DEPARTMENT FOR NANOSTRUCTURED MATERIALS K-7

Head:
Asst. Prof. Spomenka Kobe

The basic research in the Department for Nanostructured Materials focuses on inorganic materials whose specific physical properties are a consequence of their structural and chemical characteristics at the nanometer and atomic levels. The research involves natural and manufactured ceramic materials as well as metals and intermetallic compounds. The objective is to find relationships between the physical properties of a material and its structural and chemical properties, using electron microscopy techniques to reveal phenomena on the nanoscale. Macroscopic phenomena, for example, phase transformations, phase equilibria, polytypism, polymorphism, crystal growth and the development of the microstructure are all areas of particular interest.

Research in the field of intermetallic alloys was continued in two main areas: Sm-Fe-N and Nd-Fe-B based permanent magnets. The magnetic behavior of $Sm_{13.7}Fe_{86.3}$ and $Sm_{13.8}Fe_{82.2}Ta_{4.0}$ materials during the hydrogenation-disproportionation-desorption-recombination (HDDR) and subsequent nitriding processes was investigated using a specially designed vibrating-sample magnetometer (VSM) modified with a high-temperature (up to 1000 °C) vacuum-gas system. The techniques rely on detecting the moment of the ferromagnetic iron as it is formed during the disproportionation process. This method has the advantage of detecting the main product of the disproportionation reaction directly, rather than relying on secondary effects such as a drop in pressure or a change in resistance.

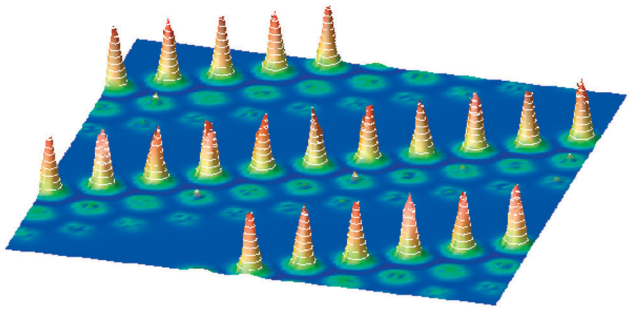

Figure 1: Calculated spin density in Pt-supported nanowires of Co atoms reveal the existence of induced magnetic moments in the substrate.

The results show that the initial absorption of hydrogen results in an increase in magnetization as well as in Tc, and that the sluggish disproportionation of the Sm_2Fe_{17} phase in the $Sm_{13.8}Fe_{82.2}Ta_{4.0}$ sample is due to dissolved Ta. Monitoring the nitriding process shows that the magnetization increases as the sample picks up nitrogen at around 250 °C. If the sample was previously HDDR-ed, the nitrogen uptake goes very smoothly whereas, if the samples are not HDDR processed, the nitrogen reaction also involves decomposition of the $SmFe_2$ and $SmFe_3$ phases. The disproportionation reaction for Nd-Fe-B type materials was investigated using our modified vibrating-sample magnetometer. Our investigations of Pr-substituted, Zr-doped Nd-Fe-B-based materials have led to the identification of a two-stage disproportionation reaction in Zr-free materials at temperatures in the range 600–650 °C. These results suggest that the standard Johnson–Mehl–Avrami–Kolmogorov (JMAK) model is inadequate for describing the process of disproportionation in these alloys, and that an extension to the JMAK theory that takes into account a critical radius for stable iron nuclei is required to explain these low values.

The evolution of microstructure during the HDDR preparation of hard magnetic powders for bonded magnets, based on Nd-Fe-Dy-B as the basic material, was studied using the transmission electron microscopy. The distribution of phases, grain size and morphology of NdH_2, Fe_2B and α-Fe in samples prepared under various conditions were investigated. It was found that certain crystallographic relationships exist between several phases, which could explain the high degree of anisotropy of the final material. The result of the applied research in the frame of a NATO SfP was the pilot production of injection moulded bonded magnets.

