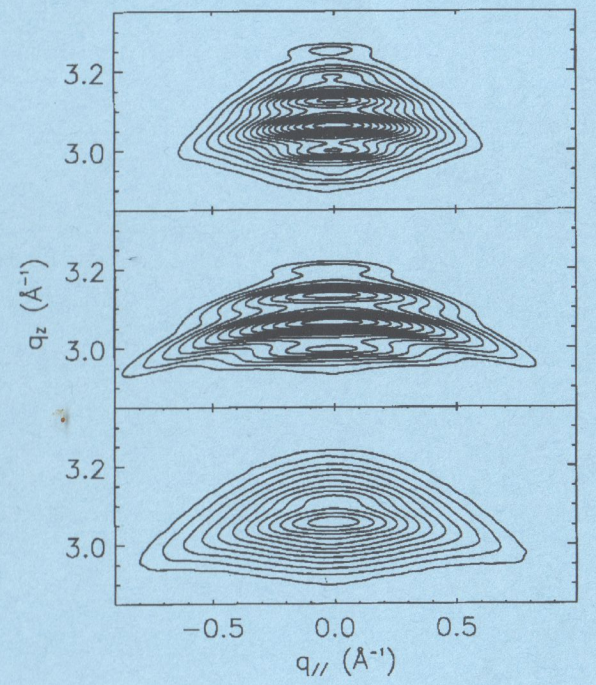
Number 13
Juni 1998

SWISS NEUTRON NEWS



Schweizerische Gesellschaft für Neutronenstreuung
Société Suisse pour la Diffusion des Neutrons
SGN / SSDN

Cover illustration:
Contour plots of the diffracted intensity near the (111) Bragg position of $[Ni_3Al|Ni]_{40}$ multilayers showing the development of coherence between the atomic planes. The spacing between the satellite peaks along the q_z-direction corresponds to the inverse of the bi-layer period of 100 Å (see abstract of S. Tixier in this issue).

Impressum:

Herausgeber: Schweizerische Gesellschaft für Neutronenstreuung

Vorstand für die Amtsperiode Januar 1998 - Januar 2001:

Präsident: Prof. Dr. K. Yvon, klaus.yvon@cryst.unige.ch
Mitglieder: Prof. Dr. S. Decurtins, silvio.decurtins@iac.unibe.ch
Prof. Dr. G. Kostorz, kostorz@iap.phys.ethz.ch
Prof. Dr. P. Schurtenberger, schurtenberger@ifp.mat.ethz.ch
Sekretär: Dr. P. Böni, boni@psi.ch

Ehrenmitglieder: Prof. Dr. W. Hälg, ETH Zürich
Prof. Dr. K.A. Müller, IBM Rüschlikon und Univ. Zürich

Rechnungsrevisoren: Dr. W. Fischer, Paul Scherrer Institut
Dr. P. Schobinger, ETH Zürich

Korrespondenzadresse: Schweiz. Ges. für Neutronenstreuung
Laboratorium für Neutronenstreuung
ETHZ & PSI
CH-5232 Villigen PSI

Tel.: 056 / 310 25 18
Fax: 056 / 310 29 39

Postcheck-Konto: 50-70723-6

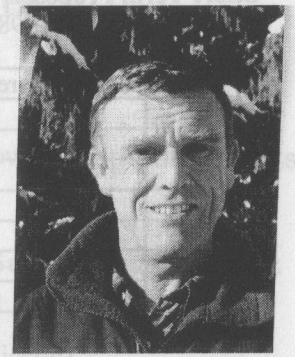
Herstellung: Druckerei PSI, A. Brun

Erscheint ca. 2 mal pro Jahr

Inhaltsverzeichnis

La page du président de la SSDN

Dear members,



as you know, the Swiss Neutron Spallation Source SINQ at the Paul-Scherrer Institute has recently made its first call for proposals. Of course, we were all very anxious to see the response. It was overwhelming, to say the least. Not less than 84 proposals came in. As expected, they originated from scientists working both in Switzerland and abroad, and they covered disciplines which ranged from physics, crystallography and materials science to chemistry and biology. Most importantly, the requests for beam time surpassed the amount of beam time available by a factor of two, on the average (see contribution on beam time allocation in this issue). This not only shows the interest that scientists take in this new neutron source, but also underlines the disparity that continues to prevail in Europe between offer and demand for neutron scattering facilities. In the meantime, the SINQ proposals have been evaluated by an international scientific advisory committee that has proposed a ranking to SINQ management. Needless to say that the evaluation was based exclusively on scientific merits, and that no considerations were given to the national origin of the proposals. All applicants should have been notified by now. Those whose project has not been retained are strongly encouraged to resubmit a project for the next call of proposals whose deadline is Oct. 15 1998.

Concerning the access to the high-flux reactor at the Institut Laue-Langevin at Grenoble, the current contract between ILL and the Swiss Federal Office of Education and Science will come to an end in December 1998. It will probably be renewed, but under changed conditions. Given that the present ILL management intends to enforce a policy of "just return", it is foreseeable that the access to ILL for scientific member countries such as Switzerland will become more difficult. On the other hand, the contracts concerning the CRG instruments D1A (powder diffractometer) and IN3 (three-axis spectrometer) will not be renewed beyond September 1998.

Please note that the Annual Meeting and General Assembly of our society will take place on Friday, November 27 1998, at the Paul Scherrer Institut in Villigen. It will coincide with the user meeting of the SINQ.

I also want to recall that the traditional Summer School on Neutron Scattering takes place in Zuoz from 8 to 14 August 1998. This years topic is "Complementarity between neutron and synchrotron X-ray scattering".

Finally, it is a pleasure to welcome all those "neutron afficionados" who have decided to become new members of our Society (see contribution on new members in this issue).

