

2001 ANNUAL REPORT



response to the rapidly expanding fields of nanotechnology and nanomaterials. All the members of the new department were previously members of Ceramics Department (K5), which was reorganised into five new units: four departments and a Centre for Electron Microscopy. The basic research in the Department for Nanostructured Materials

department at the Jožef Stefan Institute. It was formed in 2001 as a

The Department for Nanostructured Materials (K7) is a new

Head: A/Prof. Spomenka Kobe

The basic research in the Department for Nanostructured Materials focuses on inorganic materials with specific physical properties that are a consequence of their structural and chemical phenomena at the nanostructural and atomic levels. The fields of research involve natural and

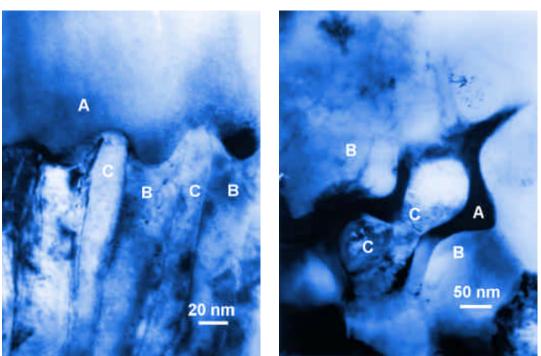
manufactured ceramic materials as well as metals and intermetallic compounds. The basis of the research is to find relationships between the physical properties of a material and its structural and chemical properties by using electron microscopy techniques to reveal phenomena on the nanoscale. Macroscopic phenomena, for example, phase transformations, phase equilibria, polytypisim, polymorphism, crystal growth and the development of the microstructure are areas of particular interest.

The research program in the field of intermetallic materials continued to build on the research of previous years with the emphasis on high-energy permanent magnets combined with the broader area of powder metallurgy. Research and development was concentrated on the following rare-earth transition-metal (RE-TM) permanent-magnet alloys: RETM₅, RE₂TM₁₇, RE₂TM₁₄B and the interstitially modified RE2TM17N3-d, all of which exhibit exceptional magnetic properties, enabling miniaturisation in many areas of their application. The use of these magnets is growing exponentially with the developments in telecommunications, computers, medicine, etc. The research into magnetic materials based on intermetallic alloys involves studies of ecologically acceptable methods for the preparation of nanocrystalline powders that can be used as a basic material for bonded magnets. It also involves the study of the rheological properties of nanocrystalline powders. By using a monomolecular layer of organic substance as a coating material the flowability of the powders was substantially

We proved the influence of planar faults and polytypic sequences on exaggerated grain growth of polycrystalline oxide materials. We developed a special analytical method with high precision for chemical composition determination of a single atomic layer (collaboration with University Bonn). Nanocrystalline magnetic powders based on RE-Fe-B with a

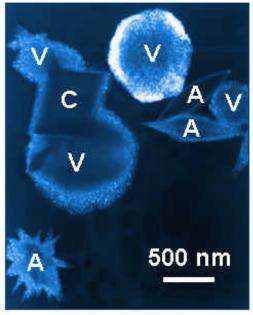
composition designed to induce highly anisotropic (87%) grain growth were prepared for bonded magnets. We reduced the size of 3 kV varistors for 30% with preserved electrical properties. As part of an EU5 project we designed and built a new closed-loop magnetic measuring system for determining permanent-magnet properties at temperatures over 400°C. This new device will allow the non-destructive characterisation of magnets at high temperatures.

