelled out due to the other atoms contributing to the valence band, especially the rare earth. Therefore, one of the reasons of the present studies has been a comparison of RNi₄Cu with the other RNi₄M compounds.

The polycrystalline RNi₄Cu compounds were prepared by induction melting of the constituent elements under a protective argon atmosphere. The RNi₄Cu compounds (R = rare earth or Y) crystallize in the hexagonal CaCu₅-type of structure, point group *P6/mmm*. R ions occupy the 1a site (0,0,0) and Ni(1) the 2c site (1/3,2/3,0). Ni(2) and Cu are statistically distributed over the 3g site (1/2,0,1/2). To verify the electronic structure of RNi₄Cu we have carried out the X-ray photoemission spectroscopy (XPS). The magnetometric measurements were performed using a MagLab 2000 System (Oxford Instruments Ltd.).

The neutron diffraction has been performed using the SV7 instrument and the wavelength was equal to 1.096 Å. The sample was contained in a cylindrical vanadium can mounted (1) in a refrigerator cryostat and (2) in a cryomagnet with external magnetic fields up to 5 T perpendicular to the horizontal diffraction plane. In 2005 the studies have been carried out for R = Nd and Pr [6]. The example of the more advanced data of R = Nd is presented below.

PrNi₄Cu and NdNi₄Cu are characterized by the Curie temperatures of 3.7 K and 11 K, respectively. The magnetic moments in $H = 9$ T were equal to $1.5\mu_B/\text{f.u.}$ at 1.8 K for PrNi₄Cu and $2.25\mu_B/\text{f.u.}$ at 4.2 K for NdNi₄Cu [4].

The lattice parameters derived from the neutron diffraction are in good agreement with the X-ray diffraction results. The diffraction patterns have been fitted with the program FULLPROF, by the full-pattern Rietveld refinement.

Fig.1a,b presents the neutron diffraction pattern for NdNi₄Cu obtained at 50 K and 4 K. Below the transition temperature of 11 K [4] only a change in the structural intensities is visible, like in the case of the previously studied NdNi₄Al [1]. It allows one to exclude any antiferromagnetic ordering, for which new reflections would appear. Following the case of NdNi₄Al, it is assumed that the magnetic moments of the Nd ions are aligned within the hexagonal plane. The magnetic moment at 4.2 K extracted from the FULLPROF analysis is $1.75\mu_B$, in good agreement with the magnetic moment determined from the magnetization measurements [4], where it was $1.77\mu_B/\text{f.u.}$ from the extrapolation of the high field part of the $M(H)$ curve to $H = 0$. Previously, in the case of NdNi₄Al we have observed a significant discrepancy between the neutron ($1.85\mu_B$) and magnetometric ($1.52\mu_B/\text{f.u.}$) result. A comparison of the calculated (LMTO) and measured (XPS) electronic structures is being carried out to explain these differences.

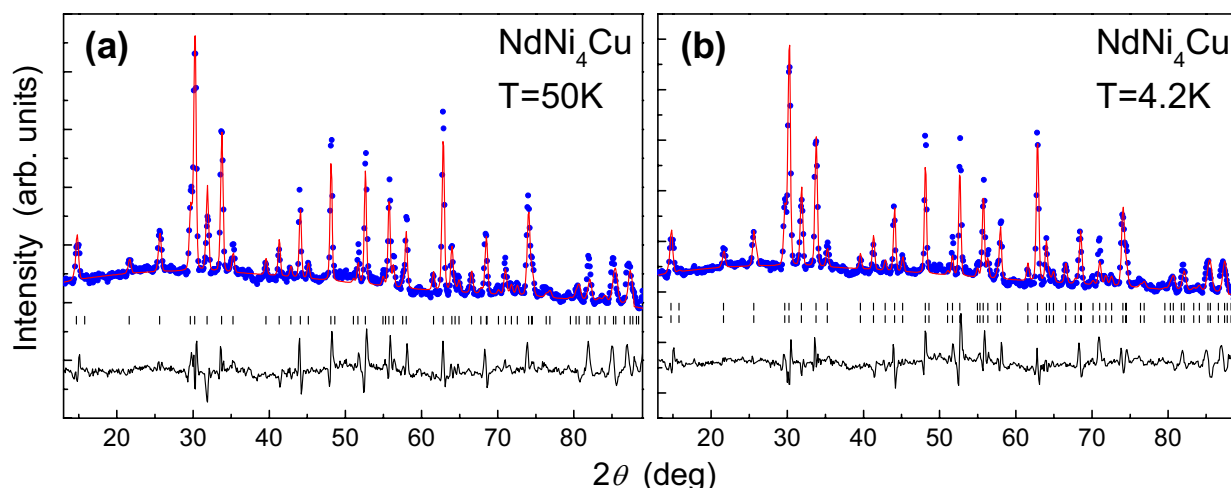


Fig.1. Neutron diffraction pattern of NdNi₄Cu at 50 K (a) and 4.2 K (b). The experimental data and the refinement are represented by dots and a line, respectively. The bottom curve represents the difference between measurements and calculations. For (b) the fit includes both the structural and magnetic contribution.

References

- [1] T. Toliński, W. Schaefer, W. Kockelmann, A. Kowalczyk, A. Hoser, Phys. Rev. B 68 (2003) 144403.
- [2] T. Toliński, A. Kowalczyk, A. Szlaferek, B. Andrzejewski, J. Kovac, M. Timko, J. Alloys Compd. 347 (2002) 31.
- [3] M. Pugaczowa-Michalska, G. Chełkowska, T. Toliński, A. Kowalczyk, phys. stat. sol. (c) 243 (2005) 309.
- [4] A. Kowalczyk, T. Toliński, B. Andrzejewski, A. Szlaferek, J. Alloys Compd. 413 (2006) 1.
- [5] W.E. Wallace, Rare Earth Intermetallics, ed. A.M. Alper, J.L. Margrave, A.S. Nowick, Academic Press, New York and London 1973, p.266.
- [6] T. Toliński et al., submitted to J. Alloys Compd.



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-04-025		
Experiment title:	Antiferromagnetic correlations in some Ce-based intermetallic compounds		
Dates of experiment:	06.04.06-09.04.06	Date of report:	18.05.2006
Experimental team: Names	Addresses		
Dr. T Toliński, Dr. A. Kowalczyk Dr. A. Hoser	Institute of Molecular Physics, PAS, Smoluchowskiego 17, 60-179 Poznań, Poland Institute of Molecular Physics, PAS, Smoluchowskiego 17, 60-179 Poznań, Poland Institut fuer Kristallographie, RWTH-Aachen, Germany		
Local Contact:	Dr. J. Walter, R. Skowronek		

Experimental report text body

Our recent investigations of the CeNi_4M compounds for $\text{M} = \text{B}$ [1], Al [2], Ga [3] and Cu [4] have revealed a contradictory behavior. From one side, the reduced effective moment extracted from the magnetic susceptibility and satellites observed in the X-ray photoemission 3d spectra suggest a strongly mixed valence behavior. Simultaneously, a low temperature minimum in resistivity and an anomaly in specific heat indicate the possibility of a Kondo system, which would exclude the mixed-valency. We have ascribed the anomalies to a Kondo impurity-like behavior, i.e. a small amount of Ce ions in incorrect crystallographic positions mimicking the classical Kondo effect for diluted systems. However, the specific heat anomaly at 6 K can be alternatively explained by a small contribution of an antiferromagnetic impurity phase - a possibility supported by a small negative paramagnetic Curie temperature (-10 K). The purpose of the neutron diffraction studies has been to confirm or to exclude the existence of any antiferromagnetic correlations.

The neutron diffraction experiment has been carried out using the SV7 instrument and the wavelength of 1.096 Å. The polycrystalline CeNi_4Cu compounds crystallize in the hexagonal CaCu_5 -type of structure, point group $P6/mmm$.

The preliminary neutron diffraction measurements were realized for temperatures of 4, 50 and 300 K. At the accessible accuracy no additional - in respect to the crystallographic - reflections could be observed going down from room temperature to 4 K, i.e. below the anomaly at 6 K for specific heat [4]. Therefore, at this stage of investigations the neutron diffraction does not support the possibility of an antiferromagnetic contribution. However, more accurate and covering temperatures below 4 K measurements are necessary.

In spite of the lack of any extra peaks, two other effects of the temperature decrease are found. First, the expected contraction of the lattice constants and, consequently, the unit cell volume is observed. The lattice constants are collected in Table 1.

Temperature	a (Å)	c (Å)
300 K	4.92197	4.01718
50 K	4.90790	4.00997
4 K	4.90696	4.01021

Table 1: Temperature dependence of the lattice parameters for the CeNi₄Cu compound.

Second, an increase of some intensities occurs, mainly for the (111), (201), (210), (301), (221) and (222) reflections. Opposite to our previous studies on RNi₄M the low angle peaks are not affected by lowering temperature. The growth of the structural intensities can be explained by a ferromagnetic ordering of the rare earth ions. The corresponding fit with FULLPROF program is displayed in Fig. 1. The obtained magnetic moment of 2.01 μ_B (assumed to lie in the hexagonal plane) is close to the free ion value (2.14 μ_B). However, the ordering would have to start already in the range 50-300 K, whereas the magnetic susceptibility indicates a paramagnetic behavior in the whole temperature range. Similar quality fit can be obtained assuming that the magnetic moment is due to Ni (usually magnetic contribution of Ni is negligible in RNi₄M compounds). It would explain a high ordering temperature, however the estimated value (>0.8 μ_B) of magnetic moment exceeds the saturation moment of Ni (0.6 μ_B). Further, higher accuracy and magnetic field assisted measurements should provide more answers to these questions.

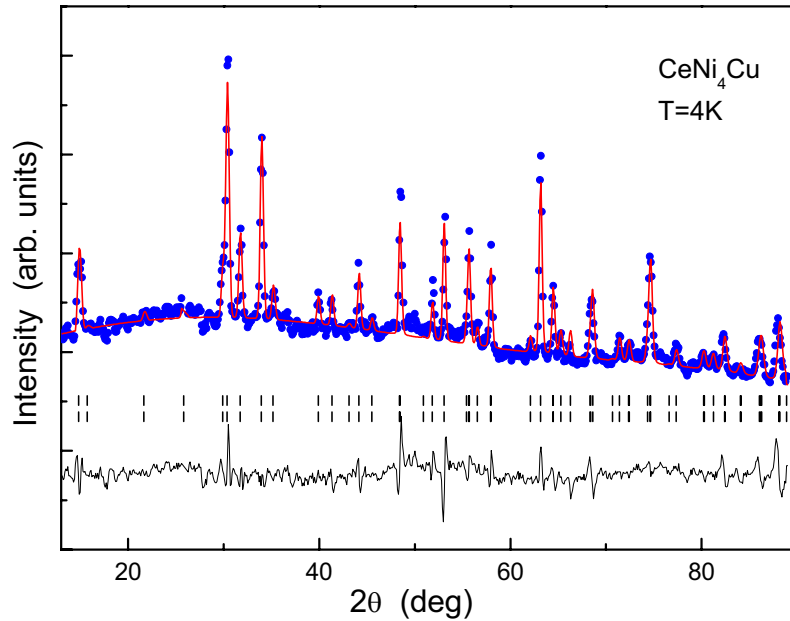


Fig.1. Neutron diffraction pattern of CeNi₄Cu at 4.2 K fitted assuming the structural and the magnetic phase with magnetic moments on the Ce ions.

References

- [1] T. Toliński, A. Kowalczyk, M. Pugaczowa-Michalska, G. Chełkowska, J. Phys.: Condens. Matter **15**, 1397 (2003).
- [2] T. Toliński, A. Kowalczyk, G. Chełkowska, M. Pugaczowa-Michalska, B. Andrzejewski, V. Ivanov, A. Szewczyk, M. Gutowska, Phys. Rev. B **70**, 064413 (2004).
- [3] T. Toliński, A. Kowalczyk, V. Ivanov, G. Chełkowska, M. Timko, Czech. J. Phys. **54**, D287 (2004).
- [4] T. Toliński, A. Kowalczyk, G. Chełkowska, A. Szewczyk, M. Gutowska, J. Phys.: Condens. Matter **18**, 3435 (2006).



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-04-026		
Experiment title:	Erfassung und Bewertung ausgewählter Vorkommen von Natursteinen und ihrer gesteintechnischen Eigenschaften		
Dates of experiment:	20.09.05-27.09.05	Date of report:	19.06.2006
Experimental team: Names	Addresses		
Hoffmann, Andreas Siegesmund, Siegfried	Geowissenschaftliches Zentrum der Universität Göttingen (GZG) Abteilung für Strukturgeologie und Geodynamik Goldschmidt Str. 3 D-37077 Göttingen		
Local Contact:	J. M. Walter, E. Jansen		

Experimental report text body

Grundlegende Voraussetzung für die Erfassung und Bewertung der gesteintechnischen Eigenschaften der Naturwerksteine ist die fundierte Zustandsbeschreibung durch petrographische, gefügekundliche und technische Kenngrößen. Durch Texturuntersuchungen, als wohl wichtigstes Gefügeelement zur Materialprognose, wurde an ausgewählten Naturwerksteinen gezeigt, ob vorhandene kristallographische Vorzugsorientierungen existieren. An dem Probenmaterial sollten Homogenitätsbereiche bestimmt werden, d.h. ob die Texturen in Intensität, Symmetrie bzw. bezüglich eines externen Referenzsystems übereinstimmen oder vollständig variabel sind.

Die Texturmessungen der Marmore zeigen insgesamt eine deutliche Orientierung der kristallographischen Achsen. Die c-Achsen sind dabei überwiegend parallel zur z-Richtung angeordnet und demonstrieren mehrheitlich ein einfaches Polpunktmaximum. Da alle Gesteine mit dieser Verteilung der Achsen eine makroskopisch deutlich erkennbare Foliation aufweisen, die in der x- und y-Richtung aufgespannt wird, sind die c-Achsen senkrecht zur Foliation der Gesteine ausgerichtet. Die Verteilung der c-Achsen in der Probe 29AH ist ebenfalls einfach ausgebildet. Die Achsen sind dabei jedoch nicht parallel zur z-Richtung angeordnet, was sich vermutlich aus der aus dem Umstand ergibt, dass das Gestein makroskopisch keinerlei erkennbare Vorzugsorientierungen aufweist. Die Probenahme konnte daher nicht unter vollständiger Berücksichtigung des externen Referenzsystems erfolgen. Das Doppelmaximum der c-Achsen bei der Probe 14AH ist eventuell auf tektonische Bewegungen zurückzuführen, da die Lagerstätte lokal durch eine intensiv ausgeprägte Scherzone beeinflusst wird.

Texturmessungen und thermische Dehnung der Marmore

An der Universität Göttingen wurden an dem Probenmaterial zentrale Elemente der Verwitterungsprozesse simuliert, wie z.B. das thermische Dehnungsverhalten der Marmore mittels eines Dilatometers.

Die Ergebnisse der thermischen Dehnung demonstrieren deutlich, dass die jeweiligen Längenänderungen von der Textur des Materials abhängen. In diesem Zusammenhang zeigt beispielsweise die Probe 01AH eine ausgeprägte Anisotropie des Dehnungsverhaltens, wobei die größten Änderungen parallel zur z-Richtung, also parallel zur bevorzugten Orientierung der c-Achsen auftreten (Abb.1a, b). Das anisotrope thermische Dehnungsverhalten ergibt sich aus den Einkristalleigenschaften des Calcits, der sich unter dem Einfluss von Temperatur in Richtung der kristallographischen c-Achse ausdehnt und parallel zur a-Achse kontrahiert. Das gleiche gilt auch für den zweiten gemessenen Texturtyp in Abb. 2.

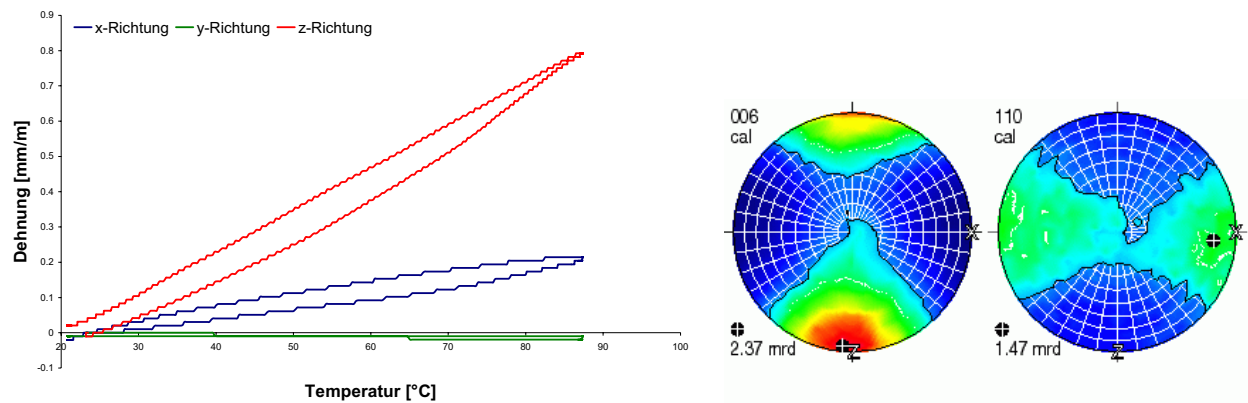


Abb. 1: Thermische Längenänderung der Probe 01AH mit maximaler Ausdehnung in z-Richtung (links). Die Richtung der maximalen Ausdehnung korrespondiert dabei mit der bevorzugten Orientierung der kristallographischen c-Achen (rechts).

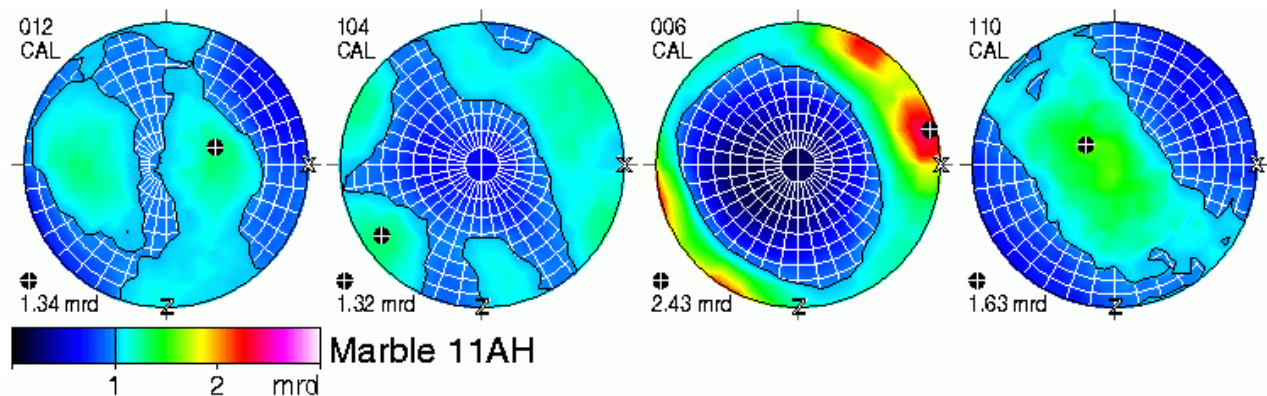


Abb. 2: Texturtyp eines Doppelmaximums eines Marmor-Naturwerksteins



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-04-027		
Experiment title:	Kinematic analysis of the Sierra Ballena Shear Zone and their meaning in the structure of the Precambrian of SE Uruguay		
Dates of experiment:	04.10.04-07.10.04 10.10.04-11.10.04 22.11.04-28.11.04	Date of report:	12.04.2006
Experimental team: Names	Addresses		
Oyhantcabal, Pedro Siegesmund, Siegfried	Departamento de Geologia, Facultad de Ciencias Universidad de la Republica Igua 4225 CP 11400 Montevideo, URUGUAY Abteilung für Strukturgeologie und Geodynamik Geowissenschaftliches Zentrum der Georg-August-Universität Göttingen (GZG) Goldschmidtstrasse 3 D - 37077 Göttingen		
Local Contact:	Dr. Jens M. Walter, R. Skowronek, E. Jansen		

Experimental report text body

Einleitung

Die „Sierra Ballena Scherzone“ ist eine horizontale Scherzone, die den Neoproterozoischen Gürtel „Dom Feliciano“ in Südamerika in zwei unterschiedliche Domänen unterteilt. Die Deformation in der Sierra Ballena Scherzone fand unter regionalen grünschieferfaziellen Bedingungen statt. Die Hauptdeformationsphase war transpressiv, in der reine Scherung vorherrschend war. Die Deformation führte sowohl zur Ausbildung von dextralen als auch von sinistralen Scherzonen und zu der Platznahme von synkinematischen Graniten. Altersdatierungen dieser granitischen Intrusionen ergeben daher ein Deformationsalter von 600 bis 580 Ma. Die Korrelation dieser Scherzone mit der Purros Scherzone des Kaoko Belts im Damara Orogen in Namibia ist derzeit gegenstand wissenschaftlicher Diskussion.

Messungen und Korrelationen

Zur Charakterisierung der Scherzonen und ihrer Deformationsmechanismen und -bedingungen, muß eine detaillierte Untersuchung der Mikrogefüge herangezogen werden. Die Mikrostrukturen im Quarz, die an granitischen Myloniten dieses Stadiums beobachtet werden können, sind charakteristisch für „dislocation creep Regime 2“. Die Polfiguren von Quarz der am SV7 gemessenen Neutronentexturen sind asymmetrische gekreuzte Gürtel und asymmetrisch einfache Gürtel mit Rhomb-*a* als aktivem Gleitsystem. Sowohl die beobachteten Mikrostrukturen als auch die Polfiguren deuten auf mittlere grünschieferfazielle Bedingungen während der transpressiven Verformung.