With my best regards

Klaus Yvon, president

Beam time allocation at SINQ for proposal round II/98

Short-Term Proposals
beam time in cycle II/98

	available	requested
DMC	32	71
DrüchaL	31	56
FOCUS	0	38
HRPT	11	15
SANS	23	55
TASP	42	121
TriCS	0	0

Long-Term Proposals
beam time in II/98, I/99, II/99, I/00

	available	requested
DMC	66	137
DrüchaL	167	288
FOCUS	49	126
HRPT	82	141
SANS	155	375
TASP	129	295
TriCS	100	98

Total requested and allocated beam time per country

	requested (days)	requested (%)	allocated (days)	allocated (%)	success rate (%)
LNS	556	30.62	314	35.40	56.48
ASQ	70	3.86	35	3.95	50.00
PSI	58	3.19	14	1.58	24.14
Switzerland	358	19.71	195	21.98	54.47
Austria	77	4.24	50	5.64	64.94
Australia	1	0.06	1	0.11	100.00
Czech Rep.	9	0.50	4	0.45	44.44
Denmark	14	0.77	10	1.13	71.43
France	128	7.05	71	8.01	55.47
Germany	98	5.40	45	5.07	45.92
Greece	2	0.11	0	0	0
Japan	47	2.59	26	2.93	55.32
Poland	29	1.60	4	0.45	13.79
Russia	288	15.85	71	8.01	24.65
Ukraine	3	0.17	0	0	0
UK	31	1.71	16	1.80	51.61
USA	67	3.69	31	3.50	46.27
total	1816	100.00	887	100.00	48.84

Announcements

Please do not forget the dates for the following important events:

6th Summer School on Neutron Scattering in Zuoz
August 8 - 14 1998
(see contribution in this issue)

Deadline for Next SINQ Proposal Round
October 15 1998

Annual Meeting for SINQ Users
and
General Assembly of the Swiss Neutron Scattering Society
November 27 1998

Meeting of the Scientific Committee for SINQ
November 28 1998

The European Spallation Source Project ESS

The European Neutron Scattering Association (ENSA) produced a report on the European Spallation Source Project ESS. The executive summary of the report is printed below; the complete report is available upon request from the Secretariat of the SGN/SSDN.

Executive Summary

The European Neutron Scattering Association ENSA is a forum of representatives, each nominated by the seventeen National Neutron Scattering Societies within Europe. Each National Society is a formal confederation of researchers who use neutron scattering methods as a tool in their scientific endeavours. An estimated 4'400 researchers are represented, via their National Societies, by ENSA.

Following extensive presentation and consultation exercises conducted by the National Societies with their members, at the request of the European Science Foundation (ESF), a large majority of European researchers have expressed their support for the building of the European Spallation Source (ESS) as being the best option to both satisfy their research needs in the future and to maintain the leading position which Europe has enjoyed globally in this field for 25 years. A digest of the National statements is included in Annex 1 and the individual National statements are presented in their entirety in Annex 2.

ENSA therefore, in its representative capacity, recognising:

- the specific power of neutron methods for the study of condensed matter covering many disciplines; and
- the relevance of microscopic information - structural, dynamic & magnetic - on a wide range of materials from pharmaceuticals, through catalysts, to superconductors, to the competitiveness of European industry; and

- the benefits of strong intra-European co-operation in maximising value for money for the European tax-payer; and
- the predicted decline in operational neutron sources over the next decade as presented by the OECD; and
- the pre-eminent position of Europe in neutron scattering techniques and utilisation, which is now threatened by American & Japanese initiatives; and
- the benefits of world-leading Centres of Excellence for the training of the highest quality scientific and technical manpower to underpin Europe's economic future in an increasingly competitive world,

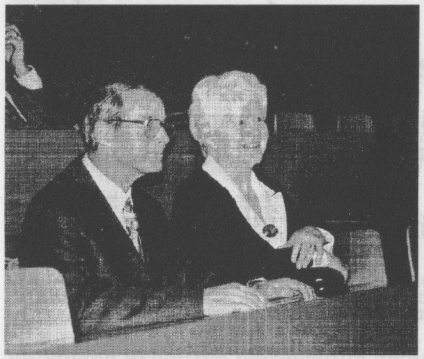
therefore wishes to express its formal position as follows:

- The European Spallation Source represents the best solution for future neutron scattering needs within Europe and ENSA strongly & unequivocally supports a positive commitment to construct it, immediately following the completion of the present R&D phase.
- Operational high flux facilities within Europe, notably ILL & ISIS, must be upgraded to fully utilise the potential of their capital investment, to maximise scientific productivity and to prototype instruments for ESS.
- Operational medium flux facilities within Europe, as well as fulfilling their principal purpose of providing high class equipment for National measuring needs, act as nurseries for the higher flux facilities, not only serving as training grounds for new young researchers but enabling prototype instrumentation and test experiments to be proven, and the future viability of these sources must be assured.

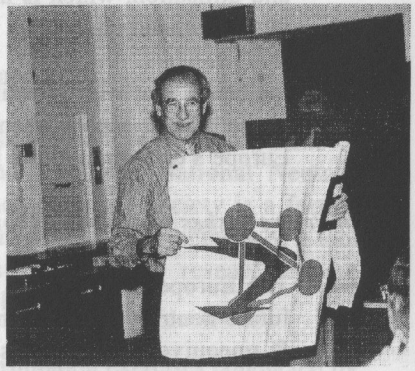
Upon these three foundation stones the future of this powerful technique will be secure. ENSA therefore urges political and scientific authorities within Europe to act both at National and International levels, working with urgency towards a positive decision on the European Spallation Source.

Workshop and Dinner in Celebration of Peter Fischer's 60th Birthday

On the occasion of Peter's birthday a workshop on neutron diffraction was held at PSI, Nov. 21 1997, with talks by Alan Hewat, Juan Rodriguez-Carvajal and Anatoly Balagurov. After the presentations Peter received from his colleagues at the LNS a copy of all his publications (a total of 322) bound into seven full sized books. After this scientific excursions the party moved to the Habsburg the ancestral castle of the Habsburgian dynasty for a cultivated dinner.