We investigated nonlinear magneto-elastic coupling in 3d transition-metal epitaxial films by applying a phenomenological theory and by calculating the electronic structure. We calculated the X-ray-magnetic-circular-dichroism (XMCD) spectra, tested the corresponding sum rules and studied the influence of the magnetic dipolar term. We investigated the magnetic properties (XMCD, magnetic moments and magnetic anisotropy) in nanowires, within the framework of the density functional theory.

The microstructure evolution during the HDDR preparation of hard magnetic powders based on Nd-Fe-Dy-B was studied using the transmission electron microscopy. It was found that certain crystallographic relationships exist between NdH_2, Fe_2B and α-Fe phases, which could explain a high degree of anisotropy of final material. We investigated the magnetic properties in nanowires within the framework of the density functional theory. The result of the applied research in the frame of a NATO SfP was the pilot production of injection molded bonded magnets.

In numerous ceramic materials with perovskite and wurtzite structure we demonstrated the influence of planar faults and polytypic sequences on exaggerated grain growth, which can be beneficially exploited for microstructure tailoring. We optimised the composition of varistor blocks used for high voltage arresters. In the field of electron microscopy we implemented Z-contrast imaging (HAADF-STEM), which enables quantitative chemical analysis of individual atomic columns based on differences in their intensities.

The nucleation and crystallisation of calcium carbonate in tap water under the influence of a magnetic field is the subject of continuing studies. Using analytical electron microscopy it was found that the nucleation of all three crystal phases is much slower under the magnetic field. The ratio of calcite, aragonite and vaterite crystal forms is also strongly influenced by the presence of the field.

In materials used as optical elements in laser nanolithography, such as various fluorides doped with rare earths, the degree of chemical homogeneity was determined using energy dispersive X-ray spectroscopy. It was found that, in the range of a few nanometers, some inhomogeneous areas exist which influence the optical properties. The presence of nanometer sized clusters with different composition was investigated with STEM/ HAADF (Z-contrast). For the TEM study of the surface of silicone wafers covered with photoresist and irradiated with high-energy laser radiation we optimized the preparation of carbon replicas.

The research program of the group for electron microscopy was primarily focused on determining the structure and chemical composition of planar faults and polytypic sequences in various polycrystalline ceramic materials, using different electron microscopy techniques. The investigations of the phenomenon of exaggerated grain growth have led us to a systematic study of grain growth in ceramic materials with perovskite and wurtzite structures. Atomic resolution transmission electron microscopy showed, that as a rule, the exaggerated grains contain polytypic faults which can either be isolated or form ordered polytypic sequences. Faults of this type can only be observed in systems where a secondary polytypic phase with incongruent decomposition exists between the main phase and the dopant. Polytypic sequences can trigger exaggerated grain growth only in a very narrow thermodynamic regime, which can be used for microstructure control.

In $SrTiO_3$ and $CaTiO_3$ perovskites with AO–excess (A=Ca,Sr,Ba), we determined the chemical composition of polytypic phases and isolated planar faults by high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM), i.e. Z-contrast imaging.

