

Nanomaterials by Severe Plastic Deformation

Proceedings of the Conference „Nanomaterials by Severe
Plastic Deformation – NANOSPD2“, December 9-13,
2002, Vienna, Austria

Edited by
Michael Zehetbauer and Ruslan Z. Valiev

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Deutsche Gesellschaft
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The cover picture symbolizes the various aspects of current research in the field of severe plastic deformation (SPD). All figures have been taken from papers of the present Proceedings of "NANOSPD2".

Bottom left position

Structure of ultrafine and nanograins developed during High Pressure Torsion of Cu as investigated by Electron Back Scatter Patternning (EBSP). With a hydrostatic pressure of 8 GPa, an von Mises equivalent strain of $\epsilon = 145$ has been reached. The side length of image is 800 nm. (paper by T.Hebesberger, A.Vorhauer, H.P. Stüwe, R.Pippan, p. 447)

Bottom right position

HRTEM micrograph of HPT deformed Ni3Al (Cr,Zr)+B at room temperature up to a shear strain ~ 800 , showing a deformation twin with atomic resolution which is typical of this deformation. The white bar represents a distance of 1nm (paper by Ch.Rentenberger, H.P.Karnthaler, R.Z.Valiev, p. 80)

Right side top

Cold rolling of Cu and Al increases their yield strength but decreases their elongation to failure (ductility). The extraordinary combination of both high strength and high ductility in nanostructured

Cu and Ti processed by SPD clearly sets them apart from coarse-grained metals (paper by R. Z. Valiev, p. 109)

Right side middle

Triple junction between 3 grains, which slide significantly relative to each other because of a macroscopic deformation of 1.3 %. Molecular Dynamics Simulation of a full 3D grain boundary network yields atomic displacement vectors with the colour determined by the magnitude of the displacement (paper by H. Van Swygenhoven, P.M. Derlet, A. Hasnaoui, p. 599)

Left side top

Repetitive Corrugation and Straightening (RCS), a new mode of Severe Plastic Deformation to achieve bulk nanomaterials (paper by N.Tsuiji, Y.Saito, S.H. Lee, Y. Minamino, p. 479)

Left side middle

Strain intensity and distribution for Equal Channel Angular Pressing (ECAP) as calculated by FEM for Aluminium alloy 5083 using the MARC™ code (paper by P.A.Gonzales, C.J.Luis, p. 251)

Background

HRTEM image of nanocrystalline and amorphous phase in Ni-50.3%-Ti after HPT processing with pressure of 6 GPa (paper by T.Waitz, V.Kazykhanov, R.Z.Valiev, H.P.Karnthaler, p. 351)

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Editorial

Dear Reader,

the present Proceedings include the papers of the conference “Nanomaterials by Severe Plastic Deformation – NANOSPD 2” which was held from December 9 to 13, 2002 in Vienna, Austria, at the Institute of Materials Physics of the University of Vienna. As many as 153 participants from 22 nations testify the high interest in the field of Severe Plastic Deformation (SPD) which has been revealing as an attractive tool to achieve ultrafine grained and nanocrystalline materials in bulk shape without defective pores and impurities. The conference offered 15 sessions on themes which spanned the spectrum from general particular physical properties of nanostructured materials to the unique ones of SPD nanomaterials, the modelling of properties and of SPD production of nanocrystalline materials, the peculiarities of nanostructures evolving during SPD investigated by electron optical techniques, X-ray diffraction and other methods, the thermostability of nanocrystalline materials, and a final session on actual and futural applications of nanomaterials from SPD. Each session has been introduced by a keynote and followed by several related oral and poster contributions. In order to guarantee a high scientific level, each contribution was peer reviewed by at least one referee, revised by the authors and finally checked by us. Of course this procedure took some time but we did our best to achieve a certain level of quality without losing too much time in publication procedure which may have been harmful to actuality of the contributions.

As a resume of the scientific outcome of the conference, at first it can be stated that since the first SPD conference held in 1999 in Moscow, nanostructured materials produced by SPD have become an extensively pursued area of research in materials science. Significantly increased research efforts can be observed in modelling and simulation of the different SPD processes, as well as in modelling of the mechanical properties of SPD materials. For the first group of tasks the simulations of strengthening under high hydrostatic pressure, the simulations of strain by several Finite Element Methods, and the texture simulations must be emphasized, while for the second type of tasks very promising simulations by Molecular Dynamics have been introduced. As a partial consequence of the improved modelling capabilities, some progress has been in bringing ECAP to a more industrial style. In this connection, other SPD techniques like Cyclic Channel Die Compression as well as Multiforging and Accumulative Roll-Bonding have been demonstrated to yield very homogeneous UFG and nanostructures. These methods may importantly complement the “classical” ones such as Equal Channel Angular Pressing (ECAP) and Torsion under Elevated Hydrostatic Pressure (HPT) especially in connection with commercial application and/or exploitation. There have been also demonstrated possibilities to combine SPD with ball milling achieving bulk samples with defect free microstructures of even nanometer scale. An increasing number of papers were concerned with the important issue being the thermostability of the SPD nanomaterials. Although this may be worse than that of conventional coarse grained materials, some specific recovery and even recrystallization treatment reveals to be highly beneficial to the ductility of material while keeping the strength on elevated level of nanomaterials. Furthermore, the research of SPD materials more and more turn to solid solutions and precipitation alloys which – in comparison with pure metals – show much smaller grain sizes as well as a markedly higher thermostability achievable by the application of SPD. There

are still left some open questions like “how is fragmentation really going on ?”, “what are the real reasons for enhanced ductility of SPD nanomaterials ?”, “which preconditions do allow for grain boundary sliding i.e. superplasticity in SPD nanomaterials ?”, which will be certainly in focus of the SPD research during the next years. Other still important tasks will be to select certain SPD methods for industrial and commercial applications, to scale them up and/or modify them in order to find the most economical solution for a continuous SPD processing.

Following the successful research activities in the field of nanostructured SPD-produced materials, various international and national projects are being proposed in this area. A number of international workshops and symposia will take place in the near future which are related to this research. During the NANO-SPD2 meeting in Vienna, an International Advisory Board (‘Steering Committee’) on NANO-SPD has formed which is supposed to coordinate the activities in the field of SPD nanomaterials. As a tool for this coordination, the Steering Committee has launched a special web-site under www.nanospd.org.

We do not want to close before having expressed our thanks to the Head of Institute of Materials Physics, Prof. Hans Peter Karnthaler for his general support and hospitality, and the provision of lecture halls. We are grateful to Mrs. Renate Seidl for having done most of the secretary work of the conference, and the ladies and gentlemen of the Atominstitut Wien for essential help in organization (Prof. Peter Wobrauschek – general advice, Maria Paukovits – booklets production, and Dipl.Ing. Shokufeh Zamini – homepage). Dr. Erhard Schafner (University of Vienna), Dipl.Ing. Anna Dubravina and Dr. Nariman Enikeev (both Ufa State Aviation Technical University) significantly contributed to the administration of these Proceedings. We should not forget to thank all the referees who kindly followed our requests for substantial help in improving the submitted manuscripts.

Many thanks also go to the sponsors of this conference i.e. the Austrian Federal Ministry of Education, Science and Culture, the Austrian Research Center Seibersdorf, the Vienna Business Agency, and the Department for Culture of the City of Vienna for their financial support which allowed many scientists from Eastern countries to participate in this conference. Last not least, we thank all the participants for coming and having contributed so many good and attractive papers which made possible that NANO-SPD2 became a remarkable success. We also look forward to seeing you at the NANO-SPD3 meeting in Fukuoka, Japan in 2005.