Peter and his wife Hedi listening to the talks.



Albert Furrer with his present for Peter's newly bought house.

Prof. W. Hälg listing all the sins of Peter's scientific youth (including the absence of any publications in 68).

Peter convincing all his guests that layered structures are very common in nature and that the rule "the larger the buckling the sweeter the taste" is actually true.

6th Summer School on Neutron Scattering: "Complementarity between Neutron and Synchrotron X-ray Scattering" August 8-14, 1998, Lyceum Alpinum, Zuoz, Switzerland

Free rooms are still available! contact R. Bercher: +41 56 310 3402

Invited Speakers
(tentative titles of topics)

Principles of neutron and synchrotron x-ray scattering	*W.E. Fischer, Villigen*
Hot topics in condensed matter research	*H.R. Ott, Zürich*
Structure determination by neutron diffraction	*E. Gray, Brisbane*
Structure determination by synchrotron x-ray diffraction	*A. Fitch, Grenoble*
Local structures	*S.J.L. Billinge, East Lansing*
Magnetic neutron and synchrotron x-ray scattering	*W.G. Stirling, Liverpool*
Magnetic dichroism	*G. Schütz, Augsburg*
Examples of inelastic neutron scattering (spin fluctuations)	*W.J.L. Buyers, Chalk River*
Examples of inelastic neutron scattering (polymer dynamics)	*D. Richter, Jülich*
Research on thin films and multilayers with neutrons and x-rays	*D. McMorrow, Risö*
SANS, SAXS and reflectometry from surfaces and interfaces	*S.K. Sinha, Argonne*
Photoemission studies of high-temperature superconductors	*C. Kim, Stanford*
Elastic and inelastic x-ray scattering from correlated electrons: a theoretical perspective	*M. Altarelli, Grenoble*
Magnetic correlations in low-dimensional systems	*H.B. Braun, Villigen*
Theoretical concepts for soft condensed matter	*V. Geshkenbein, Zürich*
Neutron beam optics	*P. Böni, Villigen*
Synchrotron x-ray beam optics	*A. Freund, Grenoble*
Summary lecture	*S.W. Lovesey, Didcot*

Convenors of Seminars

Chemical structure	*K. Yvon, Genève*
Dynamics	*B. Dorner, Grenoble*
Magnetism	*G.H. Lander, Karlsruhe*
Electronic structure	*J. Mesot, Argonne*
Surfaces and Multilayers	*S.K. Sinha, Argonne*
Materials	*H.R. Ott, Zürich*

Abstracts of Dissertations

We have decided to publish in Swiss Neutron News short abstracts of dissertations that have been finished recently. If you are interested to have the abstract of your thesis published in the Swiss Neutron News please send a hard copy on white paper A4 to the secretary of the Swiss Neutron Scattering Society.

Structural Characterisations of Sputtered Metallic Multilayers : X-ray and Neutron Scattering Studies.

S. Tixier
Laboratory for Neutron Scattering ETH and PSI
CH-5232 Villigen PSI

Structural characterisations of the metallic multilayers Ni_3Al/Ni, Ce/Fe and Ce/FeCoV were performed using x-ray and neutron scattering techniques, x-ray photoelectron spectroscopy and atomic force microscopy. Crystalline properties of the samples were studied by x-ray diffraction. Intensity contour maps were interpreted within the Born approximation. X-ray specular and diffuse reflectivity gave unique information on the roughness profiles at the interfaces of the multilayers. A more sophisticated theory, the distorted wave Born approximation, has been successfully employed to fit the diffuse scattering spectra and in particular their dynamical features.

The thin films produced at room temperature with our magnetron sputtering facility are found to have common features such as textured layers and flat interfaces. The roughness profiles are partially correlated from one interface to the next, independently of the sputtering conditions (base and Ar pressures). The vertical correlation length decreases slowly with decreasing lateral length scale of the roughness which suggests that the relaxation mechanism in the growth process is rather ineffective. An attempt is made to interpret the results in terms of local growth models.

Rather high crystalline coherence of up to 3.5 times the bilayer thickness Λ is obtained (for $\Lambda \approx 100$ Å) in the Ni_3Al/Ni multilayers. In contrast coherence is limited to the individual layer thickness in the Ce/Fe and Ce/FeCoV multilayers.

A peak in hardness of the Ni_3Al/Ni multilayers corresponding to an 88% increase when compared to bulk Ni_3Al is obtained at a bilayer thickness $\Lambda_{max} \approx 100$ Å. Above Λ_{max}, the decrease in hardness with Λ is compatible with the Hall-Petch equation. The maximum hardness is obtained at a bilayer thickness Λ_{max} which corresponds to a compromise between a small Λ and fully dense and textured layers.

Magnetic properties of the Ce/FeCoV multilayers were studied by means of polarised neutron reflectometry and Mössbauer spectroscopy. The average FeCoV moment is found to decrease with decreasing bilayer thickness. The thin multilayers with an individual thickness for FeCoV below 15 Å display perpendicular magnetic anisotropy at room temperature.

Investigation of High-Temperature Superconductors of the R-Ba-Cu-O Family: Substitution Effects and Magnetic Properties

Grit Böttger
Laboratory for Neutron Scattering ETH Zurich & PSI
CH-5232 Villigen PSI

The subject of this thesis is the investigation of substitution effects and magnetic properties of different high-temperature superconductors (HTSC) that belong to the *R*-Ba-Cu-O family. The results have been obtained mainly by elastic and inelastic neutron scattering. In some cases other experimental techniques have been used such as X-ray diffraction, magnetic susceptibility and specific heat measurements. It was a particular concern of this thesis to investigate the magnetic properties of $R_2Ba_4Cu_7O_{15-\delta}$ (*R*=Er, Dy). Because of the relatively difficult preparation of large quantities of well defined samples, limited information is presently available on $R_2Ba_4Cu_7O_{15-\delta}$ compounds. Problems connected with the synthesis of large sample quantities of superconductors with high transition temperatures T_c could be managed successfully. In the frame of this study, it was possible to carry out for the first time a detailed study on the magnetic ordering phenomena of rare earth ions R^{3+} in $R_2Ba_4Cu_7O_{15-\delta}$.