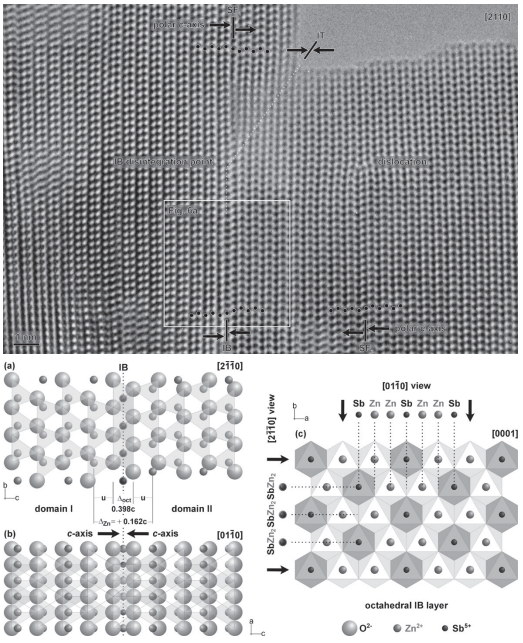

Figure 2: Reconstruction of the inversion boundary in Sb_2O_3-doped ZnO

Quantitative HAADF-STEM requires comparison between experimental and simulated images. Since the experimental images are usually distorted due to instability of the STEM unit, we developed suitable image processing algorithms that enable quantitative image correlation with the simulated images. As the first research group we quantitatively incorporated the detector noise in simulations. In systems $SrTiO_3$-CaO and $SrTiO_3$-BaO we showed that the dopant with the lower atomic number Z is always ordered at the planar fault. In the system $SrTiO_3$-Fe_2O_3 we studied the formation of precipitates in the chemical composition of the boundaries. In ZnO-Sb_2O_3, which has a wurtzite structure, we determined the structure of inversion boundaries by high-resolution transmission electron microscopy (HRTEM). For chemical analysis of sub-monolayer at the inversion boundary we have developed, in collaboration with the University of Bonn, a special analytical method for determining extremely low element concentrations at planar faults or grain boundaries. The reconstruction of structural and chemical information obtained from the inversion boundary showed that these planar faults have $ZnSb_2$ composition, where Zn and Sb atoms are completely ordered into the superstructure with 3m symmetry. These results led us to systematic investigation of microstructure developments in ZnO doped with extremely small concentration of Sb^{+3}, from 0 to a few 100s of ppm. We found that even very small amounts of Sb^{3+} caused the formation of inversion boundaries that determine further ZnO microstructure development. In ZnO-based varistor materials, doped with rare earth oxides (REO), we have studied the influence of Sb_2O_3 and Bi_2O_3/Sb_2O_3 ratio on microstructure and electrical properties of ZnO ceramics doped with Y_2O_3.

Applied research for VARSI d.o.o. continued with the development of varistor blocks for high voltage arresters for voltages up to 12 kV. By optimizing the starting composition, the binder burnout process and the sintering regime, we have achieved better final electric properties of the material. We have further collaborated with VARSI d.o.o. in developing energy varistors with different nominal voltages and shapes, for miniaturized and integrated modules for overvoltage protection. We are involved with Bosch in developing a $Sr(Ti,Fe)O_3$ oxygen sensor.

In 2002 the group for electron microscopy carried out electron microscopy analyses of inorganic and organic materials for the following customers: Jožef Stefan Institute (K5, K9, F5, F3, K3), National Institute of Chemistry, Faculty of Pharmacy, Faculty for Natural Sciences, Faculty for Mathematics and Physics, Biotechnical Faculty, Slovenian Health Society, Pulp and Paper Institute, Slovenian Restoration Centre, Lek d.d., Krka, DONIT TESNIT d.d., BIA Separations d.o.o., Swaty d.d., Premogovnik Velenje d.d., EMO Kemija, Cinkarna Celje, IRMA, KEKO OPREMA, ERICO Velenje, and EKOM.

Some outstanding publications in the year 2002

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    Structure and chemistry of (111) twin boundaries in sphalerite crystals from Trepča mines in Kosovo
    In: Proceedings, ICEM 15, 15th International Congress on Electron Microscopy, Durban, South Africa, September 1-6, 2002, [S.l.], Microscopy Society of Southern Africa, 2002, Zv. 1, p. 1075.
19. Sašo Šturm, Aleksander Rečnik, Miran Čeh
    Chemistry of planar faults in AO doped $SrTiO_3$ (A=Ca,Sr,Ba) resolved by image-WARP processing of HAADS-STEM images
    In: Proceedings, ICEM 15, 15th International Congress on Electron Microscopy, Durban, South Africa, September 1-6, 2002, [S.l.], Microscopy Society of Southern Africa, 2002, Zv. 1, pp. 495-496.
20. T. Walther, Aleksander Rečnik, Nina Daneu
    Test of a new analytical method to measure the composition of a planar fault
    In: Proceedings, ICEM 15, 15th International Congress on Electron Microscopy, Durban, South Africa, September 1-6, 2002, [S.l.], Microscopy Society of Southern Africa, 2002, Zv. 1, pp. 535-536.
21. T. Yamazaki, Aleksander Rečnik, M. Kawasaki, Miran Čeh, K. Watanabe, M. Shiojiri
    A HAADF-STEM investigation of inversion boundaries in Sb_2O_3-doped ZnO ceramics
    In: Proceedings, ICEM 15, 15th International Congress on Electron Microscopy, Durban, South Africa, September 1-6, 2002, [S.l.], Microscopy Society of Southern Africa, 2002, Zv. 1, pp. 515-516.