Vienna, September 2003

Michael J. Zehetbauer, Chairman

Ruslan Z. Valiev, Co-Chairman

Content

I Reasons to Use Nanostructured Materials	1
Unique Features and Properties of Nanostructured Materials	3
<i>Hahn, H. Technische Universität Darmstadt, Institute of Materials Science, Darmstadt (D)</i>	
Properties, Benefits and Application of Nanocrystalline Structures in Magnetic Materials	18
<i>Grössinger, R., Sato, R., Holzer, D., Dahlgren, M. Technische Universität Wien, Institut für Festkörperphysik, Wien (A)</i>	
Formation of Nanostructures in Metals and Composites by Mechanical Means	30
<i>Fecht, H.-J. University of Ulm, Center for Micro-and Nanomaterials, Ulm (D)</i>	
Scale Levels of Plastic Flow and Mechanical Properties of Nanostructured Materials	37
<i>Panin, V. E., Panin, A. V., Derevyagina, L. S. Institute of Strength Physics and Materials Science, Tomsk (Rus); Kopylov, V. I. Physical-Technical Institute of National Academy of Sciences of Belarus, Minsk, Belarus; Valiev, R. Z. Institute of Physics of Advanced Materials associated with (USA)TU, Ufa (Rus)</i>	
Hydrogen-Induced Formation of Silver and Copper Nanoparticles in Soda-Lime Silicate Glasses	44
<i>Suszynska, M., Krajczyk, L., Macalik, B. Institute of Low Temperature and Structure Research, Polish Academy of Sciences, Wrocław (Pl)</i>	
II Large Strain Cold Working and Microstructure	53
Equivalent Strains in Severe Plastic Deformation	55
<i>Stüwe, H. P. Erich Schmidt Institute of Materials Science, Austrian Academy of Sciences and Institute of Metal Physics, University of Leoben, Leoben (A)</i>	
Stage IV: Microscopic Or Mesoscopic Effect ?	65
<i>Aldazabal, J., Alberdi, J. M., Sevillano, J. Gil CEIT (Centro de Estudios e Investigaciones Técnicas de Guipúzcoa), and TECNUN (Technological Campus, University of Navarra), San Sebastián (E)</i>	
Deformation Substructure and Mechanical Properties of BCC - Polycrystals	72
<i>Firstov, S. A. Frantsevych Institute for Problems of Materials Science, Kyiv (Ua)</i>	
Micro- and Nanostructures of Large Strain-SPD deformed L12 Intermetallics	80
<i>Rentenberger, C., Karnthaler, H. P. Institute of Materials Physics, University of Vienna, Vienna (A); Valiev, R. Z. Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, Ufa (Rus).</i>	

VIII

The Nature of the Stress-Strain Relationship in Aluminum and Copper over a Wide Range of Strain.....	87
<i>Chinh, N. Q. Eötvös University, Budapest (Hu); Horita, Z. Kyushu University, Fukuoka (J); Langdon, T. G. University of Southern California, Los Angeles (USA).</i>	
Strain Hardening by Formation of Nanoplatelets	95
<i>Han, K., Xin, Y., Ishmaku, A. National High Magnetic Field Laboratory, Florida State University, Tallahassee, Fl (USA)</i>	
Modelling the Draw Hardening of a Nanolamellar Composite: A Multiscale Transition Method.....	101
<i>Krummeich-Brangier, R. Groupe de Physique des Matériaux-UMR CNRS 6634, Université de Rouen (F); Sabar, H., Berveiller, M. Laboratoire de Physique et Mécanique des Matériaux-UMR CNRS 7554, Université de Metz (F)</i>	
III Unique Features of SPD – Microstructure and Properties	107
Paradoxes of Severe Plastic Deformation.....	109
<i>Valiev, R. Z. Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, Ufa (Rus)</i>	
Investigation of Phase Transformations in Nanostructured Materials Produced by Severe Plastic Deformation.....	118
<i>Sauvage, X., Guillet, A., Blavette, D. Groupe de Physique des matériaux, UMR CNRS 6634, Université de Rouen, Saint Etienne du Rouvray (F)</i>	
Structure-Phase Transformations and Properties in Nanostructured Metastable Alloys Processed by Severe Plastic Deformation.....	125
<i>Stolyarov, V. V., Valiev, R. Z. Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, Ufa (Rus)</i>	
On the Influence of Temperature and Strain Rate on the Flow Stress of ECAP Nickel	131
<i>Hollang, L., Thiele, E., Holste, C. Institut für Physikalische Metallkunde, Technische Universität Dresden, Dresden (D); Brunner, D. Max-Planck-Institut für Metallforschung, Stuttgart (D)</i>	
The Effect of Second-Phase Particles on the Severe Deformation of Aluminium Alloys during Equal Channel Angular Extrusion.	138
<i>Apps, P. J. UMIST, Manchester (GB); Bowen, J. R. Risø National Labs (Dk); Prangnell, P. B. UMIST, Manchester (GB)</i>	
Effects of ECAP Processing on Mechanical and Aging Behaviour of an AA6082 Alloy	145
<i>Bassani, P., Tasca, L., Vedani, M. Politecnico di Milano, Dipartimento di Meccanica, Milano (I)</i>	

Influence of the Thermal Anisotropy Internal Stresses on Low Temperature Mechanical Behavior of Polycrystalline and Nanostructured Ti.....	151
<i>Bengus, V. Z., Smirnov, S. N. B. Verkin Inst. for Low Temperature Physics & Engineering Ukraine Academy of Sciences, Kharkov (Ua)</i>	
Nano- and Submicrocrystalline Structure Formation During High Pressure Torsion of Al-Sc and Al-Mg-Sc Alloys	158
<i>Dobatkin, S. V. Baikov Institute of Metallurgy and Material Science RAS, Moscow (Rus); Zakharov, V. V., Rostova, T. D. All-Russia Institute of Light Alloys, Moscow (Rus); Vinogradov, A. Yu. Osaka City University, Osaka (J); Valiev, R. Z., Krasilnikov, N. A. Ufa State Aviation Technical University, Ufa (Rus); Bastarash, E. N., Trubitsyna, I. B. Moscow State Steel and Alloys Institute (Technological University), Moscow (Rus)</i>	
Phase Transformation in Crystalline and Amorphous Rapidly Quenched Nd-Fe-B Alloys under SPD	165
<i>Gunderov, D. V., Stolyarov, V. V. Institute of Physics of Advanced Materials, USATU, Ufa (Rus); Popov, A. G., Schegoleva, N. N. Institute of Physics of Metals, UrD RAS, Ekaterinburg (Rus); Yavary, A. R. National Polytechnic Institute, Grenoble (F)</i>	
Structure and Functional Properties of Ti-Ni-Based Shape Memory Alloys Subjected to Severe Plastic Deformation.....	170
<i>Khmelevskaya, I. Yu., Trubitsyna, I. B., Prokoshkin, S. D. Moscow Steel and Alloys Institute, Moscow (Rus); Dobatkin, S. V. Moscow Steel and Alloys Institute, Moscow (Rus) and Baikov Institute of Metallurgy and Material Science, Russian Academy of Science, Moscow (Rus); Stolyarov, V. V., Prokofjev, E. A. Ufa State Aviation Technical University, Ufa (Rus)</i>	
Formation of Submicrocrystalline Structure in The Hard Magnetic Alloy Fe-15wt.%Co-25%Cr During Straining by Complex Loading.....	177
<i>Korznikova, G. F., Korneva, A. V. Institute of Metals Superplasticity Problems, Russian Academy of Sciences, Ufa (Rus); Korznikov, A. V. Austrian Research Centres, Seibersdorf Research GmbH and Institute of Metals Superplasticity Problems, Russian Academy of Sciences, Ufa (Rus)</i>	
Experimental Investigations of the Al-Mg-Si Alloy Subjected to Equal-Channel Angular Pressing.....	183
<i>Krallics, G., Szeles, Z. Budapest University of Technology and Economics, Budapest (Hu); Semenova, I. P., Dotsenko, T. V., Alexandrov, I. V. Ufa State Aviation Technical University, Ufa (Rus)</i>	