improved, which is of great importance for industrial applications. The result of optimising the chemical composition and the HDDR processing of $RE_2Fe_{14}B$ -based alloys was a highly anisotropic high-coercivity powder. Using TEM we were able to study the influence of the processing on the grains, the grain boundaries and the phase boundaries in the final microstructure, and the influence of these factors on the magnetic properties. We also made a study of the influence of various additives like Zr and Ta on the development of microstructure and the induced anisotropy in the $RE_2TM_{17}N_{3-d}$ powders processed by the HDDR technique. Analytical microscopy is one of the few methods that allow us to make such studies and due to the complicated and demanding preparation of samples for the TEM and EDXS analyses from agglomerated nanocrystalline magnetic powders we needed to develop a special preparation method. As part of an EU 5th FW project we developed and constructed a device to measure the magnetic properties of permanent magnets up to 450°C in a closed magnetic circuit, a very important achievement in the area of high-temperature RE_2TM_{17} sintered magnets. Much of the department's research is linked to international projects, these include: NATO SfP, EU 5th FW, and bilateral projects with IFW, Dresden and the University of Florida, Gainesville.

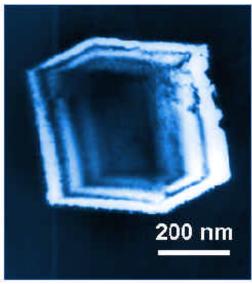


(a) TEM bright field image of partially disproportionated alloy (decomposition of Nd-Fe-B grain), (b) partially recombined Nd-Fe-B crystallite (around the NdH₂ phase) during the HDDR processing of nanocrystalline Nd-Fe-B powders (A: Nd-Fe-B, B: aFe, C: NdH₂)

The research in magnetic materials is supported by theoretical studies within the framework of ab-initio electron-structure calculations using the full-potential linearized-augmented-plane-wave (FLAPW) method. The results of these investigations are recognized as being among the leading achievements in this field. The research and development work that is financially supported by Termoelektrarna-Toplarna, Ljubljana, was continued in cooperation with a group at the Faculty of Natural Science. Using X-ray diffraction analyses, zeta-potential measurements and HRTEM analyses we systematically followed the influence of applied magnetic fields on the crystallization form of CaCO₃.



TEM micrograph (dark field) of particles of Calcite (C), Aragonite (A) and Vaterite (V) found in magnetically treated water samples.

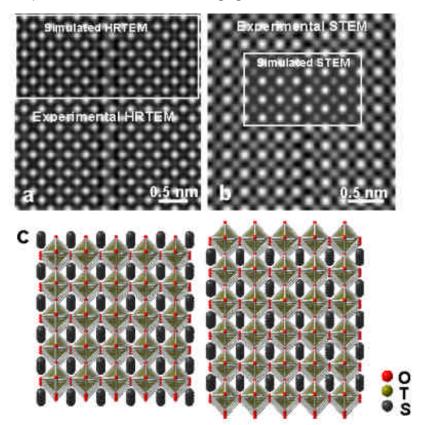


TEM micrograph (dark field) of Calcite particle in magnetically treated water samples.



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. Our recent results showed that exaggerated grain growth in polycrystalline materials could be induced by polytypic sequences in crystals. Our investigations of the phenomenon of exaggerated grain growth have led us to a systematic study of the grain growth in various ceramic materials, with particular reference to perovskites. Using atomic-resolution transmission electron microscopy we showed that, as a rule, the exaggerated grains contain polytypic faults. These faults can be either isolated or in the form of ordered polytypic sequences. The structure and chemical composition of these faults unambiguously showed that such polytypic sequences in the crystals are the result of an early phase transformation between the phase and the dopant. Faults of this type can only be observed in the systems where a secondary polytypic phase exists between the main phase and the dopant. Such a polytypic phase decomposes incongruently to a liquid phase and a different polytype with a lower ordering sequence. In the past, most of the research work of the group was aimed at structural investigations of the faults in crystals that can induce exaggerated grain growth only in a very narrow thermodynamic regime. This phenomenon enables us to control the final microstructure in many technologically important ceramic oxide systems.

In SrTiO₃ and CaTiO₃ perovskites with an AO excess (A=Ca,Sr,Ba) we investigated the chemical composition of polytypic phases and isolated planar faults by scanning-transmission electron microscopy (STEM) and incoherent Z-contrast imaging.