LECTURES - GUEST LECTURES AT FOREIGN UNIVERSITIES

1. Miran Čeh
   High-resolution HAADF STEM imaging of $(Ca,Sr,Ba)TiO_3$ perovskites: invited talk
   Graz, Forschungsinstitut für Elektronenmikroskopie und Feinstrukturforschung, 10 Jan. 2002.
2. Miran Čeh
   Microscopic investigation of AO (A=Sr,Ca,Ba) doped perovskite: invited talk
   Shanghai, Shanghai Institute of Ceramics, Chinese Academy of Sciences, 6 Dec. 2002.
3. Nina Daneu
   Grain growth control in Sb_2O_3-doped ZnO: invited talk
   Bonn, Institut für Anorganische Chemie, Anorganische Materialforschung, 27 Nov. 2002.
4. Aleksander Rečnik
   The influence of polytypic faulting on exaggerated growth of crystals: invited talk
   Stuttgart, Max-Planck-Institut für Metallforschung, 29 Oct. 2002.
5. Aleksander Rečnik
   Solving the atomic structure of inversion boundaries in Sb-doped ZnO: invited talk
   Stuttgart, Max-Planck-Institut für Metallforschung, 30 Oct. 2002.
6. Aleksander Rečnik
   Solving the atomic structure of inversion boundaries in Sb-doped ZnO: invited talk
   Oxford, Oxford University, Department of Materials, 1 Aug. 2002.

PATENTS

Patent applications

1. No. 200200031
   A process and device for monitoring the solidification of aqueous ceramic suspensions in closed molds
   Sasa Novak, Srečo Maček, Goran Dražič
   Slovenian Intellectual Property Office, Ljubljana, Slovenia, 2002

THESIS

B. Sc. Thesis

1. Ferenc Király: The influence of the dipolar interaction on the magnetic anisotropy of nanoclusters (Prof. Peter Prelovšek, Dr. Matej Komelj)

MESS SUPPORTED RESEARCH AND DEVELOPMENT GRANTS AND CONTRACTS

1. NMR measurement of magnetic fields and their biological effects
   Asst. Prof. Spomenka Kobee
2. Novel permanent magnets for high temperature applications
   Dr. Matej Komelj

Research programs

1. Powder metallurgy and intermetallic magnets
   Asst. Prof. Spomenka Kobe
2. Electron microscopy and microanalysis of materials
   Dr. Miran Čeh

INTERNATIONAL PROJECTS

1. Micrometer Scale Patterning of Protein and DNA Chips
   MICROPROTEIN
   G5RD-CT-2002-00744, 5. FP
   EC; Dr. Ion Siotis, National Hellenic Research Foundation, Athens, Greece
   Asst. Prof. Spomenka Kobe
   Dr. Goran Dražič
2. Novel Permanent Magnets for High Temperature Applications
   HITEMAG
   G5RD-CT-2000-00213, 5. FP
   EC; Dr. Dimitris Niarchos, NCSR „Demokritos“, Institute of Materials Sciences, Aghia Paraskevi, Athens, Greece
   Asst. Prof. Spomenka Kobe
   Dr. Paul McGuiness
3. Bonded Magnets Based on RE-TM Nanocrystalline Powders
   NATO SfP - Bonded Magnets, NATO SfP – 972428
   NATO; Dr. Dimitris Niarchos, NCSR “Demokritos”, Institute of Materials Sciences, Aghia Paraskevi, Athens, Greece
   Asst. Prof. Spomenka Kobe
4. ZnO Based Varistors, Doped with Rare Earth Elements
   PROTEUS
   FR-2000-2
   Prof. Bui Ai, Université Paul Sabatier de Toulouse III, Laboratoire de Génie Electrique, Toulouse Cedex, France
   Dr. Slavko Bernik
5. Characterization of Planar Faults and Boundaries on a Sub-nm Scale
   2001/04
   Prof. Wayne D. Kaplan, Technion Israel Institute of Technology, Haifa, Israel
   Dr. Aleksander Rečnik
6. Analysis of Grain Boundaries in Ceramics by HAADF Scanning Transmission Electron Microscopy and Cathodoluminiscence Microscopy
   SLO-JAP-01/03
   Prof. Hiroshi Saijo, Kyoto Institute of Technology, Kyoto, Japan
   Dr. Miran Čeh
7. Analytical Electron Microscopy of Interfaces in Ceramic Materials
   04-03
   Dr. Gu Hui, Shanghai Institute of Ceramics, Chinese Academy of Sciences, Shanghai, China
   Dr. Miran Čeh
8. Resistive Exhaust Gas Sensors on the Basis of Temperature-Independent Semiconducting Oxides; Electron Microscopy Investigations of $SrTiO_3$-Based Perovskites
   Dr. Wolfgang Menesklou, Universität Karlsruhe, Institut für Werkstoffe der Elektrotechnik (IWE), Karlsruhe, Germany
   Dr. Miran Čeh
9. Zinc Oxide Based Varistor Ceramics
   SVN 99/021
   Prof. Werner Mader, Universität Bonn, Institut für Anorganische Chemie, Bonn, Germany
   Dr. Aleksander Rečnik
10. Bonded Magnets Based on RE-TM Nanocrystalline Powders
    SVN 99/020
    Dr. K.-H. Müller, Dr. Oliver Gutfleisch, IFW Dresden, Institut für Festkörper und Werkstofforschung, Dresden, Germany
    Asst. Prof. Spomenka Kobe
    Dr. Paul McGuiness
11. Interfaces in Ceramics
    SVN 99/026
    Prof. Manfred Rühle, Max-Planck-Institut für Metallforschung, Stuttgart, Germany
    Dr. Miran Čeh
12. Cohesive Powder Fluidization Via Magnetic Excitation
    SLO-US-2001/36
    Prof. James F. Klausner, University of Florida, Gainesville, Florida, USA
    Asst. Prof. Spomenka Kobe
13. Electron Probe Microanalysis of Ceramic Materials – II
    SLO-US-2001/49
    Dr. Ryna Marinenko, National Institute of Standards and Technology (NIST), Surface and Microanalysis Science Division, Gaithersburg, MD, USA
    Dr. Slavko Bernik

NEW CONTRACTS SIGNED

1. Varistors for miniaturised and integrated search-protection devices
   VARSI d.o.o. Ljubljana
   Dr. Slavko Bernik
2. VARESTER: Miniaturised high-voltage arrester
   VARSI d.o.o. Ljubljana
   Dr. Slavko Bernik
3. MICROPROTEIN: Micrometer Scale Patterning of Protein and DNA Chips
   National Hellenic Research Foundation - NHRF, Theoretical and Physical Chemistry Institute, Athens, Greece
   Asst. Prof. Spomenka Kobe, Dr. Goran Dražič
4. Resistive Exhaust Gas Sensors on the Basis of Temperature-Independent Semiconducting Oxides
   Universität Karlsruhe, Institut für Werkstoffe der Elektrotechnik - IWE, Karlsruhe, Germany Dr. Miran Čeh
5. Morphology studies of active substances and pharmaceuthical products
   Lek d.d. Ljubljana
   Dr. Miran Čeh