After a short introduction covering historical aspects of superconductivity and the motivation for this work, the aspects of theory that are essential for the understanding of this thesis are outlined. The instruments used for neutron scattering experiments are presented, as well. In the third chapter, the preparation methods for polycrystalline samples are introduced with respect to their thermodynamical properties. In the fourth chapter it is described how neutron crystal-field spectroscopy can successfully contribute in the ongoing debate about the symmetry of the gap in HTSC. The study of the temperature dependence of crystal-field (CF) transitions in the slightly underdoped HTSC $Er_2Ba_4Cu_7O_{14.92}$ and $HoBa_2Cu_4O_8$ gives clear evidence for the opening of an electronic gap or pseudogap in the normal state far above T_c. The main observed features can be reproduced by considering a strongly anisotropic gap function with predominant *d*-wave character with a small *s*-wave component to be added. The fifth chapter reports on a systematic study on the influence of Ca doping on the structure and physical properties of orthorhombic $RBa_2Cu_3O_{7-\delta}$. A high-resolution neutron diffraction investigation of a series of polycrystalline $R_{1-x}Ca_xBa_2Cu_3O_{7-\delta}$ (*R*=Y, Er; $0.04 \le \delta \le 0.15$) samples shows remarkable structural changes induced by the partial substitution of the rare earth ion by Ca. The CF excitations of Er^{3+} in $Er_{1-x}Ca_xBa_2Cu_3O_{7-\delta}$ have been determined. It is shown that the partial substitution of R^{3+} by Ca^{2+} introduces additional hole carriers in the structure and makes the $R_{1-x}Ca_xBa_2Cu_3O_{7-\delta}$ ($0.04 \le \delta \le 0.15$) system accessible far into the overdoped region. Finally, in chapter six, the structure and the magnetic properties of the high-temperature superconductors $R_2Ba_4Cu_7O_{15-\delta}$ (*R*=Er, Dy; $0 < \delta < 1$) are discussed, resulting from a study based on neutron scattering, specific heat and magnetic susceptibility measurements. The neutron diffraction results indicate for the first time a long-range antiferromagnetic order of the Er ions in $Er_2Ba_4Cu_7O_{14.92}$.

First results from the prompt gamma-ray activation (PGA) facility at PSI

M. Crittin, J. Kern, J.-L. Schenker, H. Van Swygenhoven*
Physics Department, University of Fribourg, CH-1700 Fribourg, Switzerland
**Paul Scherrer Institute,CH-5232 Villigen, Switzerland*

During the months of October and November 1997, first measurements were performed at the Prompt Gamma Activation installation (its detection system was described briefly in the June 1997 number of Swiss Neutron News). During this period, many tests were performed showing that the installation works according to specifications. In order to build up a good data basis, several gamma-ray spectra of pure and composed elements were measured and some studies for external users were executed. The neutron focusing lens was also tested.

The performances of the Compton-suppression (CS) and pair spectrometers are illustrated in Figures 1-2. The figures show a part of the Fe gamma-ray spectrum measured during 7200 s. with the CS spectrometer (Figure 1) and with the pair spectrometer (Figure 2). The same energy domain is displayed. The sample was prepared by pressing 136 mg iron powder in a disk of 5 mm in diameter. The single and double escape peaks (due to the non-reabsorption of the annihilation photons produced in the pair creation processes) corresponding to the Fe doublet are not totally suppressed with the CS spectrometer. In contrast, only the double escape peaks are observed with the pair spectrometer. However, the CS spectrometer has a much higher efficiency (cf. Figure 3). The efficiency is defined as the ratio of the number of detected photons of a particular energy to the total number emitted by the source at that energy.

Element	This work [mg]	X-ray spectrometer [mg]
Na	0.84	0.82
Si	19.6	21.8
K	2.0	2.2
Ca	4.4	4.8
Ti	0.28	0.31
Mn	0.050	0.048
Fe	3.2	3.1

Table 1 Analytical results of a geological reference sample.

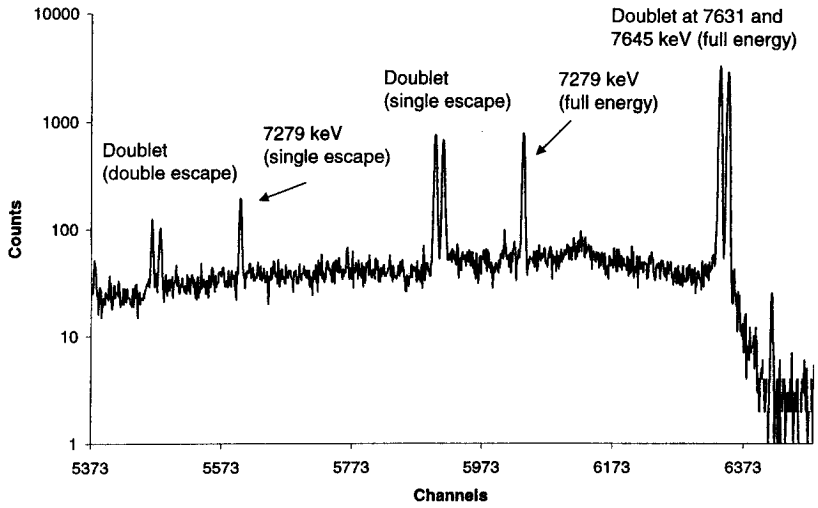


Figure 1 Part of the Fe spectrum measured with the CS spectrometer. Several peaks corresponding to the transitions in iron at 7279, 7631 and 7645 keV are observed.