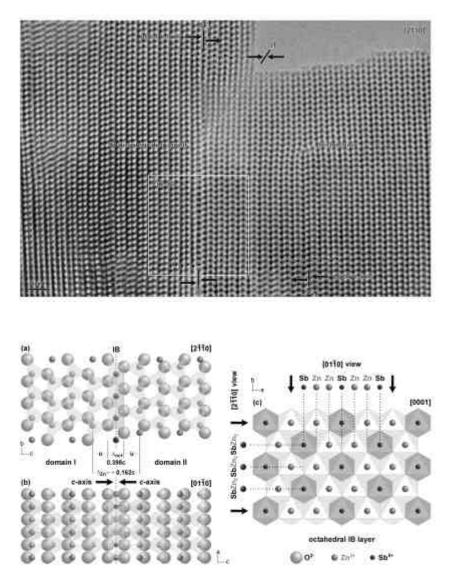


(a) Experimental and simulated HRTEM image and (b) experimental and simulated HAADF STEM image of a single Ruddlesden-Popper fault in SrO-doped SrTiO3 in [001] zone axis. (c) The structural model of the observed planar fault.

We developed suitable image-processing algorithms that enabled us to quantitatively correlate the observed images with the calculated images. We discovered that the dopant with the lower atomic number (Z) is always concentrated at the planar faults. Also, the mechanism of nucleation and growth of polytypic phases and planar faults in nonstoichiometric perovskites was determined.



In the ZnO-Sb₂O₃ system we studied the structural conditions for the formation of planar faults that induce exaggerated grain growth in ZnO. The atomic structure of these so-called inversion boundaries was studied by transmission electron microscopy. With electron microdiffraction and high-resolution transmission electron microscopy (HRTEM) we determined the structure of the inversion boundaries. For the chemical analysis of the mono-atomic layer at the inversion boundary, which apart from Zn atoms also contains Sb atoms, we have developed, in collaboration with the University in Bonn, a special analytical method for determining extremely low elemental concentrations at planar faults or grain boundaries. The method has a precision of 0.5%, which is two orders of magnitude better than commonly used techniques of comparable spatial resolution. The reconstruction of structural and chemical information obtained from the inversion boundary confirmed that these planar faults have the composition ZnSb₂, where the Zn and Sb atoms are completely ordered into a superstructure with 3m symmetry.



Reconstruction of the inversion boundary in Sb₂O₃-doped ZnO (published in J. Am. Ceram. Soc., **84**, 2001, 2657-2668, A. Recnik, N. Daneu, T. Walther, W. Mader, Structure and chemistry of basal-plane inversion boundaries in antimony oxide-doped zinc oxide)

These results led us to a systematic investigation of microstructure developments in ZnO doped with extremely small concentrations of Sb^{+3} , i.e. from 0 to a few 100 ppm. We found that even a very small amount of Sb^{3+} caused the formation of inversion boundaries that determine the subsequent ZnO microstructure development. The grains that contain inversion boundaries grew at

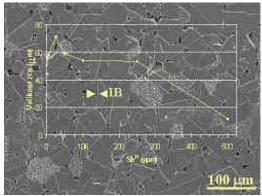


the expense of the grains without inversion boundaries until the grains with the inversion boundaries came to dominate the microstructure.

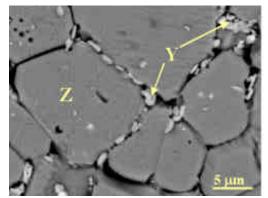
The normal grains in ZnO ceramics grew with a growth exponent of 3. The grains with the inversion boundaries have a similar growth exponent that is independent of the amount of added Sb^{+3} . As long as there are normal grains with inversion boundaries present, they grow faster, with the exponent 2. When only the grains with the inversion boundaries are present their growth is hindered and the growth exponent reaches a value of 4. When the Sb⁺³ additions exceed 250 ppm, a fine-grained spinel phase inhibits the grain growth.

In ZnO-based varistor materials doped with rareearth oxides (REOs) we have studied the influence of the ratio of Sb₂O₃ to Bi₂O₃/Sb₂O₃ on the microstructure and the electrical properties of ZnO ceramics doped with Y_2O_3 .