Mechanical Properties of AZ91 Alloy after Equal Channel Angular Pressing	190
<i>Máthis, K. Department of Metal Physics, Charles University, Prague (Cz) and Department of General Physics, Eötvös University, Budapest (Hu); Trojanová, Z., Lukác, P. Department of Metal Physics, Charles University, Prague (Cz); Lendvai, J. Department of General Physics, Eötvös University, Budapest (Hu); Rauch, E., Mussi, A. Génie Physique et Mécanique des Matériaux, INP Grenoble - ENSPG (F)</i>	
Influence of Microstructural Heterogeneity on the Mechanical Properties of Nanocrystalline Materials Processed by Severe Plastic Deformation	194
<i>Pakiela, Z., Sus-Ryszkowska, M. Faculty of Materials Science and Engineering, Warsaw University of Technology, Warsaw (Pl)</i>	
Creep Behaviour of Pure Aluminium Processed by Equal-Channel Angular Pressing.....	200
<i>Sklenicka, V., Dvorak, J., Svoboda, M. Institute of Physics of Materials, Academy of Sciences of the Czech Republic, Brno (Cz)</i>	
Low Temperature Strain Rate Sensitivity of some Nanostructured Metals	207
<i>Tabachnikova, E. D., Bengus, V. Z., Natsik, V. D., Podolskii, A. V., Smirnov, S. N. B. Verkin Inst. for Low Temperature Physics & Engineering (Ua) Academy of Sciences, Kharkov (Ua); Valiev, R. Z., Stolyarov, V. V., Alexandrov, I. V. Institute of Physics of Advanced Materials, Ufa (Rus)</i>	
IV Modelling of SPD and Mechanical Properties of SPD Materials.....	213
Importance of Disclination in Severe Plastically Deformed Materials	215
<i>Romanov, A. E. Ioffe Physico-Technical Institute, St. Petersburg (Rus)</i>	
Disclination-Based Modelling of Grain Fragmentation during Cold Torsion and ECAP in Aluminium Polycrystals	226
<i>Seefeldt, M., Houtte, P. Van K.U. Leuven, Heverlee (B)</i>	
Modeling of Deformation Behavior and Texture Development in Aluminium under Equal Channel Angular Pressing	233
<i>Baik, S. C. Technical Research Labs., Pohang Iron & Steel Co. Ltd., Pohang (ROK); Estrin, Y., Hellmig, R. J. IWW, TU Clausthal, Clausthal-Zellerfeld (D); Kim, H. S. Department of Metallurgical Engineering, Chungnam National University, Daejeon (ROK) and Technical Research Labs., Pohang Iron & Steel Co. Ltd., Pohang (ROK)</i>	
Process Modeling of Equal Channel Angular Pressing	239
<i>Kim, H. S., Hong, S. I., Lee, H. R., Chun, B. S. Chungnam National University, Daejeon (ROK)</i>	

Deformation Behaviour of ECAP Cu as described by a Dislocation-Based Model.....	245
<i>Enikeev, N. A. Institute of Mechanics, Ufa Science Centre, RAS, Ufa (Rus);</i>	
<i>Alexandrov, I. V. Institute for Physics of Advanced Materials (USA)TU, Ufa (Rus);</i>	
<i>Hong, S. I., Kim, H. S. Chungnam National University, Taejon (ROK)</i>	
Severe Plastic Deformation by ECAP in an Acommercial Al-Mg-Mn-Alloy	251
<i>González, P. A., Luis, C. Departamento de Ingeniera Mecánica, Energética y</i>	
<i>Materiales, UPNA, Campus de Arrosadía, Pamplona (E)</i>	
Evolution of Mechanical and Microstructural Properties of ECAP Deformed Copper	257
<i>Hellmig, R. J., Estrin, Y. Technische Universität Clausthal, Clausthal-Zellerfeld</i>	
<i>(D); Bowen, J. R, Juul Jensen, D. Center for Fundamental Research: Metal</i>	
<i>Structures in Four Dimensions, Roskilde (Dk); Baik, S. C. Pohang Iron & Steel</i>	
<i>Co. Ltd., Pohang (ROK); Kim, H. S., Seo, M. H. Chungnam National University,</i>	
<i>Daejeon (ROK)</i>	
A Composite Grain Model of Strengthening for SPD Produced UFG Materials.....	263
<i>Kozlov, E. V., Popova, N. A., Koneva, N. A. Tomsk State University of Architecture</i>	
<i>and Building, Tomsk (Rus); Zhdanov, A. N. Altai State Technical University,</i>	
<i>Barnaul (Rus)</i>	
Computer Simulation of Equal-Channel Angular Pressing of Tungsten by Means of the Finite Element Method	271
<i>Krallics, G. Budapest University of Technology and Economics, Budapest (Hu);</i>	
<i>Budilov, I. N., Alexandrov, I. V., Raab, G. I., Zhernakov, V. S., Valiev, R. Z. Ufa</i>	
<i>State Aviation Technical University, Ufa (Rus)</i>	
V Texture Evolution and Simulation During SPD.....	279
Texture Evolution in Severe Plastic Deformation by Equal Channel Angular Extrusion.....	281
<i>Tóth, L. S. Laboratoire de Physique et Mécanique des Matériaux, Université</i>	
<i>de Metz (F); Rauch, E. F., Dupuy, L. Génie Physique et Mécanique des</i>	
<i>Matériaux (GPM2), Institut National Polytechnique de Grenoble ENSPG,</i>	
<i>Saint-Martin d'Hères (F)</i>	
Textural Evolution during Equal Channel Angular Extrusion versus Planar Simple Shear	297
<i>Rauch, E. F. and Dupuy, L. Génie Physique et Mécanique des Matériaux</i>	
<i>(GPM2), Institut National Polytechnique de Grenoble ENSPG, Saint-Martin</i>	
<i>d'Hères (F)</i>	
Grain Refinement and Texture Formation during High-Strain Torsion of NiAl.....	303
<i>Skrotzki, W., Klöden, B., Tamm, R., Oertel, C.-G. Institut für Strukturphysik,</i>	
<i>Technische Universität Dresden, Dresden (D); Wcislak, L. HASYLAB at DESY,</i>	
<i>Hamburg (D); Rybacki, E. Geoforschungszentrum Potsdam, Potsdam (D)</i>	

XII

Severely Plastically Deformed Ti from the Standpoint of Texture Changes.....	309
<i>Bonarski, J. Polish Academy of Sciences, Institute of Metallurgy and Materials Science, Krakow (Pl); Alexandrov, I. V. Ufa State Aviation Technical University, Ufa (Rus)</i>	
Development of Crystallographic Texture and Microstructure in Cu and Ti, Subjected to Equal-Channel Angular Pressing.....	315
<i>Bonarski, J., Tarkowski, L. Polish Academy of Sciences, Institute of Metallurgy and Materials Science, Krakow (Pl); Alexandrov, I. V. Ufa State Aviation Technical University, Ufa (Rus)</i>	
VI Details of SPD Nanostructures as Investigated by Electron Microscopy.....	321
Boundary Characteristics in Heavily Deformed Metals	323
<i>Winter, G., Huang, X. Center for Fundamental Research: Metal Structures in 4D, Risø Laboratory (Dk)</i>	
Quantitative Microstructural Analysis of IF Steel Processed by Equal Channel Angular Extrusion.....	332
<i>De Messemaeker, J., Verlinden, B., Van Humbeeck, J., Froyen, L. Katholieke Universiteit Leuven, Leuven (B)</i>	
HRTEM Investigations of Amorphous and Nanocrystalline NiTi Alloys Processed by HPT	339
<i>Waitz, T., Karnthaler, H. P. Institute of Materials Physics, University of Vienna, Vienna (A); Valiev, R. Z. Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, Ufa (Rus)</i>	
Effect of Grain Size on Microstructure Development during Deformation in Polycrystalline Iron.....	345
<i>Kawasaki, K., Hidaka, H., Tsuchiyama, T., Takaki, S. Kyushu University, Fukuoka (J)</i>	
Microstructure and Phase Transformations of HPT NiTi.....	351
<i>Waitz, T., Karnthaler, H. P. Institute of Materials Physics, University of Vienna, Wien (A); Kazykhanov, V. Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, Ufa (Rus)</i>	
Types of Grains and Boundaries, Joint Disclinations and Dislocation Structures of SPD-produced UFG Materials.....	357
<i>Koneva, N. A., Popova, N. A., Ignatenko, L. N., Pekarskaya, E. E., Kozlov, E. V. Tomsk State University of Architecture and Building, Tomsk (Rus); Zhdanov, A. N. Altai State Technical University, Barnaul (Rus)</i>	

Microstructure Development of Copper Single Crystal Deformed by Equal Channel Angular Pressing.....	363
<i>Koyama, T., Miyamoto, H., Mimaki, T. Doshisha University, Kyotanabe (J); Vinogradov, A., Hashimoto, S. Osaka City University, Osaka (J)</i>	
TEM Investigations of Ti Deformed by ECAP	369
<i>Mingler, B., Zeipper, L., Karnthaler, H. P., Zehetbauer, M. Institute of Materials Physics, University of Vienna, Wien (A)</i>	
Microstructural Evolution during Severe Deformation in Austenitic Stainless Steel with Second Phase Particles.....	375
<i>Miura, H., Hamaji, H., Sakai, T. Department of Mechanical Engineering and Intelligent Systems, University of Electro-Communications, Chofu, Tokyo (J)</i>	
Structural Models and Mechanisms for the Formation of High-Energy Nanostructures under Severe Plastic Deformation	381
<i>Tyumentsev, A. N., P. Pinzhin, Yu., Litovchenko, I. Yu., Ovchinnikov, S. V. Institute of Strength Physics and Materials Technology, RAS, Tomsk (Rus); Korotaev, A. D., Ditenberg, I. A., Surikova, N. S., Shevchenko, N. V. Siberian Physicotechnical Institute, Tomsk (Rus); Valiev, R. Z. Institute of Physics of Advanced Materials Ufa State Aviation Technical University, Ufa (Rus)</i>	
Grain Refinement and Microstructural Evolution in Nickel During High-Pressure Torsion	387
<i>Zhilyaev, A. P. Universitat Autònoma de Barcelona, Bellaterra (E); Nurislamova, G. V. Institute for Physics of Advanced Materials, Ufa State Aviation Technical University, Ufa (Rus); Kim, B.-K., Szpunar, J. A. Department of Metals and Materials Engineering, McGill University, Montreal (Cdn); Baró, M. D. Universitat Autònoma de Barcelona, Bellaterra (E); Langdon, T. G. Departments of Aerospace & Mechanical Engineering and Materials Science, University of Southern California, Los Angeles, (USA)</i>	
VII Analyses of SPD Materials by Selected Physical Methods.....	393
The Meaning of Size Obtained from Broadened X-ray Diffraction Peaks.....	395
<i>Ungár, T. Department of General Physics, Eötvös University, Budapest (Hu)</i>	
Ultra Fine Grained Copper Prepared by High Pressure Torsion: Spatial Distribution of Defects from Positron Annihilation Spectroscopy.....	407
<i>Cizek, J., Prochazka, I., Kuzel, R., Cieslar, M. Charles University in Prague, Faculty of Mathematics and Physics, Prague (Cz); Islamgaliev, R. K. Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, Ufa (Rus); Anwand, W., Brauer, G. Institut für Ionenstrahlphysik und Materialforschung, Forschungszentrum Rossendorf, Dresden (D)</i>	

Anelastic Properties of Nanocrystalline Magnesium	413
<i>Trojanová, Z., Lukác, P., Stanek, M. Department of Metal Physics, Charles University, Praha (Cz); Riehemann, W., Weidenfeller, B. Department of Materials Engineering and Technology, Technical University Clausthal, Clausthal-Zellerfeld (D)</i>	
X-ray Peak Profile Analysis on the Microstructure of Al-5.9%Mg-0.3%Sc-0.18%Zr Alloy Deformed by High Pressure Torsion Straining.....	420
<i>Gubicza, J. Department of General Physics, Eötvös University, Budapest (Hu) and Department of Solid State Physics, Eötvös University, Budapest (Hu); Fátay, D., Nyilas, K., Ungár, T. Department of General Physics, Eötvös University, Budapest (Hu); Bastarash, E., Dobatkin, S. Moscow State Steel and Alloys Institute (Technological University), Moscow (Rus)</i>	
Evolution of Microstructure during Thermal Treatment in SPD Titanium	426
<i>Schafner, E. Institute of Materials Physics, University of Vienna (A) and Erich Schmid Institute of Material Science, Leoben (A); Zeipper, L. Institute of Materials Physics, University of Vienna (A) and ARC Seibersdorf Research GMBH, Seibersdorf (A); Zehetbauer, M. J. Institute of Materials Physics, University of Vienna (A)</i>	
VIII Influence of Deformation Parameters to SPD Nanostructures.....	433
The Role of Hydrostatic Pressure in Severe Plastic Deformation	435
<i>Zehetbauer, M. J., Schafner, E. Institute of Materials Physics, University of Vienna, Vienna (A); Stüwe, H. P., Vorhauer, A. Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben (A); Kohout, J. Department of Physics, Military Academy Brno, Brno (Cz);</i>	
Influence of the Processing parameters at High Pressure Torsion	447
<i>Hebesberger, T. Erich Schmid Institute of Materials Science Austrian Academy of Sciences, Leoben (A), now Voest-Alpine Stahl, GmbH; Vorhauer, A. Erich Schmid Institute of Materials Science Austrian Academy of Sciences, Leoben (A); Stüwe, H. P. Erich Schmidt Institute of Materials Science, Austrian Academy of Sciences and Institute of Metal Physics, University of Leoben, Leoben (A); Pippan, R. Erich Schmid Institute of Materials Science Austrian Academy of Sciences, Leoben (A) and Christian Doppler Laboratory for Local Analysis of Deformation and Fracture, Leoben (A)</i>	