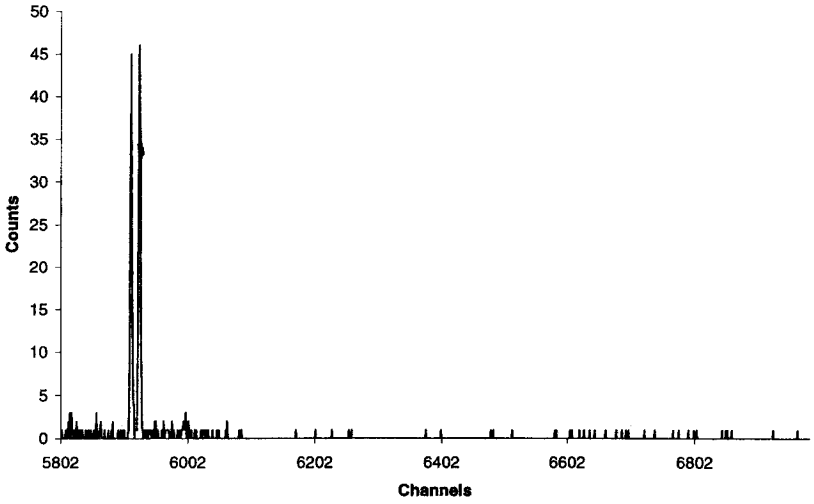


Figure 2 Part of the Fe spectrum measured with the pair spectrometer. Only the double escape peaks do appear. Note the very high peak-to-background ratio.

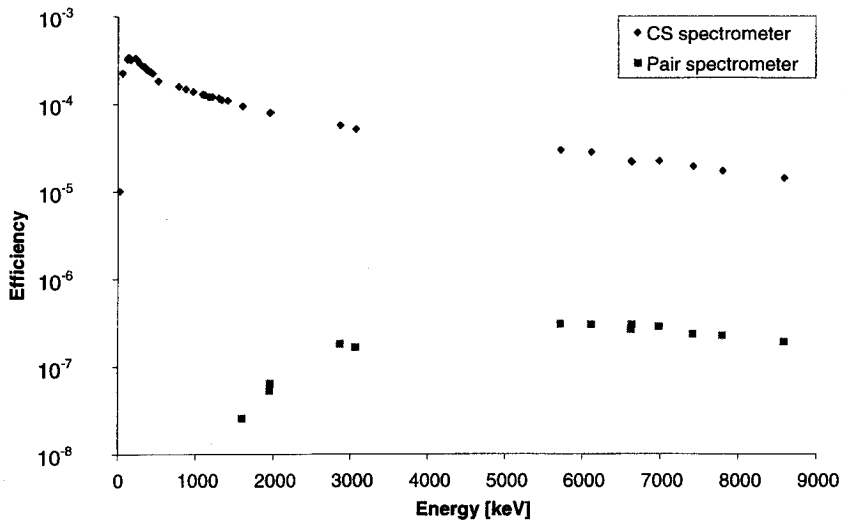


Figure 3 Efficiency curves of the CS and pair spectrometers.

Table 1 shows a comparison of a sample analysis performed with the PGA and a X-ray spectrometer. The results agree within 10%.

Various improvements to the system to further reduce the background were performed during the shut-down. The facility is specially optimized to obtain a high sensitivity for boron. Sensitivities and detection limits for selected elements will be carry out during the period 1998. Also many tests and measurements will be accomplished with the neutron lens which will be encapsulated in a new special shielding.

We are now in the position to accept proposals for measurements from external users.

Konferenzen 1998

Datum	Ort	Thema
15.-18.7.98	Paris	International Conference on "Strongly Correlated Electron Systems"
18.-23.7.98	Arlington	Meeting of the American Crystallographic Association
1.-5.8.98	Ascona	1*st* SLS Workshop on Synchrotron Radiation
6.-8.8.98	Nyköping	4*th* Int. Workshop on Quasi-Elastic Neutron Scattering
8.-14.8.98	Zuoz	6*th* Summer School on Neutron Scattering: Complementarity between Neutron and Synchrotron X-ray Scattering
16.-20.8.98	Prag	ECM-18 (European Crystallographic Meeting)
22.-25.8.98	Budapest	6*th* Europ. Powder Diffraction Conference (EPDIC-6)
25.-29.8.98	Grenoble	17*th* General Conf. of the Condensed Matter Division of EPS
30.8.-3.9.98	Dresden	15*th* Int. Workshop on Rare-earth Magnets
9.-12.9.98	Zaragoza	EMMA'98 (European Magnetic Materials and Applications Conference)
18.-22.9.98	Patras	Euroconference on "Novel Materials"
21.-23.9.98	Grenoble	Polarized Neutrons for Condensed Matter Investigations (P.N.C.M.I. 98)
29.-30.9.98	Villigen	SINQ Industrieseminar
8.-11.10.98	Matrahaza	International Symposium on Small-Angle Scattering
22.-24.10.98	Grenoble	Workshop on Particle Physics with Slow Neutrons
9.-12.11.98	Miami	43*rd* Annual Conference on Magnetism and Magnetic Materials
24.-27.11.98	Rappongi	7*th* Int. Symposium on Frontiers in Neutron Scattering Research
27.11.98	Villigen	SINQ User Meeting & Generalversammlung der Schweiz. Ges. f. Neutronenstreuung
30.11.-4.12.98	Boston	MRS Fall Meeting
9.-12.12.98	Grenoble	Neutrons and Numerical Methods

Neue Mitglieder

P. Böni

Die Anzahl der neuen Mitglieder hat im ersten Halbjahr 1998 erneut stark zugenommen. Wir begrüssen:

- V. L. Aksenov, JINR, Dubna
- W. Bronger, Technische Universität Aachen, Aachen
- Ph. Buffat, EPFL-CIME, Lausanne
- N. Cavadini, Labor für Neutronenstreuung ETH & PSI, Villigen
- R. Caciuffo, Univsità die Ancona, Ancona
- I. Ermeev, International Business Nucleonic, Moscow
- K. Funke, Universität Münster, Münster
- E. M. Iolin, Institute of Physical Energetics, Riga
- I. Ionita, Institute for Nuclear Research, Pitesti
- S. N. Ishmaev, Kurchatov Institute, Moscow
- E. Jericha, Labor für Neutronenstreuung ETH & PSI, Villigen
- J. Jolie, Université de Fribourg
- H. Kohlmann, Université de Genève, Genève
- D. Kuse, Paul Scherrer Institut, Villigen
- V. A. Nazarenko, Petersburg Nuclear Physics Institute, Gatchina
- R. Pellaux, Universität Zürich, Zürich
- G. A. Petrakovskii, Institute of Physics SB, Krasnoyarsk
- D. Protopopescu, JINR, Dubna
- A. Radulescu, IFIN-HH, Bukarest
- T. Schucan, Paul Scherrer Institut, Villigen
- F. Semadeni, Labor für Neutronenstreuung ETH & PSI, Villigen
- N. Zotov, Universität Bayreuth, Bayreuth

Zur Zeit zählt die schweizerische Gesellschaft für Neutronenstreuung 189 Mitglieder.

Mitgliederbeitrag 1998

P. Böni

Wir bitten alle Mitglieder der Schweizerischen Gesellschaft für Neutronenstreuung, den Jahresbeitrag 1998 mit anliegendem Einzahlungsschein auf unser Postcheckkonto einzuzahlen. Der Beitrag beträgt immer noch nur die Wenigkeit von CHF 10.–.

Wir bitten unsere ausländischen Kollegen, Ihren Beitrag bei Gelegenheit in bar zu bezahlen, da die Gebühren für Überweisungen oft höher sind als der Mitgiederbeitrag selbst.

Membership Fees 1998

P. Böni

We ask our foreign colleagues to pay the membership fee for 1998 in cash at a reasonable occasion because the fees for forwarding the money to our account is usually higher than our modest fee of CHF 10.–.

Martensitic Phase Transformation in Ni-Ti-Shape-Memory-Alloys

R. Gotthardt, Institut de Génie Atomique, Département de Physique,
Ecole Polytechnique Fédérale de Lausanne, Ch-1015 Lausanne,
Switzerland.

I. Introduction

The martensitic phase transformation in Ni-Ti-Shape-Memory-Alloys (SMA) transforms a high temperature cubic phase, called austenite (A), into a low temperature monoclinic phase, called martensite (M). In binary alloys, an additional rhombohedral phase, called the R-phase can be observed. These transformations depend strongly on the chemical composition and the thermo-mechanical treatment (Duerig 1990). Above the normal transition temperatures such transformation can also be induced by applied stresses.
The study of the martensitic phase transformation in SMA is of twofold interest. On the one hand there is a fundamental question because this transformation is a special type of the displacive phase transformations, which means that they take place without diffusion. On the other hand this martensitic transformation is accompanied by effects which open interesting aspects for applications:

- the shape-memory-effect,
- the superelasticity and
- the high damping capacity in the martensitic state.

These auxiliary effects are at the base of many applications going from macroscopic scale as satellite antennas or earth-quake protections, through the medium scale as fasteners and couplings to the microscopic scale as micro-gripper and thin films. Very recent and important applications of the SMA have occurred in the medical field where they are used as stents, staples or even as drug delivery devices.
In order to understand the transformation mechanisms and to control the auxiliary effects for applications it is important to know the relationship between the microstructure of the involved phases and their physical behaviour during the martensitic transformation. Therefore, to acquire this information, calorimetry measurements have been carried out for the transformation characteristics and Transmission Electron

Microscopy (TEM) observations have been made to determine the microstructure. In addition martensitic phase transformations have been studied in-situ in the microscope to observe directly the micromechanisms taking place during this transition. The martensitic phase transformation and effects of different heat treatments can also be followed by neutron diffraction. First experiments have been done in collaboration with W. Bührer (Bührer 1997).
In the following an example TEM study will be given for the case of a complicated transformation behaviour in a Ni-Ti- SMA.

II. Microstructure and Transformation Behaviour

II.1 Experimemtal procedures

In order to introduce, in a controlled way, a certain microstructure, Ni-Ti samples with a nominal composition of 48.86 at% Ti and 51.14 at% Ni were heat treated at 1173 K, quenched in water at room temperature and annealed at 793 K, both annealings were for 30 minutes under a flux of Argon gas. The samples were then quenched again into water at room temperature. The transparency for TEM observation was obtained by twin-jet electropolishing, using an electrolyte solution of 1:4 volume fraction of sulphuric acid and CH_3OH.
The observations were made in a Philips CM 20 microscope operating at 200kV allowing studies between 77 K and 373 K. The temperature variation was about 5K/min in the tansformation temperature range.

II.2 Results

A typical result of the calorimetry measurements with samples which have been treated as described in the above section is shown in fig. 1. It can be seen clearly that these treatments induce an even more structured transition (three steps during cooling), therefore this is called a multi-step-transformation. In the austenitic state (373 K) the samples are well recrystallized and contain precipitates of oblate spheroid shape. A typical microstructure is shown in fig. 2. The dimensions of these precipitates are between 300 nm and 500 nm in diameter and about 50 nm in thickness. They are identified as being of the Ni_4Ti_3 type, parallel to {111} planes of the austenitic structure and coherent with the matrix (Bataillard and Gotthardt 1995). Due to the different lattice parameter a Ni_4Ti_3-particle (fig.3a) is surrounded by a stress field as can be seen in fig. 3b by a dark contrast (dynamic imaging conditions). If the sample is treated differently, e.g. furnace cooled after 30 minutes at 1173 K, the

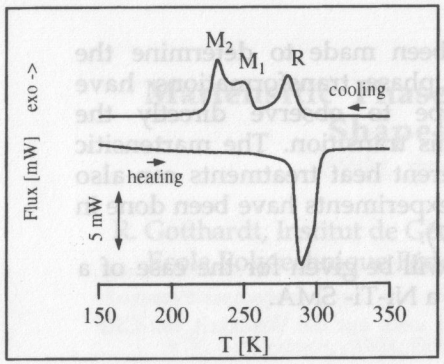


Figure 1: Calorimetry measurement of a sample heat treated at 1173K and aged at 793K, showing a multiple transformation.