The applied research with VARSI (formerly Iskra VARISTOR) was aimed at developing varistor blocks with a diameter and height of 40 mm for medium-voltage arrester applications within the EKOVARESTER project.



Influence of the amount of added Sb^{3+} on the grain size of ZnO ceramics, fired at 1200° C. In the background of the graph backscattered electron (BE) image of the microstructure of ZnO sample doped with 100ppm Sb^{3+} is given. Inversion boundaries (IB) are evident in all ZnO grains.



In the Y_2O_3 -doped ZnO- Bi_2O_3 -based varistor ceramics fine-grained Bi-Zn-Sb-Y-O-type phase (Y) uniformly distributed along the grain boundaries of ZnO (Z) contributes to the effective inhibition of grain growth which results in the fine-grained varistor ceramics with increased threshold voltage.

The starting varistor composition was modified in terms of Co, Mn and Cr content. By optimizing the sintering process in terms of the burnout of the binder, as well as the sintering regime, we have achieved better final electrical properties for the material. The obtained results enabled the online production of medium-voltage

varistors. Within the R&D phase of the project "Miniaturization of the energy varistors for 3kV voltages" we have improved the standard starting composition and the sintering procedure and increased the breakdown voltage by a factor of 1.5 to 2.



ZnO based varistor blocks developed in colaboration between IJS and VARSI and produced by VARSI.

In 2001 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,O2), National Institute of Chemistry, Faculty of Pharmacy, Faculty for Natural Sciences, Slovenian Restoration Center, Lek d.d., DONIT TESNIT d.d., BIA Separations d.o.o., Swaty d.d., FERITI d.d., Akripol d.d., Mehanika d.d., Magneti d.d. and Premogovnik Velenje d.d..

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STAFF

Researchers

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Postdoctoral Associates

Dr. Matej Komelj Dr. Boris Saje**

*engaged in pedagogical process at the University of Ljubljana **part-time employment

Ph.D. Students

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Technical Officers

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

Technical and Administrative Staff Sanja Fidler Anton Porenta, Eng.

B.Sc. THESIS

Janez Bernard: Influence of inversion boundaries on grain growth in ZnO ceramic doped with Sb₂O₃" (Prof. Stanko Pejovnik, Dr. Slavko Bernik)

VISITORS FROM ABROAD

- 1. Dr. Eamonn Devlin, National Centre for Scientific Research NCSR Demokritos, Athens, Greece, January 16 21, 2001
- 2. Dr. Christina Scheu in Klaus van Benthem, Dipl. Phys., Max-Planck-Institut für Metallforschung, Stuttgart, Germany, March 3 - 10, 2001
- 3. Dr. Oliver Gutfleisch, Institut für Festkörper und Werkstofforschung IFW, Dresden, Germany, April 9 20, 2001
- 4. Prof. Povl Olgaard, Risoe National Laboratory, Roskilde, Denmark, and Dr. Paul R. Jay, University of Ottawa, Faculty of Engineering, Ottawa, Canada, April 23, 2001
- 5. Dr. Günter Möbus, University of Oxford, Oxford, United Kingdom, May, 19 27, 2001
- 6. Prof. Bui Ai, Université Paul Sabatier, Laboratoire de Génie Éléctrique, Associé au CNRS, Toulouse, France, June 30 July 7, 2001
- 7. Dr. Masahiro Kawasaki, Jeol USA, Boston, USA, July 7 14, 2001
- 8. Dr. Thomas Walther, Institut für Anorganische Chemie, Landenszentrum für Hochleistungs-Elektronenmikroskopie NRW, Universität Bonn, Bonn, Germany, July 21 - 27, 2001
- Prof. Makoto Shiojiri, Kyoto Institute of Technology, Kyoto, Japan, Prof. Kazuto Watanabe and Takashi Yamazaki, Dipl. Ing., Tokyo Metropolitan College of Technology, Tokyo, Japan, August 24 - 30, 2001
- 10. Dr. Dimitris Niarchos and Dr. Eamon Devlin, National Centre for Scientific Research NCSR Demokritos, Athens, Greece, August, 4 5, 2001

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- 11. Prof. Wayne D. Kaplan, Technion Israel Institute of Technology, Haifa, Israel, August 19 31, 2001
- 12. Prof. Constaninos Cefalas, National Hellenic Research Foundation NHRF, Theoretical and Physical Chemistry Institute, Athens, Greece, August 27 31, 2001
- 13. Dr. Albir A. Layyous, Iscar Ltd., Materials and Ceramics Engineering, Tefen, Israel, September 10, 2001
- 14. Prof. Saijo Hiroshi and Prof. Toshiyuki Isshiki, Kyoto Institute of Technology, Kyoto, Japan, September 30 – October 5, 2001
- 15. Prof. Hui Gu, Shanghai Institute of Ceramics, Chinese Academy of Sciences, Shanghai, China, November 1 10, 2001
- 16. Dr. Wilfried Sigle, Max-Planck-Institut für Metallforschung, Stuttgart, Germany, November 13 21, 2001
- Dr. Evangelija Sarantopoulou, National Hellenic Research Foundation NHRF, Theoretical and Physical Chemistry Institute, Short Light Wavelengths Nanoapplications Laboratory, Athens, Greece, November 17 - 23, 2001
- 18. Dr. Alexander Loewe, Universität Bonn, Institut für Anorganische Chemie, Bonn, Germany, November 20 29, 2001
- 19. Yaron Kauffmann, Dipl.Ing., Amir Avishai, Dipl.Ing. and Tzipi Cohen, Dipl.Ing., Technion Israel Institute of Technology, Haifa, Israel, December 8 - 22, 2001

ORGANISATION OF CONFERENCES, CONGRESSES AND MEETINGS

- 1. 9th Conference on Materials and Technologies, Portorož, Slovenia, November 14 16, 2001 (coorganisation)
- 5th Multinational Congress on Electron Microscopy MCEM, Lecce, Italy, September 20 26, 2001 (co-organisation)
- 17th International Workshop on Rare Earth Magnets and Their Applications, 12th Symposium on Magnetic Anisotropy and Coercivity in RE-TM Alloys (membership in International Advisory Committee)



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BASIC RESEARCH PROJECTS

Powder metallurgy and intermetallic magnets P0-0506-0106/01 A/Prof. Spomenka Kobe

Electron microscopy and microanalysis of materials P0-0509-0106/01 Dr. Miran Ceh

Microstructural and surface analysis of ceramic materials J2-7613-0106/01 Dr. Slavko Bernik

Formation of septarian nodules near Gornji Štrihovec T1-0592-0106/01 Dr. Aleksander Recnik

Electron microscopy of the planar faults and boundaries in ceramics J2-0543-0106/01 Dr. Miran Ceh

NMR measurement of magnetic fields and their biological effects J2-2264-0106/01 A/Prof. Spomenka Kobe

Novel permanent magnets for high temperature applications J2-3505-0106/01 Dr. Matej Komelj

INDUSTRIAL PROJECTS

Physical treatment of sanitary and cooling water Termoelektrarna Toplarna Ljubljana Asst. Prof. Spomenka Kobe

EKOVARESTER: ZnO varistor blocks with improved energy characteristics for new generation arresters – with consideration of environment, reliability and stability of electric network Iskra Varistor, Ljubljana Dr. Slavko Bernik

Miniaturisation of the energy varistors for 3 kV voltages Iskra Varistor, Ljubljana Dr. Slavko Bernik



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INTERNATIONAL COOPERATION

Mutilateral

Bonded Magnets Based on RE-TM Nanocrystalline Powders (NATO SfP - Bonded Magnets), NATO SfP - 972428, 3311-01-837002 1999 - 2002 EC; Dr. Dimitris Niarchos, NCSR "Demokritos", Institute of Materials Sciences, Aghia Paraskevi, Attikis, Greece

A/Prof. Spomenka Kobe

HITEMAG: Novel permanent magnets for high temperature applications G5RD-CT-2000-00213 (5. FP) EC; Dr. Dimitris Niarchos, NCSR "Demokritos", Institute of Materials Sciences, Aghia Paraskevi, Attikis, Greece 2000-2003 **A/Prof. Spomenka Kobe**

Bilateral

Interfaces in Ceramics (SLO-026-99, SVN 99/026) 1999 - 2002 Prof. Manfred Rühle, Max-Planck-Institut für Metallforschung, Stuttgart, Germany **Dr. Miran Ceh**

Zinc Oxide Based Varistor Ceramics (SLO-021-99, SVN 99/021) 1999 - 2002 Prof. Werner Mader; Universität Bonn, Institut für Anorganische Chemie, Bonn, Germany **Dr. Aleksander Recnik**

Bonded Magnets Based on RE-TM Nanocrystalline Powders (SLO-020-99, SVN 99/020) 1999 - 2002 Dr. K.-H. Mueller, Dr. Oliver Gutfleisch, IFW Dresden, Intstitut für Festkörper und Werkstofforschung, Dresden, Germany A/Prof. Spomenka Kobe Dr. Paul McGuiness

Cohesive Powder Fluidization Via Magnetic Excitation (SLO-US-2001/36) January 2001 – December 2002 Prof. James Klausner, Department of Mechanical Engineering, University of Florida, Gainesville, Florida, USA **A/Prof. Spomenka Kobe** Electron Probe Microanalysis of Ceramic Materials (SLO-US-008)

1999 - 2001

dr. Ryna Marinenko, National Institute of Standards and Technology, Surface and Microanalysis Science Division (NIST), Gaithersburg, Maryland, USA

Dr. Miran Ceh

Electron Probe Microanalysis of Ceramic Materials - II (SLO-US-2001/49)

2001 - 2003

Dr. Ryna Marinenko, National Institute of Standards and Technology, Surface and Microanalysis Science Division (NIST), Gaithersburg, Maryland, USA

Dr. Slavko Bernik

PROTEUS – ZnO Based Varistors, Doped with Rare Earth Elements 2000-2002 Prof. Bui Ai, Université Paul Sabatier de Toulouse III, Laboratoire de Génie Électrique, Toulouse Cedex, France **Dr. Slavko Bernik**

Analytical electron microscopy of interfaces in ceramic materials (KIT 04-03/2002) 2000-2002 Dr. Gu Hui, Shanghai Institute of Ceramics, Chinese Academy of Sciences, China **Dr. Miran Ceh**

Analysis of Grain Boundaries in Ceramics (SLO-JAP-01/03) Kyoto Institute of Technology, Kyoto, Japan April 2001 – March 2003 **Dr. Miran Ceh**

Characterization of Planar Faults and Interfaces on a Sub-Nanometer Scale (SLO-IZR-2001/04) August 2001 – December 2002 Technion - Israel Institute of Technology, Haifa, Israel **Dr. Aleksander Recnik**



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- Marko Hrovat, Janez Holc, Zoran Samardžija, Darko Belavic The influence of firing temperature on gauge factors and the electrical and microstructural characteristics of thick-film resistors In: J. mater. sci. lett., Vol. 20, pp. 701-705, 2001.
- Marko Hrovat, Zoran Samardžija, Janez Holc, Darko Belavic Microstructural and electrical characteristics of some "overfired" thick-film resistors In: J. mater. sci. lett., Vol. 20, pp. 347-351, 2001.
- Saša Javoric, Goran Dražic, Marija Kosec A study of the crystallization of CSD-prepared La_{0.5}Sr_{0.5}CoCO₃ thin films using analytical electron microscopy

In: J. Eur. Ceram. Soc., Vol. 21, pp. 1543-1546, 2001.

- Leon Kaluža, Boris Orel, Goran Dražic, M. Kohl Sol-gel derived CuCoMnO_x spinel coatings for solar absorbers: structural and optical properties In: Sol. energy mater. sol. cells, Vol. 70, no. 2, pp. 187-201, 2001.
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- Spomenka Kobe, Goran Dražic, Paul J. McGuiness, Janez Stražišar The influence of the magnetic field on the crystallisation form of calcium carbonate and the testing of a magnetic water-treatment device In: J. magn. magn. mater., Vol. 236, pp. 71-76, 2001.
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- Marija Kosec, Barbara Malic, Janez Holc, Marko Hrovat, Mira Mandeljc, Andreja Bencan, Zoran Samardžija, Goran Dražic Interface reactions among electrodes, substrates and Pb(Zr,Ti)O₃-based films In: Acta chim. slov., Vol. 48, pp. 51-62, 2001.
- 16. Marija Kosec, Darja Murko, Janez Holc, Barbara Malic, Miran Ceh, Tilo Hauke, Horst Beige Low-temperature processing of (Pb,La)(Zr,Ti)O₃ thick films on alumina substrates: dedicated to Professor Dr. Drago Kolar in memory of this brilliant scientist and teacher In: Z. Met.kd., Vol. 92, pp. 97-104, 2001.

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17. Danjela Kušcer, Slavko Bernik, Janez Holc Subsolidus phase relations in the La₂O₃-Fe₂O₃-Al₂O₃ system

In: J. mater. res., Vol. 16, pp. 822-827, 2001.

- Paul J. McGuiness, Spomenka Kobe, I. Škulj, A. Bollero, O. Gutfleisch, E. J. Devlin, D. Niarchos Coercivity variations with Pr- and Zr-substituted NdDyFeB-based HDDR powders In: J. magn. magn. mater., Vol. 237, pp. 267-275, 2001.
- 19. Paul J. McGuiness, Spomenka Kobe, Irena Škulj Adapting the HDDR process and NdFeB-based permanent-magnet alloys for factory production In: Mater. tehnol., Vol. 35, pp. 231-236, 2001.
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- 22. Aleksander Recnik Twins in barium titanate In: Acta chim. slov., Vol. 48, no. 1, pp. 1-50, 2001.
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- 27. Angela Šurca Vuk, Boris Orel, Goran Dražic IR spectroelectrochemical studies of Fe₂V₄O₁₃, FeVO₄ and InVO₄ thin films obtained via the solgel synthesis In: J. Solid State Electrochem., Vol. 5, no. 7/8,

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28. Nguyen The Hung, Nguyen Dinh Quang, Slavko Bernik Electrical and microstructural characteristics of $ZnO\text{-}Bi_2O_3\text{-}based$ varistors doped with rare-earth exides

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Guest Lectures

- Miran Ceh Electron microscopy studies of planar faults in perovskite ceramic materials: invited talk Kyoto, Murata Manufacturing Co. Ltd., 12 Oct. 2001.
- Miran Ceh High-resolution HAADF STEM imaging of planar faults in CaTiO₃ and Sr TiO₃: invited talk Haifa, Technion-Israel Institute of Technology, Faculty of Materials Engineering, 25 Nov. 2001.
- Matej Komelj Magnetoelastic effects in epitaxial magnetic films: a combination of phenomenological theory of magnetoelasticity with ab initio calculations:

invited talk at 258. WE-Heraeus-Seminar "Electronic origin of magnetoelastic anisotropy and stress in atomic layers, Schloß Ringberg, Tagernsee, Germany, Sep. 9-12, 2001, Halle, Max-Panck-Institut für Mikrostrukturphysik, 2001.

- Aleksander Recnik The influence of polytypic faults on exaggerated growth of crystals: [invited talk] Materialwissenschaftliches Seminar Bonn, Institut für Anorganische Chemie, Anorganische Materialforschung, 22 Oct. 2001.
- Aleksander Recnik Solving the atomic structure of inversion boundaries in Sb₂O₃-doped zinc oxide: [invited talk] Materialwissenschaftliches Seminar Bonn, Institut für Anorganische Chemie, Anorganische Materialforschung, 19 Oct. 2001.