Mechanical Properties and Thermal Stability of Nano-Structured Armco Iron Produced by High Pressure Torsion.....	453
<i>Ivanisenko, Yu., Minkow, A. Division of Materials, Ulm University, Ulm (D); Sergueeva, A. V. Chemical Engineering and Material Science Department, University of California, Davis (USA); Valiev, R. Z. Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, Ufa (Rus); Fecht, H.-J. Division of Materials, Ulm University, Ulm (D) and Institute of Nanotechnology, Research Center Karlsruhe, Karlsruhe (D)</i>	
Properties of Aluminum Alloys Processed by Equal Channel Angular Pressing Using a 60 Degrees Die	459
<i>Furukawa, M. Fukuoka University of Education, Munakata (J); Akamatsu, H., Horita, Z. Kyushu University, Fukuoka (J); Langdon, T. G. University of Southern California, Los Angeles (USA)</i>	
Deformation Behaviour of Copper Subjected to High Pressure Torsion	465
<i>Dubravina, A. A. Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, Ufa (Rus) and Department of Chemical Engineering & Materials Science, University of California, Davis (USA); Alexandrov, I. V., Valiev, R. Z. Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, Ufa (Rus); Sergueeva, A. V. Department of Chemical Engineering and Material Science, University of California, Davis (USA)</i>	
Features of Equal Channel Angular Pressing of Hard-to-Deform Materials.....	471
<i>Raab, G. I., Soshnikova, E. P. Ufa State Aviation Technical University, Ufa (Rus)</i>	
IX New Methods of SPD.....	477
ARB (Accumulative Roll-Bonding) and other new Techniques to Produce Bulk Ultrafine Grained Materials	479
<i>Tsuji, N., Saito, Y., Minamino, Y. Osaka University, Suita (J); Lee, S-H Mokpo National University, Mokpo (ROK)</i>	
Optimal SPD Processing of Plates by Constrained Groove Pressing (CGP).....	491
<i>Alkorta, J., Sevillano, J. G. Centro de Estudios e Investigaciones Técnicas de Guipúzcoa y Tecnun, University of Navarra, San Sebastian (E)</i>	
Comparative Study and Texture Modeling of Accumulative Roll Bonding (ARB) processed AA8079 and CP-Al	498
<i>Heason, C. P., Prangnell, P. B. UMIST, Manchester (GB)</i>	
Nanocrystallization in Carbon Steels by Various Severe Plastic Deformation Processes	505
<i>Todaka, Y., Umemoto, M., Tsuchiya, K. Department of Production System Engineering, Toyohashi University of Technology, Toyohashi (J)</i>	

Severe Plastic Deformation by Twist Extrusion.....	511
<i>Beygelzimer, Y., Varyukhin, V., Orlov, D., Synkov, S., Spuskanyuk, A., Pashinska, Y. Donetsk Phys&Tech. Institute, Donetsk (Ua)</i>	
SPD Structures Associated With Shear Bands in Cold-Rolled Low SFE Metals	517
<i>Higashida, K., Morikawa, T. Kyushu University, Fukuoka (J)</i>	
Features of Mechanical Behaviour and Structure Evolution of Submicrocrystalline Titanium under Cold Deformation	523
<i>Mironov, S. Yu., Salishchev, G. A. Institute of Metals Superplasticity Problems, Russian Academy of Sciences, Ufa (Rus); Myshlyayev, M. M. Baikov Institute of Metallurgy and Materials Science, Russian Academy of Sciences, Moscow (Rus)</i>	
Ultra Grain Refinement of Fe-Based Alloys by Accumulated Roll Bonding	530
<i>Reis, A. C. C., Tolleneer, I., Barbé, L., Kestens, L., Houbaert, Y. Ghent University, Department of Metallurgy and Materials Science, Ghent (B)</i>	
X SPD with Ball Milling and Powder Consolidation.....	537
Mechanically Activated Powder Metallurgy : A Suitable Way To Dense Nanostructured Materials.....	539
<i>Gaffet, E. UMR 5060 CNRS / UTBM, Sévenans, Belfort (F) and GFA, GdR 2391 CNRS, Dijon (F); Paris, S., Bernard, F. LRRS UMR 5613 CNRS / Univ. Bourgogne, Dijon (F) and GFA, GdR 2391 CNRS, Dijon (F)</i>	
Characterization and Mechanical Properties of Nanostructured Copper Obtained by Powder Metallurgy	545
<i>Langlois, C., Hÿtch, M. J., Lartigue, S., Champion, Y. Centre d'Etudes de Chimie Métallurgique CECM-CNRS, Vitry-sur-Seine (F); Langlois, P. Laboratoire d'Ingénierie des Matériaux et des Hautes Pressions LIMHP-CNRS, Villetaneuse (F)</i>	
Densification of Magnesium Particles by ECAP with a Back-Pressure.....	551
<i>Lapovok, R. Ye., Thomson, P. F. CAST CRC, School of Physics and Materials Engineering, Monash University, Melbourne (Aus)</i>	
Annealed Microstructures in Mechanically Milled Fe-0.6%O Powders	558
<i>Belyakov, A., Sakai, Y., Hara, T., Kimura, Y., Tsuzaki, K. Steel Research Center, National Institute for Materials Science, Tsukuba (J)</i>	

Processing and Characterization of Nanocrystalline Aluminum obtained by Hot Isostatic Pressing (HIP).....	564
<i>Billard, S., Dirras, G., Fondere, J. P., Bacroix, B. Laboratoire des Propriétés Mécaniques et Thermodynamiques des Matériaux (LPMTM) CNRS, Institut Galilée Université Paris 13, Villetaneuse (F)</i>	
Characteristics of Nano Grain Structure in SPD-PM Processed AISI304L Stainless Steel Powder	571
<i>Inomoto, H. Ritsumeikan University, Kusatsu, Shiga (J); Fujiwara, H. Department of Environmental Systems Engineering, Kochi University of Technology, Tosayamada, Kochi (J); Ameyama, K. Department of Mechanical Engineering, Ritsumeikan University, Kusatsu, Shiga (J)</i>	
Formation of Powder and Bulk Al-Cu-Fe Quasicrystals, and of Related Phases During Mechanical Alloying and Sintering	579
<i>Kaloshkin, S. D., Tcherdyntsev, V. V., Laptev, A. I., Shelekhov, E. V. Moscow State Institute of Steel and Alloys, Moscow (Rus); Principi, G., Spataru, T. Settore Materiali and INFM, DIM, Padova (I)</i>	
Production and Consolidation of Nanocrystalline Fe Based Alloy Powders	585
<i>Rombouts, M., Froyen, L. Department of Metallurgy and Materials Engineering (MTM), Katholieke Universiteit Leuven (B); C. Reis, A. C., Kestens, L. Department of Metallurgy, Ghent University (B)</i>	
Strain Measurement in the ECAP Process.....	591
<i>Werenskiold, J. C., Roven, H. J. Norwegian University of Science and Technology, Trondheim (N)</i>	
XI Mechanical Properties and Thermostability of Nanocrystalline Structures	597
Atomistic Modeling of Strength of Nanocrystalline Metals.....	599
<i>van Swygenhofen, H., Derlet, P. M., Hasnaoui, A. Paul Scherrer Institute, Villigen (CH)</i>	
Multiscale Studies and Modeling of SPD Materials.....	609
<i>Alexandrov, I. V. Ufa State Aviation Technical University, Ufa (Rus)</i>	
Microstructural Stability and Tensile Properties of Nanostructured Low Carbon Steels Processed by ECAP.....	616
<i>Shin, D. H. Department of Metallurgy and Materials Science, Hanyang University, Ansan (ROK); Park, K.-T. Division of Advanced Materials Science & Engineering, Hanbat National University, Taejeon (ROK)</i>	

XVIII

Microstructure and Mechanical Properties of Severely Deformed Al-3Mg and its Evolution during subsequent Annealing Treatment..... 623
Morris-Muñoz, M. A., Garcia Oca, C., Gonzalez Doncel, G., Morris, D. G. CENIM, CSIC, Madrid (E)

Dependence of Thermal Stability of Ultra Fine Grained Metals on Grain Size..... 630
Stuliková, I., Cieslar, M., Kuzel, R., Procházka, I., Cízek, J. Faculty of Mathematics and Physics, Charles University, Prague, (Cz); Islamgaliev, R. K. Institut of Physics of Advanced Materials, Ufa State Aviation Technical University, Ufa (Rus)

Thermomechanical Properties of Electrodeposited Ultra Fine Grained Cu-Foils for Printed Wiring Boards..... 636
Betzwar-Kotas, A., Gröger, V., Weiss, B., Wottle, I., Zimprich, P. Institute of Material Physics, University of Vienna (A); Khatibi, G. Institute of Material Physics, University of Vienna (A) and Institute of Physical Chemistry, Material Science, University of Vienna (A)

Effect of Grain Boundary Phase Transitions on the Superplasticity in the Al-Zn system 642
López, G. A., Gust, W., Mittemeijer, E. J. Max Planck Institute for Metals Research and Institute of Physical Metallurgy, University of Stuttgart, Stuttgart (D); Straumal, B. B. Max Planck Institute for Metals Research and Institute of Physical Metallurgy, University of Stuttgart, Stuttgart (D) and Institute of Solid State Physics RAS, Chernogolovka (Rus)

Microstructure and Thermal Stability of Tungsten based Materials after Severe Plastic Deformation..... 648
Vorhauer, A. Erich Schmid-Institute of Materials Science of the Austrian Academy of Sciences, Leoben (A) and Christian Doppler Laboratory of Local Analysis of Deformation and Fracture, Leoben (A); Pippan, R. Erich Schmid-Institute of Materials Science of the Austrian Academy of Sciences, Leoben (A) and Christian Doppler Laboratory of Local Analysis of Deformation and Fracture, Leoben (A); Knabl, W. Plansee AG, Reutte (A)

Development of Microstructure and Thermal Stability of Nano-structured Chromium Processed by Severe Plastic Deformation 654
Wadsack, R. Erich Schmid Institute for Materials Science of the Austrian Academy of Sciences, Leoben (A); Pippan, R. Erich Schmid Institute for Materials Science of the Austrian Academy of Sciences, Leoben (A) and Christian Doppler Laboratory for Local Analysis of Deformation and Fracture, Leoben (A); Schedler, B. Plansee AG, Reutte (A)

XII Influence of Deformation Path to Properties of SPD Materials	661
Fatigue of Severely Deformed Metals	663
<i>Vinogradov, A., Hashimoto, S. Department of Intelligent Materials Engineering, Osaka City University, Osaka (J)</i>	
Cyclic Deformation Behaviour and Possibilities for Enhancing the Fatigue Properties of Ultrafine-Grained Metals	677
<i>Höppel, H. W., Kautz, M., Barta-Schreiber, N., Mughrabi, H. Friedrich-Alexander-Universität Erlangen-Nürnberg, Erlangen (D); Xu, C., Langdon, T. G. University of Southern California, Los Angeles (USA)</i>	
The Influence of Type and Path of Deformation on the Microstructural Evolution During Severe Plastic Deformation	684
<i>Vorhauer, A., Pippan, R. Erich Schmid Institute for Materials Science of the Austrian Academy of Sciences, Leoben (A) and Christian Doppler Laboratory for Local Analysis of Deformation and Fracture, Leoben (A)</i>	
Formation of a Submicrocrystalline Structure in Titanium during Successive Uniaxial Compression in Three Orthogonal Directions	691
<i>Salishchev, G. A., Zherebtsov, S. V., Mironov, S. Yu. Institute for Metals Superplasticity Problems, Ufa (Rus); Myshlyayev, M. M. Baikov Institute of Metallurgy and Materials Science, Moscow (Rus); Pippan, R. Institute of Material Science, Leoben (A)</i>	
XIII Features and Mechanisms of Superelasticity in SPD Materials	699
Achieving a Superplastic Forming Capability through Severe Plastic Deformation	701
<i>Xu, C., Langdon, T. G. University of Southern California, Los Angeles (USA); Horita, Z. Kyushu University, Fukuoka (J); Furukawa, M. Fukuoka University of Education, Munakata (J)</i>	
Production of Superplastic Mg Alloys Using Severe Plastic Deformation	711
<i>Horita, Z., Matsubara, K., Miyahara, Y. Kyushu University, Fukuoka (J); Langdon, T. G. University of Southern California, Los Angeles (USA)</i>	
High Strain Rate Superplasticity in an Micrometer-Grained Al-Li Alloy Produced by Equal-Channel Angular Extrusion.....	717
<i>Myshlyayev, M. M., Myshlyayeva, M. M. Institute of Solid State Physics, Russian Academy of Sciences, Chernogolovka (Rus); Kamalov, M. M. Institute of Solid State Physics, Russian Academy of Sciences, Chernogolovka (Rus) and Baikov Institute of Metallurgy and Material Science, Russian Academy of Sciences, Moscow (Rus)</i>	

Diffusion-Controlled Processes and Plasticity of Submicrocrystalline Materials.....	722
<i>R. Kolobov, Yu., Ivanov, K. V., Grabovetskaya, G. P., Naidenkin, E. V.</i> <i>Institute of Strength Physics and Materials Science, Tomsk (Rus)</i>	
Superplastic Behavior of Deformation Processed Cu-Ag Nanocomposites.....	728
<i>Hong, S. I., Kim, Y. S., Kim, H. S. Department of Metallurgical Engineering,</i> <i>Chungnam National University, Taedok Science Town, Taejon (ROK)</i>	
Features of Microstructure and Phase State in an Al-Li Alloy after ECA Pressing and High Strain Rate Superplastic Flow	734
<i>Myshlyaev, M. M. Institute of Solid State Physics, Russian Academy</i> <i>of Sciences, Chernogolovka (Rus) and Baikov Institute of Metallurgy and</i> <i>Material Science, Russian Academy of Sciences, Moscow (Rus); Mazilkin, A. A.,</i> <i>Kamalov, M. M. Institute of Solid State Physics, Russian Academy of Sciences,</i> <i>Chernogolovka (Rus)</i>	
Microstructure Refinement and Improvement of Mechanical Properties of a Magnesium Alloy by Severe Plastic Deformation.....	740
<i>Mussi, A., Blandin, J. J., Rauch, E. F. Génie Physique et Mécanique des</i> <i>Matériaux (GPM2) Saint-Martin d'Hères (F) and Institut National Polytechnique</i> <i>de Grenoble (INPG) Saint-Martin d'Hères (F)</i>	
Grain Refinement and Superplastic Properties of Cu-Zn Alloys Processed by Equal-Channel Angular Pressing	746
<i>Neishi, K., Horita, Z. Kyushu University, Fukuoka (J); Langdon, T. G.</i> <i>University of Southern California, Los Angeles (USA)</i>	
XIV Mechanisms of Diffusion Related Processes in Nanocrystalline Materials.....	753
Diffusion in Nanocrystalline Metals and Alloys – A Status Report.....	755
<i>Würschum, R., Brossmann, U. Technische Universität Graz, Institut für</i> <i>Technische Physik, Graz (A); Herth, S. Forschungszentrum Karlsruhe,</i> <i>Institut für Nanotechnologie, Karlsruhe (D)</i>	
Self-Diffusion of ¹⁴⁷ Nd in Nanocrystalline Nd ₂ Fe ₁₄ B.....	767
<i>Sprengel, W., Barbe, V., Herth, S., Wejrzanowski, T. Institut für</i> <i>Theoretische und Angewandte Physik, Universität Stuttgart, Stuttgart (D)</i> <i>and Faculty of Materials Science, Warsaw University of Technology, Warsaw</i> <i>(Pl); Gutfleisch, O. Leibniz-Institut für Festkörper- und Werkstoffforschung</i> <i>Dresden, Dresden (D); Eversheim, P. D. Helmholtz Institut für Strahlen- und</i> <i>Kernphysik, Universität Bonn, Bonn (D); Würschum, R. Institut für Technische</i> <i>Physik, Technische Universität Graz, Graz (A); Schaefer, H.-E. Institut für</i> <i>Theoretische und Angewandte Physik, Universität Stuttgart, Stuttgart (D)</i>	

Theoretical Investigation of Nonequilibrium Grain Boundary Diffusion Properties	773
<i>Perevezentsev, V. N. Blagonravov Nizhny Novgorod Branch of Mechanical Engineering Research Institute, Russian Academy of Sciences, Nizhny Novgorod (Rus)</i>	
On Annealing Mechanisms Operating in Ultra Fine Grained Alloys.....	780
<i>Sakai, T., Miura, H. Department of Mechanical Engineering and Intelligent Systems, University of Electro-Communications, Chofu, Tokyo (J); Belyakov, A., Tsuzaki, K. Steel Research Center, National Institute for Materials Science, Tsukuba (J)</i>	
XV Application of SPD Materials	787
Commercialization of Nanostructured Metals Produced by Severe Plastic Deformation Processing.....	789
<i>Lowe, T. C., Zhu, Y. T. Los Alamos National Laboratory, Los Alamos (USA)</i>	
The Main Directions in Applied Research and Developments of SPD Nanomaterials in Russia	798
<i>Fokine, V. A. Editor of the Journal "Russia and World: Science and Technology", Director General of the F&F Consulting Co., Moscow (Rus)</i>	
Developing of Structure and Properties in Low-Carbon Steels During Warm and Hot Equal Channel Angular Pressing	804
<i>Dobatkin, S. V. Baikov Institute of Metallurgy and Material Science, Russian Academy of Sciences, Moscow (Rus) and Moscow State Steel and Alloys Institute (Technological University), Moscow (Rus); Odessky, P. D. Institute of Building Constructions, Moscow (Rus); Pippan, R. Erich Schmid Institute of Material Science, Austrian Academy of Sciences, Leoben (A); Raab, G. I., Krasilnikov, N. A. Ufa State Aviation Technical University, Ufa (Rus); Arsenkin, A. M. Moscow State Steel and Alloys Institute (Technological University), Moscow (Rus)</i>	
Mechanical Properties of Severely Plastically Deformed Titanium.....	810
<i>Zeipper, L., Korb, G. ARC Seibersdorf Research GmbH, Seibersdorf (A); Zehetbauer, M., Mingler, B., Schafner, E., Karthaler, H. P. University of Vienna, Vienna (A)</i>	
Ways to Improve Strength of Titanium Alloys by Means of Severe Plastic Deformation	817
<i>Popov, A. A. Ural State Technical University, UGTU-UPI, Ekaterinburg (Rus)</i>	
Structures, Properties, and Application of Nanostructured Shape Memory TiNi-based Alloys.....	822
<i>Pushin, V. G. Institute of Metal Physics, Ural Division of Russian Academy of Sciences, Ekaterinburg (Rus)</i>	

Microstructure and Properties of a Low Carbon Steel after Equal Channel Angular Pressing 829
Wang, J., Wang, Y., Du, Z., Zhang, Z., Wang, L., Zhao, X. School of Metallurgical Engineering, Xi'an Univ. of Arch. & Tech., Xi'an (VRC); Xu, C., Langdon, T. G. Depts. of Mech. Eng. and Mater. Sci., Univ. of Southern California, Los Angeles (USA)

Formation of Submicrocrystalline Structure in Large-Scale Ti-6Al-4V Billets during Warm Severe Plastic Deformation 182 835
Zherebtsov, S. V., Salishchev, G. A., Galejev, R. M., Valiakhmetov, O. R. Institute for Metals Superplasticity Problems, Ufa (Rus); Semiatin, S. L. Air Force Research Laboratory, Wright-Patterson Air Force Base (USA)

Author Index 841

Subject Index 845

I Reasons to use Nanostructured Materials

Unique Features and Properties of Nanostructured Materials

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1 Abstract

In this introductory paper an attempt is made to give an overview of the area of nanostructured „materials irrespective of the synthesis process. The various microstructural features such as clusters or isolated nanoparticles, agglomerated nanopowders, consolidated nanomaterials and nanocomposite materials as well as all materials classes are considered. As an important component of modern research on nanomaterials a section describes the various characterization tools available. Based on these remarks some properties of nanostructured materials will be summarized emphasizing the property-microstructure relationships. Finally, a brief outlook on applications and initial industrial use of nanomaterials is presented.

2 Introduction

Nanostructures are plentiful in nature. In the universe nanoparticles are distributed widely and are considered to be the building blocks in planet formation processes. Biological systems have built up inorganic-organic nanocomposite structures to improve the mechanical properties or to improve the optical, magnetic and chemical sensing in living species. As an example, nacre (mother-of-pearl) from the mollusc shell is a biologically formed lamellar ceramic, which „exhibits structural robustness despite the brittle nature of its constituents. [1] Figure 1 shows an SEM image of a fracture surface of an abalone shell exhibiting the CaCO_3 -platelets which are se-

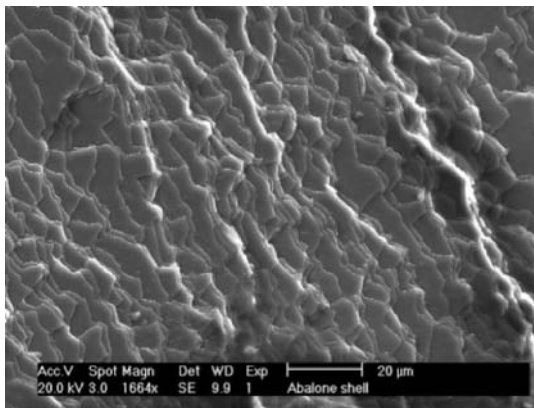


Figure 1: SEM image of a fracture surface of a Korean abalone shell showing the individual calcium-carbonate platelets separated by organic compounds.

parated by organic compounds which exhibit nanometer dimensions. These systems have evolved and been optimized by evolution over millions of years into sophisticated and complex structures. In natural systems the bottom-up approach starting from molecules and involving self organization concepts has been highly successful in building larger structural and functional components. Functional systems are characterized by complex sensing, self repair, information transmission and storage and other functions all based on molecular building blocks. Examples of these complex structures for structural purposes are teeth, such as shark teeth, which consist of a composite of biomineralized fluorapatite and organic compounds. These structures result in the unique combination of hardness, fracture toughness and sharpness, see Figure 2.

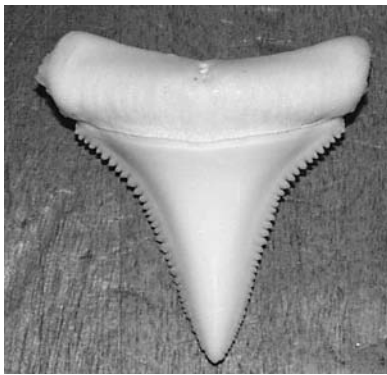


Figure 2: Example of a nanostructure found in nature: shark tooth with unique mechanical properties. The overall dimension of the tooth can reach several cm.

Another example for a biological nanostructure is opal which exhibits unique optical properties. The self cleaning effects of the surfaces of the lotus flower have been attributed to the combined micro- and nanostructure which in combination with hydrophobic groups give the surface a water and dirt repellent behavior. [2] In the past few years, numerous companies have realized products resembling the surface morphology and chemistry of the lotus flower such as paint, glass surface and ceramic tiles with dirt repellent properties. The realization that nature can provide the model for improved engineering has created a research field called „bio-,mimicking or bio-inspired materials science. It has been possible to process these ceramic-organic nanocomposite structures which provide new technological opportunities and potential for applications. [3] Other exciting results have been published such as the biomimetic growth of synthetic fluorapatite [4] in the laboratory and promising new technical applications of these nanomaterials are envisioned. [5] Other man-made nanostructures were manufactured for their attractive optical properties, such as the colloidal gold particles in glass as seen in medieval church windows.

While plentiful man made materials with nanostructures have been in use for a long time (partially without knowing it) a change of the scientific and technological approach can be identified over the past two decades. This change can be related to a few key ideas and discoveries: the idea of assembling nanostructures from atomic, molecular or nanometer sized building blocks, [6] the discovery of new forms of carbon, i.e., fullerenes [7] and carbon nanotubes, and the development of scanning probe microscopy, [8] such as scanning tunneling microscopy (STM) and atomic force microscopy (AFM). With the visionary goals many researchers world-

wide have worked intensively on the development of novel or improved synthesis methods, new and better characterization techniques and the measurement and the design of the properties of nanostructured materials. In this paper some aspects of the immensely wide field will be described. However, as the field of nanostructured materials is very broad including all classes of materials as well as composites it is not possible and not attempted to consider all developments and all research groups and industries working in this area.

3 Synthesis

The microstructure and properties of nanostructured materials depend in an extreme manner on the synthesis method as well as on the processing route. Therefore, it is of utmost importance to select the most appropriate technique for preparation of nanomaterials with desired properties and property combinations. Synthesis techniques can be divided into bottom-up and top-down approaches. The top-down approach starts with materials with conventional crystalline microstructures, typically metals and alloys, and defects such as dislocations and point defects are introduced by severe plastic deformation such as in equal channel pressing. The recrystallization of the material leads to finer and finer grain sizes and under certain processing conditions to nanostructured materials. The advantage of these approaches is the fact that bulk nanostructured materials with theoretical density can be prepared. An alternative to obtain theoretical dense materials is the pulsed electrodeposition method developed by Erb and El-Sherik which yields nanocrystalline strips, however, only with thicknesses of several hundred microns. [9] The bottom-up approach includes many different techniques which are based on liquid or gas phase processes. Classically, wet chemical processes such as precipitation and sol-gel have been employed to obtain nanoparticles, however, with the disadvantage of severe agglomeration. In the gas phase metallic and ceramic nanoparticles have been synthesized by using Inert Gas Condensation, Flame Pyrolysis (Aerosol process by Degussa) and chemical vapor based processes. The major microstructural features in preparing nanoparticles for subsequent use are: nanometer sized primary particles with narrow size distributions, minimum amount of agglomeration, good crystallinity, etc.

Two techniques, chemical vapor synthesis (CVS) in the gas phase [10] and electrodeposition under oxidizing conditions (EDOC) in the liquid phase, [11] together with the resulting microstructures will be presented in more detail and the advantages and disadvantages be discussed. CVS is based on chemical vapor deposition (CVD) for the synthesis of thin films and coatings by the decomposition of metalorganic precursors. Whether thin films are deposited by heterogeneous „nucleation or nanoparticles are formed in the gas phase by homogeneous nucleation is determined by the residence time of the precursor in the hot zone of the reactor. The most important parameters determining the growth regime and the particle size are the total pressure, the precursor partial pressure and the temperature of the reaction zone. A typical reactor set-up is shown schematically in Figure 3 with one precursor source, the hot wall reactor, the thermophoretic collector, the pumping unit and the control devices for pressure and temperature. The hot wall concept operating at reduced pressures has been successfully scaled up in a cooperation project with a large German corporation involved in the synthesis of nanopowders such as carbon black, titania and silica. [12]

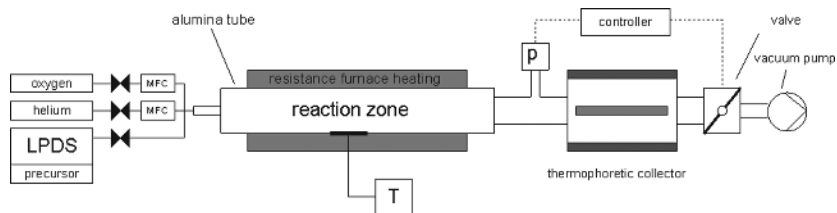


Figure 3: Schematic diagram of the major components of a CVS hot wall reactor: precursor source (liquid precursor delivery system, LPDS), hot wall reaction zone, thermophoretic particle collector, pumping system, and pressure and temperature control.

When two precursors are used, the precursor delivery can be modified in the following way:

- (1) two precursors are introduced simultaneously into the reaction zone yielding doped nanoparticles (i.e. alumina doped zirconia); [13]
- (2) two precursors are introduced into two concentric reaction tubes, reacted to form nanoparticles and then mixed in the gas phase to yield a nanocomposite structure (i.e., alumina mixed with zirconia) and
- (3) in the first reaction zone the first precursor is decomposed to form nanoparticles by homogeneous nucleation which are subsequently coated in a second reaction zone by introducing the second precursor under conditions which favor CVD deposition (i.e., alumina surface coated zirconia). [14]

The experimental set-up of case 3) can be further modified by using a plasma reaction zone with pulse option which allows the controlled functionalization with organic molecules and polymeric shells. [15,16] Figure 4 shows a high resolution electron image of polymer coated titania nanoparticles where the crystalline titania core can be clearly distinguished from the amorphous organic shell on several grains.

Further evidence of the complete coating can be obtained by surface analysis, FTIR studies and by dispersion „experiments in different organic liquids and water. The modification of the surfaces of nanoparticles allows the improvement of dispersibility in various aqueous and organic solvents which is important for many ceramic processing steps (dip- or spin-coating, slurries for ceramic processing, etc.) and for technical applications of dispersions. Additionally, the inorganic core/polymer shell structure allows the preparation of polymer nanocomposites with excellent separation between the inorganic nanoparticles.

A further variation by exact control of all synthesis parameters allows the growth of thick nanocrystalline coatings on dense and porous substrates. Depending on the substrate temperature the porosity of the coating can be changed over a wide range up to theoretical density. This intermediate stage, called CVD/CVS, has been successfully used to deposit a nanocrystalline coating of yttrium stabilized zirconia on porous anode substrates for high temperature solid oxide fuel cell applications. Figure 5 shows a high resolution scanning electron image of a coated anode substrate. [17]

The processes leading to particle formation have been modeled and simulated by many authors. The detailed description of these efforts is beyond the scope of this paper. A comprehen-