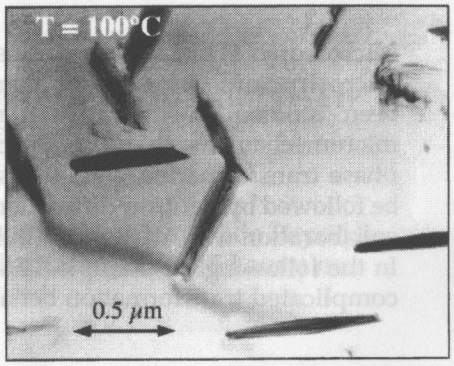


Figure 2: TEM micrograph of a sample heat treated as that in fig. 1. Precipitates lie on the {111} planes of the austenitic phase.

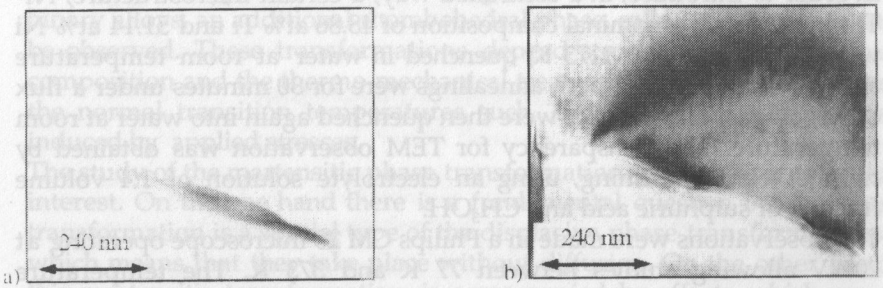


Figure 3: TEM observation of the local stress around precipitates of the Ni_4Ti_3 type. The stress field can be seen qualitatively as a dark contrast in b).

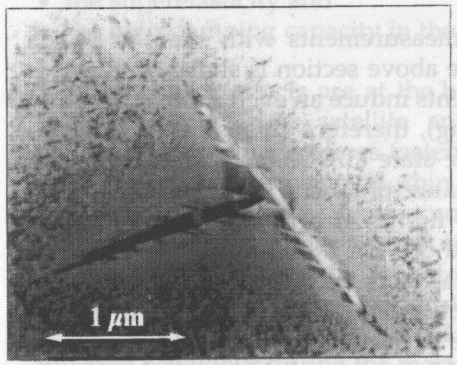


Figure 4: TEM micrographs of a sample heat treated at 1173K and furnace cooled.

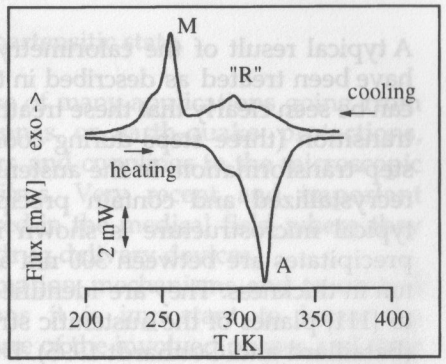


Figure 5: Calorimetry measurement of a sample heat treated as the sample in fig. 4.

microstructure is quite different as can be seen in fig. 4. The corresponding calorimety measurement is shown in fig. 5 and reveals a more complicated transition from austenite to martensite.

Figures 6a-d show the changes in microstructure of the same region that was shown in the austenitic state in fig. 2, when the specimen is cooled inside the microscope. In order to identify the different observed phases, diffraction patterns have been made and analysed for each of them. Due to the lack of space they are not shown here, but are published elsewhere (Bataillard 1996). Figure 6 shows the preferential appearance of the R-phase around the precipitates (fig. 6a and b) followed by the normal transformation from austenite to the R-phase (fig. 6c). At still lower temperatures the whole region transforms into martensite (fig. 6d). This transformation sequence can not be resolved in the calorimetry measurements (fig. 1).

The modifications in microstructure concerning the R-phase during retransformation from M to A are shown in fig. 7a-f. In fig. 7a the interface between phase M (black contrast) and phase A (grey contrast) is indicated by a white arrow. In fig. 7b and c more and more phase A is present; only the regions around and between the precipitates are still martensitic (fig. 7c and d). The remaining phase M around the particles does not transform directly into A, but transforms first to the R-phase as can be seen by the characteristic striped contrasts (fig.7d), already observed during cooling (fig.6).

III Discussions

The TEM observations of in-situ transformations in NiTi samples show clearly that the stress appearing around the small coherent Ni_4Ti_3-particles, which have been created during the annealing treatment, favours the transformation to the new phase. This transformation takes place at higher temperatures. In the case of the A-R transition, the difference is too small to be observed by calorimetry. In contrast, for the R-M transition the splitting of the transformation is large enough to be measured (fig.1, M_1 and M_2). During heating the normal retransformation sequence M->A is modified into an M-> R -> A sequence in the regions which are exposed to the stress field around the precipitates.