VISITORS FROM ABROAD

1. Dr. Oliver Gutfleisch, Institut für Festkörper und Werkstofforschung – IFW, Dresden, Germany, January 24 - 27, 2002
2. Rahmati Behnaz, B. Sc., Max-Planck-Institut für Metallforschung, Stuttgart, Germany, January 26 - February 1, 2002
3. Prof. Bui Ai, Université Paul Sabatier, Laboratoire de Génie Éléctrique, associé au CNRS, Toulouse, France and Dr. Nguyen The Hung, Hanoi University of Technology, Hanoi, Vietnam, April 11 - 15, 2002
4. Prof. Constantinos Cefalas, National Hellenic Research Foundation - NHRF, Theoretical and Physical Chemistry Institute, Athens, Greece, April 27 - May 1, 2002
5. Dr. Ryna B. Marinenko, National Institute for Standards and Technology - NIST, Surface and Microanalysis Science Division, Gaithersburg, Maryland, USA, September 6 - 14, 2002
6. Prof. Makoto Shiojiri and Prof. Saijo Hiroshi, Kyoto Institute of Technology, Kyoto, Japan, September 27 - October 4, 2002
7. Takashi Yamazaki, B.Sc., Tokyo University of Science, Tokyo, Japan, September 28 - October 4, 2002
8. Dr. Eamonn Devlin, National Centre for Scientific Research – NCSR Demokritos, Athens, Greece, October 13 - 16, 2002
9. Prof. Bui Ai, Université Paul Sabatier, Laboratoire de Génie Éléctrique, associé au CNRS, Toulouse, France, October 17 - 18, 2002
10. Prof. Constantinos Cefalas, National Hellenic Research Foundation - NHRF, Theoretical and Physical Chemistry Institute, Athens, Greece, October 21 - 25, 2002
11. Elena Tchernychova, B.Sc., Max-Planck-Institut für Metallforschung, Stuttgart, Germany, November 17 - 30, 2002

12. Prof. Wayne D. Kaplan, Technion – Israel Institute of Technology, Department of Materials Engineering, Haifa, Israel, December 1 - 8, 2002
13. Amir Avishai, B.Sc., Technion – Israel Institute of Technology, Department of Materials Engineering, Haifa, Israel, December 1 - 14, 2002
14. Yaron Kauffmann, B.Sc., Technion - Israel Institute of Technology, Department of Materials Engineering, Haifa, Israel, December 1 - 22, 2002

ORGANIZATION OF CONFERENCES AND MEETINGS

1. 17th International Workshop on Rare Earth Magnets and Their Applications, 12th Symposium on Magnetic Anisotropy and Coercivity in RE-TM Alloys, August 18 - 22, 2002, Newark, Delaware, USA (membership in International Advisory Committee)
2. 15th International Congress on Electron Microscopy, ICEM 15, Durban, South Africa, September 1 - 6, 2002 (co-organization)
3. 10th Conference on Materials and Technologies, Portorož, Slovenia, November 13 - 15, 2002 (co-organization)

STAFF

Researchers

1. Dr. Slavko Bernik
2. Dr. Miran Čeh**
3. Dr. Goran Dražić
4. **Asst. Prof. Spomenka Kobe**, Head**
5. Dr. Paul John McGuiness
6. Dr. Aleksander Rečnik

Postdoctoral associates

7. Dr. Matej Komelj
8. Dr. Boris Saje***

Postgraduates

9. Nina Daneu, B. Sc.
10. Vesna Šrot, B. Sc.
11. Sašo Šturm, B. Sc.
12. Kristina Žužek Rožman, M. Sc.

Technical officers

13. Medeja Gec, B. Sc.
14. Zoran Samardžija, B. Sc.

Technical and administrative staff

15. Sanja Fidler, B. Sc.
16. Anton Porenta, Eng.

** Part-time faculty member
*** Member of industrial or other organisation