Eine zweite Deformationsphase war „strike-slip“ artig mit sinistraler, dominanter einfacher Scherung. Ar/Ar Alterbestimmungen an Muskovit der mylonitischen Quarzite zeigen dieses zweite Ereignis in zwei verschiedenen

Altersgenerationen an. Das Alter dieser zweiten Deformationsphase liegt zwischen 579 und 581 Ma. Die Mikrostrukturen des Quarz zeigen den Übergang von „dislocation creep Regime 2“ zu „dislocation creep Regime“ 3 an. Die Textur Polfiguren zeigen ein *c*-Achsen Maximum in Y-Richtung, mit Prismen-*a* als Hauptgleitsystem (siehe Abb. 3). Diese Charakteristika zeigen eine Deformation bei oberen grünschiefer- bis unteren amphibolitfazieller Metamorphosebedingungen an (~500 °C). Die schiefe Lage der *c* und *a* Achsen Maxima in den Polfiguren relativ zur Scherebene im Gelände, kann als ein Nachweis für transpressive Verformung betrachtet werden (Abb. 1).

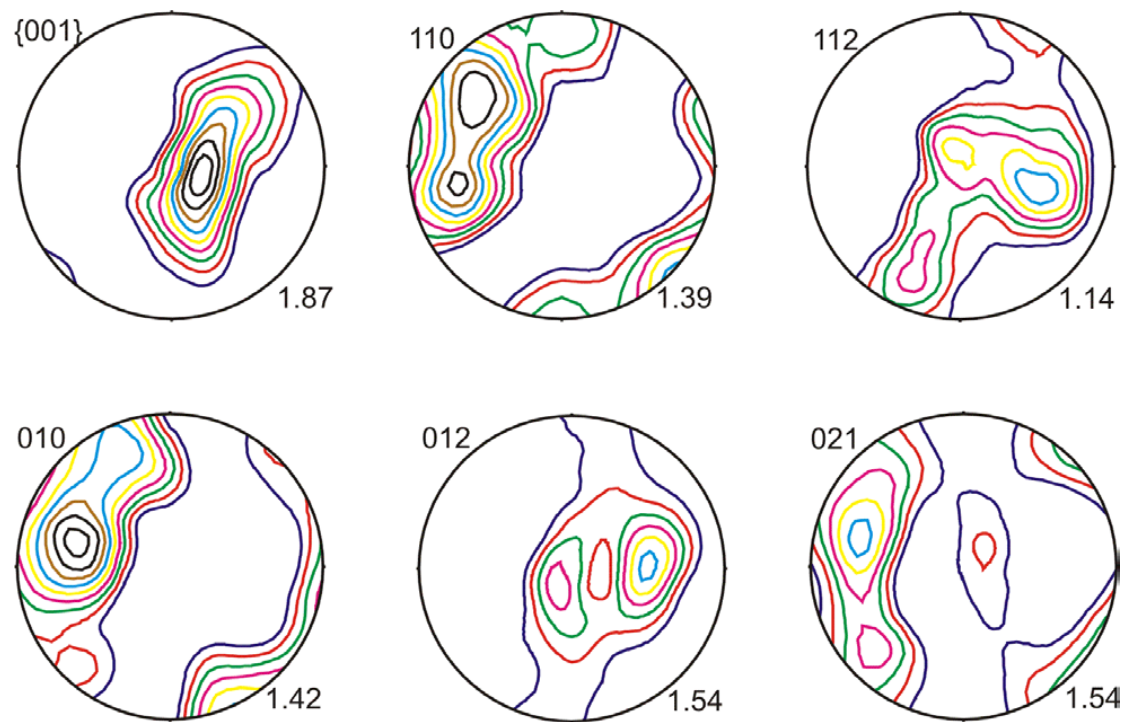


Abb 1: Quarzpolfiguren zeigen transpressive Deformationsgeometrien durch die schiefe Lage der *c*- und *a*-Achsen relativ zur horizontalen Scherebene. Das *c*-Achsen Maximum in Y-Richtung zeigt das Prismen-*a* Hauptgleitsystem bei oberen grünschiefer- bis untere amphibolitfaziellen Bedingungen



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-04-028		
Experiment title:	Texturen dolomitischer Itabirite		
Dates of experiment:	10 days	Date of report:	26.06.2006
Experimental team:			
Names	Addresses		
H. Siemes	Institut für Mineralogie und Lagerstättenlehre RWTH Aachen Bunsenstr. 8, 52056 Aachen		
C. Rosière	Instituto de Geosciencias Universidade Federal de Minas Gerais Av. Antonio Carlos, 6627 CEP 31270-901 Belo Horizonte, MG, Brasilien		
Local Contact:	J.M. Walter, E. Jansen, R. Skowronek		

Experimental report text body

Itabirite is a metamorphic variety of iron formations from the Itabira group that is a iron-rich stratigraphic unit from the Paleoproterozoic Minas supergroup in the Quadrilátero Ferrífero Mineral District. The itabira Group contains two units: i) the lower Cauê Formation characterized by the predominance of quartz itabirite, composed mainly of quartz and iron oxides (hematite and magnetite/martite) with local compositional varieties comprising silicates (chlorite/clinochlore, talk and amphiboles) and carbonates (mainly dolomite), and ii) the upper Gandarela formation comprising mainly dolomite which grades into the Cauê formation with the development of Fe-rich dolomite and dolomitic itabirite. The units of the Itabira Group may also grade into the underlying Batatal Formation. This unit comprises mainly phyllites of variable compositions but also dolomites and volcanic/volcanoclastic rocks.

Three main compositional types of itabirite could be discriminated in the in the Itabira group:

Quartz- Itabirite - quartz and iron oxides

Dolomitic Itabirite - dolomite, quartz, iron oxides and minor silicates

Silicate-rich/Amphibolitic Itabirite - silicates (chlorite/clinochlore, talk, amphiboles), iron oxides, some quartz and dolomite.

The present lithological varieties in the Itabira group are product of metamorphic mineral changes so it is not possible to find a direct relation to sedimentary facies, such as in anchimetamorphic sequences in several basins as the Transvaal-Griqualand West in South Africa and the Hamersley basin in Australia. Iron formations present a more or less regular banding due to primary variations in the composition related to sedimentation and diagenetic transformations and this structure remain more or less well preserved during metamorphism or may be transposed by deformation. Mineralogy and fabric, otherwise, reflect changing conditions during diagenesis, hydrothermal mineralization, metamorphism and deformation.

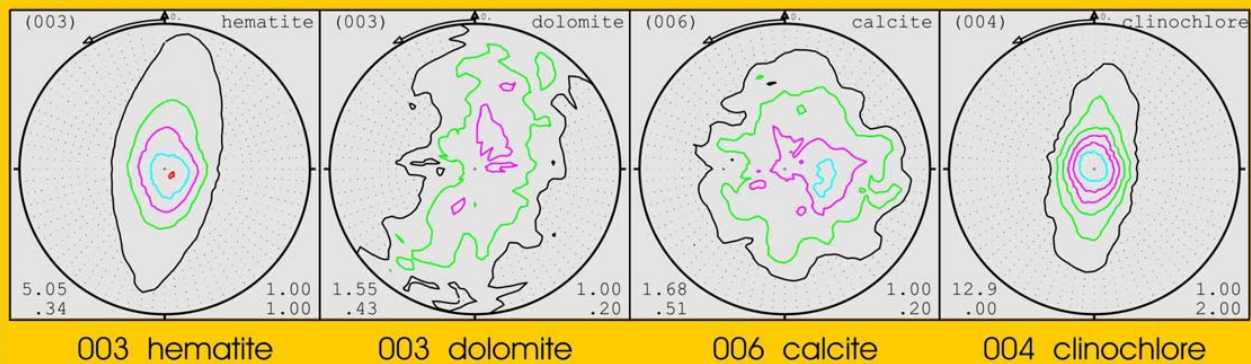
In the itabirites most hematite crystals are product of metamorphic recrystallization, dolomite is considered to be a product of diagenetic change of sedimentary carbonates and silicates which are probably always formed by metamorphic (solid state) reactions.

The metamorphic grade and intensity of deformation in the Quadrilátero Ferrífero increases eastwards and the degree of preferred orientation of hematite also increases with the development of oriented hematite platelets named specularite which texture changes according to the characteristics in intensity of deformation undergone by the rock.

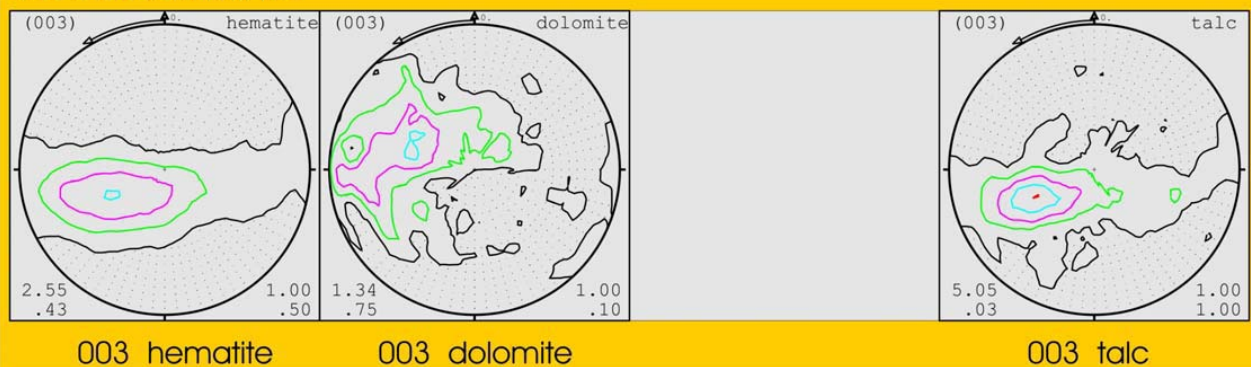
The first attempt to understand the deformation behavior of hematite and the types of texture developed was accomplished in monomineralic (hematitic) high-grade ores where gangue minerals were leached out or substituted by hydrothermal fluids during the mineralization processes.

The determination of the partial texture of several mineralogical components from dolomitic itabirite, namely, hematite, dolomite, chlorite (clinocllore) and talk indicate that they were deformed under the effect of a single deformation plan, but with the development of entirely different degrees of preferred orientation. Hematite presents similar texture as phyllosilicates but carbonates present a weaker texture.

Aguas Claras Mine



Aboboras Mine



For details see references e.g.:

- Bascou, J., Raposo, M.I.B., Vauchez, A., Egydio-Silva, M., (2002) Titanohematite lattice preferred orientation and magnetic anisotropy in high-temperature mylonites. *Earth and Planetary Science Letters* 198, 77-92.
- Jansen, E., Schäfer, W., Kirfel, A. (2000) The Jülich neutron diffractometer and data processing in rock texture processing. *Journal of Structural Geology* 22, 1559-1564.
- Rosière, C.A., Siemes, H., Quade, H., Brokmeier, H.-G., Jansen, E.M. (2001) Microstructures, textures and deformation mechanisms in hematite. *Journal of Structural Geology* 23, 1429-1440.
- Siemes, H., Klingenberg, B., Rybacki, E., Naumann, M., Schäfer, W., Jansen, E., Rosière, C.A. (2003) Texture, microstructure, and strength of hematite ores experimentally deformed in the temperature range 600° to 1100°C and at strain rates between 10^{-4} and 10^{-6} s^{-1} . *Journal of Structural Geology* 25, 1371-1391.



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-04-029		
Experiment title:	Low and high-temperature behaviour of kyanite. Thermoelastic constants and structural evolution		
Dates of experiment:	24.01.05-30.01.05 12.04.05-14.04.05 10.06.05-27.06.05	Date of report:	12.04.2006
Experimental team: Names	Addresses		
Gatta, Giacomo Diego Nestola, Fabrizio	Bayerisches Geoinstitut Universität Bayreuth Universität Str. 30 D-95447 Bayreuth Germany		
Local Contact:	Dr. Jens M. Walter, R. Skowronek		

Experimental report text body

Introduction

Kyanite is one of the three Al_2SiO_5 polymorphs (with andalusite and sillimanite). The fields of stability and the thermodynamic properties of the Al_2SiO_5 polymorphs play a crucial role of these minerals in the fields of the metamorphic petrology. In addition, the Al_2SiO_5 polymorphs are object of interest in the industrial applications of refractory materials. By calcinations, these minerals convert to mullite ($\text{Al}_6\text{Si}_2\text{O}_{13}$) and silica (SiO_2) at temperatures in excess of 1200-1400°C at ambient pressure, (Harben, 1999). Refractory materials that contain the Al_2SiO_5 polymorphs and/or synthetic mullite include e.g. insulating brick, firebrick and kiln furniture. In addition, the interlocking grain structure of kyanite and mullite applies ancillary mechanical strength to refractory and other non-refractory ceramic materials. Other end uses of kyanite and related materials include electrical porcelain, floor and wall tiles, foundry use, sanitary ware, and other related products. Despite the petrologic importance and the industrial use of kyanite, sillimanite and andalusite, the relative fields of stability of the Al_2SiO_5 polymorphs are not completely clear because of the small change in thermodynamic properties associated to the polymorphic phase transition (Winter and Ghose, 1979; Kerrick, 1990; Bohlen et al., 1991).

Measurements and Results

The high-temperature (HT) behaviour of kyanite (Al_2SiO_5) was investigated by means of *in-situ* neutron powder diffraction at the powder diffractometer SV7 in Jülich in a temperature range of room temperature up to 1200°C. Within the investigated T -range, no phase transition has been observed. The axial and volume thermal expansion coefficient ($\alpha_j = l_j^{-1} \cdot \partial l_j / \partial T$, $\alpha_V = V^{-1} \cdot \partial V / \partial T$), calculated by weighted linear regression through the data points, are: $\alpha_a = 5.5(2) \cdot 10^{-5}$, $\alpha_b = 5.9(2) \cdot 10^{-5}$, $\alpha_c = 5.18(8) \cdot 10^{-6}$, $\alpha_V = 7.4(1) \cdot 10^{-3} \text{ } ^\circ\text{C}^{-1}$, with $\alpha_a : \alpha_b : \alpha_c = 1.06 : 1.14 : 1.00$. All the three angles of the kyanite lattice show a slight decrease with T , with $\partial\alpha/\partial T = -2(2) \cdot 10^{-5}$, $\partial\beta/\partial T = -4(1) \cdot 10^{-5}$, $\partial\gamma/\partial T = -10(2) \cdot 10^{-5} \text{ } ^\circ\text{C}^{-1}$. The magnitudes of the principal unit-strain coefficients (ϵ_1 , ϵ_2 , ϵ_3) and the

orientations of the thermal strain-ellipsoids, between the ambient temperature and each measured T , were calculated. The magnitude and the orientation of all the three unit-strain coefficients are almost maintained constant with T . At $T-T_0=1177^\circ\text{C}$, $\varepsilon_1^a = 76(2)^\circ$, $\varepsilon_1^b = 70(2)^\circ$, $\varepsilon_1^c = 38(3)^\circ$, $\varepsilon_2^a = 49(3)^\circ$, $\varepsilon_2^b = 66(3)^\circ$, $\varepsilon_2^c = 127(4)^\circ$, $\varepsilon_3^a = 135(3)^\circ$, $\varepsilon_3^b = 31(3)^\circ$, $\varepsilon_3^c = 91(2)^\circ$ with $\varepsilon_1:\varepsilon_2:\varepsilon_3 = 1.57:1.29:1.00$. The structural refinements, performed at 23, 600, 650, 700, 750, 800, 850, 900, 950, 1050 and 1200°C allowed the description of the structural evolution and the main T -induced deformation mechanisms.

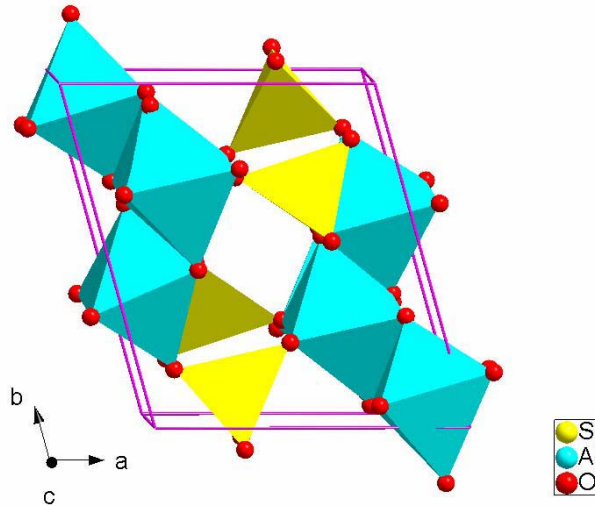


Fig. 1. The crystal structure of kyanite

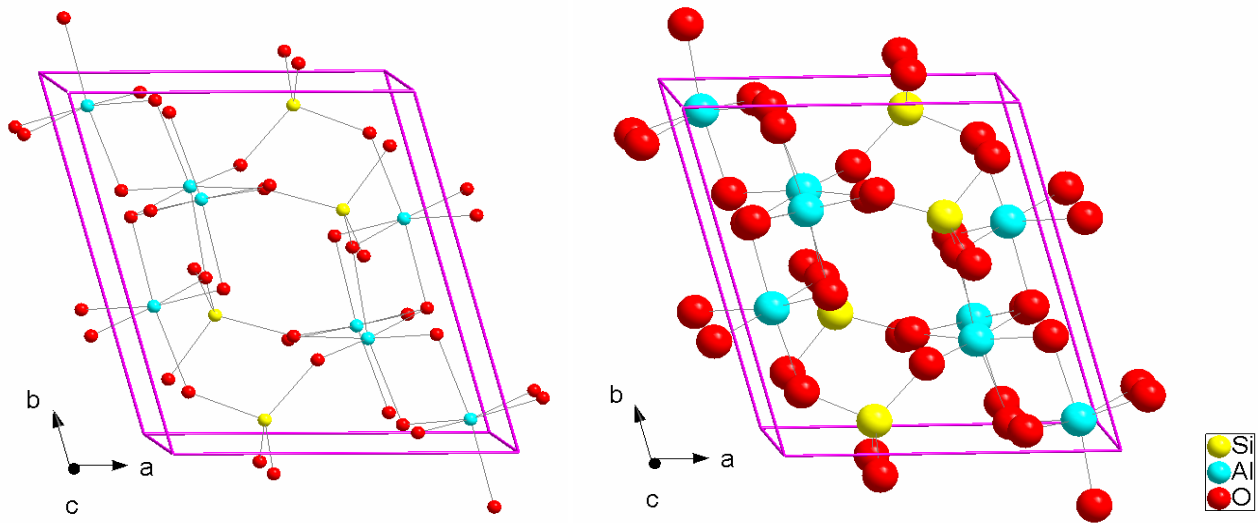


Fig. 2 . Clinographic view of the crystal structure of kyanite based on the structure refinements at room and 1200°C

References

- [1] Winter, J.K and Ghose, S. (1979) *American Mineralogist*, **64**, 573-586.
- [2] Kerrick, D.M. (1990) Reviews in Mineralogy, Volume 22, *Mineralogical Society of America*, Washington, D.C., pp. 406.
- [3] Bohlen, S.R., Montana, A. and Kerrick, D.M. (1991) *American Mineralogist*, **76**, 677-680.



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-05-001		
Experiment title:	Texture of hematite ore deformed to high strains in torsion		
Dates of experiment:	12 days	Date of report:	16.02.2006
Experimental team:			
Names	Addresses		
H. Siemes *	Institut für Mineralogie und Lagerstättenlehre * Förderung DFG Si209/36-1 RWTH-Aachen, D-52056 Aachen		
B. Klingenberg			
E. Rybacki	Geoforschungszentrum Potsdam Projektbereich 3.2: Rheology and Tectonophysics D-14474 Potsdam		
M. Naumann			
K. Kunze	Geologisches Institut, ETH-Zürich CH-8092 Zürich		
Local Contact:	J.M. Walter, E. Jansen		

Experimental report text body

Einführung

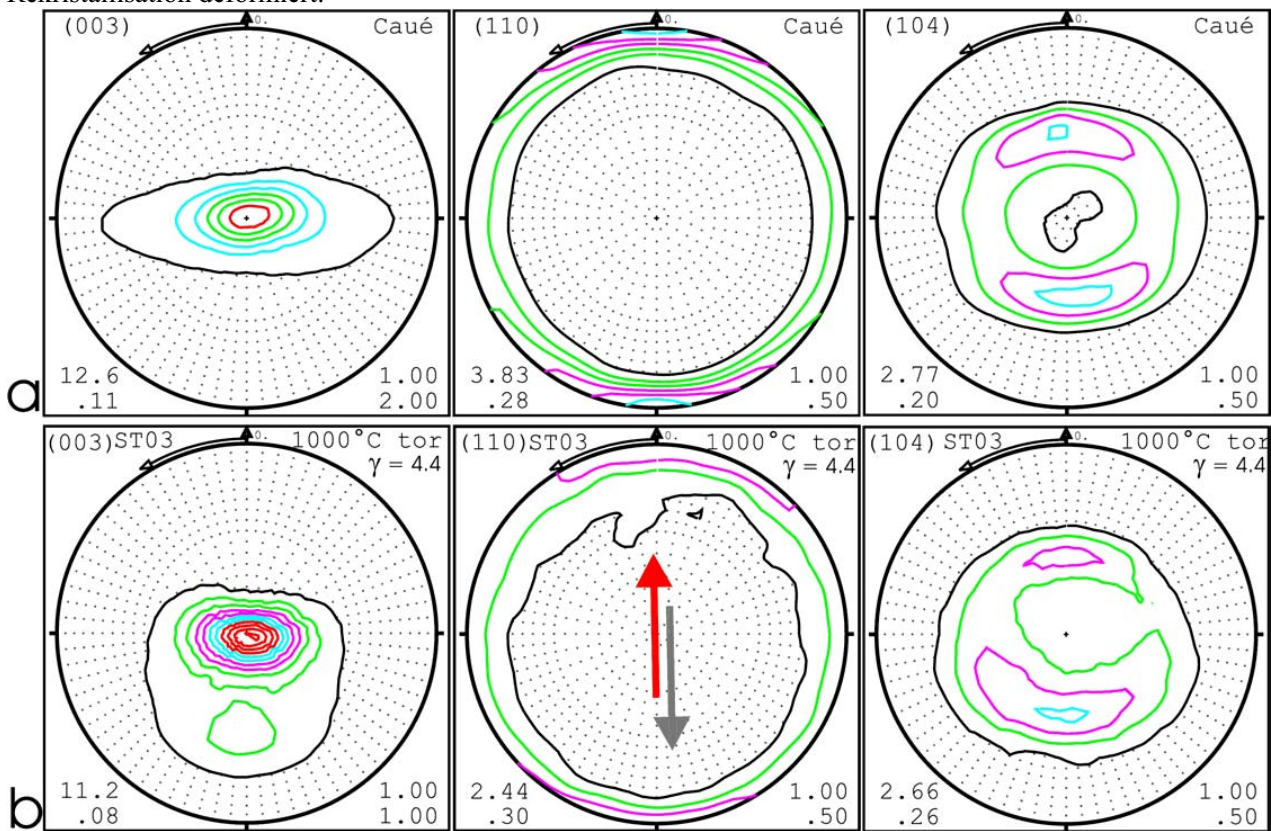
Die Beziehungen zwischen der Mikrostruktur und der kristallografischen Vorzugsorientierung (Textur) von Hämatiterzen des Eisernen Vierecks in Brasilien und von anderen Lokalitäten wurden in vielen Veröffentlichungen behandelt, z.B. Quade et. al. 2000, Rosière et al. 2001, Bascou et al. 2002. Polfiguren dieser Hämatiterze zeigen kreisförmige bis elliptische c-Achsen-Maxima, die um den Pol der Foliation liegen. Die Pole der Prismenflächen liegen auf Großkreisen in der Foliationsebene und die Maxima auf diesen fallen mit der Lineation zusammen (Abb. a). Die Entstehung dieser Regelung wird auf Scherverformungsprozesse zurückgeführt. Bei experimentellen Stauchversuchen an polykristallinem Hämatit gab es Anzeichen der Bildung eines c-Achsenmaximums senkrecht zur Kompressionsrichtung (Siemes et al. 2003). Da jedoch zu vermuten war, dass Gleiten parallel zur Basis des Hämatits Ursache der Regelung ist, wurden Scherexperimente an polykristallinen Hämatitaggregaten konzipiert.

Konzeption der Versuche

Zunächst wurden drei Torsionsversuche in einer Hochdruck-Hochtemperatur-Apparatur (Paterson & Olgaard, 2000) konzipiert, um zu prüfen, ob und wie diese Versuche sich mit Hämatiterz realisieren lassen. Als Versuchsmaterial wurde ein Hämatiterz von Sishen, Südafrika, gewählt, das fast unregelmäßig ist und eine Kristallitgröße von 5-60 Mikrometer aufweist (Siemes et al. 2003). Proben mit 14 mm Durchmesser und 10 mm Länge wurden für die Torsionsversuche hergestellt. Eine Schutzhülle aus Silber-Palladium in einem Eisen-jacket, das die Probe vom Druckmedium Argon trennt, erwies sich als die geeignete Versuchsanordnung, um Hämatit mit nur geringer Magnetitbildung zu verformen. Bei Temperaturen von 1000°C bis 800°C wurden bei 400 MPa allseitigem Druck, mit einer Torsionsrate von $6 \cdot 10^{-5} \text{ s}^{-1}$ in 30-Stunden-Versuchen Scherverformungen von 3,8 bis 4,4 erreicht.

Erste Ergebnisse der Versuche bei 1000°C

Der Hämatit ist bei 1000°C zu einem gleichförmigen polygonalen Korngefüge rekristallisiert, das sich auch bei natürlich verformten Erzen findet. Die Korngrößen weisen dabei keine Unterschiede über die Probenquerschnitte auf, obwohl die finite Scherverformung von Null im Zentrum der Probe bis zum maximalen Wert an der Peripherie variiert. Neutronentexturmessungen (Jansen et al. 2000) im Probenbereich mit der stärksten Verformung ergaben für die (0003)-Polfigur ein zentrales, elliptisches Maximum im Pol der Scherebene. In der (11-20)-Polfigur befindet sich auf dem Grundkreis ein Maximum, das die Orientierung der Scherrichtung angibt. Die (10-14)-Polfigur, die zwei bananenförmige Maxima (Quade 1988) unterschiedlicher Höhe aufweist, lässt auf den Schersinn schließen (Abb. b). Diese Polfiguren stimmen weitgehend mit Polfiguren natürlich verformter Hämatiterze überein. Im Gegensatz zum homogenen Korngefüge besitzt die Textur einen grossen Gradienten von nahezu regellos im Zentrum (undeformiert) zu stark geregelt am Rand (maximaler Shear-Strain) wie EBSD-Messungen (Kunze et al. 1993) zeigen. Die Beobachtungen unterstützen die Vermutung, dass Hämatiterz bevorzugt durch Versetzungsgleiten auf $\{0001\}\langle 11-20 \rangle$ mit simultaner dynamischer Rekristallisation deformiert.



(0003)-, (11-20)- und (10-14)-Polfiguren a) natürlich verformtes Hämatiterz, Caué, Minas Gerais, Brasilien, b) experimentell verformtes Hämatiterz aus dem Randbereich der tordierten Probe, Polfiguren parallel zur Scherebene, Scherrichtung etwa NS, Schersinn N.

Danksagung

Die Autoren danken der Deutschen Forschungsgemeinschaft und den Mitarbeitern der beteiligten Institute.

Literatur

- Bascou, J., Raposo, M.I.B., Vauchez, A., Egydio-Silva, M., 2002. Earth and Planetary Science Letters 198, 77-92.
 Jansen, E., Schäfer, W., Kirfel, A., 2000. Journal of Structural Geology 22, 1559-1564.
 Kunze, K., Wright, S.I., Adams B.L. and Dingley D.J., 1993. Textures and Microstructures 20, 41-54.
 Paterson, M.S., Olgaard, D.L. 2000. Journal of Structural Geology 22, 1341-1358.
 Quade, H., 1988. Textures and Microstructures, 8-9, 719-736
 Quade, H., Rosière, C.A., Siemes, H., Brokmeier, H.-G., 2000. Zeitschrift für angewandte Geologie, Sonderheft 1, 155-162.
 Rosière, C.A., Siemes, H., Quade, H., Brokmeier, H.-G., Jansen, E.M., 2001. Journal of Structural Geology 23, 1429-1440.
 Siemes, H., Klingenberg, B., Rybacki, E., Naumann, M., Schäfer, W., Jansen, E., Rosière, C.A., 2003. J. Struct. Geol. 25, 1371-1391.



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-05-002		
Experiment title:	Magnetic and Mineral Fabrics in Synthetic Marbles		
Dates of experiment:	72 days	Date of report:	16.05.2006
Experimental team: Names	Addresses		
Leiss, Bernd Hirt, Ann Schmidt, Volkmar Piguet, P. Burlini, Luigi	Geowissenschaftliches Zentrum der Universität Göttingen (GZG) Abteilung Strukturgeologie und Geodynamik Goldschmidt Str. 3 D-37077 Göttingen Institut für Geophysik, ETH-Hoenggerberg, CH-8093 Zürich, Switzerland Geologisches Institut, ETH Zurich, CH-8092 Zürich, Switzerland		
Local Contact:	Jens M. Walter		

Experimental report text body

Introduction

The anisotropy of magnetic susceptibility (AMS) of rocks reflects the preferred orientations of minerals. Therefore AMS is a quick and easy way to characterize rock fabrics (Hrouda 1982, Borradaile 1988); the obtained result is also called the magnetic fabric of the rock. The method has been often used to measure the orientation of ferromagnetic minerals, mainly magnetite, but in recent studies it has been increasingly used to measure textures of paramagnetic minerals as phyllosilicates (Lüneburg et al. 1999, Cifelli et al. 2004). AMS of calcite textures A further application is the measurement of diamagnetic textures, especially calcite textures. Calcite is suitable for the AMS method, because it has a high magnetic anisotropy with the minimum susceptibility along the crystallographic c-axis. Therefore a preferred orientation of the c-axes, which can be induced by deformation, generates a magnetic fabric. The relationship between AMS and deformation of marbles has been investigated in laboratory (Owens & Bamford 1976) and field studies (de Wall et al. 2000). However, the diamagnetic fabric of calcite is relatively weak and easily overprinted by paramagnetic and ferromagnetic phases. In natural carbonate rocks the ferromagnetic and the paramagnetic subfabrics should be separated to assure a correct interpretation of the AMS. This separation can be made by torque measurements in high fields at different temperatures

Textures of artificial calcite-muscovite aggregates

To test how well AMS reflects the actual mineral texture, we produced a series of artificial calcite-muscovite aggregates. Powders made from Carrara marble and muscovite single crystals were mixed in different proportions and compacted uniaxially at room temperature to obtain a texture. The samples were further compacted hydrostatically to reduce the porosity and to improve the mechanical properties. The samples show a c-axis preferred orientation of the calcite and muscovite along the compaction direction (Fig. 1). The textures

were measured with different methods as AMS, X-ray diffraction and neutron diffraction and the results are compared. Sample volumes of more than 11 cm³ were used to measure AMS as well as neutron diffraction; the same specimens could be used for both methods. Low- and high-field AMS measurements were made on all samples. The high-field measurements reflect more accurately the weak dia/paramagnetic fabrics. A nearly perfectly oblate AMS ellipsoid with minimum susceptibility axis sub-parallel to the compression direction developed even at very low pressures, and the principle axes of the AMS ellipsoid are co-axial with the calcite fabric ellipsoids. A general increase in AMS is observed with an increase in compaction. The quantitative correlation between the strength of the texture and the AMS will be investigated in the future. Moreover the separation of magnetic sub-fabrics will be tested on these artificial samples. The first results show that AMS can reflect the textures of the individual phases in a multiphase rock.

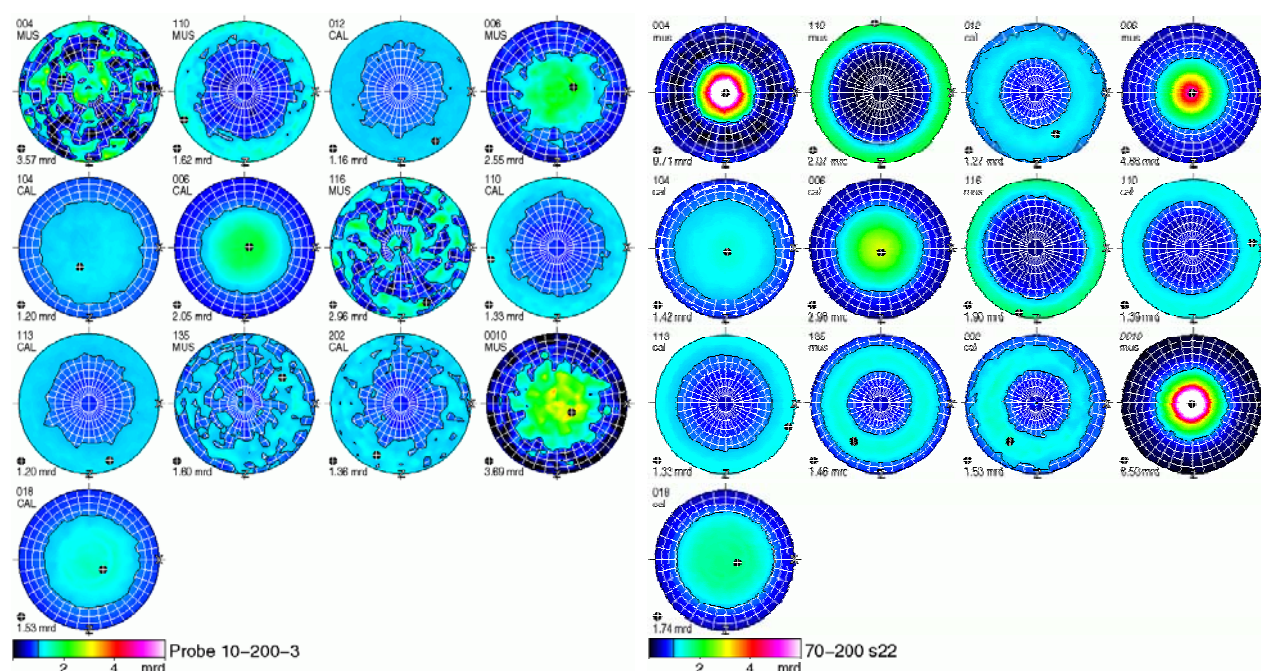


Figure 1: Examples of neutron diffraction pole figures (equal-area, lower hemisphere projection). Compaction direction is normal to the pole figure. Left: 10 % muscovite content, right: 70 % muscovite content

References:

- Borradaile GJ (1988) Magnetic-susceptibility, petrofabrics and strain. *Tectonophysics* 156:1–20
- Cifelli F, Mattei M, Hirt AM & Gunther A (2004) The origin of tectonic fabrics in ‘undeformed’ clays: The early stages of deformation in extensional sedimentary basins. *Geophys Res Lett* 31: Art. No. L09604
- De Wall H, Bestmann M & Ullemeyer K (2000) Anisotropy of diamagnetic susceptibility in Thassos marble: A comparison between measured and modeled data. *J Struct Geol* 22:1761–1771
- Hrouda F (1982) Magnetic-anisotropy of rocks and its application in geology and geophysics. *Geophysical Surveys* 5:37–82
- Lüneburg CM, Lampert SA, Lebit HD, Hirt AM, Casey M & Lowrie W (1999) Magnetic anisotropy, rock fabrics and finite strain in deformed sediments of SW Sardinia (Italy). *Tectonophysics* 307:51–74
- Owens BH & Bamford D (1976) Magnetic, seismic, and other anisotropic properties of rock fabrics. *Phil Trans R Soc Lond Ser A* 283:55–68



Experimental Report of Neutron Scattering Experiments at the FRJ-2 Reactor

Proposal number:	SV7-05-003		
Experiment title:	Investigation of $\text{La}_{1-x}\text{Sr}_x\text{MnO}_3$ samples by powder diffractometer		
Dates of experiment:	14.03.05-20.03.05	Date of report:	22.06.2006
Experimental team: Names	Addresses		
Eugene A. Gorelik	ISM, IFF, Forschungszentrum Jülich		
Local Contact:	Dr. Jens M. Walter		

Experimental report text body

Introduction

The aim of experiment was to check the results of preliminary measurements of LSMO samples made by SV7 and D20 (ILL) powder diffractometer. The strange behaviour of the temperature dependences of integral intensity were found in diffraction experiments made by D20 in Grenoble (see fig.1)

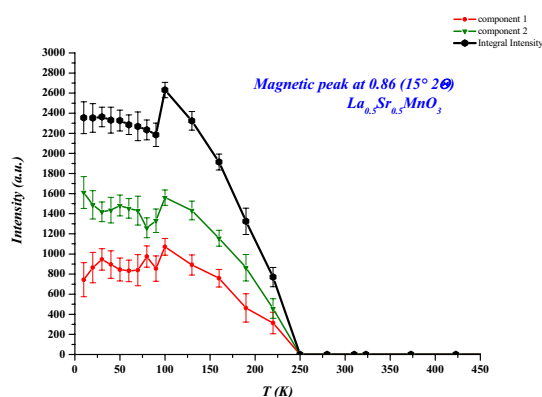


Fig. 1 Temperature dependence of integral intensity of peak (001)

Measurements and Results

Result from that it was interesting to make some new experiment to obtain answers to following questions:

1. Is this the real effect (visualisation of Jan-Teller effect) or it is an artefact of D20 powder diffraction machine (ILL)?
2. What are the exact temperatures of start and end points of effect?
3. How the curves behave around critical temperatures?

To find that information was decided to make neutron powder diffraction measurement of $\text{La}_{1-x}\text{Sr}_x\text{MnO}_3$ samples with x equally 50, 52.5, 55, 57.5 and 60% in a q -range from 0 to 3.5 and in the following temperature areas: 90 – 120 K (step 5 K), 190 – 280 K (step 10 K), 300 – 400 K (step 10 K). Also it was interesting to make preliminary measurements of two new samples with x equally 80 and 90% in a full range of q -values and temperatures.

We had some serious problems with the instrument during the measurements. It was not perfectly calibrate after re-equipment. As a result we had an extremely high background and some additional peaks on the pattern (see fig.2)

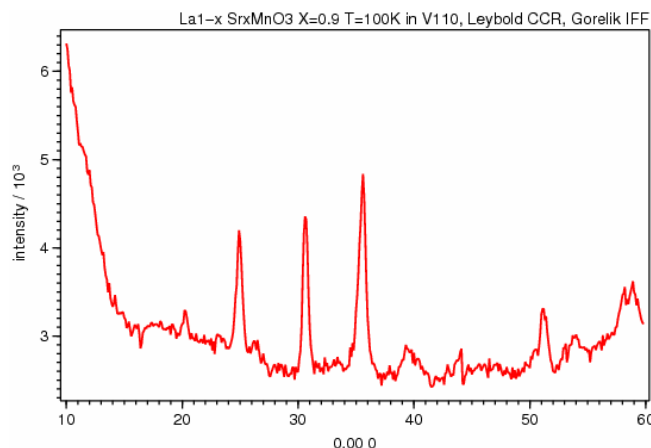


Fig.2 Neutron diffraction of LSMO sample $x = 90\%$. $T = 100$ K duration of measurements 3 h.

At least only one sample ($x = 90\%$) was measured. The results of this powder diffraction can not be used for following modelling and answers for the questions of experiment also could not be found. To obtain that answers was made another one experiment by DNS instrument. It shows that the gap in temperature dependence of integral intensity of LSMO samples is an instrumental artefact of D20 and does not connected with anyone physical effect.



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-05-004		
Experiment title:	Neutron diffraction analysis of silver coins from the Himyarite-Sabean Empire		
Dates of experiment:	34 days	Date of report:	17.05.2006
Experimental team: Names	Addresses		
Kirfel, Armin Kockelmann, Winfried, Yule, Paul	Mineralogisch-Petrologisches Institut der Universität Bonn Poppelsdorfer Schloss D-53115 Bonn Rutherford Appleton Laboratory, ISIS Facility, ROTAX, Chilton, Didcot, Oxon, OX11 0QX United Kingdom Völkerkundemuseum München		
Local Contact:	Jens M. Walter, R. Skowronek		

Experimental report text body

Introduction

The Himyarite, later Himyarite-Sabean empire in the southern part of the Arabic peninsula lasted from about the first century A.D. till the end of the sixth century when it fell under the control of the caliphate of Medina. During the period of its existence coins were issued typically of 10 - 15 mm diameter and carrying heads as motifs on both sides. These coins are expected to consist of pure silver, and the peculiar shape of them, i.e. their irregular and concave (scaphate) appearance (Fig. 1), implies that their patterns were not prepared by casting in a mould, but rather by striking or deep-drawing with mechanical force. Other coins are flat and exhibit bubbles which indicate casting. Although the coins significantly testify to a period during which little real historic information is available, surprisingly they have never been studied fully so far, e.g. by means of physical methods. It is the aim of our study to non-destructively determine the metallurgical composition, e.g. the phase abundance of Ag and eventually other metals like Cu, and to obtain information about the manufacturing methods, both also as a function of age.



Fig. 1) Examples of himyarite silver coins: flat with bubbles (left) and concave (right)

Measurements and Results

In order to assess these questions, 10 coins of different ages and locations have been selected from the collection held by the Staatliche Museum für Völkerkunde in Munich in order to determine their phase compositions by neutron diffraction. This is the only appropriate technique if the object(s) of interest are not to be damaged at all by the very method of investigation. Furthermore neutron diffraction was chosen as the appropriate method to obtain data from the complete sample volume. Additionally density measurements have been conducted and correlated to the phase refinements from the neutron powder diffraction data.

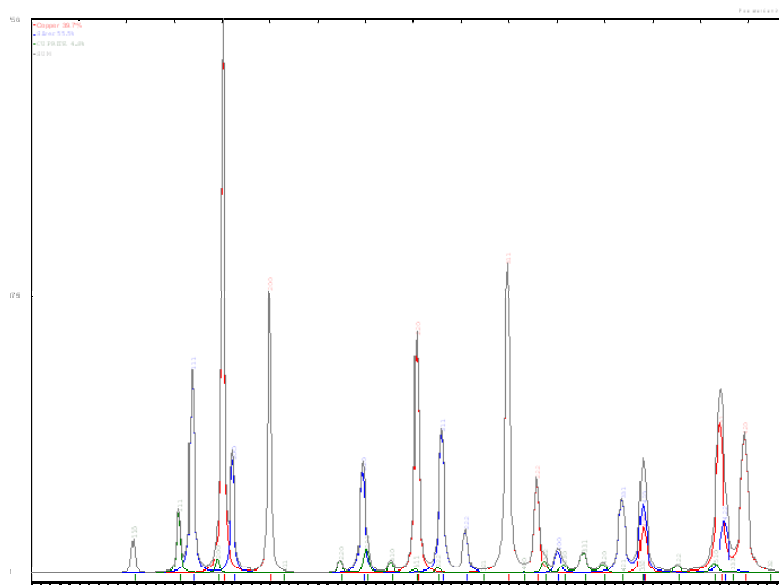


Fig. 1: Rietveld refinement of silver coin for phase compositions

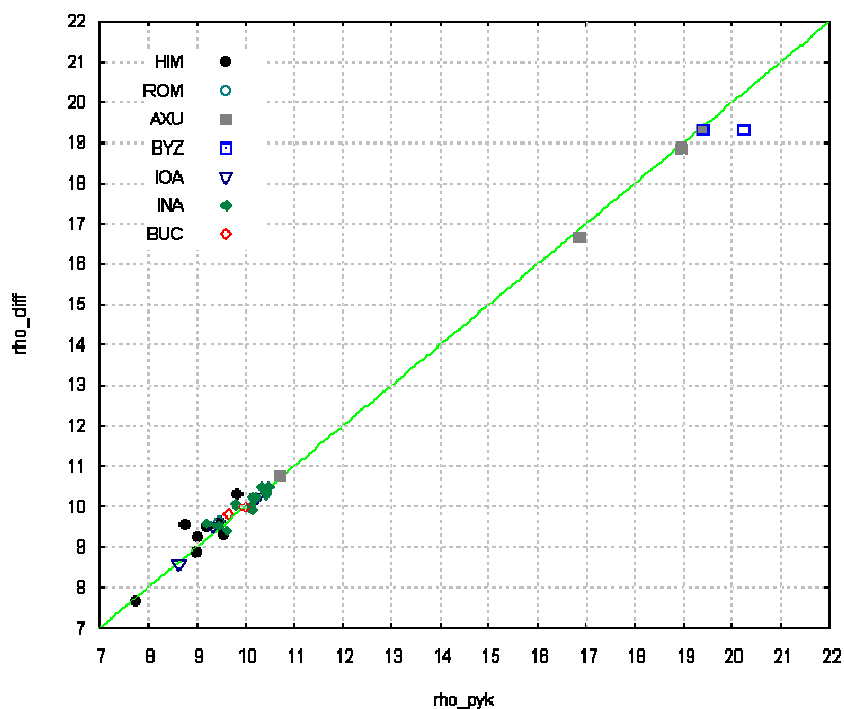


Fig. 2: Density measurements correlated with phase analysis



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-05-005		
Experiment title:	Crystal fabric of lava flows and domes by textural neutron diffraction analysis		
Dates of experiment:	30.08.05, 12.01.06 18.04.06-26.04.06	Date of report:	12.04.2006
Experimental team: Names	Addresses		
Fiori, Fabrizio Albertini, Gianni Iezzi, Gianluca Ventura, Guido	Dipartimento di Scienze Applicate ai Sistemi Complessi, Sez. Scienze Fisiche – Università Politecnica delle Marche, Via Brecce Bianche, 60131 Ancona (Italy) Dipartimento di Fisica e Ingegneria dei Materiali e del Territorio – Università Politecnica delle Marche, Via Brecce Bianche, 60131 Ancona (Italy) Dip. Scienze della Terra, Università di Chieti (Italy) Osservatorio Vesuviano, Ercolano, Napoli (Italy)		
Local Contact:	Dr. Jens M. Walter, E. Jansen R. Skowronek		

Experimental report text body

Introduction

For the fabric determination and its variation on different volcanic rocks it is important to obtain important insights on the kinematical processes underwent by both lavas and pyroclastic flows, during their ascent and emplacement processes across and on the Earth (Smith, 2002). Basically crystals can be regarded as passive strain markers for both dilute (not-interacting) crystal-poor bearing lavas (Manga, 1998; Castro et al., 2002; Iezzi and Ventura, 2002; Canon-Tapia and Castro, 2004) or (small to strong) interacting crystal-bearing lavas (Shelley, 1985; Wada, 1992; Ventura et al., 1996). The theoretical assumption to use crystals as passive strain markers implies, that they must be undeformable and not-growing solid particles during magma deformation. The spatial alignment of crystals can therefore be used such to reconstruct the strain path underwent by a lava and/or pyroclastic rocks during their emplacement.

Measurements and results

Since long time the determination of volcanic crystal fabric has been mainly carried out using microscopic (optical and electronic) observations and AMS measurements. Taking in account that, we have carried out several neutron texture diffraction experiments at the SV-7 diffractometer, with twofold aims: a) introduce a new and completely independent analytical tool for crystal fabric determination in the modern volcanology science and b) to elucidate the crystal fabric parameters of several (mainly effusive) volcanic rocks.

Thus crystal fabric analysis allows to constraint and in principle also to reconstruct the deformation path suffered by a lava or by a smaller portion of it. Anyway despite its potential applications, its use have been relatively poor adopted for volcanic rocks. The few studies which report crystal fabric analyses for effusive magmatic rocks have been carried out using optical thin section observations or AMS measurements. However

all these studies take advantages from particular and extreme favourable situation of cropping exposure, such as the possibility to determine the section containing the shear direction or the shear plane (Ventura et al., 1996) or the situation on which is possible to reconstruct the crystal orientation in space due to the extremely low crystallinity of the rocks and also to their smaller dimensions (tens of μm) (Manga, 1998; Castro et al., 2002; Canon-Tapia and Castro, 2004). Moreover AMS data can be used only for certain mineral association, as a function of their chemistry and their spatial arrangements (continuous vs. not-continuous network Fe-bearing phases).

By contrast, neutron diffraction crystal textural analysis can be used for most types of volcanic rocks. It has the advantage to analyse a large rock volume (order of cm^3 units) directly in 3D and to be selective for any mineral phase hosted in the rock specimen under investigation (Schäfer, 2002). For the fabric determination it is of capital importance to constraint and link the measured crystallographic orientation (CPO) for each mineral phase (see Fig. 1) with the crystal shape orientation in the rock. In any case is important to stress that the majority of silicate magmatic minerals have crystal shapes currently from medium to high anisotropic. Thus is generally rather easy to determine which crystal direction correspond to a well determined crystallographic direction.

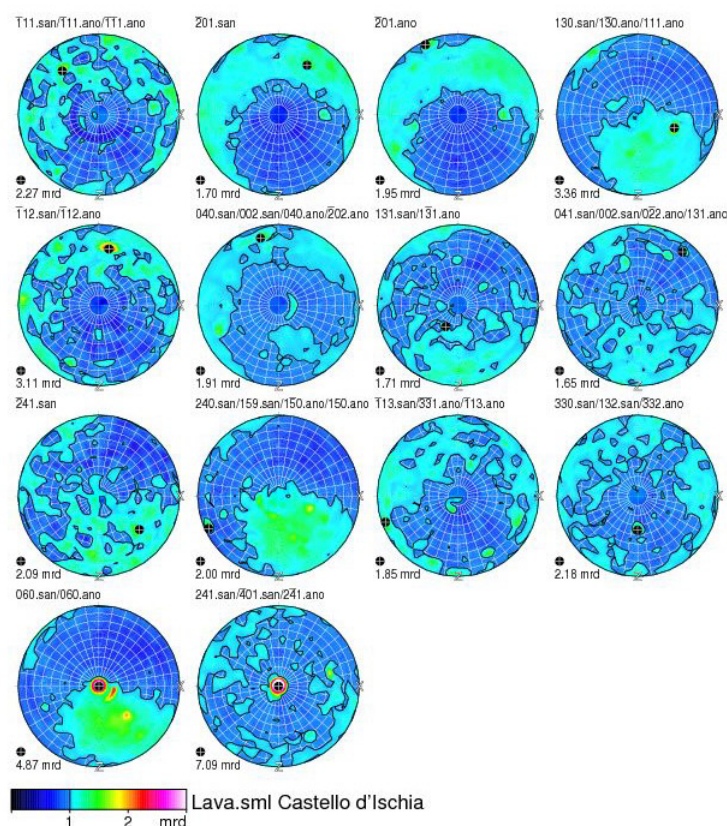


Fig.1 Texture measurement of a lava from Castello d'Ischia

References

- [1] Canon-Tapia E., Castro J. (2004). *Journal of Volcanology and Geothermal Research*, 134: 169-182.
- [2] Castro J., Manga M., Cashman K. (2002). *Earth and Planetary Science Letters*, 199: 211-226.
- [3] Iezzi G., Ventura G. (2002). *Earth and Planetary Science Letters*, 200: 33-46.
- [4] Manga M. (1998). *Journal of Volcanology and Geothermal Research*, 86: 107-115.
- [5] Schäfer W. (2002). *European Journal of Mineralogy*, 14: 263-290.
- [6] Shelley D. (1985). *Journal of Volcanology and Geothermal Research*, 25: 69-79.
- [7] Smith J.V. (2002). *Earth-Science Reviews*, 57: 279-297.
- [8] Ventura G., De Rosa R., Colletta E., Mazzuoli R. (1996). *Bulletin of Volcanology*, 57: 555-562.
- [9] Wada Y. (1992). *Journal of Volcanology and Geothermal Research*, 49: 119-126.



Experimental Report
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at the FRJ-2 Reactor

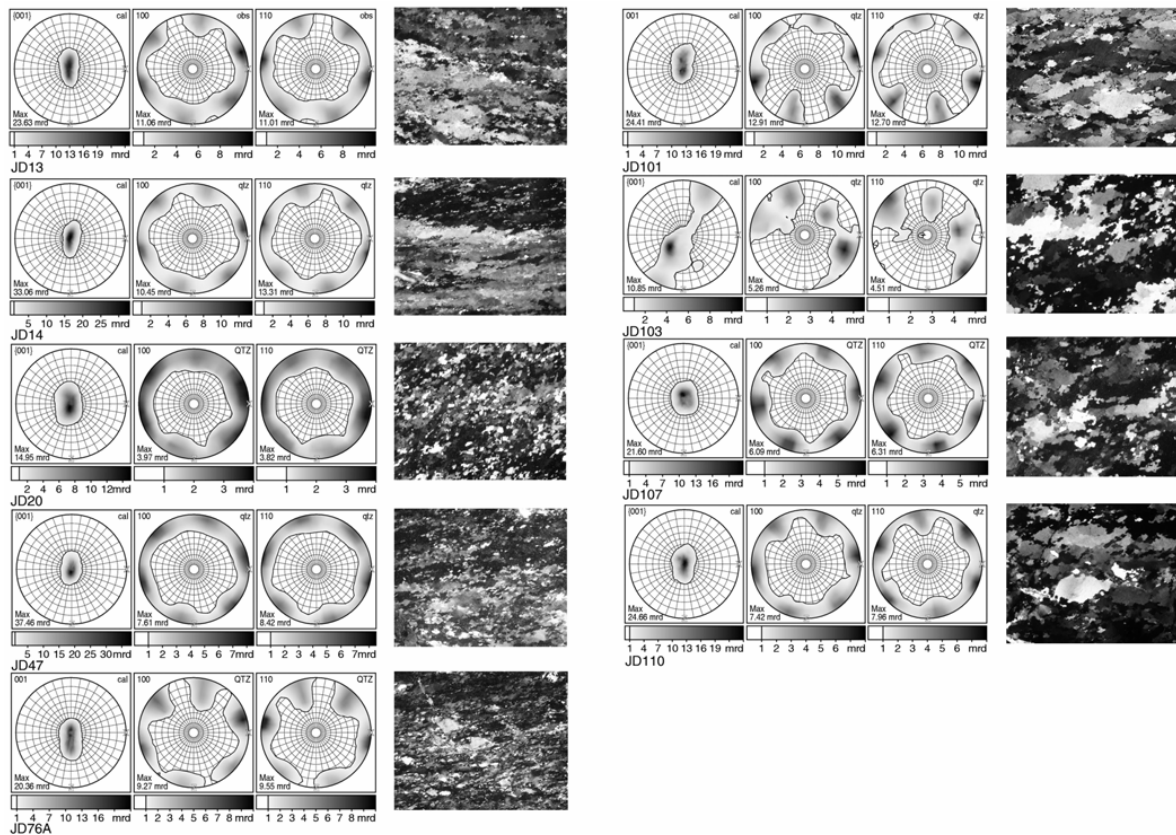
Proposal number:	SV7-05-006		
Experiment title:	Neutron texture goniometry on quartzitic mylonites of a Permian detachment fault at the base of the Collio basin, northern Italy		
Dates of experiment:	04.03.05-07.03.05 23.06.05-27.06.05 19.08.05-20.08.05	Date of report:	14.06.2006
Experimental team: Names	Addresses		
N. Froitzheim J.F. Derks	Geologisches Institut der Universität Bonn, Nußallee 8, D-53115 Bonn		
Local Contact:	J.M. Walter		

Experimental report text body

In the area of the Alpine Orobic anticline (Southern Alps), a large-scale extensional detachment fault was identified that separates Hercynian basement in the core of the anticline from overlying Permian clastic sediments and volcanic rocks (Collio Formation). The basement consists mainly of paragneiss (Morbegno gneiss) which is intruded by plutons, known as Val Biandino Granodiorite and Valle Biagio Granite. The emplacement of plutons was probably coeval with and genetically related to deposition of volcanics in the Collio Formation in the late Sakmarian to middle Artinskian, c. 287-280 Ma ago. The Collio Formation is everywhere in the study area separated from the basement by a layer of cataclasite. The Morbegno Gneiss underlying the cataclasite is a mylonite with a strong foliation and lineation. The foliation is parallel to the cataclasite layer and therefore also to the basement/Collio contact. The intensity of the mylonitic deformation is strongest near the top and decreasing downward. The thickness of the mylonitic zone is about 100 metres. The Val Biandino Granodiorite is partly affected by the same mylonitisation as the Morbegno Gneiss, but also contains locally mylonite xenoliths. These observations indicate overall contemporaneity between mylonitisation and granodiorite intrusion.

Mylonitisation happened under higher greenschist-facies conditions near the intrusions, and under lower greenschist-facies conditions away from them (biotite and chlorite zone, respectively). While plagioclase is only brittlely deformed, quartz is always dynamically recrystallised. In some samples, quartz grain shapes suggest recrystallisation by grain-boundary migration (e.g., sample JD 103). Other samples have a uniform, very small grain size, suggesting subgrain rotation as the dominant recrystallisation mechanism (e.g., sample JD 20). In thin sections parallel to the X-Z plane of the finite strain, abundant shear-sense criteria indicate top-to-the-SE shearing, both in the biotite zone and the chlorite zone mylonites. Textures of nine almost monomineralic quartz mylonites have been analysed. All diagrams are upper-hemisphere Wulff projections. All textures except one (JD 103) are very similar and surprisingly clear and regular. {c} forms a single

maximum parallel to the foliation and perpendicular to the stretching lineation, i.e. parallel to the strain Y axis. This maximum is strong and partly round, partly slightly elongated in the plane perpendicular to the stretching lineation. The shear sense is given by the {m} and {a} pole figures. Both {m} and {a} pole figures show three maxima distributed on a great circle parallel to the X-Z plane. These maxima are not symmetric with respect to the foliation trace, but rotated anticlockwise in the case of {m} and clockwise in the case of {a}. The strongest of the {a} maxima, representing the dominant slip direction, is rotated about 10° to 20° clockwise from the stretching direction. The textures are clear evidence for predominant prism <a> slip. The obliquity indicates dextral (top-to-the-SE) shear sense. Prism <a> slip becomes dominant in quartz mylonites under temperatures above c. 500° C. Temperatures above 500° C during the mylonitisation of the Morbegno gneiss are in line with the observed stability of biotite. As demonstrated above, the mylonitic shearing was broadly synintrusive. Therefore, heat from the rising melt body may have caused the elevated temperatures. JD 103 is the only sample with a different texture. The thin section shows strongly sutured and interlocking quartz grain boundaries suggestive of grain-boundary migration recrystallisation. Biotite is completely replaced by chlorite, whereas it was stable during deformation in the other samples. In JD 103, {c} is distributed on an oblique single girdle suggesting top-to-the-SE shearing. Two maxima are on this girdle, one near the periphery, and a stronger one about 40° away from the centre of the pole figure. The weaker {c} maximum close to the periphery may result from the activity of basal <a> slip. In this case, the texture may represent deformation under lower temperatures than for the other samples, which is in line with the observed replacement of biotite by chlorite. The fault marked by the cataclasite layer at the basement/Collio contact accommodated strong exhumation of the footwall: It juxtaposes footwall mylonites initially deformed under high temperatures (above c. 500° C) against hanging-wall volcanics erupted at Earth's surface. We therefore interpret the fault as an extensional detachment fault and call it "Grassi detachment fault". The mylonites formed during an early stage of its activity. They were transported upward by the continuing tectonic unroofing of the footwall as a metamorphic core complex. Therefore we assume that the brittle Grassi detachment fault had the same kinematics, top-to-the-SE, as the mylonites.





Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-05-007		
Experiment title:	Pilot-Studie für Neutronentexturgoniometrie-Messungen an ultramafischen Gesteinen		
Dates of experiment:	08.03.05-09.03.05	Date of report:	11.05.2006
Experimental team: Names	Addresses		
Grimmer, Jens Leiss, Bernd	Geologisch-Paläontologisches Institut, Ruprecht-Karls-Universität Heidelberg, Im Neuenheimer Feld 234, D-69120 Heidelberg Geowissenschaftliches Zentrum der Universität Göttingen (GZG) Abteilung Strukturgeologie und Geodynamik Goldschmidt Str. 3 D-37077 Göttingen		
Local Contact:			

Experimental report text body

Einleitung und Fragestellung

Ultramafische Gesteine in hochgradig metamorphen Kristallindecken von Kollisionsorogenen sind wichtige Indikatoren dafür, dass die Versenkung kontinentaler Krustengesteine bis in den oberen Mantel erfolgte. Diese ultramafischen Gesteine wurden vermutlich während der Subduktion bzw. der Exhumierung tektonisch in die Krustengesteine eingeschuppt. Die wahrscheinlich auftriebsgesteuerte Exhumierung dieser Kristallingesteine bringt dabei Fragmente des lithosphärischen Mantels in krustale Niveaus. Texturelle Untersuchungen an diesen Mantelgesteinen geben wesentliche Aufschlüsse über Deformationsmechanismen, -richtungen und -temperaturen, die wiederum wichtige Informationen über die orogenen Prozesse liefern.

Die untersuchte Probe 005b stammt aus einem dunitischen Bereich des Kittelfjäll-Peridotit, der linsenförmig in einer Kristallindecke aus Gneisen, Glimmerschiefern und Amphiboliten Platz genommen hat. Diese Kristallindecke gehört zur Seve-Einheit der schwedischen Zentralkaledoniden. Die Kristallingesteine der Seve-Einheit repräsentieren den ehemaligen distalen passiven Kontinentalrand Balticas, der im frühen Paläozoikum subduziert und nachfolgend exhumiert wurde.

Die Analyse der kristallographischen Vorzugsorientierung der Probe 005b mit der Neutronentexturgoniometrie dient einerseits der Quantifizierung der Olivin-Textur sowie andererseits dem Vergleich mit AMS-Messungen. Der Zusammenhang zwischen dem gesteinsmagnetischen Gefüge und kristallographischen Vorzugsorientierungen ist weder qualitativ noch quantitativ verstanden und ein wesentliches methodisches Ziel. Die Anwendung der Neutronenbeugung ist bei dieser Probe Voraussetzung, da sie Korngrößen bis zu 1 mm aufweist.

Ergebnisse

Die Ergebnisse der Neutronentexturgoniometrie-Messungen und der Gesteinsmagnetik sind in Abb. 1, 2 und 3 dargestellt. Die Neutronentexturgoniometrie-Messungen ergeben eine deutliche kristallographische

Vorzugsorientierung (Abb. 1). Es zeigt sich eine sehr gute Übereinstimmung der (100)-Richtung mit der Richtung der größten Hauptsuszeptibilität ($k_{\max}$) bzw. der (001)-Richtung mit der Richtung der kleinsten Hauptsuszeptibilität ($k_{\min}$). Die Ergebnisse zeigen eine signifikante kristallplastische Deformation, die sich durch Neutronentextur-goniometrie-Messungen und gesteinsmagnetische Methoden quantifizieren lassen. Für weitere und tiefer gehende quantitative Vergleiche zwischen beiden Methoden sowie eine regionalgeologisch-tektonische Interpretation ist allerdings ein wesentlich umfangreicherer Datensatz notwendig.

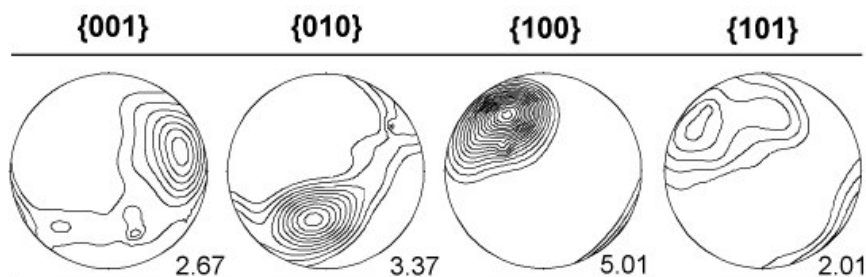


Abb. 1: Polfiguren der Probe 005b. Die quantitative Textur wurde auf Basis der experimentellen Polfiguren mit Hilfe der Texturkomponentenmethode bestimmt.

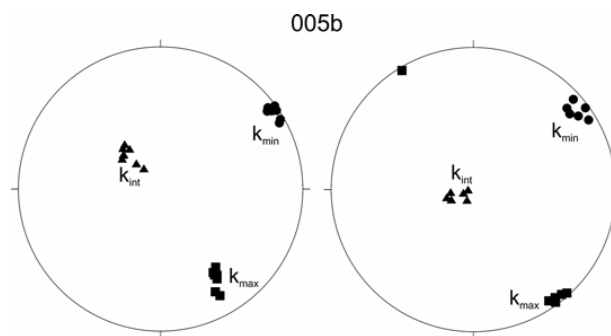


Abb. 2: Die Hauptsuszeptibilitäten ($k_{\max}$, k_{int} , $k_{\min}$) der Probe 005b wurden bei Raumtemperatur (links) und bei niedrigen Temperaturen (rechts), d.h. Temperaturen unterhalb des Verwey-Übergangs ($<-155^{\circ}\text{C}$) gemessen. Die Tieftemperaturmessung diente zur Hervorhebung des paramagnetischen Olivin-Gefüges, da aufgrund geringer retrograder Überprägung Magnetit gebildet wurde, dessen Einfluss auf das Gesamtgefüge (links) hierdurch nahezu eliminiert werden konnte.

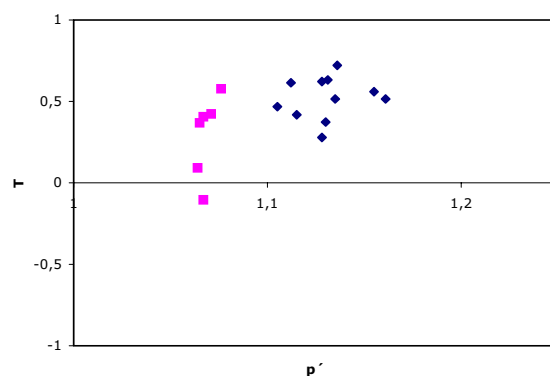


Abb. 3: Jelinek-Diagramm der Probe 005b mit Angabe von Formfaktor T und dem korrigierten Grad der Anisotropie der magnetischen Suszeptibilität P' . P' streut wesentlich geringer, wenn der Einfluss des ferrimagnetischen Magnetits unter niedrigen Temperaturen ($<-155^{\circ}\text{C}$) nahezu eliminiert ist. Insgesamt ist P' mit Werten von 1,064 bis 1,076 relativ gering.



Experimental Report of Neutron Scattering Experiments at the FRJ-2 Reactor

Proposal number:	SV7-05-008:		
Experiment title:	Neutron Diffraction on new Carbodiimides		
Dates of experiment:	21.04.05-25.04.05 09.05.05-12.05.05 25.10.05-01.11.05 12.12.05-13.12.05 13.02.06-16.02.06	Date of report:	28.6.2006
Experimental team: Names	Addresses		
Dr. Müller, Paul Prof. Dr. R. Dronskowski, Dr. Liu, Xiaohui	Institut für Anorganische Chemie der RWTH Aachen Landoltweg 1, 52056 Aachen		
Local Contact:	Dr. Jens M. Walter		

Experimental report text body

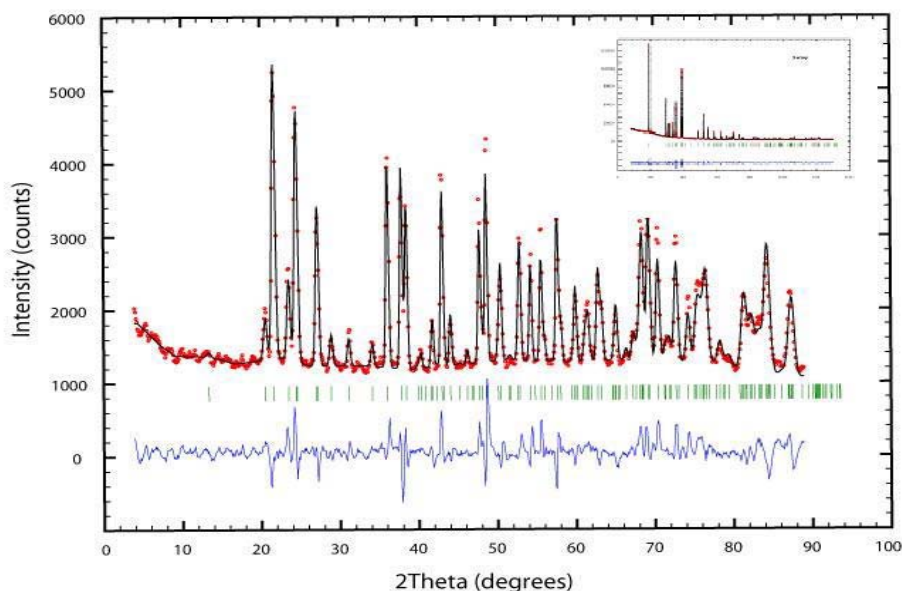
Neutron data on a powdered sample of CuNCN were collected using the SV7 diffractometer with $\lambda = 1.09590$ Å radiation. The crystal structure eventually arrived at is based on a Rietveld refinement with a data set of 138 reflections using a Gaussian profile for temperatures of 293 and 4 K. The residual values referring to the data from 4 K and 293 K arrive at 0.050/0.044 (R_p), 0.068/0.063 (R_{wp}), and 0.11/0.13 (R_{Bragg}) for a total of 14 variables. The final structural parameters are given in Table 1 while an overview of the Rietveld refinement at 4 K is shown in Figure 1.

The crystal structure of CuNCN derived from neutron diffraction corroborates the earlier X-ray diffraction analysis [1]. The Cu^{2+} ions form cationic sheets parallel to the ab face. Each Cu^{2+} ion is coordinated by six nitrogen atoms in the spirit of a typical first-order Jahn–Teller effect. The NCN^{2-} units run parallel with the c axis. The volume of the unit cell decreases slightly with temperature, which indicates that no structural phase transition happens down to 4 K. The complex anion is practically linear ($\text{N–C–N} = 177.4(9)^\circ$) with approximate $D_{\infty h}$ symmetry at 4 K but slightly more bend ($\text{N–C–N} = 173.2(9)^\circ$) at 293 K which stands for C_{2v} symmetry but is still very close to $D_{\infty h}$. All bond lengths and angles are apparently unaffected by changes in temperature.

Table 1. Structural parameters and conventional reliability factors for CuNCN at temperatures of 4 K and 293 K obtained from the Rietveld refinement of the neutron powder diffraction data.

	$T = 4\text{ K}$	$T = 293\text{ K}$
Space group	<i>Cmcm</i>	<i>Cmcm</i>
a (Å)	2.9916(5)	2.9915(5)
b (Å)	6.1482(9)	6.191(1)
c (Å)	9.406(1)	9.411(1)
V (Å ³)	173.01(4)	174.27(5)
Cu on $4a$ (0, 0, 0)		
B_{iso} (Å ²)	0.58(7)	1.63(9)
N on $8b$ (0, y , z)		
y	0.3802(7)	0.3806(7)
z	0.3810(4)	0.3808(4)
B_{iso} (Å ²)	1.02(5)	1.79(6)
C on $4c$ (0, y , 1/4)		
y	0.3847(15)	0.3925(14)
B_{iso} (Å ²)	0.80(9)	1.4(1)
R_p , R_{wp} , R_{Bragg}	0.050, 0.068, 0.11	0.044, 0.063, 0.13

Fig. 1. Observed, calculated, and difference intensities for the neutron-diffraction Rietveld refinement of CuNCN at 4 K. The vertical markers show the positions of the Bragg reflections used to simulate the pattern and right above is the XPD refinement.



References

- [1] X. Liu, M. Wankeu, H. Lueken, R. Dronskowski, Z. Naturforsch. **60b**, 593-596 (2005).



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-05-010		
Experiment title:	Extinction measurements on high purity copper		
Dates of experiment:	15.08.05-28.08.05 02.09.05-03.09.05 05.10.05	Dates of experiment:	14.12.2005
Experimental team: Names	Addresses:		
Jesús Palacios G.	Escuela Superior de Física y Matemáticas Instituto Politécnico Nacional Edif.. 9 de la UP ALM Av. Instituto Politécnico Nacional S/N 07739 México City		
Local Contact:	Dr. Jens Walter, Ekkehard Jansen, Rolf Skowronek		

Experimental report text body

Introduction

Primary and secondary extinction in X-ray and neutron diffraction of crystals has been widely investigated since it affects measurements intended to determine crystal structures, including charge densities and thermal parameters. Extinction is present also in diffraction of polycrystals, especially when annealed and with high texture intensity. However, there have been only a few systematic studies aimed to determine the extent of its influence. For the quantitative texture analysis this is important. Thus, the aim of this investigation is to quantify the influence of this phenomenon for the special case of pure cold rolled and also recrystallized copper samples. Here the results of 96% cold rolled and annealed high purity copper samples are reported.

Measurements and Results

In the first sample, due to its deformed condition, coherent domains are small and do not contribute to primary extinction and thus we expect mainly secondary extinction. In the annealed sample primary extinction could also be present.

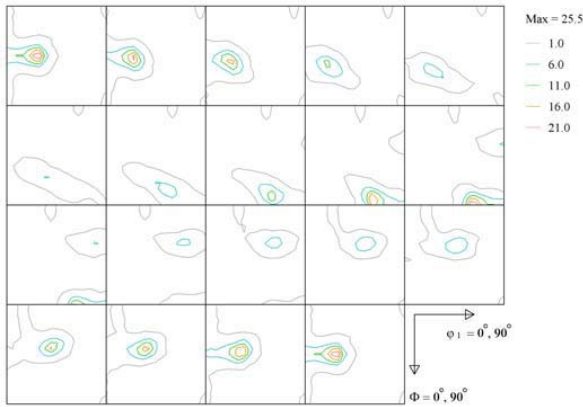
As well known, primary and secondary extinction affect strong reflections more strongly, and so, a difference should be observed when the Orientation Distribution Function (ODF) obtained through strong reflections ((111), (200) and (311)) and that obtained from weaker reflections ((220), (222) and (331)) are compared. Extinction also depends on wavelength and here it is also intended to search if this effect is also observable. Pole figures (111), (200), (220), (222), (311), (331), (400), (420), (422), (440), (531) and (620) were measured with wavelength $\lambda_1 = 2.332 \text{ \AA}$, $\lambda_2 = 1.166 \text{ \AA}$ and $\lambda_3 = 0.777 \text{ \AA}$. Unfortunately intensities for the latter were small and statistically not relevant for high Miller index reflections, i.e. from reflection (331) on. Partial or total peak overlapping also prevented the use of large Miller index reflections with λ_2 for ODF evaluation. With the first wavelength only the first two reflections could be measured. Overlapping of reflections could be partially

avoided by the use of a graphite filter.

According to the value of the position angle Ω of the Eulerian Cradle, a given reflection angle 2θ can appear at a lower or at a higher angle than Ω , or coincide with it. A comparison of these equivalent ODFs obtained by pole figures measured with λ_2 from reflections (111), (200) and (311), allow a consistency test and a measure of the accuracy of our measurements. In our case this was of the order of 5%. A comparison of these ODFs with the one obtained with λ_3 for the deformed sample gave a difference of 6%, which is actually within the measuring error range. An ODF from high Miller indexes however could not be obtained for the cold rolled sample, due to the low available intensities. The annealed sample shows a strong cubic texture, and when measured with λ_3 , a 30% increment of pole density could be observed between the ODFs determined from low intensity reflections and strong reflections. Such a large increase in pole density is not likely to be caused by secondary extinction alone. Also an increase of 11% could be observed when compared with corresponding λ_2 values. Fig. 1 shows the ODF measured with λ_2 when each used reflection coincided with Ω . Figs. 2 show the ODFs with λ_3 for the annealed sample, from strong reflections (Fig. 2a), and from weak reflections (Fig. 2b). Fig.3 shows the ODFs with λ_2 for the annealed sample, from strong reflections.

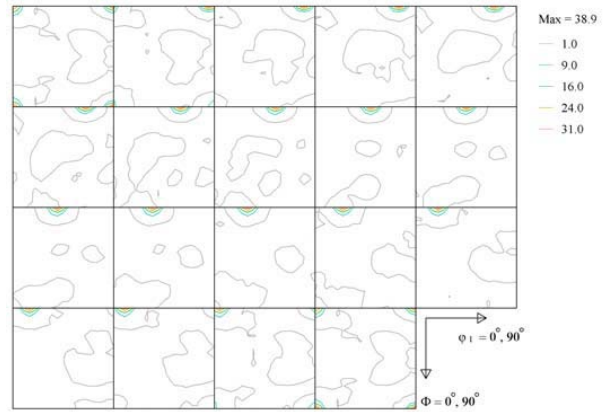
Although further evaluations are needed, it can be concluded that:

- For low or moderate texture, wavelength dependence lies within error range. For strong texture, it amounts to 11%.
- Due to the large pole density values obtained from weak reflections, as compared with those obtained from strong reflections, primary, as well as secondary extinction are present in the strong reflections.



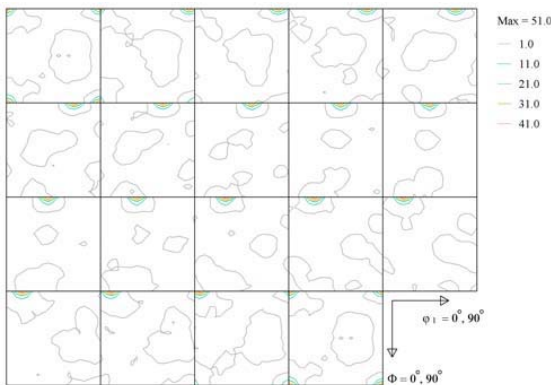
2EQ

Fig. 1. ODF of 96% cold rolled sample obtained with λ_2 from reflections (111), (200) and (311).



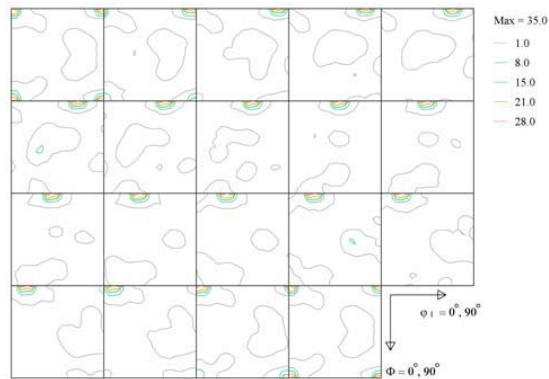
3NE_AN

Fig.2 a) ODF of annealed sample obtained with λ_3 from strong reflections



3NE_HI_AN

Fig. 2 b). ODF of annealed sample obtained with λ_3 from weak reflections



2EQ_AN

Fig. 3. ODF of annealed sample obtained with λ_2 from strong reflections



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-05-011		
Experiment title:	Neutron diffraction study of the NaNbO₃ submicron powder		
Dates of experiment:	29.08.05-02.09.05 27.09.05-06.10.05	Date of report:	29.06.2006
Experimental team: Names	Addresses		
Pithan, Christian Shiratori, Yosuke Magrez, Arnould	IFF, Forschungszentrum Jülich Laboratory for Nanostructures and Novel Electronic Materials Institute of Physics and Complex Matter École Polytechnique de Lausanne		
Local Contact:	Dr. J. M. Walter		

Experimental report text body

As indicated in the original proposal, particle size-induced phase transitions in the (K_xNa_{1-x})NbO₃ powders have to be clarified. So far size-induced phase transitions in (K_{0.5}Na_{0.5})NbO₃ and NaNbO₃ powders have been reported, respectively [1,2].

1. Temperature tuning neutron diffraction study for (K_{0.5}Na_{0.5})NbO₃

(K_{0.5}Na_{0.5})NbO₃ transforms from a monoclinic symmetry (M type structure) towards a triclinic one (T type structure) by reducing the particle size below a critical size of 100nm. This feature is surprisingly accompanied by an expansion of the lattice. From XRPD refinements, several space groups are possible for both symmetries (P2 or Pm for M type and P1 or P-1 for T type) when refining the crystal structure. Raman spectroscopy and nuclear site group analysis of the Raman active normal modes supported P1 as possible space group for fine powders while this method was not sufficient to extract the true space group for the coarse powder, M type structure [1]. As reported in the FRJ-2 Experimental Reports 2004 [3], refinements of the neutron diffractions concluded Pm and P1 to be the more suitable space groups for the M and T type structures, respectively.

Results: Neutron diffraction patterns, which have been recorded for both types of powders at various temperatures (-150 – 800 °C), were analyzed. We could see successive structural phase transitions for both powders. For the coarse powder (particle size > 200 nm), a drastic spectral change corresponding to the transition from monoclinic to cubic phases was confirmed at around 450 °C. On the other hand, phase transition behavior for the fine powder (particle size < 200) was more diffused.

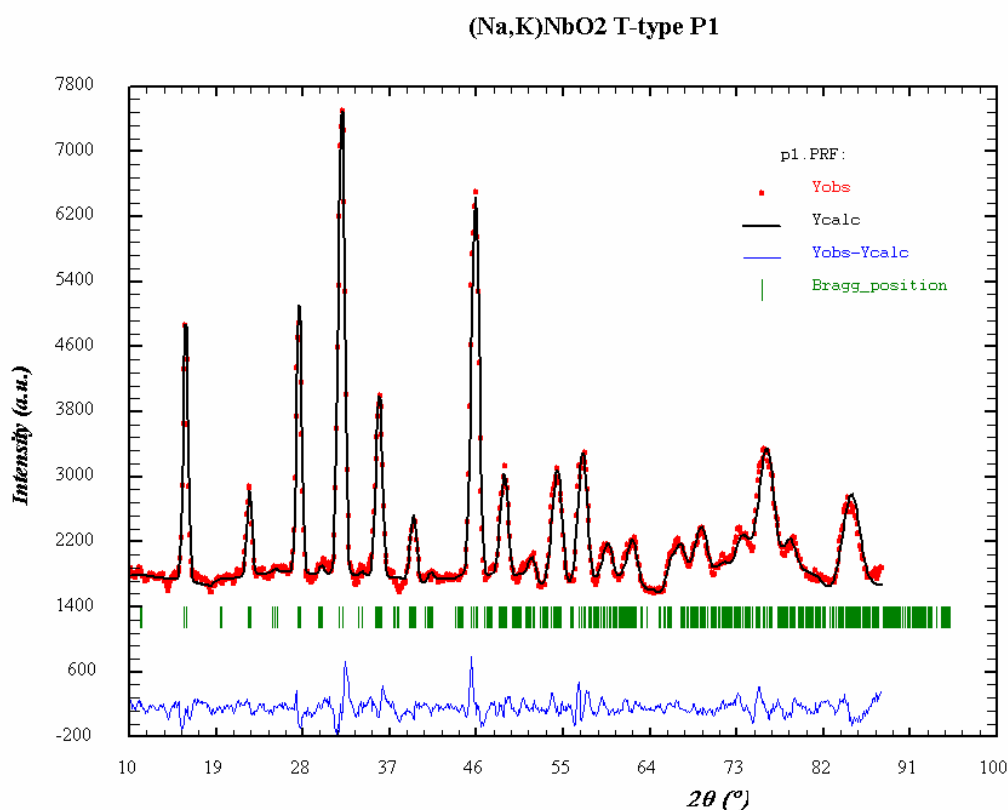
2. Neutron diffraction study for the NaNbO₃ submicron powder at various temperatures

NaNbO₃ showed a unique phase transition behavior [2]. Coarse powders take an orthorhombic Pbcm structure. At an averaged particle size of around 600 nm, the space group transforms to Pmc2₁, which shows an enhanced

piezoelectric property, accompanied by lattice expansion. In the intermediate particle size range (200 – 400 nm), the $Pmc2_1$ symmetry still holds but the lattice volume decreases with decreasing particle size. Finally, fine powders consisting of particles with an average diameter of 70 nm present a structure described in a $Pmma$ space group, which has a centrosymmetric structure. This structure assigned for the fine powders has the most compact reduced cell.

Recent temperature tuning Raman spectroscopy revealed that the piezoelectric submicron powder undergoes successive phase transitions toward a more symmetric phase on heating from -150 °C up to 450 °C and takes at least four polymorphs within this range. Transition points seem to be about -100, 120 and 340 °C from the overview of Raman spectra and these phases are new. In order to elucidate the crystallographic structures of these different phases, neutron diffraction measurements at various temperatures (-150, -50, 20, 50, 100, 150, 200, 300 and 400 °C) and their refinements are necessary. We believe that the mechanisms of the successive phase transitions and the Curie temperature for the piezoelectric phase can be revealed by the combination of temperature tuning Raman spectroscopy and neutron diffraction.

Results: Neutron diffraction patterns for the submicron NaNbO_3 powder indicated that at least three types of phases exist within the measuring temperature region.



References

- [1] Y. Shiratori, A. Magrez, C. Pithan, "Phase transformation of KNaNb_2O_6 induced by size effect", Chem. Phys. Lett. 391 (2004) 288.
- [2] Y. Shiratori, A. Magrez, J. Dornseiffer, F.-H. Haegel, C. Pithan, R. Waser, "Polymorphism in micro-, submicro- and nano-crystalline NaNbO_3 ", J. Phys. Chem. B (2005) in press.
- [3] R. Skowronek, J. Walter, C. Pithan, Y. Shiratori, A. Magrez, "Neutron diffraction study of nanocrystalline and microcrystalline $(\text{K}_{0.5}\text{Na}_{0.5})\text{NbO}_3$ powders", in The FRJ-2 Experimental Reports 2004, ed. T. Brückel, D. Richter, R. Zorn, Forschungszentrum Jülich GmbH, p. 33.
- [4] C. Pithan, Y. Shiratori, J. Dornseiffer, F.-H. Haegel, A. Magrez, R. Waser, "Microemulsion mediated synthesis of nanocrystalline $(\text{K}_x\text{Na}_{1-x})\text{NbO}_3$ powders", J. Cryst. Growth. 280 (2005) 191.



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

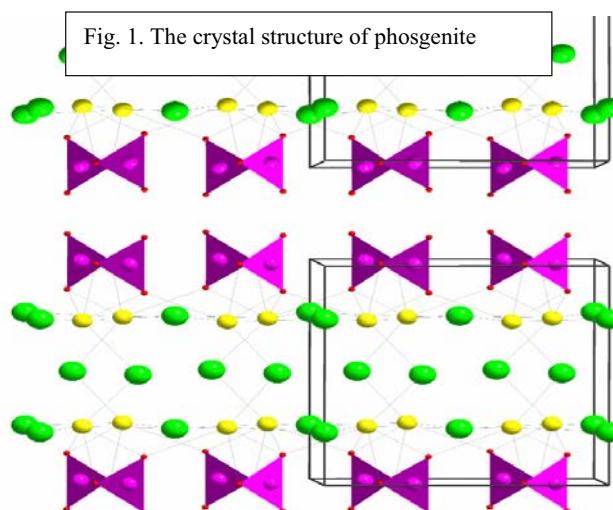
Proposal number:	SV7-05-012		
Experiment title:	High-temperature/Low-temperature structural behavior of phosgenite, $\text{Pb}_2\text{CO}_3\text{Cl}_2$		
Dates of experiment:	09.12.05-12.12.05 18.02.06-26.02.06 06.03-06-12.03.06	Date of report:	10.05.2006
Experimental team: Names	Addresses		
Gatta, G. Diego	Dipartimento di Scienze della Terra Università degli Studi di Milano I-20133 Milano Italy		
Local Contact:	Dr. J. Walter, R. Skowronek		

Experimental report text body

Phosgenite is a rare mineral consisting of lead chlorocarbonate $[\text{Pb}_2\text{Cl}_2(\text{CO}_3)]$. It forms from the oxidation of lead bearing minerals, such as when galena (PbS) comes in contact with carbonated and chlorinated waters. Usually natural phosgenite is found associated with cerussite (PbCO_3), anglesite (PbSO_4), galena and limonite ($\text{Fe}_2\text{O}_3 \cdot n\text{H}_2\text{O}$). The crystal structure of phosgenite was first refined by Gruseppetti and Tadini (1974). The structure (unit-cell: $a = 8.1600 \text{ \AA}$, $c = 8.8830 \text{ \AA}$, $Z=4$; space group: $P4/mbm$) consists of continuous layers parallel to the c -axis where large Pb-polyhedra lie, which are bridged by CO_3^{2-} units, see Fig. 1.

The structural arrangement in parallel planes is in fact reflected by the good cleavage parallel to (001).

The coordination number of the Pb-site in the crystal structure of phosgenite is 7:3 chlorines and 4 oxygens belonging to the CO_3 groups (Fig. 1). The interest in the stabilities of carbonate minerals within the Earth, considered as a buffer for the long-term cycling of CO_2 between the atmosphere, oceans and the solid Earth, gave rise to a relevant number of high-pressure and high-temperature experiments on natural C-bearing phases as calcite (low pressure- CaCO_3), aragonite (high pressure- CaCO_3), dolomite $[\text{MgCa}(\text{CO}_3)_2]$, strontianite (SrCO_3), witherite (BaCO_3), otavite (CdCO_3),



rhodochrosite (MnCO_3) and cerussite (PbCO_3) (Redfern, 2000). However, a few studies have been devoted to Cl, F and OH-bearing carbonates. In particular, no study was devoted to the low and high temperatures and high pressure behaviour of phosgenite and, as a consequence, the main thermodynamic parameters of this mineral are still completely unknown.

In this study, we proposed to investigate the high/low-temperature crystal chemistry of a natural phosgenite using neutron diffraction. Neutron experiment was required because of the crystal chemistry of this material consists of heavy element (*i.e.* Pb) and light element (*i.e.* C) which makes any high pressure X-ray powder diffraction data not useful for reliable structural refinements.

We performed a data collection at room condition (295K), at low-temperature (down to 4K) and at high-temperature (up to 473K) by neutron powder diffraction. The structural refinement of phosgenite at room condition confirms the topological asset described by Gruseppetti and Tadini (1974) with a tetragonal lattice. In Fig. 2 is reported the Rietveld full-profile fit. However, at between 295 and 433K the structure appear undergoes to a phase transition and the new high-temperature phase appear to be better described with a triclinic lattice with : $a=8.305389 \text{ \AA}$, $b= 8.016596 \text{ \AA}$, $c= 9.941740 \text{ \AA}$, $\alpha =83.036^\circ$, $\beta=106.055^\circ$, $\gamma=91.315^\circ$ and $V=631.381 \text{ \AA}^3$. In Fig. 3 is shown the LeBail full-profile fit of the HT-phosgenite at 433K. The work is in progress to refine the crystal structure of the triclinic HT-phase.

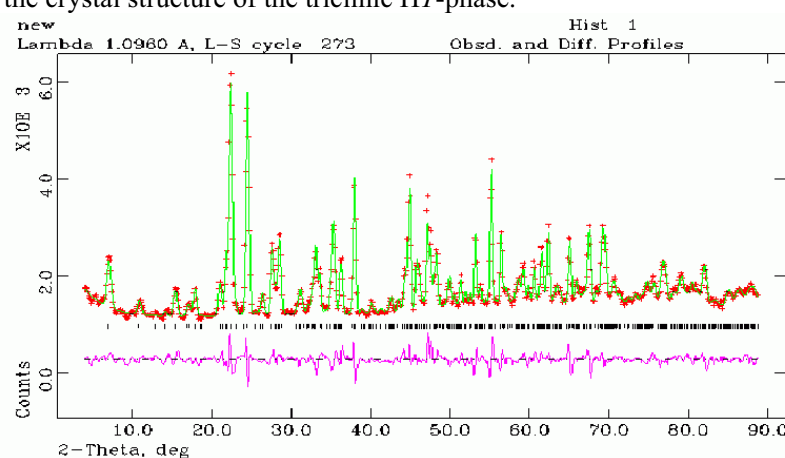


Fig. 2 Rietveld full-profile fit of phosgenite at 295K

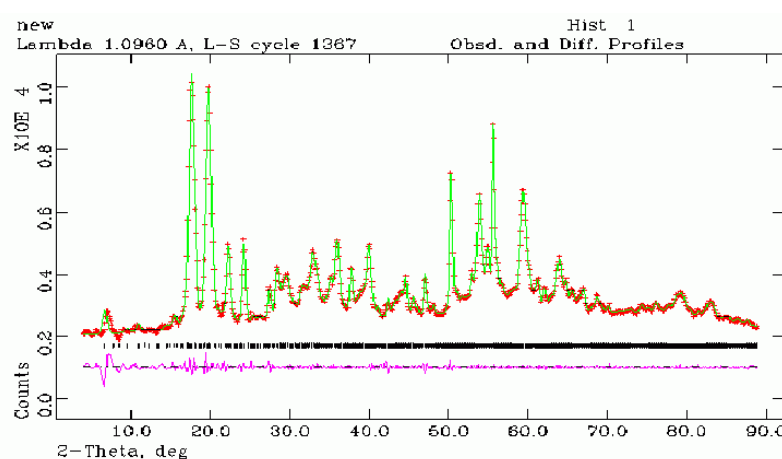


Fig. 3 LeBail full-profile fit of phosgenite at 433K

References

- Gruseppetti, G. & Tadini, C. (1974). *Tshermarks Min. Petr. Mitt.*, **21**, 101-109.
Redfern, S.A.T. (2000). *Rev. In Min. Geoch.*, **41**, 289-308.



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-05-013		
Experiment title:	Neutron Diffraction on Ni-Carbodiimides		
Dates of experiment:	25.10.05-01.11.05	Date of report:	28.6.2006
Experimental team: Names	Addresses		
Dr. Müller, Paul Krott, Manuel Prof. Dr. R. Dronskowski, Dr. Liu, Xiaohui	Institut für Anorganische Chemie der RWTH Aachen Landoltweg 1, 52056 Aachen		
Local Contact:	Dr. Jens M. Walter		

Experimental report text body

Ni(HNCN)₂, is a fine green, air stable powder. X- Ray diffraction experiments on powdered samples led to space group *Pnnm*, crystallographic data are given in tables 1 and 2.

Table 1. Crystallographic data for Ni(HNCN)₂.

Formula:	Ni(HNCN) ₂		
Crystal system, color and form:	Orthorhombic, green powder		
Lattice parameters:	$a = 6.468 \text{ \AA}$ $b = 8.768 \text{ \AA}$ $c = 3.236 \text{ \AA}$		

Table 2. Positional parameters parameter for Ni(HNCN)₂.

atom	Wyckoff-site	<i>x</i>	<i>y</i>	<i>z</i>
Ni	2 <i>a</i>	0	0	0
N1	4 <i>g</i>	0.708	0.48	0
C	4 <i>g</i>	0.185	0.639	0
N2	4 <i>g</i>	0.087	0.768	0

Although our earlier experiments on $\text{Cu}_4(\text{NCN})_2\text{NH}_3$ had shown, that it is possible to determine the H-positions with a powder sample without the use of deuterated samples, we now realized that the use of non-deuterated compounds results in a rather poor powder pattern (cf. fig.1) with the typical background of a hydrogen- containing sample.

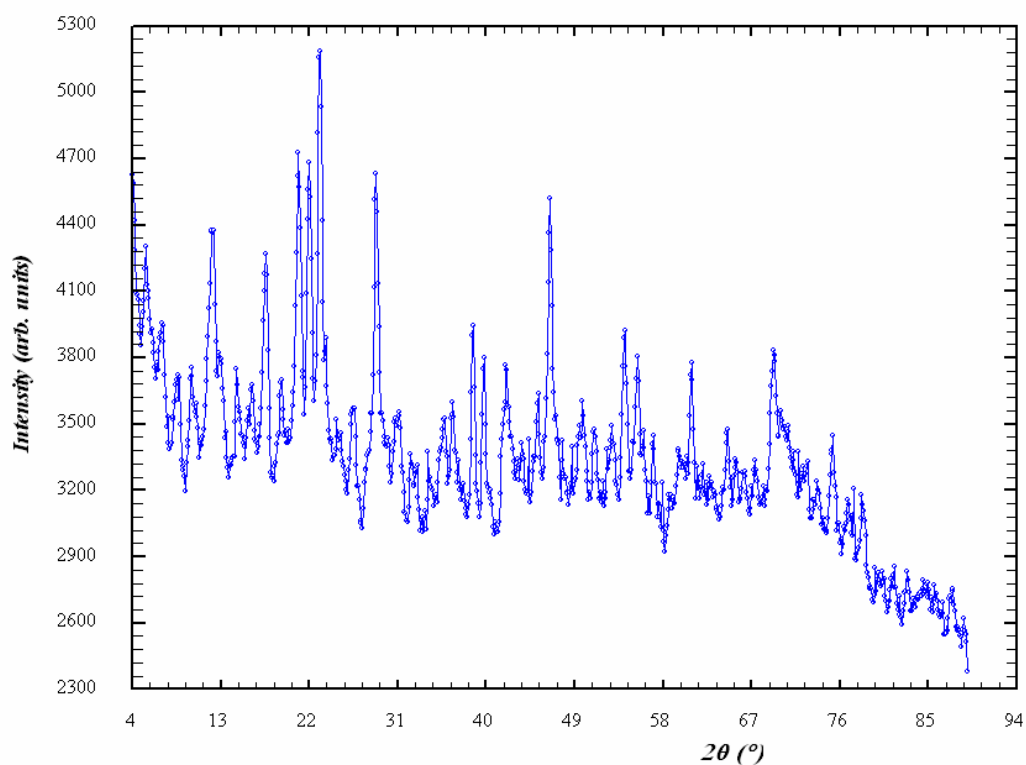


Fig. 1 Neutron powder diagram of $\text{Ni}(\text{HNCN})_2$ at 298K

The reflections of the powder pattern can be indexed by the orthorhombic cell, but the intensities are too poor to allow the determination of the hydrogen position with profile refinement calculations. Therefore we started additional experiments to grow single crystals for X-ray diffraction experiments to get more information about the crystal structure.



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-05-14		
Experiment title:	Neutron diffraction analysis of silver coins from the Himyarite-Sabean Empire		
Dates of experiment:	51 days	Date of report:	17.05.2006
Experimental team: Names	Addresses		
Kirfel, Armin, Kockelmann, Winfried Yule, Paul	Mineralogisch-Petrologisches Institut der Universität Bonn Poppelsdorfer Schloss 53115 Bonn Rutherford Appleton Laboratory, ISIS Facility, ROTAX, Chilton, Didcot, Oxon, OX11 0QX United Kingdom Völkerkundemuseum München		
Local Contact:	Dr. Jens M. Walter, Rolf Skowronek		

Experimental report text body

1. Introduction

This investigation is a follow-up project from the proposal SV7-05-004, where silver coins from the Himyarite and later Himyarite-Sabean empire in the southern part of the Arabic peninsula were investigated. In these first measurements 10 coins have been measured by neutron diffraction to determine the phase compositions and to correlate these with density measurements. Due to the auspicious results from the first measurements we measured another set of 40 coins with the same aim as in the last campaign. The peculiar shape of the coins, i.e. their irregular and concave (scaphate) appearance, implies that their patterns were not prepared only by casting in a mould, but rather by striking or deep-drawing with mechanical force. However, other coins are flat and exhibit bubbles which indicate casting.

Measurements and results

The coins were selected to cover a broader range over time and locations to determine their variation in composition in more detail. Furthermore selected coins were also measured in the texture diffractometer SV7 to enlighten the manufacturing techniques further more. For these measurements flat and concave coins were selected to gain information about the technique used to obtain concave coins as describe above. Another question addressed by the selection of sample coins for texture analysis was, whether the manufacturing technique had changed over time and may it possible to identify counterfeited coins in the variation of manufacturing techniques in the same period of time.

Due to the form anisotropy of the coins, it was not possible to measure complete pole figures of the coins, which would in this case also show form texture. Therefore only distinct ϕ -rotations in certain χ -tilts were selected for the measurements. From the resulting cascading diffraction patterns as a function of the orientation,

it is possible to obtain qualitatively by the comparison of the different peak heights, whether the coin was casted into a mould from molten material or worked by mechanical force. Casted coins show only distinct singular peak heights of single crystallites (see Fig. 1, 3), whereas mechanically worked coins show a classical bulk texture distribution, which varies in a wavy distribution depending on the measured angle (see Fig. 2).

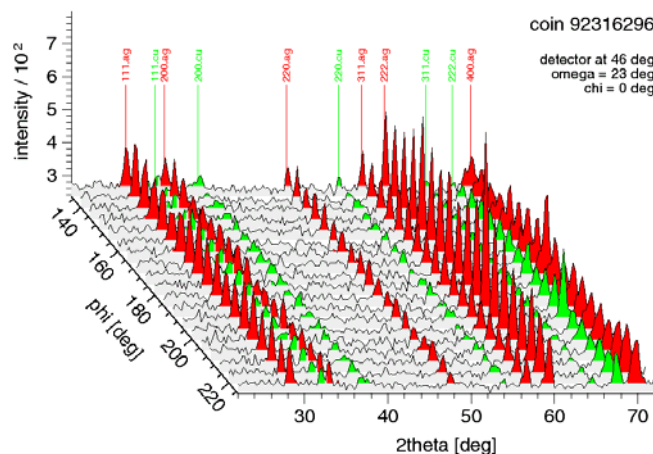


Fig. 1: Casted hymeriade silver coin with single crystal peaks

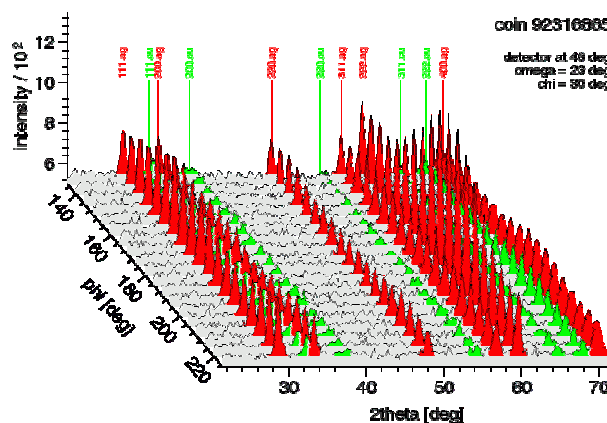


Fig. 2: mechanically worked flat silver coin

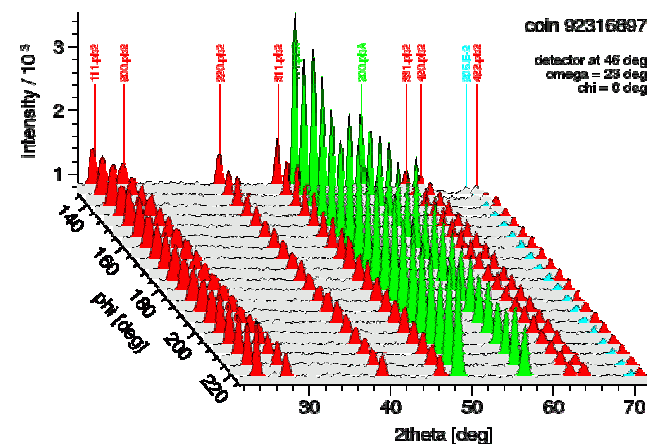


Fig. 3: Casted flat silver coin with single crystal peaks

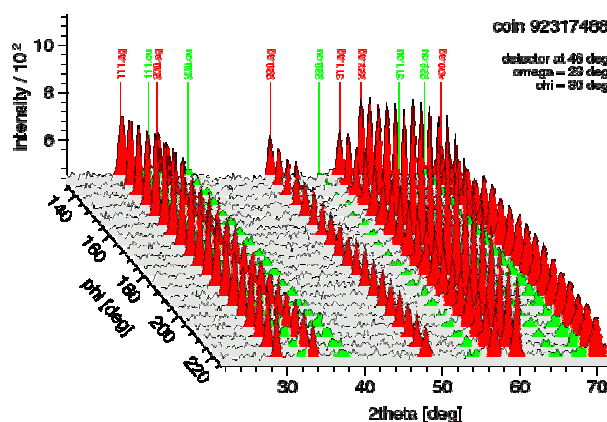


Fig. 4: mechanically worked concave silver coin



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-05-015		
Experiment title:	Low-and high field magnetic and mineral fabrics of carrara marbles		
Dates of experiment:	29.11.05-18.12.05 14.04.06 27.04.06-28.04.06	Date of report:	18.05.2006
Experimental team: Names	Addresses		
Otto, Michael Leiss, Bernd	Geowissenschaftliches Zentrum der Universität Göttingen Abteilung Strukturgeologie und Geodynamik Goldschmidt Str. 3 D-37077 Göttingen		
Local Contact:	Jens Walter, Ekkehard Jansen		

Experimental report text body

Introduction

The anisotropy of magnetic susceptibility (AMS) is a time-efficient method to describe crystallographic preferred orientations of rocks and has been applied in a wide field of sedimentary, metamorphic and magmatic geology. The method, however, suffers from limitations which mainly result from the interference of diamagnetic, paramagnetic and ferromagnetic fabrics (de Wall 2005) — the term ferromagnetism is used in a wider sense here, including e.g. ferrimagnetism. The AMS is an integral parameter which describes a crystallographic preferred orientation as an ellipsoid. The quantitative correlation of the AMS with the crystallographic preferred orientations should help to allow a closer view at the applicability and the limitations of the AMS analysis (see also Schmidt et al. 2006 a, b).

Measurements

Schmidt et al. (2005, 2006 a,b) developed a method for the separation of paramagnetic from diamagnetic fabric. It is achieved by the comparison of room-temperature and low-temperature (77 K) measurements using the high-field torque method. This method was applied and compared to neutron textures on synthetically-deformed calcite-mica samples. To test the application of the new methods on naturally deformed rocks, mica-bearing calcite marbles and mylonites from the Alpi Apuane in Italy and dolomite mylonites from the Damara Orogen in Namibia were selected. Selection criteria were varying mica contents and varying intensities/types of their crystallographic-preferred orientations (CPOs). Quantitative texture analyses were carried out by means of the ‘powder and texture diffractometer SV7’ at the research reactor Jülich 2 of the Research Center Jülich (FRJ-2) in Germany. Due to a low absorption coefficient of neutrons in condensed matter, neutron diffraction allows a volume related quantitative texture analysis of the sample cylinders which were also used for AMS measurements. Only this strategy allows a direct correlation of CPOs with the AMS. The 30 samples analysed cover a large variety of texture types. The carbonate phases show single c-axis maxima (Fig.1), covering a

range from weak to very strong intensity maxima. Other samples show distinct to weak c-axis double maxima (Fig. 2). Furthermore, some samples show partially developed girdle distributions with moderate to weak intensity. In one case, dolomite displays a completely developed girdle distribution. The mica phases show c-axis preferred orientations covering a range of very weak to very strong single maxima.

Results

The results of this study are based on a large variety of fabric types of carbonate-mica marbles and mylonites, i.e. varying mica content, grain sizes, grain shapes, types and intensities of the crystallographic preferred orientation. The presented first correlations of the AMS and CPO for the single mineral phases in general demonstrate a good matching. Regarding the comparison of texture types and the AMS, limitations are possible. While single c-axis maxima and girdle-like c-axis distributions can be also distinguished by the AMS, it is obvious that distinguishing between these types and the double c-axis type is not possible at the present stage.

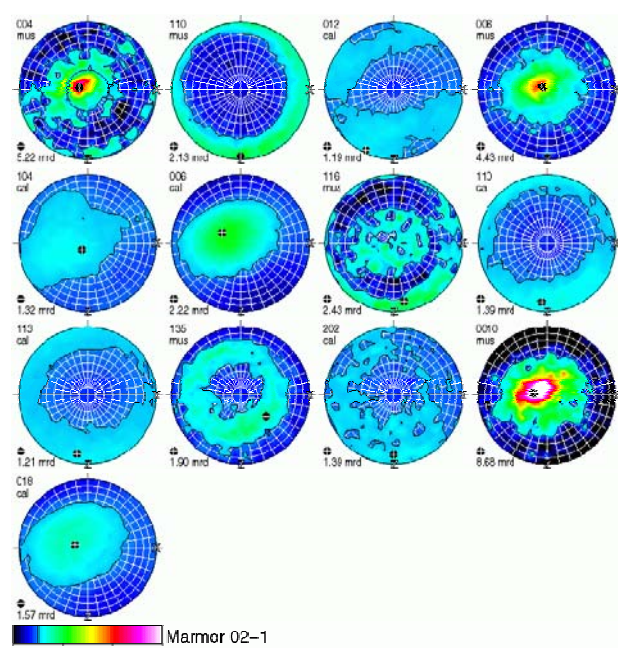


Fig. 1 Single c-axis maxima of calcite and muscovite

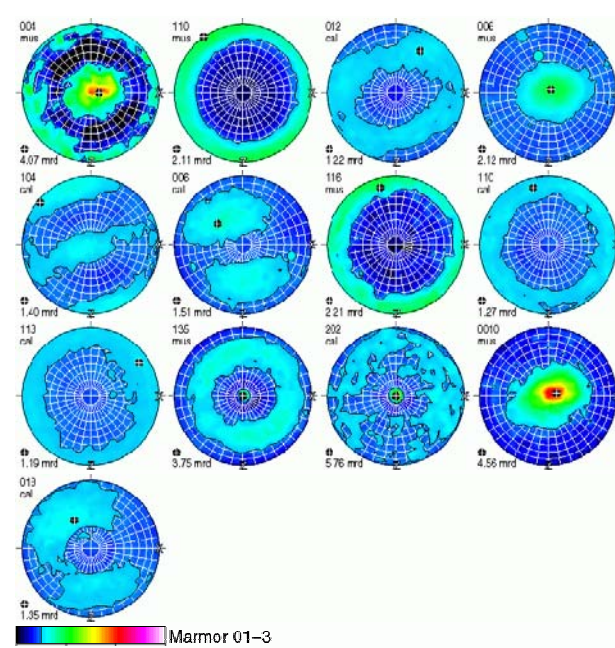


Fig. 2 Double calcite c-axis maximum and single mica c-axis maximum

References

- De Wall, H (2005) Die Anisotropie der magnetischen Suszeptibilität — eine Methode zur Gefügeanalyse. Z. d. dt. Geol. Ges. 155, 287–298
- Martin-Hernandez F & Hirt AM (2001) Separation of ferrimagnetic and paramagnetic anisotropies using a high-field torsion magnetometer. Tectonophysics 337, 209–221
- Hrouda F (1982) Magnetic-anisotropy of rocks and its application in geology and geophysics. Geophysical Surveys 5, 37–82
- Lowrie W (1989) Magnetic Analysis of Rock Fabric. In: James, DE The Encyclopedia of Solid Earth Geophysics, 698–706
- Schmidt, V, Hirt, AM, Burlini, L & Leiss, B (2005) Crystallographic-Preferred Orientations and Anisotropic Magnetic Susceptibilities in Experimentally Produced Calcite Samples: Deformation Mechanics, Rheology and Tectonics 2005, Zürich, pp 190
- Schmidt V, Hirt AM & Rosselli P (2006a) Separation of magnetic subfabrics by high-field, low-temperature torsion measurements. 11. Symposium “Tektonik, Struktur- und Kristallingeologie”
- Schmidt V, Hirt AM, Burlini L, Leiss B & Walter JM (2006b) Measurement of calcite crystallographic preferred orientations by magnetic anisotropy and comparison to diffraction methods. 11. Symposium “Tektonik, Struktur- und Kristallingeologie”



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-05-016		
Experiment title:	The crystal structure of methyl bromide clathrate at T=4K		
Dates of experiment:	27.01.06-29.01.06	Date of report:	20.5.2006
Experimental team: Names	Addresses		
M. Prager R. Skowronek	Forschungszentrum Jülich Inst. F. Mineralogie, Universität Bonn		
Local Contact:	E. Jansen, J. Walter		

Experimental report text body

Clathrates are a fascinating class of materials: a framework of matrix molecules forms cages which allow incorporating simple molecules in identical environments which thus experience the same matrix effect. The combination of different host and guest molecules allows a huge variety of clathrates to be produced. Water clathrates with water soluble guest molecules like tetrahydrofuran can be prepared easily. A recently grown interest concentrates on the methane water clathrates [1] which are only stable in a certain pressure-temperature regime. This 'burnable ice' exists in nature and may form one of the largest energy reservoirs on earth.

From a fundamental point of view methane represents a prototype of a non polar guest molecule. Methyl halides are interesting and different probes of the clathrate cages compared to methane because it interacts with the host surface via dipolar interaction. We already studied the methyl iodide clathrate. To incorporate this non water-soluble molecule to form the clathrate $\text{CH}_3\text{I} \times 17\text{H}_2\text{O}$ a special preparation technique [2] was developed. The formation of the clathrate becomes evident by the transformation into a transition gel state and an increase of the melting point to $T_m \sim 4^\circ\text{C}$. The system crystallizes in the cubic II structure [3]. On this basis very detailed high resolution spectra gave much very important information [4]. Here we extend these studies to methyl bromide clathrate, which is reported to crystallize in the cubic I structure [5]. The prepared $\text{CH}_3\text{Br} \times 5.75\text{D}_2\text{O}$ had a melting point $T_m = 11^\circ\text{C}$.

A sample with deuterated matrix was prepared in an analogous way as methyl iodide clathrate. Neutron diffraction was applied using the single crystal side of SV7. The wavelength used was $\lambda = 2.332\text{\AA}$. According to literature $\text{CH}_3\text{Br} \times 5.75\text{H}_2\text{O}$ should be of the cubic I structure with a lattice parameter of $\sim 11.8\text{\AA}$. The sample was mounted cold on the precooled head of a CCR cryostat and cooled to the required temperatures. Fig. 1 shows a typical diffraction pattern. All observed Bragg peaks can be indexed within the cubic I structure. A lattice parameter of $a = 11.9045\text{\AA}$ is found at a sample temperature $T = 10\text{K}$ in agreement with the cubic I structure.

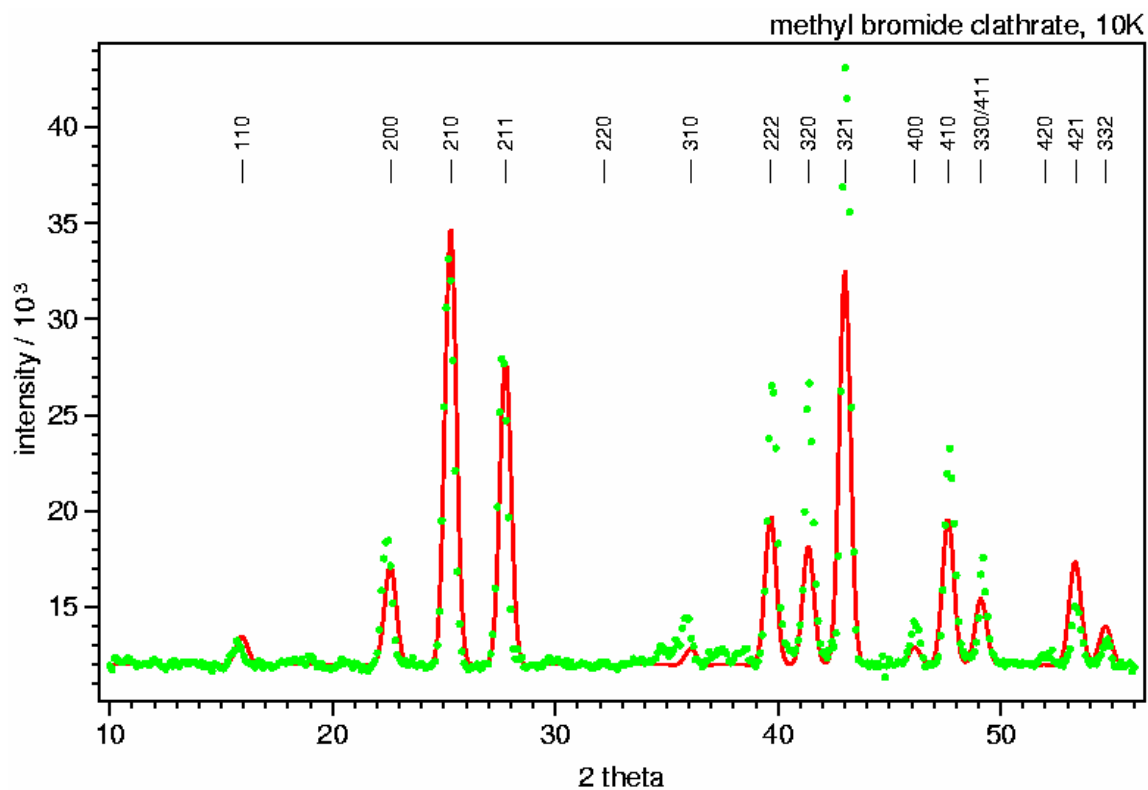


Fig. 1 Diffractogram of $\text{CH}_3\text{Br} \cdot 5.75\text{D}_2\text{O}$. Sample temperature $T=10\text{K}$. Miller indices characterize each peak.

- [1] C. Gutt, W. Press, A. Hüller, J. Tse, H. Casalta, J. Chem. Phys. **114**,4160(2001)
- [2] C. Albayrak, Dissertation, Aachen 1988
- [3] Experimental reports 2003 of FRJ2, report SV7-02-016
- [4] M. Prager, J. Pieper, A. Buchsteiner, A. Desmedt, J. Phys.: Condens. Matter **16**,7045(2004)
- [5] G.A. Jeffrey in „Inclusion compounds”. Vol. 1, p. 135ff, J.L. Atwood et al editors, Academic Press, London, 1984



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-06-001		
Experiment title:	Investigations on the magnetic structure of MnNCN		
Dates of experiment:	11.02.06-15.02.06	Date of report:	13.06.2006
Experimental team: Names	Addresses		
Dr. Müller, Paul Krott, Manuel Prof. Dr. R. Dronskowski, Dr. Liu, Xiaohui	Institut für Anorganische Chemie der RWTH Aachen Landoltweg 1, 52056 Aachen		
Local Contact:	Dr. Jens M. Walter		

Experimental report text body

MnNCN, the first carbodiimide of a magnetic transition metal, has been synthesised and characterized by X-Ray diffraction. Lattice constants are $a = b = 3.357(1)\text{\AA}$, $c = 14.339(2)\text{\AA}$ Spacegroup $R\bar{3}m$.

Table 1. Positional parameters and isotropic displacement parameters for MnNCN with standard deviations in parentheses.

Atom	Lage	x	y	z	$B (\text{\AA}^2)$
Mn	$3a$	0	0	0	0.1(1)
C	$3b$	0	0	$\frac{1}{2}$	1.5(7)
N	$6c$	0	0	0.589(3)	0.4(4)

Magnetic measurements show Curie-Weiss behaviour above 150 K and a Néel point about 28 K. We performed neutron diffraction at RT and below the transition point and could prove the existence of a magnetic ordering. Fig 1 shows the experimental results.

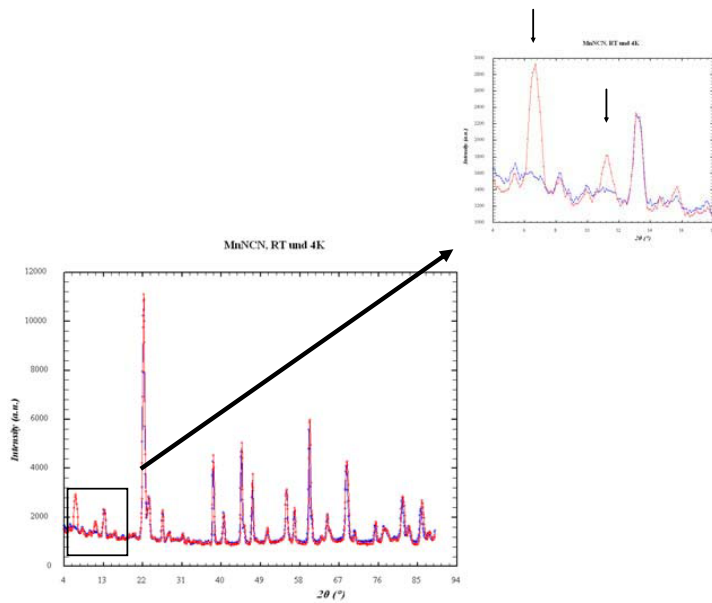


Fig. 1 Neutron diffraction diagrams of MnNCN at RT and 4K.

Due to the weak magnetic reflections we were not able up to now to present the complete magnetic structure. Fig 2 shows a profile fitting that results from a doubling of the c-axis and an orientation of the magnetic moments perpendicular to the c-axis. In consecutive manganese layers the spins are rotated by 60° in the a-b plane.

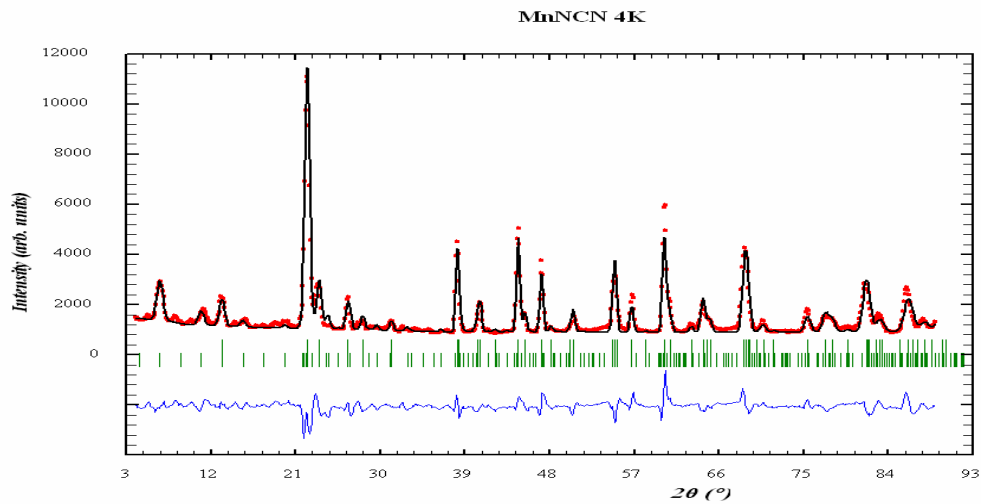


Fig. 2 Rietveld refinement of MnNCN at 4.2K



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-06-002		
Experiment title:	Textural development of quartzitic mylonites from the Pohorje ultrahigh-pressure unit (Slovenia)		
Dates of experiment:	06.01.06-13.01.06	Date of report:	14.06.2006
Experimental team: Names	Addresses		
N. Froitzheim J. Pleuger	Geologisches Institut der Universität Bonn, Nußallee 8, 53115 Bonn		
Local Contact:	J.M. Walter		

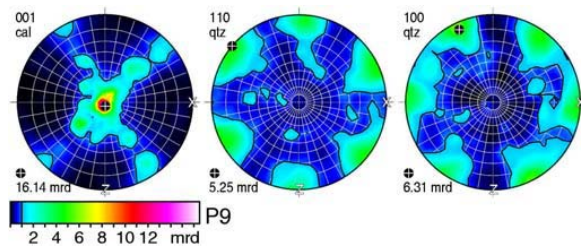
Experimental report text body

Two samples of mylonitised quartzite from the Pohorje mountains in northeastern Slovenia have been analysed in the present project. The Pohorje mountains are the southeasternmost extension of the Alps and expose nappes of the Lower and Upper Central Austroalpine units. The Lower Central Austroalpine complex experienced Cretaceous (c. 90 Ma) high-pressure metamorphism as testified e.g. by the well-known eclogites of Koralpe and Saualpe in southeastern Austria. In the Lower Central Austroalpine unit of Pohorje (Pohorje nappe), evidence for ultrahigh-pressure metamorphism, reaching conditions of c. 40 kbar and 900° C (Janák et al., 2006), has recently been discovered in metabasic and metaultrabasic rocks. The metabasic and metaultrabasic rocks are contained as lenses within medium- to high-grade micaschists, gneisses and amphibolites of the Pohorje nappe. Probably in the Early Miocene, a mostly granodioritic to tonalitic pluton intruded into the Pohorje nappe and is now exposed in central parts of the Pohorje mountains. In the Koralpe, most of the exhumation of high-pressure rocks occurred until the end of Cretaceous and was accompanied by strong deformation and retrograde metamorphic reequilibration in the surrounding rocks. In the Pohorje nappe, much deformation in the country rocks of the (ultra)high-pressure rocks probably also happened during the Upper Cretaceous. However, there is also evidence for east- to northeast-vergent extensional shearing during the Miocene which led to the final exhumation of the Pohorje nappe as a metamorphic core complex.

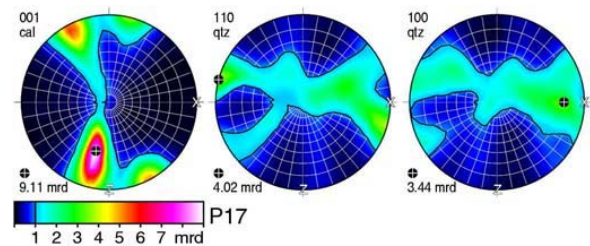
For the samples P9 and P17, the textures do not correspond to the macroscopically visible mineral lineations. Therefore, we rotated the textures around the strain Z axis, i.e. the normal to the mylonitic foliation, so that the strongest {a} maximum fell onto the periphery of the pole figure and the {c} maximum within the foliation plane parallels Y. These rotations are based on two assumptions. First, <a> was the only important slip direction in slip systems that acted during the formation of our textures. One of the {a} maxima should then align with the direction of shear, i.e. normal to Y and near to X. Second, in the case of initially randomly distributed crystallographic axes, homogeneous deformation should, irrespective of the flow type, produce

textures with twofold internal symmetry, either rotational with respect to Y (P9) or with the shear plane as a mirror plane (P17). The resulting texture of P9 yields an incomplete cross girdle distribution of {c} with a strong absolute maximum in the centre of the pole figure. Both {m} and {a} pole figures show three almost equally strong maxima distributed on a great circle parallel to the X-Z plane. The texture is only slightly asymmetric with respect to the strain axes, indicating only a weak component of rotational shear. The shear sense is dextral, i.e. top-to-the-SE (155°) in geographic coordinates. The P9 texture suggests intracrystalline deformation of quartz by dominant prism <a> slip. This slip system becomes dominant in quartz mylonites under temperatures above c. 500° C (Stipp et al., 2002), i.e. in the amphibolite facies. P17 shows two {c} maxima close to Z. The stronger and the weaker one are rotated by about 15° and 25° towards X in a clockwise and anticlockwise sense, respectively. {m} and {a} are unequally strong disposed each on two girdles perpendicular to the {c} maxima. Both {c} maxima and related {m} and {a} orientations would indicate opposite shear senses, which may be conjugate. The net shear sense is dextral (306° in geographic coordinates) as most obviously visible from the inclination of the main {m} and {a} girdles with respect to X. However, also in this case the rotational component of strain was quite weak. The position of the {c} maxima close to the periphery of the pole figure indicate basal <a> slip, possibly with some additional influence of rhomb <a> slip. These slip systems are active under lower temperatures than prism <a> slip, i.e. below 500° C.

In the present state of our study, more structural fieldwork has to be carried out to understand the kinematics of the Pohorje nappe and to interpret the textures in terms of this history. In a mapping project which will be carried out this summer, the large-scale structural history will be investigated and more samples for the comparison of textures with petrological P-T estimates will be collected. So far, we conclude that the shear senses deduced from P9 and P17 are not compatible with the reported kinematics of Miocene extension but rather with Cretaceous deformation. The fact that the shear senses of P9 and P17 are nearly opposite and that the textures formed under amphibolite- and greenschist-facies deformation, respectively, suggests a polyphase Cretaceous deformation history of the Pohorje nappe.



Texture of P9: mylonitic foliation is oriented S225/28, the reconstructed strain X direction is L155/10 (to the right).



Texture of P17: mylonitic foliation is oriented S345/44, the reconstructed strain X direction is L306/37 (to the right).

References:

- Janák, M., Froitzheim, N., Vrabec, M., Krogh Ravna, E.J. & de Hoog, J.C.M. (2006): Ultrahigh-pressure metamorphism and exhumation of garnet peridotite in Pohorje, Eastern Alps. - *Journal of metamorphic Geology* 24, 19-31.
- Stipp, M., Stünitz, H., Heilbronner, R. & Schmid, S.M. (2002): The eastern Tonale fault zone: a „natural laboratory“ for crystal plastic deformation of quartz over a temperature range from 250 to 700 °C. - *Journal of structural Geology* 24, 1861-1884.



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-06-003		
Experiment title:	Textural development of quartzitic mylonites of the western Rila Massif, western Bulgaria		
Dates of experiment:	13.01.06-15.01.06 17.02.06-18.02.06	Date of report:	14.06.2006
Experimental team: Names	Addresses		
N. Froitzheim J. Pleuger C. Tückmantel M. Neisen	Geologisches Institut der Universität Bonn, Nußallee 8, D-53115 Bonn		
Local Contact:	J.M. Walter		

Experimental report text body

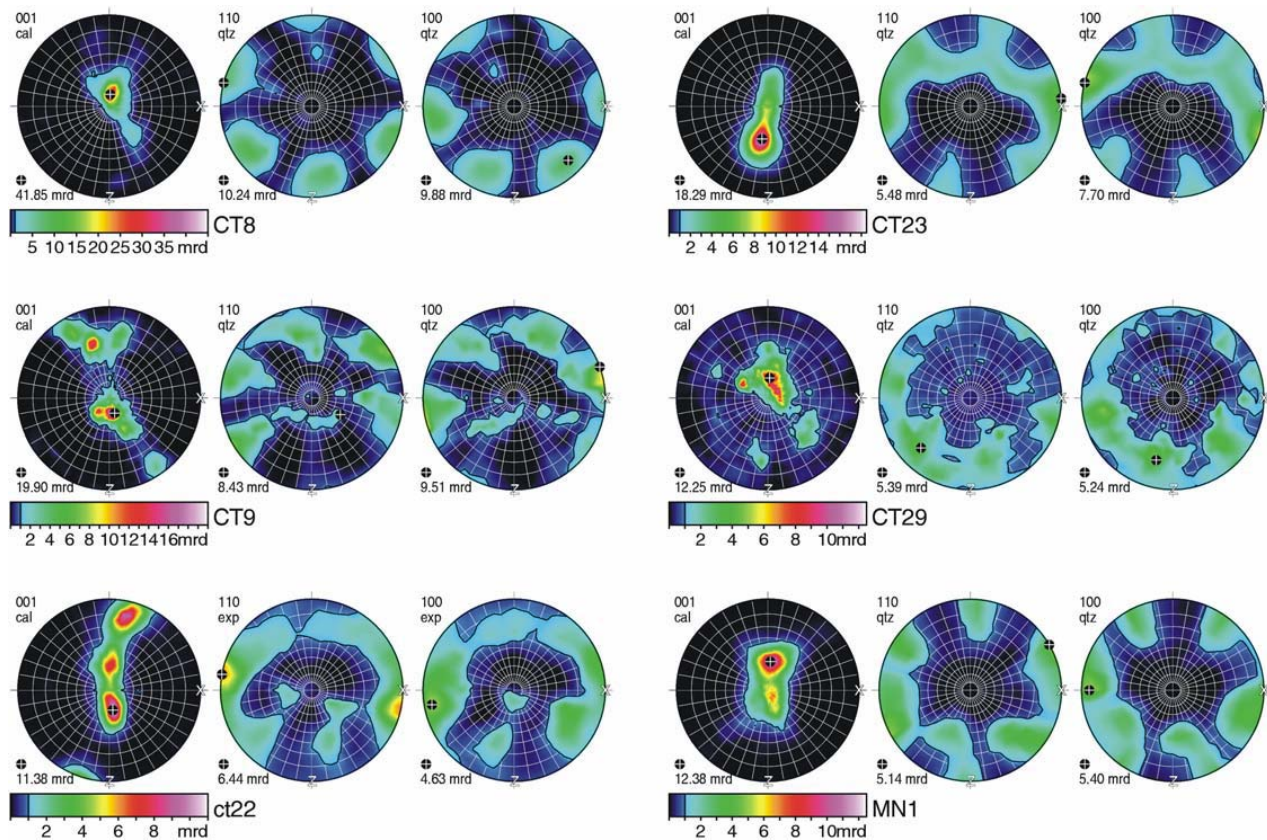
The Rhodope complex is part of the Alpine orogen and formed by metamorphic and magmatic rocks representing the deep levels of a collisional orogen. Structural and geochronological work has shown that the Rhodope complex is a stack of nappes, including Hercynian basement, assembled and metamorphosed during the Alpine orogeny. The metamorphic rocks include orthogneiss, paragneiss and schist, metabasic and ultrabasic rocks, calcsilicates, and marble. Recently, evidence for ultrahigh-pressure metamorphism was found in the Central and Eastern Rhodopes. The most widespread regional (Alpine) metamorphism is amphibolite-facies to locally granulite-facies with anatexis. Pelitic schists in the Rila Mountains give evidence that the amphibolite facies was reached during decompression. The complex is the site of voluminous Eocene to Early Miocene syn- and postcollisional magmatism and of postcollisional extension leading to the formation of detachment faults, metamorphic core complexes, and rift basins. Cenozoic sedimentary rocks of the western Rhodope complex occur in rift structures and are interfingering with volcanic rocks related to the Tertiary plutonism.

A most prominent extensional structure is the Gabrov Dol detachment fault that separates the greenschist-facies ophiolitic Frolosh unit in the hanging wall from the mostly amphibolite-facies and gneissic Ograzhden unit in the footwall. In the course of two diploma mapping works in the Rila Massif, six mylonitised quartzites (among many other specimens) have been sampled from three shear zones within the Ograzhden unit in order to gain information about strain geometry, temperature, and shear senses from their textures. This information shall contribute to a better understanding of the complex extensional deformation history and a possible thermal influence by syntectonic plutonism.

In three of the textures, CT8, CT29, and MN1, {c} forms a single maximum close to the strain Y axis which is parallel to the foliation and perpendicular to the stretching lineation. In these cases, {a} forms three maxima in the X-Z plane the strongest of which lies more or less close to X. These textures are evidence for prism <a> slip

which operates in the amphibolite facies above 500 °C. The other three textures also show influence of prism $\langle a \rangle$ slip in that the $\{c\}$ maximum extends through the centre of the diagram. In CT23, the position of the absolute $\{c\}$ maximum between the centre and the periphery of the pole figure probably indicates strong additional rhomb $\langle a \rangle$ slip. In CT9 and CT22, $\{c\}$ is distributed over incomplete single girdles. Such textures are commonly interpreted to be formed by the combined activity of basal $\langle a \rangle$, rhomb $\langle a \rangle$, and prism $\langle a \rangle$ slip under greenschist-facies conditions. The inclination of the single girdles with respect to the foliation plane (X-Y) indicates a strong rotational component of shear which is sinistral for CT9 and dextral for CT22. For CT9, the same shear sense is also displayed by the position of the strongest $\{m\}$ maximum c. 20° away from X on the periphery in an anticlockwise sense, and for CT22 by the strongest $\{a\}$ maximum c. 10° away from X in a clockwise direction.

In the further course of the study, the textures will be compared to grain-shape analyses of the same samples and petrological observations on other rock types. Our preliminary interpretations of the neutron textures are in line with field observations that in higher parts of the Ograzhden unit extensional faults with considerable displacement led to significant exhumation of the units below. The textures and petrological observations on thin sections independently show that faulting in the investigated shear zones took place partly under amphibolite-, partly under greenschist-facies conditions, while final stages of exhumation were accommodated by brittle faulting.





Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-06-004		
Experiment title:	Texture of hematite ore deformed to high strains in torsion		
Dates of experiment:	17 days	Dates of experiment:	26.06.2006
Experimental team:			
Names	Addresses		
H. Siemes * B. Klingenberg	Institut für Mineralogie und Lagerstättenlehre RWTH-Aachen, D-52056 Aachen		* Förderung DFG Si209/36-1
E. Rybacki M. Naumann	Geoforschungszentrum Potsdam Projektbereich 3.2: Rheology and Tectonophysics D-14474 Potsdam		
K. Kunze	Geologisches Institut, ETH-Zürich CH-8092 Zürich		
Local Contact:	J.M. Walter, E. Jansen, R. Skowronek		

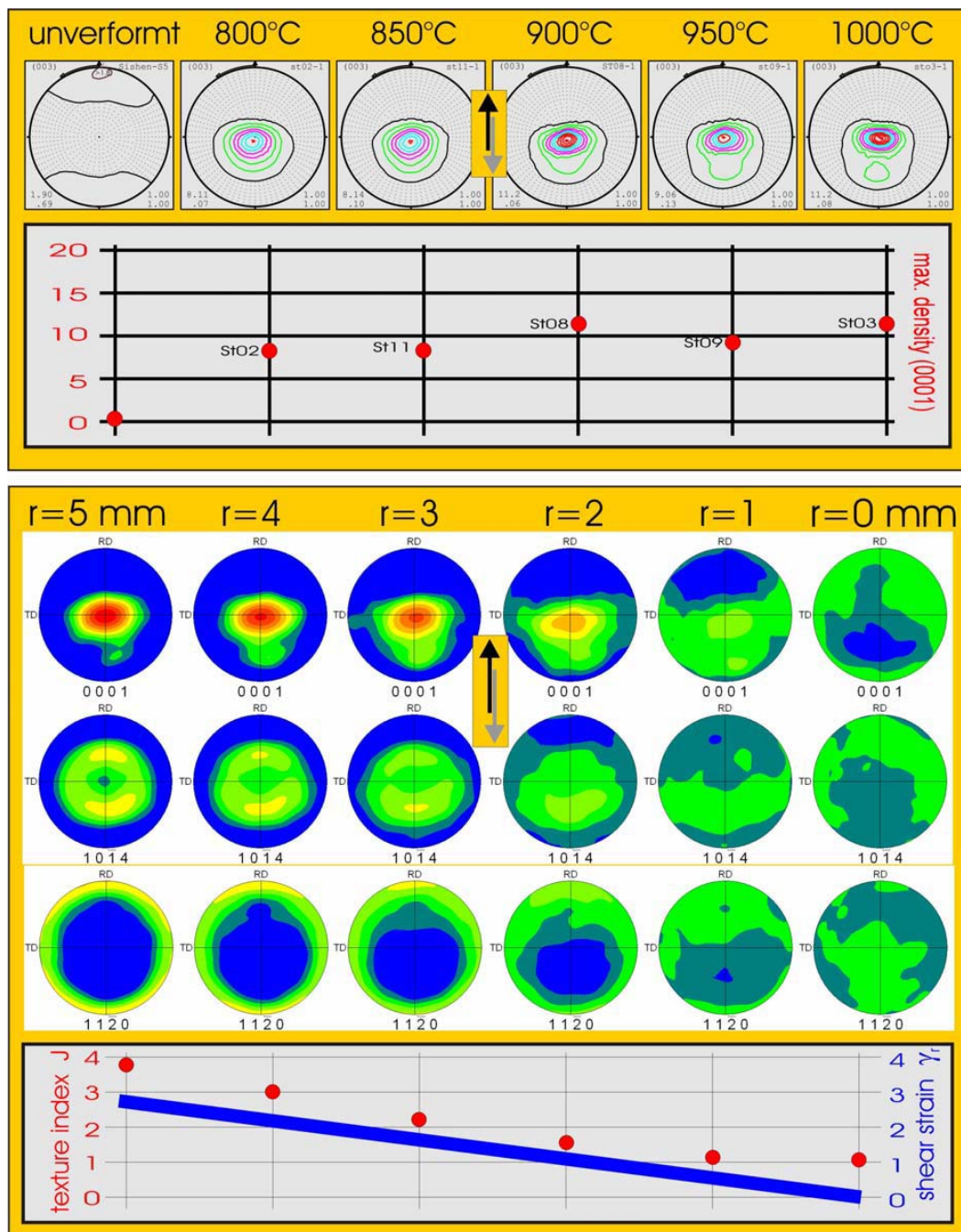
Experimental report text body

Dieser Bericht ist eine Ergänzung zu SV7-05-001

Verformungsversuche an feinkörnigem Hämatit (Fe_2O_3) wurden in einer Paterson-Apparatur mit Torsionseinrichtung (Paterson & Olgaard, 2000) bei Temperaturen zwischen 800 und 1000°C gefahren. Die Scherverformungen lagen bei allen Versuchen zwischen $\gamma=4,4$ und $\gamma=4,6$ und die dabei erreichten maximalen Festigkeiten zwischen 81 und 38 MPa. Der Hämatit ist bei allen Temperaturen zu einem gleichförmigen polygonalen Korngefüge rekristallisiert, das sich auch bei natürlich verformten Erzen findet. Die Korngrößen, die mit steigender Temperatur zunehmen, weisen dabei keine Unterschiede über die Probenquerschnitte auf, obwohl die finite Scherverformung von Null im Zentrum der Probe bis zum maximalen Wert an der Peripherie variiert. Neutronentexturmessungen (Jansen et al. 2000) im Probenbereich mit der stärksten Verformung und EBSD-Messungen (Kunze et. al. 1993) ergaben für die Polfigur des 0003-Reflexes ein zentrales, elliptisches Maximum nahe dem Pol der Scherebene. Die Höhe des 0003-Maximums nimmt dabei von 8,1 bis 11,2 [m.u.d.] (siehe obere Abbildung) mit steigender Temperatur zu. Der Texturindex bei der 1000°C-Probe steigt im Probenquerschnitt von 1,1 im Zentrum auf 3,8 an der Peripherie (siehe untere Abbildung) an. Auf dem peripheren Gürtel der $\{11\bar{2}0\}$ -Polfigur befindet sich ein Maximum, das die Orientierung der Scherrichtung angibt. Die $\{10\bar{1}4\}$ -Polfigur, die zwei bananenförmige Maxima (Quade, 1988) unterschiedlicher Dichte und Ausdehnung aufweist, lässt auf den Schersinn schließen. Diese Polfiguren stimmen weitgehend mit Polfiguren natürlich verformter Hämatiterze überein.

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Literatur

- Bascou, J., Raposo, M.I.B., Vauchez, A., Egydio-Silva, M., 2002. Earth and Planetary Science Letters 198, 77-92.
- Jansen, E., Schäfer, W., Kirfel, A., 2000. Journal of Structural Geology 22, 1559-1564.
- Kunze, K., Wright, S.I., Adams B.L. and Dingley D.J., 1993. Textures and Microstructures 20, 41-54.
- Paterson, M.S., Olgaard, D.L. 2000. Journal of Structural Geology 22, 1341-1358.
- Quade, H., 1988. Textures and Microstructures, 8-9, 719-736
- Quade, H., Rosière, C.A., Siemes, H., Brokmeier, H.-G., 2000. Zeitschrift für angew. Geologie, Sonderheft 1, 155-162.
- Rosière, C.A., Siemes, H., Quade, H., Brokmeier, H.-G., Jansen, E.M., 2001. J. Structural Geology 23, 1429-1440.
- Siemes, H., Klingenberg, B., Rybacki, E., Naumann, M., Schäfer, W., Jansen, E., Rosière, C.A., 2003. J. Struct. Geol. 25, 1371-1391.



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	SV7-06-005		
Experiment title:	The crystal structure of methyl chloride clathrate at T=200K		
Dates of experiment:	30.04.06-01.05.06	Date of report:	20.5.2006
Experimental team: Names	Addresses		
M. Prager R. Skowronek	Forschungszentrum Jülich Inst. F. Mineralogie, Universität Bonn		
Local Contact:	E. Jansen, J. Walter		

Experimental report text body

We have continued our study of methyl halide clathrates. Methyl halides are interesting and different probes of the clathrate cages compared to methane clathrate [1] because it interacts with the host surface via dipolar interaction. To incorporate the non water-soluble methyl halide molecules into water a special preparation technique was developed for $\text{CH}_3\text{I} \times 17\text{H}_2\text{O}$ [2]. In this case the formation of the clathrate becomes evident by the transformation into a transition gel state and an increase of the melting point to $T_m \sim 4^\circ\text{C}$. The preparation technique was applied by us in analogue manner to the chloride compound. The crystal structures cited in the literature [3] were confirmed for the methyl iodide and bromide clathrates [4,5]. So far high resolution neutron scattering tunneling spectra and the densities of states were obtained for the methyl iodide [6] and methyl fluoride [7] clathrates. Based on the structures the detailed high resolution spectra gave important information on the potential surfaces of the cages [6,7]. Here we finish the structural work by investigating methyl chloride clathrate, which is in the literature is reported to crystallize either in the cubic I or cubic II structure [3].

A stoichiometric mixture of 1 CH_3Cl and 5.75 D_2O was used. The two-phase liquid was mixed mechanically and solidified with a new melting point $T_m \sim 19^\circ\text{C}$. The sample was mounted cold on the head of a CCR cryostat precooled to 275K and cooled to the required temperatures. Neutron diffraction was applied using the single crystal side of SV7. The wavelength was $\lambda = 2.332\text{\AA}$. Sample temperatures $T_s = 10\text{K}$ and 200K were used. For the predicted cubic I structure a lattice parameter is $\sim 11.8\text{\AA}$. Fig. 1 shows the diffraction pattern measured at $T_s = 200\text{K}$. Compared to the diffraction pattern of methyl bromide clathrate which is cubic I [5] the rather empty spectrum begins with lines at much higher scattering angles only and cannot represent a classical clathrate structure. A search for the unit cell lead to a indexing in the hexagonal space group $\text{P6}_3\text{mc}$ with lattice parameters $a = 4.504\text{\AA}$ and $c = 7.333\text{\AA}$. These are the lattice parameters of hexagonal ice Ih. It was confirmed in an experiment on V3 of HMI that the methyl chloride is pumped off in the present experiment and we were left with pure hexagonal ice.

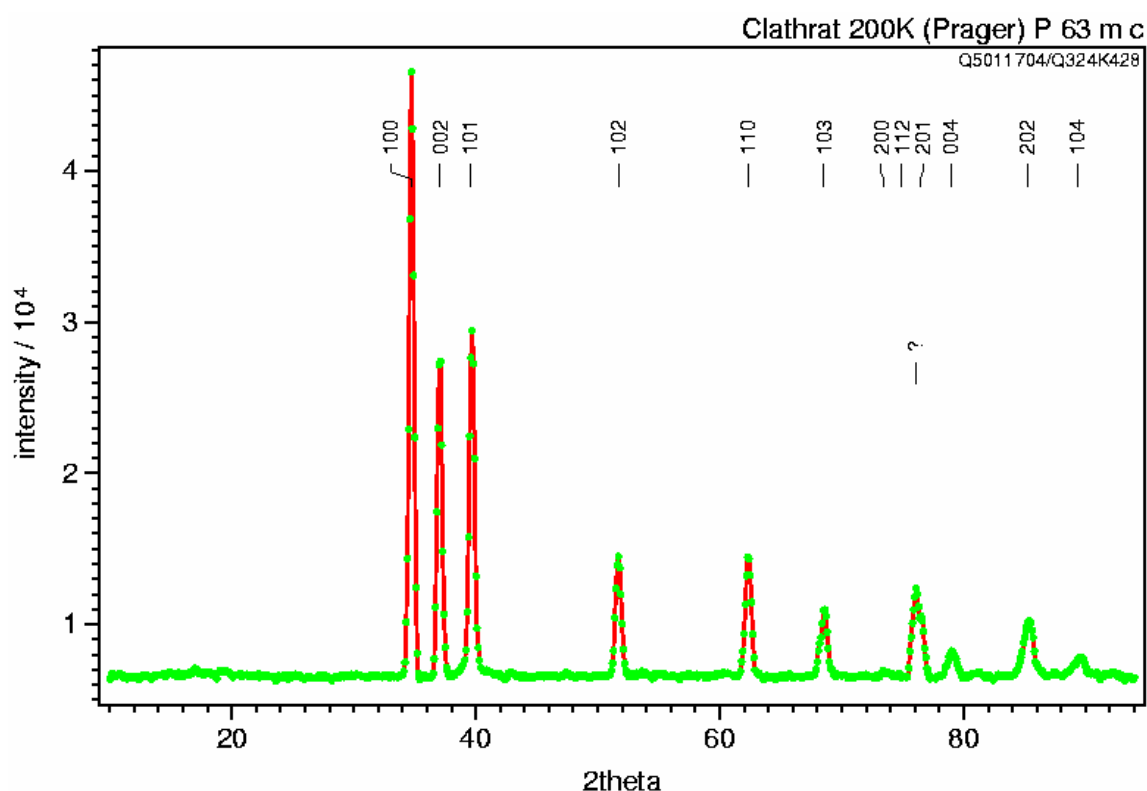


Fig. 1 Diffractogram of D₂O. Sample temperature T=200K. Miller indices characterize each peak. A minor uncertainty represents the doublet line deforming the 201 peak.

- [1] C. Gutt, W. Press, A. Hüller, J. Tse, H. Casalta, J. Chem. Phys. **114**,4160(2001)
- [2] C. Albayrak, Dissertation, Aachen 1988
- [3] G.A. Jeffrey in „Inclusion compounds”. Vol. 1, p. 135ff, J.L. Atwood et al editors, Academic Press, London, 1984
- [4] Experimental reports 2003 of FRJ2, report SV7-02-016
- [5] This volume, report SV7-05-016
- [6] M. Prager, J. Pieper, A. Buchsteiner, A. Desmedt, J. Phys.: Condens. Matter **16**,7045(2004)
- [7] M. Prager, J. Baumert, W. Press, M. Plazanet, J.S. Tse, D.D. Klug, Phys. Chem. Chem. Phys. **7**,1228(2005)