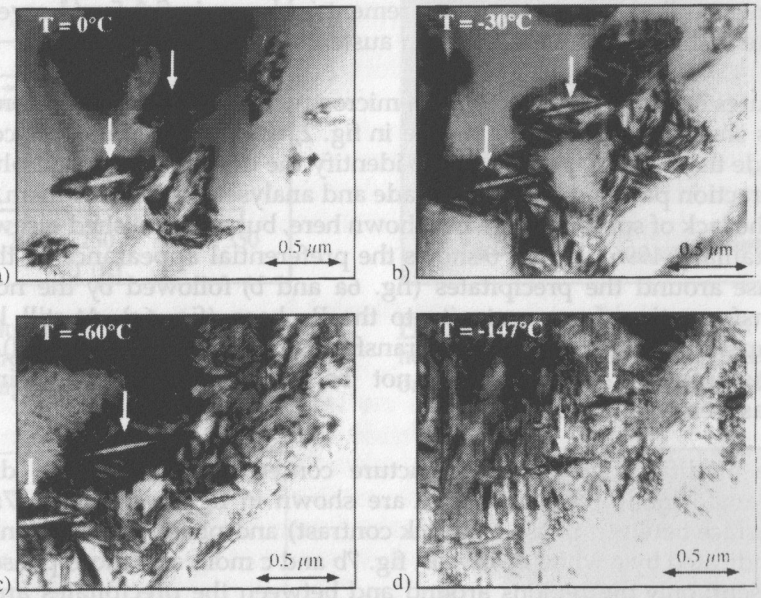


Figure 6: Sequence of micrographs showing the transformation of austenite to R-phase (a-c) and the final martensitic phase (d).

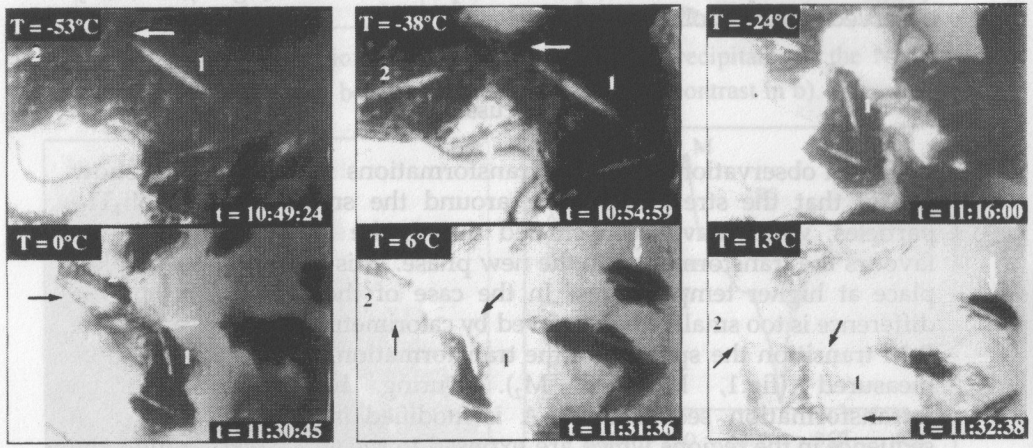


Figure 7: Sequence of micrographs taken from a video film showing the retransformation from martensite to austenite. In the stress field around the precipitates, the retransformation passes through the formation of R-phase.

IV Literature

T.W. Duerig, « Engineering Aspects of Shape Memory Alloys », Ed.: T.W. Duerig, K.N. Melton, D. Stöckel and C.M. Wayman, Butterworth-Heinemann Ltd., London, (1990).

L. Bataillard and R. Gotthardt, "Influence of Thermal Treatment on the Appearence of a Three Step Martensitic Transformation in NiTi", Journal de Physique IV, supplém. Journ. de Phys. III, 5, (1995) p. C8-647.

L. Bataillard, PhD-Thesis Nr.1518, Ecole Polytechnique Fédérale de Lausanne, Lausanne, Switzerland, (1996).

W. Bührer, D. Rubio, R. Gotthardt, L. Bataillard, and C. Ritter, « Crystal Structures and Phase Transitions of the Shape Memory Alloy Ni 50.5 Ti 49.5 », Neutronenstreuung Annual Progress Report 1996, LNS-190, (1997), p.45.

R.Grüebler, W. Bührer, R. Gotthardt and M. Reehuis, « Long-Range Order in the Nearly Equiatomic Shape-Memory Alloy Ni 50.5 Ti 49.5 », ibido, p.46

Schweizerische Gesellschaft für Neutronenstreuung (SGN)
Société Suisse pour la Diffusion des Neutrons (SSDN)

Anmeldeformular

Name:.....

Vorname:.....

Akad. Titel:.....

Geschäftsadresse:.....

.....

.....

Telefon:.....

Telefax:.....

Telex:.....

E-Mail:.....

Privatadresse:.....

.....

.....

Telefon:.....

Zustelladresse: Geschäft/Privat ? (Nichtzutreffendes streichen)

Datum:

.....

Unterschrift:

.....

Bitte senden an:

Sekretariat SGN, c/o Laboratorium für Neutronenstreuung, 5232 Villigen PSI

(Jahresbeitrag: Fr. 10.--)

SINQ **Swiss Spallation Neutron Source** **Paul Scherrer Institute**	**Paul Scherrer Institute (PSI)** SINQ Scientific Coordination Office WHGA/147, CH-5232 Villigen PSI, Switzerland Phone: +41 56 310 2087, Fax: +41 56 310 2939 Email: SINQ@psi.ch, Web: www.psi.ch/sinq

EXPERIMENT REQUEST

Proposal number

[ ] **Short term proposal (next allocation period)**	[ ] **Long term proposal (2 years)**

Proposer *(to whom correspondence will be addressed)*
Name and first name:
Address:

Phone:
Fax:
Email:

Co-proposer:
Name: Address: *(if different from above)* Phone/Fax/Email:

Sample description

Substance and formula:

Mass: Size:

[ ] Polycrystalline [ ] Single crystal [ ] Multilayer [ ] Liquid [ ] Gas

Sample Container: Space group: Unit cell: a= b= c=

Hazard

Is there any danger associated with the sample or sample environment?
[ ] No [ ] Yes [ ] Uncertain If yes or uncertain, please give details of the risks associated:

Experimental details

Instrument	**Days**	**Sample cond.:** Temp., Pressure, Magn. field	**Exp. cond.:** E, ΔE, λ, Δλ, Q, ΔQ

Requested dates:	**Unacceptable dates:**

Title of Experiment:

Research funded by:

Scientific background/Aim of experiment: *(Please restrict to the space given within this box!)*

I certify that the above details are complete and correct.
Date: Signature of proposer: