



**RESEAU FRANÇAIS DE
MECANOSYNTHESE**

Lettre N°88

Juillet 2002

**188 Groupes de Recherche
(dont 114) à l'étranger / 34 Pays)**

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**Do Not Forget the Annual Subscription Fees
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A check will be done during the Summer !!

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(à retourner à l'adresse suivante - to be sent at the following address) :

Eric GAFFET

CNRS UMR5060 « Métallurgies et Cultures »

Nanomaterials Research Group

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Nom/Name :Prénom / First Name :

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désire adhérer au Réseau Français de Mécanosynthèse / want to become a member of the French Mechanical Alloying Network

Chèque ci joint / Check enclosed in the amount of **20 Euros (20€)**

The check has to be to the order to : Réseau Français de Mécanosynthèse

Le site web du RFM est :

<http://www.bls.fr/amatech>

Rubrique Pages Sciences et Techniques pour l'Ingénieur (Rubrique Sciences)

⇒ vous y trouverez les anciennes lettres du RFM (accessible par Adobe Acrobat), les statuts du RFM ainsi que les annonces concernant les JRFM'2001 et quelques éléments mis à jour régulièrement concernant les derniers résultats dans ce domaine.

Congress and School Announcements

Workshop

Gordon Research Conference on Granular and Granular-Fluid Flow

Plymouth, NH, USA June 30 - July 5, 2002

<http://sol.rutgers.edu/~shinbrot/gordon2002/gordon2002.html>

ICSTR

INTERNATIONAL CONFERENCE ON SOLVO-THERMAL REACTIONS

July 22-26, 2002 - Hilton East Brunswick / East Brunswick, New Jersey,

More information on this meeting can be found at <http://www.ICSTR.rutgers.edu/>

or by contacting Professor Richard E. Riman at Rutgers University via
riman@email.rci.rutgers.edu/732-445-4946(v)/732-445-6262 (f).

RQ11

Rapidly Quenched and Metastable Materials

25-30 August 2002

Department of Materials, University of Oxford, UK

Contact: RQ11 Conference Organiser, Beggars Roost, Channels End Road,

Comworth Bedford MK44 2NS, U.K.

Tel: +44 (0) 1234 378862

Fax: +44 (0) 1234 376219

E-mail: mailto:rq11@materials.ox.ac.uk

Website: <http://www.materials.ox.ac.uk/rq11>

10th European Symposium on Comminution

Heidelberg from 2-5 September 2002.

Org. European Federation of Chemical Engineering

Full information available at <http://www.comminution2002.de>

8th ICCPS

8th International Conference on Ceramic Processing Science

Hamburg - Sept. 2nd - 5, 2002.

The conference will focus on novel processing of advanced structural and functional

ceramics and ceramic composites. The program will favor the most recent

developments in this presented in only 10 topical sessions:

1. New Concepts for Economic Production of Powders of High Purity, Reactivity and Ease of Handling
2. Novel Powder Processing and Non-Conventional Shaping (Nanoprocessing, Cellular Structures, etc.)
3. Solution Processing (Thin Film Deposition, Soft Solution, Polymer-Derived, etc.)
4. Biomimetic Structuring (Biotemplates, Biomineralization, etc.)
5. Novel Reaction Forming (Controlled SHS, Reactive Casting, in situ Processing, etc.)
6. Computer-Controlled Shaping and Structuring (Rapid Prototyping, Solid Free-Forming, Controlled Heterogeneities, etc.)
7. Tailoring of Synergy Ceramic Microstructures (LTCC, Self-Sensing Devices, Smart Structures, MEMS, etc.)
8. Grain Boundary Engineering (Grain-Boundary-Free Microstructures, Directed Eutectics, Advanced Electroceramics, etc.)
9. Micromechanics of Composite Synthesis (Transient and Residual Stresses, Constrained Sintering, etc.)
10. COST 528: Chemical Solution Deposition of Thin Films

website : <http://www.tu-harburg.de/gk/8th-ICCPS>

ISMANAM-2002

International Symposium on

Metastable, Mechanically Alloyed and Nanocrystalline Materials

Seoul, Korea, 8-12 September, 2002.

Web site : <http://anu.andong.ac.kr/~ismanam/>

L. A. C. A. M. E - 2. 0. 0. 2

EIGHTH LATIN AMERICAN CONFERENCE ON APPLICATIONS OF THE MÖSSBAUER EFFECT PANAMA, 22-27, SEPTEMBER, 2002.

E-mail: mailto:lacame2000@fisica.ciens.ucv.ve

<http://www.up.ac.pa/Eventos/lacame2002/inicio.htm>

Matériaux 2002

Tours - France

21- 25 Octobre 2002

Website : <http://www.materiaux2002.net>



E_mail : materiaux@materiaux2002.net

Les JRFM'2002 seront intégrées dans le cadre du Congrès
Matériaux 2002 (Tours – France, du 21 au 25 Octobre 2002)

Poudres et Matériaux Nanostructurés, du fondamental aux applications industrielles

Symposium 1 :

Website : <http://www.materiaux2002.net> : E_mail : materiaux@materiaux2002.net

ICAMP 2002

2nd International Conference on Advanced Materials Processing -
2nd to 4th December 2002 in Singapore.

You are invited to submit papers to the conference secretariat at

Denis Lam - ICAMP 2002 Secretariat
Integrated Meetings Specialist Pte Ltd
114 Middle Road - #05-02 Lee Kai House
Singapore 188971

E-mail: icamp@inmeet.com
Tel: (65) 6226 3069 - Fax: (65) 6226 3016

More detailed information can be found in ICAMP web site
<http://www.eng.nus.edu.sg/PACentre/icamp>



Cooperative Research on Related Areas

France (12/04/2001)

Le portail Internet "France Contact" a été lancé: ce portail s'adresse aux chercheurs étrangers séjournant ou ayant séjourné en France et permettra le suivi et l'animation du réseau que constituent les milliers de chercheurs étrangers ayant effectué un séjour scientifique au sein des établissements et des organismes de recherche français:

Website : <http://www.francecontact.net>

Europe (6/03/2001)

The ESF, on the recommendation of the scientific Standing Committee for Physical and Engineering Sciences (PESC), will support, in fields related to PESC's remit, approximately 10 ESF Exploratory Workshops to be held in 2002.

Each workshop will allow 20-25 leading European scientists to explore novel ideas at the European level with the challenging aim to "spearhead" new and preferably inter-disciplinary areas of research.

In specific terms, PESC's 2001 Call is for workshop proposals on R&D subjects which are NOVEL AND PREFERABLY INTERDISCIPLINARY and which concern emerging fields within any of the following areas: chemistry, physics, mathematics, information sciences, fundamental engineering sciences, materials sciences, and technologies research in these areas.

The PESC Call is available at <http://www.esf.org/physical/WorkshopCalls/Call2001.htm>



**Job Vacancies, Ph D Position and, Post Doc Position
Requests – Proposals**

From Assoc. Prof. Gerardo F. Goya
(on the 7th June 2002)

A postdoctoral position is available at the Physics Department, University of São Paulo, Brazil.

The position is for one year (with renewal up to three years), and the salary is about R\$ 34,400.00/year (13,600.00 USD). Candidates must be interested in developing a research project focused on Mechanosynthesis of Magnetic Materials and magnetic characterization.

Applications including a CV, publication list, and names of three references should be sent by e-mail, before August 30, 2002 to:

Assoc. Prof. Gerardo F. Goya,
Departamento de Física dos Materiais
Instituto de Física
Universidade de São Paulo
e-mail: goya@if.usp.br
fax +55 11 3091 6984
tel +55 11 3091 6885

From Dr.-Ing. Adrian JIANU (Rostock University, Physics Dept.)
On 7th June 2002

The Chair of Physics of New Materials of the University of Rostock is seeking outstanding young (35 years old or less) scientists to work on a new project of advanced materials synthesis under extreme pressure and temperature conditions.

The two fellowships (24 months each) are offered in the frame of the Marie Curie Development Host scheme of the 5th European Community Framework Programme.

A Ph.D. in Material Science or related sciences (Solid State or Applied Physics, Chemistry, etc) or at least 4 years of full-time research experience at a post-graduate level is required.

Eligible candidates should be nationals or residents of an European Community Member or Associated States other than Germany.

For further details please visit the website :

<http://www.physik1.uni-rostock.de/user/agburkel/jobs.html>

or please contact for :

Dr. A. Jianu,
phone: 0049-381-4981731,
e-mail: jianu@physik1.uni-rostock.de

Please send applications with C.V. and list of publications to: _

Prof. Eberhard Burkel
Rostock University, August-Bebel-Str., 55,
018055 Rostock, Germany,
tel: 0049-381-4981727,
fax:0049-381-4981726,
e-mail: burkel@physik1.uni-rostock.de

Proposal on 4 June 2002



MATERIALS SCIENTIST

Crown Cork & Seal is the world's leading packaging company. It consists of the best-established packaging companies in the UK, USA, France, Germany, and many other countries, successfully merged into a dynamic family of packaging professionals. In Europe we may be more familiar as CarnaudMetalbox, the largest manufacturer of metal and plastics domestic and retail packaging, such as food and beverage cans and PET bottles. Worldwide, the Company has a turnover of \$7 billion, with 36,000 employees and operations in 51 countries.

Corporate Technologies, based in Wantage UK, and Chicago USA, is the research, development and engineering arm of Crown Cork & Seal, providing technical support to our Businesses and Project Teams.

Our Materials Group, based in Wantage, South Oxfordshire, has a vacancy within the packaging performance and corrosion failure analysis team, for a Materials Scientist with suitable experience in electrochemistry and corrosion. An integral part of the role will involve the integration of electrochemical approaches to corrosion problem solving, together with advanced imaging and analytical techniques (S.E.M, AFM, ESCA etc).



Candidates should have a good first degree, or PhD, in the appropriate discipline with either some relevant industrial experience or an interest in packaging. They will be reliable team players who can communicate at all levels, be self-motivated and demonstrate an ability to translate a sound, scientific knowledge base into practical advantage in a commercially driven environment.

Applications (full cv or SAF) should be sent to:
Graduate Recruitment & Training Manager,
Crown Cork & Seal,
Downsview Road,
Wantage, Oxon: OX12 9BP
e-mail: grad.careers@eur.crowncork.com

Proposal from 13/05/2002
POSTDOCTORAL RESEARCH FELLOW

Department of Materials and Process Engineering - School of Science and Technology - (Fixed term for 2 years)

We are seeking a postdoctoral research fellow for a 2-year fixed term contract, starting July 2002. The position involves research in processing, consolidating and developing titanium-based metallic and metal-ceramic composite powder materials, including titanium based metal-ceramic nanocomposites. The appointee will also assist the project leader in project planning and postgraduate supervision.

The candidate should either have a PhD degree in materials science and engineering, or expect to have completed a PhD by the time of appointment. Previous research experience and a strong publication track record in titanium-based metallic materials, mechanical alloying or powder metallurgy will be an advantage.

The position is funded by a New Economy Research Fund (NERF) grant from the Foundation for Research, Science and Technology.

The Department of Materials and Process Engineering hosts the Waikato Centre for Advanced Materials (WaiCAM) and the website for the department is <http://mape.waikato.ac.nz>

The salary range is NZ\$46,564 - \$52,666 per year.

Applications close on Friday, 7 June 2002.

Further information and an application form are available from; www.waikato.ac.nz/hrm/ or the Human Resource Management Division, The University of Waikato, Private Bag 3105, Hamilton, phone 64-7-8384003, fax 64-7-8560135, email; hrm@waikato.ac.nz.

The University is committed to providing equal opportunities for all.

Proposal from 30/04/2002
Queen's University - Department of Mechanical Engineering
Post Doctoral Research Position in - Nuclear Materials

The Department of Mechanical Engineering, Queen's University, invites applications for a post-doctoral research position to work in the NSERC Industrial Research Chair program in Nuclear Materials under the direction of the Chair-holder, Prof. Rick Holt. The chair program is co-funded by OPG Inc., COG Inc. and Nu-Tech Precision Metals Inc.

The purpose of the Chair is to further the fundamental understanding of nuclear materials and to develop the applied technology required by industry in this area. This includes radiation-induced deformation of nuclear materials (especially zirconium alloys), its relationship to manufacturing variables, crystallographic texture and microstructure, and other related topics.

Candidates must have completed a Ph.D. in metallurgical engineering, metallurgy, materials science or a related field.

The successful candidate will be an experienced electron-microscopist with expertise in TEM (specimen preparation techniques, bright field, dark field and weak beam imaging, EDX, CBED and SADP analysis). Expertise with SEM, especially orientation analysis using electron back scattering would be an asset.

The successful candidate will investigate the microstructure and microtexture of zirconium alloys using electron microscopy, and will assist with the supervision of post graduate students working in this area. Publication of the results is an expected outcome.

Applicants should send their resumés to:
Prof. R.A. Holt
Materials Engineering Group
Department of Mechanical Engineering



Nicol Hall
Queen's University
Kingston ON K7L 3N6
holt@me.queensu.ca

Proposal from 24/04/2002

POST-DOCTORAL RESEARCHER

Nanoelectronics at the Quantum Edge

Postdoctoral Research Assistant in Scanning Probe Microscopy and Related Techniques

Grade RAIA / Salary: £17,626 - £26,491 / Job Ref: DJ02/023

Oxford and Cambridge Universities are working together with Hitachi Europe Ltd to produce radically new devices for future computing, in a project jointly funded by a Foresight Link Award from the Department of Trade and Industry and Hitachi Europe Ltd. The project brings together research in physics, chemistry, materials science and electronics engineering to make prototype structures for advanced conventional computing and for the new field of quantum computing. See www.nanotech.org. Applications are invited for a postdoctoral position in scanning probe microscopy and related techniques, for atomic and electronic structural characterization of endohedral fullerenes and single walled carbon nanotubes. This is available until September 2004.

Candidates should have a good first degree and completed PhD or equivalent in a relevant physical science, a track record of innovative and effective experimental research using atomic resolution scanning probe microscopy, and evidence of quality publications in international peer reviewed journals and presentations at national and international seminars and conferences. They should have proven skills in maintaining a fundamental academic and research overview of the structural characterization of nanomaterials. Flexibility, excellent verbal and written communication skills in English, and the ability to work independently and in a team within an agreed time-scale are essential.

Before submitting an application, candidates should obtain further particulars available from The Deputy Administrator (Teaching), Department of Materials, University of Oxford, Parks Road, Oxford OX1 3PH (email: posts@materials.ox.ac.uk), or telephone 01865 273750 quoting reference: DJ02/023. The closing date for applications is 31 May 2002 and interviews are currently planned for the week beginning 17 June. Further information on the Department may be found on the web-site: <http://www.materials.ox.ac.uk>

See also <http://www.ox.ac.uk/staff.html>

Proposal from 4/04/2002

PhD CORUS Industrial CASE Studentship. Oxford University

Supported by EPSRC and CORUS, for postgraduate research on the structure, properties and processing of aluminium alloys or steel. The studentship will pay the holder a stipend of at least £11,000 per year, plus University and College fees.

Applicants should send a curriculum vitae and names of two academic referees to Kay Sims, Secretary to the Director of Graduate Studies, Department of Materials, University of Oxford, Parks Road, Oxford OX1 3PH, UK; tel: (01865) 273682; fax: (01865) 273783; e-mail: kay.sims@materials.ox.ac.uk

Proposal from 11/03/2002

Announcing Ph.D. and postdoc positions:

A Ph.D. and a postdoc position is available in a joint project of the Fritz-Haber-Institut der Max-Planck-Gesellschaft (Matthias Scheffler, <http://www.fhi-berlin.mpg.de/th/th.html>) and The Pennsylvania State University (Henry C. Foley, <http://fenske.che.psu.edu/Faculty/Foley/index.html> and Kristen Fichthorn, <http://fenske.che.psu.edu/Faculty/Fichthorn/index.html>).

Theme:

The role of nano-porous carbon in dehydrogenation and oxidation catalysis

Project summary:

This is a highly interdisciplinary project involving, e.g. extensive density-functional theory calculations and Statistical Mechanics simulations (with DFT derived parameters).

The catalytic production of styrene is one of the most important processes in chemical industry (a key process for making most plastics). Recently it could be shown that the typically employed iron-oxide catalyst is in fact not the active material, but the true catalyst is formed during the induction period: The material that is actually doing the catalysis apparently is "nano porous carbon". This consists of strained and twisted graphite sheets that have a lot of defects (in particular five-fold rings). http://www.fhi-berlin.mpg.de/th/Slides/Scheffler_transparencies_pdf/npc-2002.pdf summarizes some aspects of our recent work.

The planned research may start with an analysis of the chemical reactivity of nanotubes of different diameter and of the different regions of nano porous carbon. At a later step it is planned to model the dynamics of the flow of steam + ethylbenzene at such carbon structures, and the process of ethylbenzene dehydrogenation.

Where:

The student/postdoc will spend some time in Berlin and some time in the US. Details will be decided along the progress of the work.

We are looking for computational physicists, chemists, or chemical engineers. Good background in electronic structure theory, thermodynamics, and statistical mechanics is important

Please send your application material to:

Matthias Scheffler

Fritz-Haber-Institut phone : ++49-30-8413 4711

der Max-Planck-Gesellschaft fax : ++49-30-8413 4701

Faradayweg 4-6 e-mail: scheffler@fhi-berlin.mpg.de

D-14 195 Berlin-Dahlem / Germany

WWW: <http://www.fhi-berlin.mpg.de/th/th.html>



Proposal from 8/03/2002

The Laboratory for Neutron Scattering (ETH Zurich & PSI Villigen) has an open position for a scientist to work in the field of powder neutron diffraction. The position is on a contractual basis and has a duration of 2-3 years with an option for prolongation. The starting date is November 1, 2002.

Research Scientists (Physicists, Chemists, Crystallographers) are invited to apply for this position

• Your tasks:

- Responsibility for the operation and further development of a powder neutron diffractometer at the spallation source SINQ at PSI Villigen.
- Co-operation with guest scientists in their experiments at SINQ.
- Performance of your own research projects.

• Your profile:

- You are a graduated research scientist (PhD) with some years' experience in the field of neutron scattering, particularly with neutron diffraction.
- You have some practical knowledge of computing and cryogenics.
- You are willing to work in a team and to communicate (establishing a professional relationship with guest scientists) as well as to work flexible hours.

• For further information

please contact Prof. Dr. A. Furrer, phone: +41-56-3102088, fax: +41-56-3102939, e-mail: albert.furrer@psi.ch

Please send applications with C.V., a list of publications and the names of two academic referees no later than by April 30, 2002, to: Prof. Dr. A. Furrer, Laboratory for Neutron Scattering, CH-5232 Villigen PSI, Switzerland.



SPECIFIC ANNOUNCEMENT

FROM N. LYAKHOV (ON 7TH JUNE 2002)

PROCEEDINGS OF THE INTERNATIONAL CONFERENCE

"FUNDAMENTAL BASES OF MECHANOCHEMICAL TECHNOLOGIES"

ARE AVAILABLE IN ENGLISH ON-LINE.

YOU MAY VISIT THE WEB PAGE AT

[HTTP://WWW-PSB.AD-SBRAS.NSC.RU/CSDE.HTM](http://www-psb.ad-sbras.nsc.ru/csde.htm)

BEGIN WITH JULY 1ST.

[154] FABRICATION OF HOLLOW ALUMINA MICROSPHERES VIA CORE/SHELL STRUCTURE OF POLYMETHYLMETHACRYLATE/ALUMINA PREPARED BY MECHANOFUSION

Kato T. Ushijima H. Katsumata M. Hyodo T. Shimizu Y. Egashira M. - Journal of Materials Science. 37(11):2317-2321, 2002

Core/shell microspheres of polymethylmethacrylate coated with Al₂O₃ or a mixture of Al₂O₃ and SiO₂ nanoparticles have been successfully prepared by a mechanofusion system. The microspheres were then subjected to removal of PMMA by firing in air, followed by sintering at 1600degreesC for 3 h. The shell structure was destroyed after the sintering when the shell layer consisted of only Al₂O₃. The addition of SiO₂ was found to be effective for maintaining the original shell structure, while the shell size expanded slightly and a hole was formed in each hollow microsphere during the removal process of PMMA.

[153] MICROSTRUCTURES AND MECHANICAL BEHAVIOR OF BULK NANOCRYSTALLINE GAMMA-NI-Fe PRODUCED BY A MECHANOCHEMICAL METHOD

Qin XY. Lee JS. Lee CS. - Journal of Materials Research. 17(5):991-1001, 2002

The microstructures and mechanical behavior of bulk nanocrystalline gamma-Ni-xFe (n-Ni-Fe) with x = similar to 19-21 wt%, synthesized by a mechanochemical method plus hot-isostatic pressing, were investigated using microstructural analysis [x-ray diffraction, energy-dispersive spectroscopy, light emission spectrum, atomic force microscopy (AFM), and optical microscopy (OM)], and mechanical (indentation and compression) tests, respectively. The results indicated that the yield strength of n-Ni-Fe (d similar to 33 nm) is about 13 times greater than that of conventional counterpart. The change of yield strength with grain size was basically in agreement with Hall-Petch relation in the size range (33-100 nm) investigated. OM observations demonstrated the existence of two sets of macroscopic bandlike deformation traces mostly orienting at 45-55degrees to the compression axis, while AFM observations revealed that these bandlike traces consist of ultrafine lines. The cause for high strength and the possible deformation mechanisms were discussed based on the characteristics of microstructures and deformation morphology of n-Ni-Fe

[152] STRUCTURE AND HYPERFINE INTERACTIONS IN MECHANOSYNTHESIZED IRON-MOLYBDENUM ALLOYS

Jartych E. Karolus M. Oleszak D. Zurawicz JK. Sarzynski J. Budzynski M. - Journal of Alloys & Compounds. 337:69-75, 2002

X-ray diffraction and Mossbauer spectroscopy were used to study the structure and hyperfine interactions in Fe₈₀Mo₂₀ and Fe₅₀Mo₅₀ prepared by mechanical alloying. Two solid solutions, i.e. Fe(Mo) and Mo(Fe) with b.c.c. lattice were formed during milling of Fe₈₀Mo₂₀. Mossbauer spectra revealed different magnetic arrangements in these solid solutions. In the case of Fe₅₀Mo₅₀ no amorphisation was observed, as literature data suggest. During mechanosynthesis of Fe₅₀Mo₅₀ a paramagnetic Mo(Fe) solid solution was probably formed

[151] PROPERTIES OF A NEW NANOSIZED TIN SULPHIDE PHASE OBTAINED BY MECHANOCHEMICAL ROUTE

Balaz P. Takacs L. Ohtani T. Mack DE. Boldizarova E. Soika V. Achimovicova M. - Journal of Alloys & Compounds. 337:76-82, 2002

The paper deals with the properties of a new 4H-SnS₂ phase (N-phase) synthesized together with berndtite 4H-SnS₂ by a mechanochemical route from elemental tin and sulphur. The surface and bulk properties of SnS₂ phases were verified by surface area measurements, particle size analysis, electron microscopy, XRD, Mossbauer spectroscopy and chemical dissolution methods. The particles of synthesized product are 12-18 nm in size and during milling, secondary particles (aggregates) are formed that are 20-60 [µm in size. The particle size and a high developed surface area (7-21 m² g⁻¹) greatly influence the solid-liquid reaction and solid transformations of mechanosynthesized tin sulphide products

[150] STUDIES OF ALKALINE SOLID POLYMER ELECTROLYTE AND MECHANICALLY ALLOYED POLYCRYSTALLINE Mg₂Ni FOR USE IN NICKEL METAL HYDRIDE BATTERIES

Mohamad AA. Mohamed NS. Alias Y. Arof AK. - Journal of Alloys & Compounds. 337:208-213, 2002

Ni-MH cells comprising PVA-KOH solid polymer electrolyte and mechanically alloyed Mg₂Ni as the negative electrode have been fabricated. The alkaline solid polymer electrolyte with PVA:KOH wt.% ratio of 60:40 exhibits the highest room temperature ionic conductivity of 8.5 x 10⁻⁴ S cm⁻¹. This sample is mostly amorphous. The cell was charged at a constant current of 10 mA and discharged at 0.1 mA. The discharge characteristics improved upon cycling and the plateau voltage maintained above 1.2 V for similar to 10 h.



[149] BARIUM TITANATE DERIVED FROM MECHANOCHEMICALLY ACTIVATED POWDERS

Kong LB. Ma J. Huang H. Zhang RF. Que WX. - Journal of Alloys & Compounds. 337:226-230, 2002

A high-energy ball milling process was applied to a mixture with equal molar ratio of BaCO₃ and TiO₂. X-ray diffraction data indicated that there was no reaction happening during the milling process although the particle size was greatly reduced. The effect of post-annealing on the phase formation and grain growth of BaTiO₃ was investigated by thermal analysis (DTA/TGA). X-ray diffraction (XRD) and scanning electron microscopy (SEM). No reaction was observed in the sample annealed at 600degreesC. Ba₂TiO₄ was found as the mixture annealed at 700degreesC. An 800degreesC-annealing led to single phase BaTiO₃ with a cubic structure. The temperature at which BaTiO₃ phase was derived is lower than required by the solid-state reaction process, which could be attributed to the refined grains/particles of the mixture as a result of high-energy ball milling. Tetragonal structured BaTiO₃ was formed at 1150degreesC to, ether with a sharp increase in grain size

[148] ENHANCING LOW PRESSURE HYDROGEN STORAGE IN SODIUM ALANATES

Meisner GP. Tibbetts GG. Pinkerton FE. Olk CH. Balogh MP. - Journal of Alloys & Compounds. 337:254-263, 2002

We investigated the dehydrogenation of NaAlH₄ and the reversible low-pressure rehydrogenation from NaH to Na₃AlH₆. Highly purified NaAlH₄ requires relatively high temperatures to decompose to NaH and long durations to rehydride to the Na₃AlH₆ phase in hydrogen gas. However, any degradation of the purity of this material, whether through ball milling with diamond powder or ball milling with diamond Plus Al powders, mixing the purified material with Pt powder, or doping with a Ti organometallic compound, lowers the decomposition temperature and facilitates rehydriding the product NaH+Al to Na₃AlH₆. Diamond ball milling of NaAlH₄ seems to be the best of these procedures; it substantially decreases the decomposition temperatures, with significant dehydrogenation starting at 180 degreesC rather than 250 degreesC for the Purified material, and with formation of NaH substantially complete at 235 degreesC rather than 290 degreesC. Rather surprisingly, it also facilitates rehydrogenation from NaH+Al to Na₃AlH₆. Similarly, NaAlH₄ doped with Ti according to the recipe of Bogdanovie lowers the decomposition temperatures and improves the hydrogenation kinetics for the low pressure transition from NaH+Al to Na₃AlH₆. Pressure-composition isotherms show that the rehydrogenation of the resulting NaH+Al decomposition phases into the Na₃AlH₆ intermediate phase at pressures below 3.6 MPa is similar for the diamond ball milled and Ti-doped material. Diamond ball milling NaAlH₄ with excess Al did not improve the rehydrogenation kinetics.

[147] AMORPHIZATION AND GRAIN SIZE EFFECT ON MILLED PBTIO3 STUDIED BY RAMAN SCATTERING AND VISIBLE PHOTOLUMINESCENCE EMISSION

Lanciotti F. Pizani PS. Campos CEM. Leite ER. Santos LPS. Carreno NLV. Longo E. - Applied Physics a (Materials Science Processing). 74(6):787-789, 2002

Raman scattering and photoluminescence (PL) studies on milled PbTiO₃ are presented in this paper. The results suggest that the visible PL emission could be related to both the localized states in the interface between the amorphous layer and the crystalline core and the amorphous layer itself. The Raman spectrum of PbTiO₃ milled for a long time showed the vibrational density of states, and a detailed analysis of the soft mode allowed us to conclude that the PbTiO₃ crystalline core did not experience any structural phase transition.

[146] STRUCTURAL AND MAGNETIC CHARACTERIZATION OF HIGH-COERCIVE BALL-MILLED HARD MAGNETIC (SMCO5) PLUS ANTIFERROMAGNETIC (NIO) COMPOSITES

J Sort, J Nogues, S Surinach, JS Munoz, E Chappel, F Dupont, G Chouteau, MD Baro - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 465-471 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Ball-milling induces high values of microstrain and a rapid decrease of the crystallite size in SmCo₅ powders. However, milling is found to be less aggressive to SmCo₅ when it is milled together with a ceramic antiferromagnet, such as NiO. In addition, as the milling proceeds, SmCo₅ particles are progressively surrounded and soldered to NiO, finally becoming embedded in an anti ferromagnetic matrix. This microstructure, along with the local heating during the milling, favors the existence of antiferromagnetic-ferromagnetic exchange interactions, which result in an enhancement of the room-temperature coercivity, H-C. Moreover, values of squareness ratio, M-R/M-S (remanence/saturation magnetizations), very close to 1 are found in SMC₅+NiO ball-milled for 1 h, without need of particle alignment previously to measurement. The cooperative effects of ferromagnetic-ferromagnetic and anti ferromagnetic-ferromagnetic exchange interactions can account for this latter effect.

[145] MOSSBAUER SPECTROSCOPY STUDY OF FESI2 POWDERS PREPARED BY WATER ATOMIZATION

K Tokumitsu, M Wada - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 473-478 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

The structures of a water atomized and a mechanically milled FeSi₂ powder were studied with X-ray diffraction, scanning electron microscope and Fe-57 Mossbauer spectroscopy. X-ray diffraction pattern showed that a water atomized powder was constituted of alpha-FeSi₂, and epsilon-FeSi. The solidification structure like a dendrite was observed at the surface and the inside of their particles. And this powder changed to the nanostructured beta-FeSi₂, by mechanical milling without a thermal treatment. Fe-57 Mossbauer spectrum for a atomized powder was fitted with three paramagnetic doublets corresponded to



alpha-FeSi₂ and epsilon-FeSi. The spectrum for a mechanically milled atomized powder was fitted with two doublets of beta-FeSi₂.

[144] STRUCTURAL CHANGE AND FE-57 MOSSBAUER SPECTROSCOPY STUDY OF MECHANICALLY ALLOYED FE₂C POWDER

K Tokumitsu, M Umemoto- METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 479-484 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

The structural change of Fe₂C by mechanical alloying of elemental powders was studied with X-ray diffraction and Fe-57 Mossbauer spectroscopy. X-ray diffraction pattern changed from bcc alpha-Fe to the orthorhombic Fe₃C in the first stage of milling and to the hexagonal phase in the second stage. Amorphous phase was not obtained. Fe-57 Mossbauer spectra changed from alpha-Fe, H-int.=330 kG, to a ferromagnetic sextet, H-int.=205kG, in the first stage and split into two ferromagnetic ones, H-int.=165 and 210kG in the second stage. The atomic distance between Fe atoms for the hcp phase prepared by mechanical alloying was 2.57Angstrom, which was larger than that of the high pressure hcp epsilon-Fe.

[143] MAGNETIC AND TRANSPORT PROPERTIES OF MECHANOSYNTHESIZED FeCr₂S₄ SULFOSPINEL

Authors GF Goya - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 491-496 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

The FeCr₂S₄ sulfospinel has been synthesized from the elemental components, by high-energy ball milling. The magnetic properties of the resulting crystallites (mean diameter d = 120+/-10 nm) have been studied by means of ac and dc susceptibility, Mossbauer and transport measurements. Magnetization curves showed a para- to ferrimagnetic transition of FeCr₂S₄ phase at T-C = 165(3) K, and a cusp at T-L approximate to 70 K. The evolution of Mossbauer spectra at different temperatures indicates the existence of a disordered surface layer, and its thickness was estimated to be t = 8(2) nm. Transport measurements show that the onset of the insulator-metal (IM) transition at T = 241 K, a similar to 30% higher temperature than previous reports. In contrast, the 'colossal' magnetoresistance (CMR) effect observed in bulk FeCr₂S₄ is greatly reduced.

[142] INFLUENCE OF THE B CONTENT ON THE STRUCTURAL AND MAGNETIC PROPERTIES OF Fe₆₀Mn₁₀Al₃₀-xBX PREPARED BY MECHANICAL ALLOYING

MM Rico, J Sort, S Surinach, JS Munoz, JM Greneche, GAP Alcazar, MD Baro - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 497-502 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

High energy ball milling of Fe, Mn, Al and B powders has been used to prepare iron-based alloys, with nominal composition Fe₆₀Mn₁₀Al₃₀-xB_x, with x = 2, 4, 6, 8, 10. For comparison, alloys with the same composition have also been prepared by arc-melting. The effect of the B content on the microstructure and magnetic properties of the products has been studied by X-ray diffraction, scanning electron microscopy, vibrating sample magnetometry and magnetic thermogravimetry. After milling for 20 h a Fe-Mn-Al solid solution with a bcc structure is formed. As the B content is increased, a decrease in the bcc cell parameter is found. Moreover, the appearance of new (Fe-Mn)(₂)B phase becomes more evident for increasing B content. In addition, some superlattice peaks corresponding to the bcc phase are observed in the arc-melt samples. The alloys are found to be ferromagnetically soft. Two magnetic transitions are observed in ball-milled samples when the temperature is increased. The first one, which is associated to a magnetic transition in the bcc phase, is found to shift to higher temperatures as B content is raised. Changes in the Fe-Fe atomic distances in the bcc phase when changing B content are likely to be responsible for this magnetic behavior.

[141] STRUCTURE OF FE-AG SUPER-LAMINATES FABRICATED BY REPEATED ROLLING AND MECHANICALLY ALLOYED FE-AG POWDER

T Yoshioka, M Yasuda, H Miyamura, S Kikuchi, K Tokumitsu - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 503-508
INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

It is known that the Fe-Ag system is immiscible both in solid and in liquid. The maximum solubility of Ag in alpha-Fe is 0.0002at.% and the solubility of Fe in Ag is 0.0065at.%. This immiscible feature is favorable for the fabrication of nano-scaled multilayers material by repeated rolling, because that both Ag and Fe components are mutually insoluble in the interface between Ag and Fe layers. We studied the structure for both Fe-Ag super-laminate fabricated by cold rolling and mechanically alloyed (MA) powder by the use of X-ray diffraction, electron microscopy, Fe-57 Mossbauer spectroscopy and magneto-resistance (MR) measurement. MA powder with the composition of Fe-18.7at.%Ag corresponding to Fe-Ag super-laminates composed of ten thousands layers in 50µm in thickness was prepared. The Mossbauer spectra indicate that Ag and Fe atoms are immiscible in the super-laminates, but mixed slightly in MA powder. So lattice parameter of alpha-Fe slightly increased from 2.866Angstrom to 2.885Angstrom with milling time for Fe_{81.3}Ag_{18.7} powder. Hyperfine magnetic field decreased from 330kG to 327.5kG for the powder, but it did not change as 330kG for the super-laminates. The



magnetic spin direction for the super-laminates was near parallel to the rolled surface. MR effects for the super-laminates have both GMR (giant magneto- resistance) and AMR (anisotropic magneto-resistance) effects.

[140] HIGH ENERGY MILLING OF SI-DOPED TITANIUM ALUMINIDES - GENERAL PROBLEMS AND POTENTIAL APPLICATIONS

G Fanta, R Bohn, T Klassen, R Bormann - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 521-528 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Fully dense submicrocrystalline intermetallic/ceramic composites based on the system Ti-Al-Si are prepared by high energy milling and subsequent hot isostatic pressing (HIP). To avoid interstitial impurities like oxygen or nitrogen, all handling and processing is conducted under vacuum or controlled gas atmospheres (Ar or Ar-5 vol.% H₂). In the first part of the present study, argon pick-up during high energy milling is investigated - a general problem that has to be considered when preparing materials by powder metallurgical processing routes. Annealing experiments above the HIP temperature of the Ti-Al-Si-composites lead to the evolution of pores. Analytical and microstructural investigations demonstrate that the formation of this voids is caused by entrapped Ar atoms (concentration: similar to 60 mug/g). Upon annealing, Ar can diffuse, leading to a rearrangement of small Ar-filled nanopores and a coarsening of the pores. The overall density of the components is not affected by this process. By further increasing the temperature, the interior gas pressure of the pores may exceed the flow stress of the material and thus contribute to an inflating of the samples. In this case. The increase in porosity is confirmed by a drop in density. Tension tests on heat treated specimens elucidate the effect of (nano-)pores on the mechanical behavior. Finally, solutions for avoiding argon pick-up and porosity will be suggested. The second part of this study deals with hot-forming of submicrocrystalline intermetallic/ceramic composites. Due to the ultrafine grained microstructure these materials show excellent mechanical properties with regard to easy workability. During hot-forming, finely dispersed silicides along the grain boundaries of the equiaxed gamma-TiAl grains prevent coarsening of the matrix. Therefore, the deformation temperatures for processing techniques like extrusion or isothermal forging can be kept about 200 degreesC below those for conventional gamma-TiAl alloys. This shows that high energy milling is an attractive processing route for the synthesis of easily workable precursor materials.

[139] MICROSTRUCTURAL EVOLUTION AND MECHANICAL FLOW OF 6061 AL DURING EQUAL CHANNEL ANGULAR PRESSING

BS Moon, MH Seo, YS Kim, KH Lee, HS Kim, SI Hong - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 577-582 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

In this study, the microstructural evolution and mechanical flow of underaged and peak-aged 6061 aluminum alloys processed by equal channel angular pressing (ECAP) were investigated. The inhomogeneity of plastic flow is appreciably influenced by the hardening behavior of underaged and peak-aged 6061 al alloys. The corner gap was developed more pronouncedly in the underaged alloy than in the peak-aged alloy, consistent with the prediction by finite element analysis. The difference of the die corner filling behavior of underaged and peak-aged Al alloys can be related to the hardening behavior, which is strongly influenced by the difference of precipitates. One interesting observation is that some beta' precipitates are slightly curved or fragmented after ECAP. Precipitates in aged Al alloys are gradually fragmented and are likely to be dissolved after several passes of ECAP.

[138] MECHANICAL ACTIVATION OF GRANITE ROCK DUST: STRENGTH DEVELOPMENT IN PORTLAND CEMENT CONCRETE AND LIME MORTARS

PC Lessard, M HavensCook - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 589-596 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Approximately 2.5 kW-h/kg of energy are required to sufficiently activate granite powder so that it meets the ASTM requirement for mineral admixtures. Scanning electron microscopy/energy dispersive spectroscopy analysis shows evidence that a pozzolanic reaction occurs from the milled granite dust and an amorphous hydrated calcium silicate product is formed. X-ray diffraction, scanning electron microscopy/electron dispersive spectroscopy, particle size, surface area, and thermal characteristics (Thermogravimetric and differential scanning calorimetry) are described for milled and unmilled powders and mortar cube specimens. When used as a partial portland cement replacement in concrete, the compressive strength increases linearly with milling intensity.

[137] BULK SHAPE MEMORY NITI WITH REFINED GRAIN SIZE SYNTHESIZED BY MECHANICAL ALLOYING

Authors WC Crone, AN Yahya, JH Perepezko - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 597-602 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Shape memory alloys (SMAs) constitute a unique class of materials that undergo a reversible phase transformation allowing the material to display dramatic stress-induced and temperature-induced deformations which are recoverable. Nickel Titanium (NiTi) SMA has been synthesized with refined grain size by a mechanical alloying process. The SMA is produced



through a cold rolling fabrication strategy that yields nanocrystalline product structures. By starting with a stacked sandwich array of individual alloy components and subjecting this sample arrangement to repeated rolling and folding, a metal-metal multilayer composite is created. After the layer thickness is reduced to the nanoscale by multiple cold rolling and folding passes, an alloying and intermediate phase formation reaction is initiated. X-ray diffraction profiles confirm that the primary phase present is intermetallic NiTi phase, which is the phase that displays shape memory behavior. The presence of superelastic behavior and the shape memory effect in the product material is established by mechanical testing. At this refined grain size, the hysteresis stress observed is exceptionally small.

[136] MECHANOSYNTHESIS OF HYDROGEN STORAGE ALLOYS

GX Liang, J Huot, R Schulz - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 603-607 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

In the last decade, the mechanical alloying and mechanical grinding techniques have been used extensively to synthesise or modify hydrogen storage materials in an attempt to improve their hydrogen sorption properties. Since hydrogen diffusion through grain boundaries is usually much faster than in the bulk and since calculations show that the H/M ratio in metallic clusters containing hydrogen can reach high values when the size of clusters is reduced to the nanometer scale, one was hoping that ball milled nanocrystalline metal hydrides could have better absorption/desorption kinetics as well as improved storage capacity. The kinetics and the activation are improved in most cases but we usually observe a loss in capacity in as-milled AB₂, AB₅ and AB₂ hydrogen storage systems. For Mg-based hydrides, however, the loss of capacity is negligible. The loss of storage capacity is usually attributed to structural disorder which could be topological or chemical in nature. For instance, the loss of capacity and the change in the equilibrium plateau pressure in ball-milled nanocrystalline FeTi was attributed to amorphous grain boundary phases. Fortunately, this loss of capacity can be recovered by thermal treatment at relatively low temperature. In this paper, we report and compare the hydrogen storage properties of mechanically treated low and high temperature metal hydride systems and discuss the influence of structural disorder, microstrain and grain size on properties.

[135] ELECTROCHEMICAL PROPERTIES OF NANOCRYSTALLINE SN-BASED ALLOYS FOR LITHIUM BATTERIES

JH Ahn, YJ Kim, CJ Choi - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 609-614 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Tin-containing nanocrystalline Mg₂Sn and Ni₃Sn₄ were prepared by ball milling to see their electrochemical properties as a new anode material for lithium ion batteries. Electrochemical tests demonstrated that the initial charge-discharge capacities are very high for both materials. Although the capacity faded rapidly after the first cycle, reversible insertion-extraction of lithium occurred after the second cycle. Both capacity and cycle life of Ni₃Sn₄ were markedly improved for the ball-milled electrode as compared with the as-cast alloy electrodes indicating that the nanocrystalline structure of the starting materials plays an important role in enhancing lithium storage performance.

[134] SYNTHESIS OF B2-STRUCTURED MG ALLOYS BY MECHANICAL ALLOYING, AND THEIR HYDRIDING PROPERTIES

Authors S Hwang, C Nishimura - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 615-620 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Binary B₂-structured magnesium alloys were produced by ball milling magnesium with silver, lanthanum or palladium elemental powder in equiatomic quantities. MgLa showed the best hydrogen absorption characteristics among the as-milled alloys. Magnesium-lanthanide alloys have recently attracted much attention due to their optical switching and metal to semiconductor transitional properties with hydrogen absorption, and hence, hydriding behaviour of MgLa is of a great interest. Pressure-composition-temperature (PCT) and X-ray diffraction measurements showed that MgLa dissociates upon hydriding between 300 and 400°C. Lanthanum hydride was formed initially at very low hydrogen pressure (< 10⁻⁵ MPa) and then magnesium hydride was formed. Van't Hoff plot showed that pressure plateaus observed at various temperatures corresponded to that of magnesium. MgAg and MgPd intermetallic alloys were observed to form at a rapid rate during milling, consistent with their high negative enthalpy of formation. However, these alloys absorbed practically no hydrogen. Adding ternary elements such as titanium, vanadium and palladium to MgAg showed some improvement in hydrogen absorption.

[133] H₂-SENSING PROPERTIES OF NANOCRYSTALLINE SnO₂ POWDER PRODUCED BY MECHANOCHEMICAL MILLING

U Kersen - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 633-638 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Nanocrystalline tin dioxide powders have been produced by mechanochemical milling followed by heat treatment and subsequent preparation of SnO₂ thick-films. X-Ray powder diffraction was used to investigate the structure of the materials.



Crystallite sizes in the range from 15 to 20 nm (determined from XRD line broadening) were obtained. Heat treatment of the milled powder resulted in the formation of tetragonal and orthorhombic SnO₂ phases, which was confirmed by XRD analysis. The optimal parameters for the milling time, rotation speed and heat treatment were determined before this work and used in the current work. The gas-sensing properties of tin dioxide can be modified by adding of La₂O₃ as surface catalyst. La₂O₃ was added by dipping sintered tin dioxide films into varying concentrations of La(NO₃)₃.6H₂O solution. The response of the prepared thick-films to HA in the concentration range from 0.5 to 20 ppm in air, revealed that at a temperature of 250 degreesC, undoped SnO₂ sensors respond to H₂S much stronger than La₂O₃-doped SnO₂ sensors do.

[132] SYNTHESIS OF BIOMATERIALS BY MECHANOCHEMICAL TRANSFORMATION

G Gonzalez, R Villalba, A Sagarzazu- METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 645-650- INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Mechanochemical solid-phase reactions of different kinds of constituent powder mixtures were investigated with the aim of obtaining Hydroxyapatite (HAp). It was found that from mixtures of Ca(OH)₂ and P₂O₅ or (NH₄)₂HPO₄ milled for different periods and calcined at 800 degreesC is possible to obtain HAp of average particle size of 200 nm. The process was followed by XRD, IR and TEM. It is observed that amorphization begins at 17 hours of milling. The material completely recrystallizes and transforms to HAp and calcium phosphate by heating at 800degreesC for 4 h.

[131] REACTIVE FABRICATION OF NANOCRYSTALLINE TITANIUM CARBIDES BY MECHANICAL ALLOYING

XK Zhu, BC Cheng, KY Zhao, QS Lin, XQ Zhang - PROGRESS IN SELF - PROPAGATING HIGH - TEMPERATURE SYNTHESIS (Series: KEY ENGINEERING MATERIALS), 2002, Vol 217, pp 77-82 - 1ST SINO-RUSSIAN WORKSHOP ON SELF-PROPAGATING HIGH-TEMPERATURE SYNTHESIS; BEIJING, PEOPLES R CHINA. SEPTEMBER 21-24, 2000

nanocrystalline titanium carbide (TiC) powders have been investigated through mechanically induced self-propagating reaction (MSR) mechanism. The process was performed using a high-energy ball mill under water-cooled and air ambient. It was found that the occurrence of the MSR was related with ambient temperature. The progress of the reaction for the Ti-C system has been followed by means of X-ray diffraction, scanning electron microscopy and laser particle sizer. Analysis of the product shows that the TiC of nanocrystalline size (20nm) and fine particle (average particle diameter 5.64 um) can be synthesized at about 2h. With further milling, The crystalline size decreased to 7nm after milling 10h and the average particle diameter increased to 40.61 um after milling 28h. The lattice distortion was relatively low. The broadening of the diffraction peaks mainly ascribed to crystalline refinement. The lattice constants of the product TiC decreased during subsequent milling. The products have porous character of the SHS products.

[130] NANOSTRUCTURES IN AL-BASED ALLOYS AFTER SEVERE PLASTIC DEFORMATION

SV Dobatkin, VV Zakharov, RZ Valiev - RECRYSTALLIZATION AND GRAIN GROWTH, VOLS 1 AND 2, 2001, pp 509-514 - 1ST JOINT INTERNATIONAL CONFERENCE ON RECRYSTALLIZATION AND GRAIN GROWTH; AACHEN, GERMANY. AUGUST 27-31, 2001

The structure and microhardness after severe plastic deformation (SPD) in torsion under high pressure of Al-Cu-Mg alloy 2024, binary Al-Sc alloys with contents of Sc-0,14%, 0,4% and 0,55%, and also industrial Al-Mg-Sc alloys 01515, 01535, 01545 and 01570 are investigated. At SPD of quenched alloy 2024 with supersaturated solid solution nanocrystalline structure with the size of a grain 50 nm is obtained due to strain aging. At SPD of binary Al-Sc alloys the only submicrocrystalline structure is formed. Addition more than 3% Mg in Al-Sc alloy results in formation of nanostructure during SPD. The minimal size of a grain - 40 nm is revealed in alloy Al-5,9%Mg-0,3%Sc.

[129] NANO- AND SUBMICROCRYSTALLINE LOW-CARBON STEELS OBTAINED BY SEVERE PLASTIC DEFORMATION

SV Dobatkin, PD Odessky, NA Krasilnikov, GI Raab, VN Konenkova - RECRYSTALLIZATION AND GRAIN GROWTH, VOLS 1 AND 2, 2001, pp 543-548 - 1ST JOINT INTERNATIONAL CONFERENCE ON RECRYSTALLIZATION AND GRAIN GROWTH; AACHEN, GERMANY. AUGUST 27-31, 2001

The opportunity of ultra-fine grained structure formation in low-carbon steels during cold and warm severe plastic deformation (SPD) in torsion under pressure 6GPa and equal channel angular (ECA) pressing is investigated. At cold SPD of 0,09CMnSi and 0,1CMnVTi steels the nanoscale structure is formed: oriented like-cell fragments and grains. The deformation of initial quenched steels with nonequilibrium structure results in the greater refinement of structure, than deformation of initial hot-rolled state. At warm ECA pressing of 0,21CMnSiV and 0,25CMnSi steels the mixed structure is formed: dynamically recovered and submicrocrystalline with the grain sizes 300...500 nm. Such partially submicrocrystalline structure causes a high-strength state: YS > 1000 MPa, EL = 10%.

[128] PRODUCTION OF THERMOELECTRIC MATERIALS BY MECHANICAL ALLOYING - EXTRUSION PROCESS

JM Simard, D Vasilevskiy, F Belanger, J LEcuyer, S Turenne - TWENTIETH INTERNATIONAL CONFERENCE ON THERMOELECTRICS, PROCEEDINGS, 2001, pp 132-135 - 20TH INTERNATIONAL CONFERENCE ON THERMOELECTRICS (ICT 2001); BEIJING, PEOPLES R CHINA. JUNE 8-11, 2001

The extrusion process offers one of the greatest prospects for the industrial production of Bi₂Te₃ based thermoelectric alloys. When coupled with mechanical alloying, this process promises substantial cost savings because of its ability to deliver net shape components, leading to a reduction in the so-called kerf losses and higher material yields. It is also well known that materials produced using one of the various powder processing routes have greater mechanical strength when compared to



conventionally grown alloys, leading to an improved reliability, We have produced both p ($\text{Bi}_{2-x}\text{Sb}_x\text{Te}_3$) and n-type ($\text{Bi}_2\text{Te}_3\text{-ySey}$) alloys using mechanical alloying and extrusion and have studied the structure, composition and thermoelectric properties of these alloys. By carefully controlling every step of the process we have found that it is possible to produce rods of thermoelectric material, for both p and n-type alloys, which have good properties. Figures of merit of up to 3.35×10^{-3} K⁻¹ for p-type alloys and 2.8×10^{-3} K⁻¹ for n-type alloys have been obtained. A fivefold improvement in the mechanical properties over conventional melt grown material has also been observed.

[127] THE MECHANOCHEMICAL SYNTHESIS OF THERMOELECTRIC MATERIAL IRON DISILICIDE BASED

EY Belyaev, GA Suchkova, AI Ancharov, OI Lomovsky, VI Maly - TWENTIETH INTERNATIONAL CONFERENCE ON THERMOELECTRICS, PROCEEDINGS, 2001, pp 218-220 - 20TH INTERNATIONAL CONFERENCE ON THERMOELECTRICS (ICT 2001); BEIJING, PEOPLES R CHINA. JUNE 8-11, 2001

The iron disilicide is the cheapest thermoelectric material available for mass production of generators. The problem is a low efficiency of iron disilicide. The increasing of efficiency can be distinguished by means of doping of iron disilicide, making composite material based on, diminishing the grain size of iron disilicide ceramics. The mechanical alloying of elements is the most convenient method of iron silicide production. The goal of our work was to improve the properties of iron disilicide ceramics by method of nonconventional multicomponents doping. The mechanochemical synthesis was performed by means of high energy planetary ball mill ISSCM construction. The balls acceleration was 600 m/sec². The compositions of synthesized powders was follows: $\text{Fe}_{1-x}\text{D}_x\text{Si}_{2-y}\text{D}'_y$, where D, D' was Co, Al, B, Cr, Nd and x,y was from 0 to 12 at%. Synthesized powders has a nanoscale grain sizes and phases composition were the mixtures FeSi_2 and FeSi phases. The powders compacting was performed by conventional pressing under 2-4 t/sm² and by explosive compacting under varied condition. Compacted samples were sintered in vacuum 10^{-6} torr at 1150degreesC and annealed at 800degreesC. The properties of powders and ceramics were investigated by X-Ray diffraction on Synchrotron Irradiation. The thermoelectric properties were examined also, and compared with the properties of conventional materials.

[126] BIOACTIVE ORGANIC-INORGANIC COMPOSITE PREPARED BY MECHANOCHEMICAL METHOD

SB Kim, SB Cho, CJ Cho, YJ Kim, HS Jeon, TH Lee, Y Hwang - BIOCERAMICS 14 (Series: KEY ENGINEERING MATERIALS), 2002, Vol 218-2, pp 295-298 - 14TH INTERNATIONAL SYMPOSIUM ON CERAMICS IN MEDICINE (BIOCERAMICS-14); PALM SPRINGS, CALIFORNIA. NOVEMBER 14-17, 2001

A bioactive sol-gel derived powder/PEEK(TM) composite was prepared by combination of mechanochemical and hot press method for the repairing of bone defects. After milling for 30mins., fine-grained bioactive powder was well dispersed on the surface of the PEEK(TM) powder. It was also shown that the novel composite formed bonelike apatite on its surface after soaking into simulated body fluid for 2 weeks. This result indicates that the composite bone to living bone when implanted into bone defects. From the results, it was suggested that combination of mechanochemical and hot press method is effective technique for fabricating bioactive organic/inorganic composite for repairing of bone defects.

[125] SINTERING OF INJECTION MOLDED NANO-STRUCTURED TUNGSTEN HEAVY ALLOY POWDER COMPACTS

X Qu, J Fan, B Huang - HEAT TREATING, VOLS 1 AND 2, PROCEEDINGS, 2000, pp 985-988 - ANNUAL CONFERENCE ON HEAT TREATING; ST LOUIS, MISSOURI. OCTOBER 9-12, 2000

A powder injection molding processing was established in this study. The nano-composite powder with composition of 90W-7Ni-3Fe (wt.%) was prepared by mechanical alloying with a planetary ball mill. The molding and sintering behaviors of the nano-composite powder were investigated and compared with those of the conventional mixed powder. The experimental results show that milling significantly increase the maximum powder loading, improve the homogeneity of the feedstock and greatly enhance the sintering process. Even when sintered at 1350 similar to 1450 degreesC without liquid phase existing, the samples exhibit full density, very fine grains (similar to 3 μ m), high tensile strength and almost no sintering distortion.

[124] NANOSTRUCTURE SYNTHESIS AND AMORPHIZATION DURING COLD ROLLING

JH Perepezko, RJ Hebert, RI Wu - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 11-20 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Many forms of materials processing involve departures from full equilibrium conditions, but during processing the thermodynamic constraints usually apply locally at interfaces as a metastable equilibrium. With deformation-induced alloying the stored energy due to defects, grain refinement and solute supersaturation is a measure of the level of metastability that is crucial to consider in microstructure analysis. Systematic study of the deformation structure patterns that develop during the repeated cold-rolling of initial elemental multilayer arrays offers an effective approach to the examination of the mechanisms involved in deformation alloying, nanostructure synthesis and amorphization. For a given alloy the comparison between structure synthesis by melt processing and deformation processing can also provide useful insight. For some systems such as the bulk amorphous Zr-Al-Cu-Ni both methods yield equivalent amorphous states. In marginal glass-forming alloys such as Al-Sm solid-state and melt processing yield distinctly different amorphous phases. In other systems deformation-induced nanocrystal synthesis can be observed from an amorphous initial structure. These developments represent a major level of microstructure control that impacts the structural performance and stability.

[123] SIGNIFICANCE OF THE HEAT OF MIXING FOR THE AMORPHIZATION OF MULTILAYERS BY DEFORMATION PROCESSING

RJ Hebert, JH Perepezko - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 21-26 - INTERNATIONAL SYMPOSIUM ON



METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

With the recent improvements in severe plastic deformation (SPD) and cold-rolling (CR), deformation processing has become an alternative to rapid-solidification for the processing of bulk-metastable structures. Further progress is linked to the prediction of glass-forming systems as well as to the understanding of the deformation-induced transformation reactions. Cold-rolling experiments on systems with different mixing enthalpy values, including Al-Pt, Al-Pd, Al-Ni and Al-Cu, indicate that the heat of mixing is fundamental to the reactions that occur during the processing. However, cold-rolling of Al-Hf demonstrates that a large negative heat of mixing alone is not sufficient for rolling-induced amorphization reactions. SEM cross sectional images of Al-Hf and Al-Pt systems reflect a connection between the layer refinement and the amorphization reaction. Complete amorphization is only possible with a layer refinement to a nm-thickness level. In addition to a large negative heat of mixing, a rapid structural refinement is therefore necessary for rolling-induced amorphization reactions. Measurements of the interfacial area per unit volume highlight the difference between the layer-refinement of cold-rolled Al-Pt and Al-Hf samples.

[122] CONSOLIDATION OF PARTIALLY AMORPHOUS AL-FE-ZR ALLOYS

CAD Rodrigues, DR Leiva, KR Cardoso, CS Kiminami, WJ Botta - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 33-38 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

We present in this work the preparation of partially amorphous Al-Fe-Zr alloy powders by mechanical alloying and their consolidation by hot-extrusion. Elemental powder mixture corresponding to the composition Al₉₀Fe₇Zr₃ Was mechanically alloyed with milling power of 10:1 and time varying from 5h to 100h. Milling for 60h resulted in a powder alloy microstructure composed mostly of nanocrystalline Al and amorphous phase. Heat treatment of such powder resulted in the formation of Al and also the equilibrium intermetallic Al₃Zr and Al₁₃Fe₄ phases. The alloy powders have been hot extruded at 450degreesC and the microstructure of the consolidated alloy was very fine and composed also by the three equilibrium phases; Al, Al₃Zr and Al₁₃Fe₄. This fine microstructure resulted in yield stress values in compression tests at room temperature in the range of 780MPa, associated with elongation to fracture of 10%.

[121] FORMATION OF GLASSY Ti50Cu20Ni24Si4B2 ALLOY BY HIGH-ENERGY BALL MILLING

LC Zhang, J Xu - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 47-52 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Under high-energy ball milling, elemental powder mixtures with a nominal composition of Ti₅₀Cu₂₀Ni₂₄Si₄B₂ can be amorphized via solid state reaction. A well-defined glass transition before crystallization appears in the obtained amorphous phase during heating in a differential scanning calorimeter. A wide supercooled liquid region, AT, is detected being about 57 K. Crystallization of the ball-milled amorphous phase was performed through one step to form the metastable (Ni, Cu)Ti phase with cubic structure and an unknown phase, which is a eutectic-type transition, followed by subsequent grain growth. The reduced glass transition temperature of the alloy was determined to be 0.56.

[120] PHASE EVOLUTION AND MICROSTRUCTURAL CHARACTERISATION OF HIGH-ENERGY BALL MILLED AL-SI-FE-NI ALLOYS

RD SaLisboa, MNRV Perdigao, CS Kiminami, WJ Botta - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 59-64 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

The effects of milling power and milling time on phase evolution during high-energy ball milling, were investigated for the Al₅₀Si₃₀Fe₁₅Ni₅ alloy. Elemental powders were milled in a SPEX 8000 apparatus and the resulting powders were characterised by X-ray diffraction (XRD) and differential scanning calorimetry (DSC). Partially amorphous powders, together with the (Al,Si)₇Ni₃ intermetallic compound, were obtained in early stages of milling. Silicon remained unreacted throughout long milling times, with particle size diminishing down to nanometric levels. Long milling times resulted in a single amorphous phase, though highly contaminated by the milling tools. Milling time and milling power showed a compensatory effect on the nature of phases obtained.

[119] VISCOSITY OF MECHANICALLY ALLOYED AMORPHOUS ZR-CU-AL-NI MATRIX COMPOSITES IN THE SUPERCOOLED LIQUID REGION

S Deledda, J Eckert, L Schultz - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 71-76 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

We report on the viscosity in the supercooled liquid region of mechanically alloyed Zr₅₅Cu₃₀Al₁₀Ni₅ glassy matrix composites containing increasing volume fractions of CaO or ZrC as a second phase. Results obtained by parallel plate rheometry are compared with single-phase amorphous Zr₅₅Cu₃₀Al₁₀Ni₅ containing small carbon or calcium additions (up to 4 at.%). This allows an estimation of the effect of different matrix compositions, resulting from limited particle dissolution processes occurring upon milling, on the viscous flow behaviour of the supercooled liquid and on the amount of free volume



of the glassy phase. Once dissolution processes are taken into account, the contribution of second phase particles on the overall viscosity is discussed considering the Einstein equation for the flow of mixtures.

[118] ELECTRICAL DISCHARGE ASSISTED BALL MILLING: A PROMISING MATERIALS SYNTHESIS AND PROCESSING METHOD

A Calka, D Wexler - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 125-134 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Novel devices for reactive ball milling, incorporating high voltage, low current electrical discharges were constructed and their application for materials synthesis and processing investigated. The effects of low frequency, high voltage electrical impulses during milling on fracturing, nitriding and mechanical alloying were studied for a range of metal and ceramic powders. Samples prepared by both conventional milling techniques and electrical discharge assisted milling were examined and compared using standard techniques of metallography, x-ray diffraction and electron microscopy. Reactive milling experiments were performed under different combinations of glow (cold) and spark (hot) discharge conditions and were found to result in completely different reaction paths for the same reacting species. Glow discharge milling generally promoted formation of metastable and nanostructural products and the enhancement of reactions, such as nitration of solids in nitrogen gas. Spark discharge milling was found to promote different reactions, including the direct formation of new phases, such as NiSi, from elemental ingredients, reduction reactions, including formation of magnetite from hematite and reduction of TiN and Si₃N₄, and rapid solid-liquid reactions, including the direct formation silicon carbide from elemental Si and toluene. Spark discharge milling also promoted formation of product in more stable microcrystalline form. For brittle, low electrical conductivity materials, including hematite, ilmenite and alumina, it was found that the electrical discharges associated with this milling method significantly speeded up fracturing, the fracture mechanism involving the bulk breakdown of individual powder particles. For conductive metals, fracturing was found to occur via chipping and shaving from the surface of particles. Discharge assisted milling was particularly useful for the enhancement of reactions of elemental powders with molecular nitrogen, boron and with hydrocarbon liquids. This resulted in increases in reaction rate, changes in the reaction route and significant extension of the range of materials that can be treated in this way. For metals such as Ti and Zr, nitration in nitrogen gas could be greatly speeded up. Other metals/metalloids, including Si, Ge, Fe, and Al, which are either more difficult or impossible to process by conventional milling in molecular nitrogen, were found to react with N₂ using electrical discharge milling. Within the mill, the electrical discharges cause molecular breakdown of the controlled atmosphere, the formation of monatomic gasses and, depending on the species present and discharge conditions, formation of specific types of plasma in the proximity of powder particles. For discharge milling in a gas atmosphere, processes have similarities with both conventional ion/plasma surface engineering techniques and direct plasma processing of powder particles. However, it is the combined process of electrical discharge and milling which separates it from other techniques and allow new reaction routes to be explored.

[117] ATMOSPHERIC AND MILLING-DEVICE EFFECTS ON THE ACTIVATION ENERGY FOR CRYSTALLIZATION OF A PARTIALLY AMORPHIZED NI-MO ALLOY

R MartinezSanchez, I EstradaGuel, D JaramilloVigueras, SD DelaTorre, C GaonaTiburcio, J GuerreroPaz - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 135-140 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

The Ni-Mo system processed through mechanical alloying (MA) technique has been widely investigated. For the composition Mo-47 at % Ni partially amorphous phase was obtained after 36 h milling time. Fe contamination was detected in the samples milled in a stainless steel container. Longer milling times have an important effect in the crystallization temperature. The peak temperature was shifted to lower temperatures as increases the milling time. Milling device, milling container, Fe and oxygen have an important effect in the rate of amorphization and in the activation energy (Q) for crystallization. Q values for samples milled under air atmosphere are lower than those observed in samples milled in argon atmosphere.

[116] MODELING OF FAST MICROCONTACT INTERACTIONS OF SOLIDS DURING MILLING, MECHANICAL ALLOYING AND MECHANICAL ACTIVATION BY DYNAMIC NANOINDENTATION TECHNIQUE

YI Golovin, VI Ivolgin, VV Korenkov, AI Tyurin, BY Farbe - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 141-146 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Initial stages of milling, mechanical alloying and mechanical activation processes involve fast local plastic deformation in "hot spots" on contacting surfaces. However, very little is known about dynamic mechanical properties of materials in localized microcontacts. A new pulse nanoindentation technique is developed to investigate dynamics and micromechanisms of plastic deformations in the above-described conditions. The nanohardness tester allowed in-situ recording of local plastic deformation with high spatial and time resolution (similar to 1 nm and similar to 50 μs accordingly). It was also capable of recording electrical polarization of the indentation zone with a time resolution of similar to 100 ns. It is established that the process of the indenter penetration under pulse loading conditions can be described by several distinct stages (as many as



four in some cases) differing in kinetics and activation parameters. Relative contributions of non-equilibrium point defects and dislocations in each stage are discussed.

[115] EFFECT OF SINTERING CONDITIONS ON THE MARTENSITIC TRANSFORMATION IN A MA AND SPS SINTERED CU-50 AT.% CO ALLOY

L DiazBarrigaArceo, E Orozco, K Tsuchiya, M Umemoto, VM LopezHirata - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 147-152 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

The martensitic transformation was studied in a mechanically alloyed and then spark plasma sintered Cu-50 at.% Co alloy. The mechanical alloying of this alloy composition produced the formation of a supersaturated solid solution with nanometric grains. The martensite start temperature was determined to be below room temperature by means of electrical resistance measurements during cooling of sintered samples. The martensite start temperature decreased with the copper addition, compared to that of pure cobalt. Additionally, this temperature also decreased as the sintering pressure increased.

[114] CONSOLIDATION OF MECHANICALLY ALLOYED FE-27AT%AL AT HIGH PRESSURE

G Gonzalez, L DAngelo, J Ochoa, D Bonyuet - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 153-158 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

In the present work consolidation of nanocrystalline mechanically alloyed powders of Fe-27at% Al supersaturated solid solution after different milling times, were carried out under different hydrostatic pressures. A study of the effect of pressure and temperature on the material consolidation is presented. Higher densification is obtained for the highest pressure and sintering temperature. It was found that the combination of high pressure and high temperature (2 GPa-600degreesC) results in a more homogeneous microstructure with smaller grain size.

[113] THE INFLUENCE OF MILLING INTENSITY ON MECHANICAL ALLOYING

Authors G Gonzalez, L DAngelo, J Ochoa, B Lara, E Rodriguez - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 159-164 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

The frequency of vibration of the mill affects directly the milling intensity and therefore the ball to powder impact energy, this parameter has an influence on the alloy formation and on the particle size. In this work we have modified a Wig-L-Bug Spex mill in order to control the vibrating frequency. These results are compared with those obtained for the same milling periods with different ball size and also with milling in a conventional horizontal Wig-L-Bug and in a Spex 8000. The ball to powder ratio was kept constant for all the cases at 8:1. The ball motion was studied by observation with a high speed video camera through a transparent vial and the ball velocity calculated. The impact energy (the kinetic energy transfer to the powder at each collision event) was calculated and the vial frequency was changed to cover a significant impact energy range. Specific relations between vial frequency, impact velocity, impact energy, alloy formation and particle size with milling time were deduced. The materials were characterized by X-ray diffraction, and scanning electron microscopy for each milling condition. The milling periods were between 15 min and 5 h. The particle size and alloy formation depended strongly on the impact energy. Nanometer particle size and complete alloy formation were reached in shorter periods for higher intensities, for long periods the particle size was not affected by milling intensity.

[112] STRUCTURAL AND CHEMICAL CHARACTERIZATION OF CU-AG AND NI-AG NANOCOMPOSITES SYNTHESIZED BY HIGH-ENERGY BALL-MILLING

S Zghal, P Bhattacharya, R Twesten, F Wu, P Bellon - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 165-174 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Mechanical alloying is widely used to synthesize nanostructured materials. In the case of alloys with positive heat of mixing, it is also sometimes possible to force a solid solution by low temperature milling (e.g., Cu-Ag), or to stabilize a nanocomposite by intermediate temperature milling (e.g., Cu-Ag or Ni-Ag). In order to gain insight on the fundamental understanding of these driven phase transformations, and in order to be able to optimize these nanostructures, a systematic study on the steady-states reached by temperature-controlled high energy ball-milling has been undertaken. In addition to standard x-ray diffraction and scanning calorimetry measurements, field ion microscope atom probe (AP), transmission and transmission scanning electron microscopy (TEM, STEM) are used to characterize the ball-milled powders. It is shown that the Cu-Ag microstructure after low temperature milling can exhibit a pronounced texture, with a complex microstructure. At higher milling temperature dynamic recrystallization leads to small equiaxed nanograins that are randomly oriented. In the Ni-Ag case, the absence of texture in the Ni-rich phase, as well as the very small grain size, suggest that full mixing of the species cannot take place because of the differences in mechanical properties of the terminal phases.

[111] MECHANICALLY ALLOYED (FE0.5CU0.5)(100-X)ZR-X ALLOYS

P Crespo, P Marin, P Agudo, MC Alocen, A Hernando, A GarciaEscorial, J Eckert, S Roth, L Schultz - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM),



2002, Vol 386-3, pp 175-180 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Alloys with nominal composition $(\text{Fe}_{0.5}\text{Cu}_{0.5})_{100-x}\text{Zr}_x$ ($7 \leq x \leq 17$) have been synthesized by high-energy ball milling of elemental powders. X-ray diffraction patterns show that the alloys with a Zr content below 13 at.% take a face centered cubic (fcc) structure with an average crystallite size around 6 nm. In all cases, the fcc structure is expanded with respect to that of fcc-Cu, suggesting the dissolution of Fe and Zr into the fcc lattice. For higher Zr content, the x-ray patterns show a broad halo typical of amorphous alloys. Room temperature Mossbauer spectroscopy confirms the alloy formation and indicates that the Fe environments in the nanocrystalline and amorphous alloys are quite similar. Thermomagnetization measurements point out that the alloys in both structural states are ferromagnetic with a Curie temperature ranging between 240 and 305 K, depending on the composition and structure.

[110] EVOLUTION OF STRUCTURE IN THE DISORDERED NANOCRYSTALLINE FE75SI25 ALLOY UNDER HEAT TREATMENT

EP Yelsukov, VM Fomin, OM Nemtsova, DA Shklyayev - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 181-186

The evolution of structure of the ground Fe-Si alloy towards equilibrium during heating-up with a heating rate of 20K/min, and during isothermal annealing at $T = 600\text{K}$, was studied by differential thermal analysis, X-ray diffraction and Mossbauer spectroscopy. Seven stages of structure evolution were found.

[109] AMORPHIZATION AND REACTIVITY OF SILICON INDUCED BY MECHANICAL TREATMENT

Authors AN Streletskii, AV Leonov, IV Beresteskaia, SN Mudretsova, AF Majorova, PJ Butyagin - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 187-192 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

The kinetics of amorphization of silicon as well as the reactivity of mechanically disordered silicon with oxygen and hydrogen were measured under mechanical treatment of silicon in the atmosphere of argon, hydrogen, or oxygen. The mechanical treatment of silicon in an inert atmosphere is accompanied by the formation of two fractions (coarse and fine) of nanoparticles along with the formation of the amorphous phase. The kinetics of grain sizes changing for both fractions were analyzed. One may conclude that the decrease of grain size below the critical value of about 4 nm is accompanied by transition of the material into the amorphous phase. The amount of amorphous phase for the maximum dose, ($D=500 \text{ kJ/g}$) exceeds 40%. The amorphous phase crystallization was accompanied by the exothermic thermal effects at the temperatures of 560-700 degreesC. During the mechanical treatment in the atmosphere of active gases, an irreversible absorption of oxygen and hydrogen is observed. The value of chemisorption of hydrogen is comparable to the number of surface atoms measured by the BET method. It does not prevent the formation of an amorphous phase. Chemisorption of oxygen depends on the gas pressure. At high gas pressure, the number of sorbed oxygen atoms reaches similar to 50% of the total number of silicon atoms and is comparable with the number of atoms in the amorphous phase. One can assume that mostly oxygen forming oxide with the atoms in the amorphous phase.

[108] FORMATION AND STABILITY OF METASTABLE PD(ZR) SOLID SOLUTION DEVELOPED DURING BALL MILLING AND/OR HEAT TREATMENT OF PD3ZR

GL Katona, M KisVarga, DL Beke - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 193-197 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Formation of nanocrystalline solid solution induced by ball milling and/or subsequent annealing of the Pd3Zr intermetallic compound was studied. The single phase initial compound was milled in a medium energy vibrating mill under vacuum for various time periods. The structural characterization was made by X-ray diffraction with monochromatic $\text{CuK}\alpha$ radiation. After a short milling time (46 h) the Pd3Zr compound transformed to nanocrystalline one without disordering. By annealing this ball milled material, the formation of Pd(Zr) solid solution was observed by X-ray diffraction. During heat treatment at increasing temperatures the phase fraction of Pd(Zr) reached a maximum between 800 and 900 degreesC, then it decreased and finally the reflections of disordered phase disappeared from XRD spectrum at 1330 degreesC indicating the return to the single phase ordered state. However, while the above results are in accordance with that of Weissmuller and Ehrhardt [1], we also observed that ball milling of Pd3Zr for longer periods resulted in direct transformation of increasing fraction of the ordered phase to nanocrystalline Pd(Zr) solid solution. The amount of this phase increased with milling time and with annealing temperature, and the disordered state proved to be metastable even at high temperatures. The stabilization of solid solution phase is explained by the segregation at grain boundaries of the nanocrystalline material and by oxidation at interfaces/grain boundaries during the preparation and annealing processes.

[107] CHARACTERIZATION OF ALLOYED CEMENTITE PRODUCED BY MECHANICAL ALLOYING AND SPARK PLASMA SINTERING

M Umemoto, ZG Liu, DY Liu, H Takaoka, K Tsuchiya - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 199-204 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001



Following our recent successful fabrication of bulk cementite (Fe,C, O) by mechanical alloying (MA) and subsequent spark plasma sintering (SPS), the influence of Cr, Mn, V, Mo, Ti, Ni and Si additions on the structure and properties of cementite bulk has been studied. It has been found that Cr, Mn, V and Mo can form alloyed cementite containing more than 5% of the alloying element and stabilize it, while Ti, Ni and Si destabilize or even completely suppress the formation of cementite. The hardness and Young's modulus of the alloyed cementite were increased in the order of V, Cr, Mn and Mo. The contribution of alloying elements to the hardness and Young's modulus was found to correlate well with their enhancement of bonding strength with iron atoms. At higher temperature, the alloyed cementite showed a higher hardness and compression strength. The large effect of Mo and V can be explained by misfit parameter of atomic radii. Thermal expansion coefficient of all the cementite shows an abnormally low value just below Curie temperature due to magnetostriction.

[106] STRUCTURAL INVESTIGATIONS OF CUZR PREPARED BY MECHANICAL ALLOYING

A AlHajry, M AiAssiri, S AiHeniti, S Enzo, J Hefne, A AShahrani, AE AiSalami - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 205-210 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Mechanical alloying (MA) technique has been used to prepare a series of Cu58Zr42 samples as a function of milling time. This has been done to study the amorphization reaction in this system. Cu58Zr42 has the best X-ray visibility of compositional short range order (CSRO) of CuxZr1-x system. X-ray diffraction was used to investigate the structural changes during the amorphization reaction. A complete amorphous phase has occurred after 4 h MA time. No diffraction peaks from the intermetallic CuZr phases were observed throughout the amorphization reaction. The crystallization temperature and the activation energy for crystallization of the amorphous CuZr alloy were obtained by differential scanning calorimetry (DSC) measurements.

[105] A NEUTRON SMALL-ANGLE SCATTERING STUDY OF THE HfPd SYSTEM

A AlHajry, M AiAssiri, S Enzo, N Cowlam, J Hefne, L Jones, F Delogu, H Brequel - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 211-216 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

The HfPd system exhibits a fast amorphization reaction. A series of specimens has been prepared by mechanical alloying (MA). X-ray diffraction (XRD) and small angle neutron scattering (SANS) were used to characterise the structural transformations during the amorphization reaction. Contrast-matching technique was used to isolate the powder surface effects and obtain the scattering from the bulk of the specimens. The structural transformations observed by the wide-angle measurements are reflected in the large-scale structure and this was detected by SANS. A strong SANS intensity profile is obtained for the parent specimen relative to those obtained for the MA treated specimens. More interesting is that the SANS intensity profiles for both the amorphous HfPd obtained by MA and that obtained by fast quenching (FQ) are almost identical, indicating similar structure on a large scale. However, the structure of particles within the amorphous phase obtained by MA is of more convoluted surfaces compared to those produced by FQ. Graphs of the $\log[I(Q)]$ vs. $\log(Q)$ are linear over a wide range of scattering vector Q indicating the fractal nature of the structure in these specimens. The fractal dimensions D were obtained as a function of MA time.

[104] SYNTHESIS AND CHARACTERIZATION OF NICKEL, COBALT AND IRON RANEY CATALYSTS PREPARED BY MECHANICAL ALLOYING AND ALKALINE LEACHING

Authors J AngelesIslas, M ArzolaMendoza, BH Zeifert, JG CabanasMoreno, HA Calderon, H YeeMadeira, R Zamorano - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 217-222 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Raney Ni, Co and Fe catalysts have been produced by mechanical alloying (MA) of elemental powder mixtures followed by leaching in aqueous alkaline solutions, with the resulting product being a fine and highly porous solid. The precursor alloys were prepared from Al50Ni50, Al65Ni35, Al50Co50, Al60Co40, Al40Fe60 and Al50Fe50 (at. Pct.) elemental mixtures. X-ray diffraction revealed that Al3Ni2, AlNi, AlCo and AlFe intermetallic phases were produced by MA. After leaching, the phases present in the materials changed mainly from Al3Ni2 to Ni and NiO; from AlCo to fcc Co and CoO; from AlFe to Fe2O3 and bcc Fe. Only the AlNi phase was not leached to any significant degree. The catalysts produced in this way had specific surface areas in the range 70 to 140 m²/g. In the case of the nickel catalyst derived from the Al3Ni2 intermetallic produced by MA, electron spin resonance measurements indicated a peculiar behavior which may be explained by a large fraction of Ni atoms occupying grain boundary sites.

[103] NIAL-AL2O3 NANOCOMPOSITE POWDERS FORMED BY REACTIVE MILLING

D Oleszak - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 223-228 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Reactive milling processes were performed for NiO + Al powder mixture in order to obtain "in situ" composite with NiAl matrix reinforced with alumina. A combustion type of reaction was observed and the formed particles exhibited a polyhedron morphology and size about 150 μm. Prolonged milling led to particle size refinement down to 10 μm and change of



morphology into spherulitic. The Hall-Williamson analysis revealed an anisotropy in crystallite size and lattice strain. Adding powders of products (NiAl ore Al₂O₃) allowed to avoid a combustion type of reaction.

[102] DIAMOND SYNTHESIS BY MECHANICALLY INDUCED DETONATION

AR Torosyan, VS Baghdasaryan, VG Martirosyan, SG Aloyan, HG Balayan, HV Navasardyan, SA Barseghyan - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 229-233 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

The process of diamond powder synthesis by mechanically induced detonation of exothermic solid-phase reactions is investigated. It is determined that the exothermic energy of the reactions and the energy saved during the mechanical activation of some solid-phase [MeO-Me-1] type systems allow to synthesize diamond and diamond-like substances under dynamic conditions.

[101] CHEMOMECHANICAL EFFECTS ACCOMPANYING MECHANOCHEMICAL REACTIONS AND CREEP

EM Gutma - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 235-244 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Mechanochemical phenomena are often interpreted through the mechanochemical effect (MCE) manifesting itself by a change in the chemical reaction rate assisted by mechanical stresses in solids. However, as it was shown using non-equilibrium thermodynamics, there exists inevitably a conjugated cross-effect that I have called chemomechanical effect (CME). It manifests itself by a change in the mechanical state of a solid and plasticity increase due to the influence of chemical (electrochemical) reaction on its surface causing an additional dislocation flux. Earlier the existence of CME was directly confirmed by hardness measurements under the conditions of anodic polarization or crystalline material etching. Now we analyze CME relevant to the problems of creep and fracture in solids. For a quantitative description of MCE and CME and experimental verification, one should proceed from basic kinetic equations for the reaction rate and creep rate, respectively. To explain the mechanism of CME it is interpreted as the phenomenon caused by heterogeneous nucleation and action of new sources of dislocations resulting from heterogeneous surface dissolution. The role of surface conditions in plastization of a solid is discussed, and differences of CME from other surface effects (e. G., Rehbinder effect) are shown. The new kind of stress corrosion called corrosion creep is demonstrated for Mg and Mg alloys as a consequence of CME and MCE autocatalytic interaction. The next problem will be to find CME in amorphous materials (e.g., metal glasses) where MCE has been already found experimentally.

[100] PREPARATION OF DISPERSER CERAMIC MATERIALS BY SOFT MECHANOCHEMICAL SYNTHESIS

Authors EG Awakumov, LG Karakchiev, AA Gusev, OB Vinokurova - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 245-249 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Specific features of soft mechanochemical synthesis as a method to prepare dispersed ceramic materials are considered. The advantages of soft mechanochemical synthesis are demonstrated using the preparation of ZrTiO₄, Al₂TiO₅ and ZrSiO₄ dispersed powders as examples. It is shown that the soft mechanochemical synthesis allows obtaining nano-sized particles close in size to the particles obtained by sol-gel method. The existence of optimal composition range has been demonstrated in which the synthesis occurs with highest efficiency.

[99] NEW MECHANOCHEMICAL METHOD FOR METAL COATING

AR Torosyan, JR Tuck, AM Korsunsky, SA Bagdasaryan - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 251-256 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

A novel approach for metal surface modification and finishing is proposed and developed based on the in situ mechanochemical processing of metal oxides in reducing media. As typical examples of the new coating process, mechanochemical deposition of pure chromium and titanium coatings on steel and aluminum substrates was chosen. The possible mechanisms of the formation of the mechanochemically deposited layer under impact conditions and some mechanical properties have been investigated. We identify the so-called welding mechanism as the most probable mechanism for coating formation under mechanical impact conditions. Hardness of the coating was chosen as a key property, since it is indicative of the work-hardened state of the material in the layer, the grain size and hence the coating strength. Hardness was measured by multiple load indentation, allowing the variation with of hardness with depth to be studied. The data was interpreted using the work-of-indentation model, and suggests that the coating shows a considerable hardness increase compared to the bulk property of the same material, but also a moderate increase compared to the substrate. The coating is therefore thought likely to provide improved wear resistance.

[98] MECHANOCHEMICAL REDUCTION OF COPPER SULFIDE

P Balaz, L Takacs, JZ Jiang, V Soika, M Luxova - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 257-262 -



INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

The mechanochemical reduction of copper sulfide with iron was induced in a Fritsch P-6 planetary mill, using WC vial filled with argon and WC balls. Samples milled for specific intervals were analyzed by XRD and Mossbauer spectroscopy. Most of the reaction takes place during the first 10 min of milling and only FeS and Cu are found after 60 min. The main chemical process is accompanied by phase transformations of the sulfide phases as a result of milling. Djurleite partially transformed to chalcocite and a tetragonal copper sulfide phase before reduction. The cubic modification of FeS was formed first, transforming to hexagonal during the later stages of the process. The formation of off-stoichiometric phases and the release of some elemental sulfur by copper sulfide are also probable.

[97] MECHANOCHEMICAL TREATMENT OF SCRAP TIRE RUBBER -

M Magini, F Cavaliere, F Padella - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 263-268 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

The Mechanical Alloying (MA) process can be attractive as a low environmental impact technology having potential applications in several fields. In this direction, in the present work, we describe the use of high energy ball milling technique on ground rubber as a mean for scrap tire rubber recycling. Vulcanised rubber powder was successfully devulcanised under the application of impact mechanical energy. Progress of mechanochemical de-cross-linking at different milling times was monitored by measuring extracted sol fractions and cross-linking densities of milled samples. The effect of the presence of an antioxidant agent on the milling process, was evaluated comparing solubility and cross-linking density of samples milled with and without the agent.

[96] PRODUCTION OF NANOSTRUCTURED COO, CO3O4 AND NIO PARTICLES BY MECHANICALLY INDUCED REACTIONS

G GarciaPacheco, JG CabanasMoreno, F CruzGandarilla, H YeeMadeira, M Umemoto - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 281-286 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

In the present work, the oxidation reactions: $\text{CO} + (1)/\text{O}-2(2) \rightarrow \text{CoO}$, $3\text{CoO} + (1)/\text{O}-2(2) \rightarrow \text{Co}_3\text{O}_4$ and $\text{Ni} + (1)/\text{O}-2(2) \rightarrow \text{NiO}$ were induced by mechanical milling. These oxidation reactions were carried out under air atmosphere and with the addition of methanol as a process control agent to avoid excessive agglomeration of the original metallic powders. Characterization of product phases has been done using x-ray diffraction, scanning electron microscopy, transmission electron microscopy and microanalysis by energy dispersive x-ray spectrometry. Oxidation of cobalt produced CoO and CO₃O₄ phases; the addition of methanol delayed the formation of the CO₃O₄ phase. For nickel, the product phases were NiO and a residual amount of Ni. Cobalt powders were more easily oxidized than Ni powders, probably as a result of their smaller initial particle size. The resulting crystallites in the oxide particle had sizes smaller than 20nm in both cases.

[95] REACTION KINETICS AND MICROSTRUCTURAL EVOLUTION DURING THE HEATING OF HIGH ENERGY BALL MILLED AL-TiO2 AND AL-V2O5 COMPOSITE POWDERS

Authors DL Zhang, DY Ying, G Adam - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 287-292 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Solid state reactions and corresponding microstructural evolution of the powder particles during heating Al/TiO₂ and Al/V₂O₅ composite powders produced by high energy ball milling have been studied. It is demonstrated that establishment and refining of the composite structure of the powder particles through milling strongly enhances the reaction kinetics. The enhancement is likely due to the creation of a large area of Al/oxide interfaces and the reduction of the inter-particle spacing of the oxide particles inside the powder particles. Al₃Ti or Al₃V or other Al rich Al_xV_y phase is the first phase to be formed from the reaction between Al and TiO₂ or V₂O₅ which starts at a temperature close to the melting point of Al. When the Al/V₂O₅ composite structure is very fine, gamma-Al₂O₃ may also form from the initial reaction. The powders must be heated to above 900degreesC in order for alpha-Al₂O₃ to nucleate. At temperatures above 1000degreesC, Al₃Ti is transformed into Ti(Al,O) by reacting with TiO₂.

[94] SOLID STATE REACTIONS BETWEEN V2O5 AND C DURING MECHANOCHEMICAL PROCESSING AND HEATING OF V2O5/C COMPOSITE POWDERS

DL Zhang, G Adam - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 293-298 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Solid state reactions between V₂O₅ and C activated by mechanical Milling and by heating the high energy mechanically milled V₂O₅/C composite powders have been investigated. It has been found that during both milling and heating processes, the reduction of V₂O₅ by carbon proceeds in three steps: Step 1: V₂O₅ --> VO₂, Step 2: VO₂ --> V₂O₃ and Step 3: VO₂ --> V₄C₃. Mechanical milling is capable of activating the first two step reactions, although at different times. Heating of the mechanically milled powders activates the different step reactions at different temperature. The first step reaction occurs at



around 400degreesC, the second step reaction occurs at approximately 720degreesC, while the third step reaction occurs at about 1000degreesC. The V2O5/C mole ratio in the starting powder has little effect on the first two step reactions, but significantly enhances the kinetics of the third step reaction. It is believed that this enhancement was due to a large area of V2O5/C interface formed with increasing amount of carbon in the powder. It has also been observed through thermal analysis using DTA and TGA that the third step reaction is highly endothermic which explains why it cannot be activated by mechanical milling.

[93] SPARK PLASMA SINTERING OF ALUMINA-CR AND -NB COMPOSITES

Authors SD DelaTorre, DE Garcia, N Claussen, R Janssen, Y Nishikawa, H Miyamoto, R MartinezSanchez, A GarciaL, D RiosJara - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 299-303 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Attrition-milled Nb-50vol%Al2O3 and Cr-50vol%Al2O3 powders have been densified from 13 10 to 1450degreesC in about 10min using the spark plasma sintering (SPS) process applying 45MPa of pressure. The milling intensity as conducted on the metal-Al2O3 powders has been found to play a fundamental role determining not only the degree of densification but also the final nature of the composites thus processed. Although the longer milling time conducted on the precursor powders has lead to a material of finer microstructure, the formation of hydroxides hinders its densification capability and strength. The maximum fracture strength developed in these composites has been attained in the Nb-Al2O3 material reaching 825MPa. The SPS densification process of these cermets is discussed.

[92] FORMATION OF NANOCRYSTALLINE FERRITE IN FE-0.89C SPHEROIDITE BY BALL MILLING

M Umemoto, ZG Liu, Y Xu, K Tsuchiya - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 323-328 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Nanocrystalline ferrite has been produced in eutectoid carbon steel with spheroidite structure through heavy deformation of ball milling. It was found that nanocrystallization of ferrite occurred as wavy band structure on the surface region of particles during ball milling, where localized heavy deformation takes place, of 0.1 mum thickness and several mum length. The volume fraction of nanocrystalline bands increases with increasing milling time until complete nanocrystallization. Together with the nanocrystallization of ferrite, the dissolution of cementite was observed. The nanocrystalline ferrite reveals a totally different morphology, microstructure and mechanical properties from the work-hardened ferrite. By annealing the deformed specimens, recrystallization was observed in the work hardened regions, while in the nanocrystalline ferrite region only a weak grain growth was observed instead of recrystallization.

[91] PROCESSING AND PROPERTIES OF DENSE CU NANOCOMPOSITES

EY Gutmanas, A Trudler, I Gotman - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 329-334 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Nanocrystalline copper powder and Cu-TiN blends were prepared via oxidation of micron/submicron Cu powders followed by attrition milling of the Cu oxide formed with or without TiN nanoparticles. This was followed by reduction of the oxide to Cu metal and cold sintering/high pressure consolidation in air or in vacuum, The materials obtained were further annealed at 200 to 800degreesC in order to study the thermal stability of microstructure. Microstructure characterization was performed by X-ray diffraction, scanning electron microscopy with energy dispersive analysis (SEM/EDS) and high resolution SEM (HRSEM). The microhardness and yield stress of as-consolidated and annealed nanoscale Cu-based materials were measured. The described processing route yielded near fully dense nanocrystalline copper and Cu-TiN nanocomposites. The mechanical properties of materials obtained were significantly higher than those of conventional Cu. Based alloys. Annealing at > 400degreesC of Cu and Cu-TiN specimens cold sintered in air resulted in a decreased density and lower mechanical properties not accompanied by any noticeable growth of the nanograins. At the same time, no density or mechanical properties decrease was observed after similar anneals for specimens cold sintered in vacuum. The expansion of air-consolidated Cu-based materials is assumed to be due to the presence of entrapped air in the nanopores. Cu based nanocomposites can be used for high strength wear resistant electric contacts or for fabrication of bearings where combination of high hardness/strength with high thermal conductivity is important.

[90] RECENT DEVELOPMENTS IN MECHANOCHEMICAL NANOPARTICLE SYNTHESIS

PG McCormick, T Tsuzuki - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 377-386 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

The results of recent investigations of the mechanochemical synthesis of CaCO3, Cr2O3 and Nb2O5 nanopowders are reported. With all three materials studied, it is shown that the volume fraction of the matrix phase is crucial to the formation of separate, unagglomerated particles. With Cr2O3 and Nb2O5, amorphous particles were formed by mechanochemical reaction and low temperature heat treatment was required for crystallization. It is shown that, as a 'bottom up' process,



mechanochemical processing enables the building up nanoparticles through solid-state chemical reaction in a microscopically uniform environment, leading to the formation of nanoparticles with narrow size distributions.

[89] MICROSTRUCTURAL INVESTIGATION OF GRAIN STABILITY IN CRYOMILLED INCONEL 625

KH Chung, J Lee, R Rodriguez, DH Shin, EJ Lavernia - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 403-408 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

The grain growth behavior of nanocrystalline Inconel 625 powders prepared by cryomilling (mechanical milling under a liquid nitrogen environment) was investigated using X-ray diffraction (XRD) and transmission electron microscopy (TEM). The average grain size of powders after 8 hours cryomilling was 22 nm. Along with this fine structure, ultrafine NiO and Cr₂O₃ oxide particles were distributed in the cryomilled material with average size of 3 nm. It was found that the grain size remain under 250 nm after 4 hours heat treatment at 800°C, which correspond to T/T(m) similar to 0.65. The cryomilled Inconel 625 showed improved grain stability compared to that of conventional Inconel 625 and cryomilled pure-Ni, due to the particle pinning of grain boundary by the oxide particles in addition to solute drag.

[88] THERMALLY STABLE NANOCRYSTALLINE AL-MG ALLOY POWDERS PRODUCED BY CRYOMILLING

Authors F Zhou, R Rodriguez, EJ Lavernia - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 409-414 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Low energy mechanical attrition under liquid nitrogen (i.e., cryomilling) was used to produce nanocrystalline Al-Mg alloy powders. The microstructural evolution during cryomilling of spray-atomized Al-7.5Mg (wt.%) alloy powders was characterized by using X-ray diffractometry and scanning electron microscopy. Cryomilling reduced the grain size to similar to 20 nm and produced a supersaturated face-centered cubic (fcc) solid solution having approximately 7.5 wt.% Mg in solution. The formation of nanocrystalline microstructure in the Al-Mg samples was associated with the total lattice strain of fcc Al, resembling that of other conventional milling processes with high or low milling energy levels. The nanocrystalline Al-Mg alloy powders were found to exhibit a high degree of thermal stability at elevated temperatures.

[87] MODELLING OF EQUAL CHANNEL ANGULAR PRESSING FOR ULTRAFINE-GRAINED METALS

HS Kim, MH Seo, SI Hong, HR Lee, BS Chun, KH Lee - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 421-426 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

This paper presents a brief overview of the recent developments in equal channel angular pressing (ECAP) for manufacturing bulk ultrafine grained (including nanocrystalline) metals, being done in the authors' laboratory. The deformation behaviour and mechanical properties of equal channel angular pressed metals are investigated using numerical techniques. The properties of the ECAP processed materials are strongly dependent on the shear deformation behaviour during plastic deformation, which is controlled mainly by die geometry, material properties, and process conditions. The plastic deformation behaviour of the materials during the ECAP process with a frictionless condition was investigated using the commercial two-dimensional rigid-plastic finite element code (DEFORM2D). The inhomogeneous strain distribution within the workpiece was analysed. Due to the faster flow of the outer part compared to the inner part within the main deforming zone, the lesser shear zone in the outer part of the workpiece occurs. The prediction of the deformation inhomogeneity is compared with the experimental data. A lumped heat transfer analysis that ignores the temperature inhomogeneity of the workpiece was made in order to investigate the temperature rise of the workpiece during ECAP. As a novel process for commercial applications, equal channel multi-angular pressing (ECMAP) is analysed, focusing on the high strain generation by one pass and the deformation homogenisation of materials during the pressing.

[86] SPIN DYNAMICS OF NANOSTRUCTURED LA₂/3CA₁/3MNO₃

GF Goya, HR Rechenberg, MR Ibarra - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 433-438 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Single phase, ultrafine La_{0.7}Ca_{0.3}MnO₃ particles have been produced from polycrystalline material by mechanical grinding, consisting of crystallites of mean diameter $D = 10(4)$ nm. The para- to ferromagnetic transition found in bulk samples at $T-C = 254(2)$ K is barely observable in milled samples. Zero-field-cooling (ZFC) curves exhibit a maximum at T approximate to 45 K, below which irreversible behavior appears. The real part of the ac magnetic susceptibility $\chi'(T)$ showed a frequency-dependent cusp at T-m approximate to 50 K, indicating a low-temperature (reentrant) spin glass phase. The normalized shift per frequency decade was $\Delta T(m)/(T-m \Delta \log \omega) = 0.008$, close to the values for canonical spin-glasses like AuMn and CuMn. We show that Arrhenius or Vogel-Fulcher models for a thermally activated process yield unphysical results, whereas the correct determination of the freezing temperatures $T-f(\omega)$, based on the criterion $\tan \phi = \chi'' / \chi' = \text{constant}$, solves this inconsistency. From the obtained $T-f(\omega)$ values, a dynamical analysis according to conventional critical slowing down gave $T-g = 47.4(4)$ K and $z\nu = 11.8(2)$ for SG transition temperature and dynamic exponent, respectively. These results indicate a spin-glass transition at finite temperature. Resistance measurements in zero and applied fields show semiconducting behavior, and the colossal Magnetoresistance Effect (CMR) observed in bulk



samples is virtually absent. From the R(T) dependence it is inferred that those mechanisms that originate the MR at low fields, such as tunneling and spin-dependent scattering seems not to be present. We discuss this behavior and compare it with other data on related La-Sr-Mn-O and La-Ca-Mn-O systems, both bulk and nanostructured.

[85] DISORDERED MAGNETISM AT THE GRAIN BOUNDARY OF PURE NANOCRYSTALLINE IRON

A Hernando, P Crespo, MS Flores, L DelBianco, F Briones - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 447-453 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

Nanocrystalline iron was synthesized by two different processing techniques in order to study the influence of the magnetic properties of grains boundaries on the macroscopic magnetic behavior. First, nanocrystalline Fe powders with an average crystallite size of around 12 nm were obtained by high-energy ball milling. Two different components are resolved by Mossbauer spectroscopy, which are attributed to the crystallites and to the grain-boundary region. The results of dc and ac susceptibility, remanence and magnetic relaxation measurements indicate a transition from a high-temperature ferromagnetic state to a low-temperature disordered regimen. The effect has been connected with the order-disorder transition of the spin located at the grain boundary. Secondly, we report on transport and magnetic measurements on Fe thin films grown by sputtering. A surprising minimum in the low-temperature thermal dependence of the resistance is observed. Since such anomaly completely disappears under the action of a magnetic field it is inferred that it originates from spin-dependent scattering arising from the disordered grain boundaries. All results clearly indicate that below a critical temperature, both systems behave as a random assembly of weakly interacting magnetic units.

[84] MAGNETISM AND STRUCTURE OF NANOPARTICLES AND MESOSCOPIC SYSTEMS

AY Yermakov, MA Uimin, AA Mysik, AY Korobeinikov, AV Korolyov, NV Mushnikov, T Goto, VS Gaviko, NN Schegoleva - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2002, Vol 386-3, pp 455-464 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS; ANN ARBOR, MICHIGAN. JUNE 24-29, 2001

In the present report the analysis of the finite-size effect on the magnetic properties and structures of nanoparticles and microclusters is given. The formation of non-homogenous structure in the process of mechanical alloying of immiscible components (Cu-Co, Cu-Fe) allows these samples to be used as model objects to verify the fundamental magnetic behavior as a function of the particle size. Abnormal magnetic characteristics (parameters of the shifted hysteresis loop, the susceptibility, etc.) in magneto-ordered state (Ni, Cu-Co, Cu-Fe and others) have been observed for the nanoscale range particles synthesized by different methods (gas condensation method, mechanical alloying etc.) and have been characterized using low and high magnetic fields (up to 40 T). It has been shown that the size of particles enriched by 3d-metal decreases as the concentration of 3d-metal is reduced, The particle size of pure Ni strongly influences the magnetic hysteresis behavior, The shifted hysteresis loop and irreversibility of the magnetization at high magnetic field have been observed. We discuss the magnetic properties of these nanoparticles taking into account the exchange interaction between non-collinear magnetic structure of the surface layer and collinear structure of the particle core.

[83] LDPE-G-MAH PREPARED THROUGH SOLID-PHASE MECHANOCHEMISTRY AND ITS COMPATIBILIZING EFFECTS ON HDPE/CACO3

Wang Q. Chen HQ. Liu Y. - Polymer-Plastics Technology & Engineering. 41(2):215-228, 2002.

In this paper, a self-designed pan-type mechanochemical reactor was used to achieve solid-phase grafting of maleic anhydride (MAH) onto low density polyethylene (LDPE), which is a new method for realizing functionalization of polyethylene (PE) and can overcome the disadvantages of traditional chemical grafting reactions. The pulverization and stress degradation of LDPE in mechanochemical process was investigated, The grafting copolymer LDPE grafted MAH (LDPE-g-MAH) obtained through the method was confirmed by Fourier transform infrared spectroscopy and differential scanning calorimetry (DSC) analysis, and the grafting rate of MAH onto LDPE could reach 1.4%. The compatibilizing effects of the grafting copolymer on high density PE and calcium carbonate system (HDPE/CaCO₃) were also studied. The results of scanning electron microscopy observation and mechanical properties tests showed that the prepared LDPE-g-MAH could improve the compatibility between polymer matrix and inorganic fillers obviously, and enhance the impact strength and elongation at break of the composites to a great degree

[82] SINTERING OF COMPOSITES WITH INTERMETALLIC MATRIX [GERMAN]

Schubert T. Bohm A. Kieback B. Guthier V. Scholl R. - Materialwissenschaft und Werkstofftechnik. 33(4):200-203, 2002

The present paper reports on basic investigations of the controlled reaction sintering of MoSi₂-based materials, including particle reinforced composites. It is shown, that certain amounts of preformed silicide phase in the powder mixtures are useful to achieve closed porosity by pressureless sintering of green compacts. This partial phase formation of alphaMoSi₂(2) can already take place during the mechanical treatment of the elemental powder mixtures by milling. In this context the role of volume changes linked with the phase formations are discussed

[81] PREPARATION OF MOSI2-X(X = B, AL, NB) ALLOYS BY MECHANICAL ALLOYING AND PULSE DISCHARGE SINTERING PROCESS

Park YH. Shan AD. Hashimoto H. - Materials Transactions. 43(3):352-354, 2002

Mechanical alloying and pulse discharge sintering (MA-PDS) process was employed to fabricate MoSi₂ alloys with additions of Al, B or Nb alloy elements. Microstructure and mechanical properties of these alloys were investigated. An exothermic behavior was identified during milling. This process is related to the reaction of each element to synthesize



intermetallic MoSi₂. The MoSi₂ alloys fabricated by MA-PDS process showed a very fine microstructure compared to that sintered from the commercial MoSi₂ alloy powders. Significant hardness increase was found due to the refinement of the binary MoSi₂ microstructure. Alloys made from powders milled in air show higher hardness compared to those in Ar gas. This was because of the oxides formed during milling process

[80] THERMALLY ENHANCED GLASS FORMATION IN MULTILAYERED CU₃₃ZR₆₇ COMPOSITE PARTICLES

El-Eskandarany MS. Inoue A. - Materials Transactions. 43(3):608-610, 2002

A single phase of glassy Cu₃₃Zr₆₇ powders has been synthesized by post-annealing the mechanically alloyed Cu₃₃Zr₆₇ composite powders at 685 K for 1.8 ks. The heat of glassy phase formation was measured directly, and found to be -2.59 kJ/mol. The glassy powders transform to big-cube CuZr₂ metastable phase (E9 structure) upon heating to 760 K. This metastable phase does not withstand against the temperature increasing during the DSC analysis and therefore transforms completely into the most stable phase of tetragonal CuZr₂ (C16 structure) at 918 K.

[79] PULSED CURRENT SINTERING OF AMORPHOUS TITANIUM ALLOY POWDER SYNTHESIZED BY MECHANICAL ALLOYING PROCESS [JAPANESE]

Kobayashi K. Matsumoto A. Nishio T. Ozaki K. - Journal of the Japan Institute of Metals. 66(3):155-158, 2002

Ti-2 at%Fe-10 at% Si, Ti-4 at% Fe-10 at% Si and Ti-6 at% Fe-10 at%/Si powders were synthesized by mechanical alloying (MA) of pure Ti, Fe and Si powders using a planetary ball milling for 720 ks. After milling for 720 ks. the amount of collectable powder decreased with increasing Fe content in the MA powder. The formation of an amorphous phase was observed at all compositions in this study after 720 ks milling. The crystallization temperature of Ti-2 at% Fe-10 at% Si powder synthesized by MA for 720 ks was higher than that of other MA powders. Ti-2 at%Fe-10 at%Si powder prepared by milling for 720 ks, which contained amorphous phase, was consolidated using a pulsed current sintering (PCS) apparatus under a high pressure. The compact consolidated at 673 K under a pressure of 1500 MPa was almost densified with the retention of amorphous phase. The pulsed current sintering under a high pressure is a powerful technique to consolidate amorphous powder into a bulk material.

[78] THERMAL STABILITY OF MECHANICALLY ALLOYED ZR-CU-AL-NI GLASS COMPOSITES CONTAINING ZrC PARTICLES AS A SECOND PHASE

Deledda S. Eckert J. Schultz L. - Scripta Materialia. 46(1):31-35, 2002

Mechanically alloyed Zr₅₅Cu₃₀Al₁₀Ni₅ metallic glass matrix composites powders containing up to 30 vol% of ZrC particles were investigated by differential scanning calorimetry. It is shown that the supercooled liquid behaviour is not affected by the presence of the particles, but changes in the thermal stability suggest deviations in the overall composition of the glassy matrix

[77] DETERMINATION OF ELASTIC MODULUS OF NANOCRYSTALLINE IRON AND TITANIUM BY MEANS OF ACOUSTIC MICROSCOPY

Cao HS. Hunsinger JJ. Elkedim O. - Scripta Materialia. 46(1):55-60, 2002

It has been shown that mechanical milling is a very suitable and versatile method to produce nanocrystalline iron and titanium. Hot consolidation produces nearly fully dense compacts. The mechanical characteristics of the materials have been determined with use of high frequency acoustic microscopy. Compared to conventional metals, hardness was enhanced and elastic moduli were similar.

[76] CHARACTERIZATION AND PERFORMANCE ANALYSIS OF SILICON CARBIDE ELECTROLYTE MATRIX OF PHOSPHORIC ACID FUEL CELL PREPARED BY BALL-MILLING METHOD

Dheenadayalan S. Song RH. Shin DR. - Journal of Power Sources. 107(1):98-102, 2002

The effect of ball milling in making a silicon carbide slurry for the electrolyte matrix of a phosphoric acid fuel cell (PAFC) was studied by measuring the zeta potential and the particle-size distribution, and by analyzing cell performance. The ball-milled slurry gives a better particle distribution than the conventional mechanical-stirring method, and the particle distribution of the slurry depends on balling time and pH, which is confirmed by zeta potential. A single cell with a ball-milled electrolyte matrix also displays high performance. It is concluded that the ball-milling method is preferable to the mechanical-stirring procedure for preparing silicon carbide slurries.

[75] EFFECT OF BALL MILLING ON STRUCTURAL AND ELECTROCHEMICAL PROPERTIES OF (PEO)(N)(LiX=LICF₃SO₃ AND LiBF₄) POLYMER ELECTROLYTES

Shin JH. Lim YT. Kim KW. Ahn HJ. Ahn JH. - Journal of Power Sources. 107(1):103-109, 2002

Polymer electrolytes consisting of poly(ethylene oxide) (PEO) and lithium salts, such as LiCF₃SO₃ and LiBF₄ are prepared by the ballmilling method. This is performed at various times (2, 4, 8, 12 h) with ball:sample ratio of 400:1. The electrochemical and thermal characteristics of the electrolytes are evaluated. The structure and morphology of PEO-LiX polymer electrolyte is changed to amorphous and smaller spherulite texture by ball milling. The ionic conductivity of the PEO-LiX polymer electrolytes increases by about one order of magnitude than that of electrolytes prepared without ball milling. Also, the ball milled electrolytes have remarkably higher ionic conductivity at low temperature. Maximum ionic conductivity is found for the PEO-LiX prepared by ball milling for 12 h, viz. 2.52 x 10⁻⁴ S cm⁻¹ for LiCF₃SO₃ and 4.99 x 10⁻⁴ S cm⁻¹ for LiBF₄ at 90 degreesC. The first discharge capacity of Li/S cells increases with increasing ball milling time. (PEO)(10)LiCF₃SO₃ polymer electrolyte prepared by ball milling show the typical two plateau discharge curves in a LUS battery. The upper voltage plateau for the polymer electrolyte containing LiBF₄ differs markedly from the typical shape.

[74] EFFECT OF BALL MILLING ON MORPHOLOGY OF CUP-STACKED CARBON NANOTUBES



Kim YA. Hayashi T. Fukai Y. Endo M. Yanagisawa T. Dresselhaus MS. - Chemical Physics Letters. 355(3-4):279-284, 2002

The effect of grinding on cup-stacked carbon nanotubes is studied. The application of a bending force on the sidewall of such carbon nanotubes, that is higher than the Van der Waals force acting between the cups, results in the shortening of carbon nanotubes and the formation of nano-barrels exhibiting an increased number of accessible active sites (represented by the ends of graphitic planes) and anisotropic properties at both ends.

[73] BARIUM TITANATE SCREEN-PRINTED THICK FILMS

Stojanovic BD. Foschini CR. Pavlovic VB. Pavlovic VM. Pejovic V. Varela JA. - Ceramics International. 28(3):293-298, 2002.

Barium titanate (BT) thick films were prepared from mechanically activated powders based on BaCO₃ and TiO₂. After homogenization and milling in a high-energy vibro mill, the powders were calcined at 700 degreesC for 2 h by slow heating and cooling rates. A thick film paste was prepared by mixing BT fine powders with small amount of low temperature sintering aid and organic binder. The thick films were screen-printed on alumina substrates electroded with Ag-Pd. The BT films were sintered at 850 degreesC for 1 h. The thickness was 25-75 μm depending of number of layers. The microstructure of thick films and the compatibility between BT layers and substrate were investigated by SEM Results of dielectric property measurements are also reported

[72] HYDROPHOBIC MECHANOCHEMICAL TREATMENT OF METALLIC SURFACES - WETTABILITY MEASUREMENTS AS A MEANS OF ASSESSING HOMOGENEITY

Roucoules V. Gaillard F. Mathia TG. Lanteri P. - Advances in Colloid & Interface Science. 97(1-3):179-203, 2002

Hydrophobicity, lubrication and anticorrosion properties of steel substrates have been obtained by a deposition of thin film (i.e. by mechanochemical treatment) at room conditions. Stearic acid and paraffin were chosen as reactive molecules. Different abrasive powders were selected to generate active sites on the treated surfaces for adsorption of the reactive molecules and then, the results were compared. The surfaces were analyzed by reflection-absorption infrared spectroscopy (RAIRS). The results emphasize that, a thick layer of mixed stearic acid/paraffin was deposited onto the metallic surface after the treatment. After hexane rinsing we could only detect a very thin layer of oriented stearic acid molecules chemically adsorbed onto the metallic surface and which engages strong interactions with it. Whereas, RAIRS only provides molecular analysis, the XPS technique was complementary for discriminating the different surfaces. It was possible to show differences in thickness as well as in coverage according to the size and shape of abrasive particles. Furthermore, we could conclude that deposit layer is not uniform. Defects were always present and were dependent on abrasive powders used. Then wettability was assessed as a way to test the homogeneity of thin films generated by the mechanochemical treatment. In agreement with theoretical data, receding contact angle was very dependent on the defects in the deposited film. If holes or aggregates were increased in the deposit layer, the receding contact angle was decreased while advancing contact angles and equilibrium contact angles remained constant. A very important point for technological applications was that the homogeneity of the deposited film was governed by abrasive powder involved in mechanochemical treatment and contact angle values were a direct measurement of the homogeneity of surfaces generated by mechanochemical treatment.

[71] OXIDE PHASE FORMATION UPON MECHANOCHEMICAL ACTIVATION OF AMORPHOUS ZIRCONIUM HYDROXIDE

Kutnetsov PN. Kuznetsova LI. Zhizhaev AM. Kolesnikova SM. Pashkov GL. Boldyrev VV. - Russian Journal of Inorganic Chemistry. 47(3):393-397, 2002

The effect of mechanical activation of amorphous zirconium hydroxide in a centrifugal planetary mill on the formation of oxide phases is studied as a function of the activation medium and the mechanical load value. Pulsed mechanical loading results in the formation of a nanosized metastable phase of tetragonal zirconia in the mechanochemical apparatus at ambient temperatures. Mechanochemical synthesis occurs by a consecutive-parallel route through the formation of the stable monoclinic phase of zirconia. The latter, under pulsed mechanical loading, undergoes an enantiotropic phase transition to the metastable tetragonal phase. Stabilization of the metastable phase is due to the small grain size of the mechanically milled powder.

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[70] IMPACT CHAOS CONTROL AND STRESS RELEASE - A KEY FOR DEVELOPMENT OF ULTRA FINE VIBRATION MILLING

Wang SL. - Progress in Natural Science. 12(5):336-341, 2002

Through our previous experimental and analytical studies, it has been discovered that the key for the development of vibration milling is the impact chaos control and stress release. The necessities for the chaos control and stress release are: (i) to strictly eliminate the sub-harmonics; (ii) to control the super-harmonics to a lower level and (iii) to load the system compressively with relatively higher period, in order that the vibration energy can be absorbed by the particles effectively and sufficiently, A new vibration model for ultra fine milling is proposed, which has wide applications in preparing ultra fine particles.

[69] COMPARATIVE ANALYSIS OF THE MECHANISMS AND KINETICS OF MECHANICAL ALLOYING IN THE SYSTEMS FE(75)X(25) (X = SI, C)

Elsukov EP. Dorofeev GA. Konygin GN. Fomin VM. Zagainov AV. - Physics of Metals & Metallography (English Translation of Fizika Metallov i Metallovedenie). 93(3):278-288, 2002



Mossbauer spectroscopy, X-ray diffraction, and thermomagnetic measurements were used to study in detail the mechanical alloying of iron powders with carbon and silicon in the atomic proportion 75:25. Differences in the sequence and kinetics of solid-state reactions caused by differences in the atomic radii of the components were found. The microscopic mechanisms of deformation-induced atomic mixing are proposed.

[68] CHARACTERISTIC OF MECHANICALLY MILLED TiO₂ POWDERS

Hu JF. Qin HW. Sui ZG. Lu HL. - Materials Letters. 53(6):421-424, 2002

The phase transition of TiO₂ from the anatase to the high-pressure srilankite can be induced by the mechanical milling processes. Accompanying the mechanical milling, the lattice distortion of the milled anatase TiO₂ occurs. The photocatalyst efficiency of the srilankite phase is poorer than that of the anatase phase.

[67] OXOALKYLATION OF PYRAZINE WITH 1-METHYLCYCLOALKANOLS UNDER MECHANICAL ACTIVATION [RUSSIAN]

Nikishin GI. Sokova LL. Makhaev VD. Petrova LA. Kapustina NI. - Khimiya Geterotsiklicheskikh Soedinenii. (4):559-561, 2002

[66] DISORDERING AND ELECTRONIC STATE OF COBALT IONS IN MECHANOCHEMICALLY SYNTHESIZED LiCoO₂

Kosova NV. Anufrienko VF. Larina TV. Rougier A. Aymard L. Tarascon JM. - Journal of Solid State Chemistry. 165(1):56-64, 2002

Mechanical activation (MA) combined with heat treatment at moderate temperatures was used to prepare disordered and highly dispersed LiCoO₂ starting from the mixtures of various cobalt precursors (CoOOH, Co(OH)₂, and Co) and LiOH. X-ray powder diffraction and IR spectroscopy were used to investigate the phase composition and the crystal structure of as-prepared samples, while the electronic state of cobalt ions was characterized by diffuse reflectance electron spectroscopy. MA of the LiOH + CoOOH mixture led to the formation of LT-LiCoO₂ with a cubic spinel-related structure. Heat treatment at 600 degreesC of the latter resulted in the formation of HT-LiCoO₂ with a hexagonal layered structure similar to ceramic LiCoO₂. However, as-prepared HT-LiCoO₂ is characterized by Co octahedra less perfect than those of ceramic LiCoO₂. All MA-LiCoO₂ samples are exclusively described by localized d electrons

[65] SONOCHEMICAL SYNTHESIS OF TUNGSTEN SULFIDE NANORODS

Nikitenko SI. Koltypin Y. Mastai Y. Koltypin M. Gedanken A. - Journal of Materials Chemistry. 12(5):1450-1452, 2002.

Amorphous WS₂ has been prepared by ultrasound irradiation of W(CO)₆ solution in diphenylmethane (DPhM) in the presence of a slight excess of sulfur at 90 degreesC under argon. Heating the amorphous powder at 800 degreesC under argon yields WS₂ nanorods and their packings. The average size of WS₂ nanorods was found to be 3-10 nm and 1-5 μm in thickness and length, respectively. The prepared WS₂ has been characterized by elemental analysis, X-ray powder diffraction measurements, FTIR spectroscopy, differential scanning calorimetry, thermogravimetric analysis, transmission electron microscopy, scanning electron microscopy, atomic force microscopy and energy dispersive X-ray analysis

[64] THERMAL EXPANSION AND SPECIFIC HEAT OF MECHANICALLY MILLED FCC Fe₇₂Pt₂₈

Takano H. Hikichi S. Maeda J. Murayama S. Hoshi K. - Journal of Magnetism & Magnetic Materials. 239(1-3 Special Issue SI):60-62, 2002

Thermal expansion of mechanically milled FCC Fe₇₂Pt₂₈ was measured by X-ray diffractometry. For FCC Fe₇₂Pt₂₈ milled for 120 h the volume increased between 80 and 323 K with decreasing temperature. It was concluded that the volume expansion in such wide temperature range is related to the distribution of the Curie temperature T_C suggested by specific heat measurement.

[63] NiFe₂O₄ NANOPARTICLES FORMED IN SITU IN SILICA MATRIX BY MECHANICAL ACTIVATION

Zhou ZH. Xue JM. Wang J. Chan HSO. Yu T. Shen ZX. - Journal of Applied Physics. 91(9):6015-6020, 2002

Nanocrystalline nickel ferrite (NiFe₂O₄) particles were successfully synthesized in situ in an amorphous silica matrix by mechanical activation at room temperature. Phase development in the amorphous precursors, derived via a modified sol-gel synthesis route, with increasing mechanical activation time was studied in detail by employing transmission electron microscopy, x-ray diffraction, and Raman spectroscopy. NiFe₂O₄ nanoparticles of 8.05 nm in mean particle size with a standard deviation of 1.24 nm, which were well dispersed in the silica matrix, were realized by 30 h of mechanical activation. The phase formation of nanocrystalline NiFe₂O₄ particles involves the nucleation of Fe₃O₄ in amorphous silica at the initial stage of mechanical activation, followed by the growth of nickel ferrite by incorporation of Ni²⁺ cations into Fe₃O₄. Their magnetic anisotropy, surface spin disorder, and cation distribution are investigated by considering both the strain imposed by silica matrix and the buffer effect during mechanical activation.

[62] MAGNETIC PROPERTIES AND MICROSTRUCTURE OF LaCo₅-BASED POWDERS PRODUCED BY MECHANICAL MILLING AND SUBSEQUENT ANNEALING

Okumura H. Zhang F. Hadjipanayis GC. - Journal of Applied Physics. 91(9):6147-6153, 2002

The magnetic and structural properties of LaCo₅-based La_xCo_{100-x} (x=17-22) alloys have been studied in powders synthesized by mechanical milling and subsequent annealing. A record high coercivity (over 1.7 T) with a M_r/M_s ratio about 0.7 (measured at room temperature) has been obtained in La₂₂Co₇₈ powders. Their microstructure consists of major LaCo₅ phase grains with the grain size of about 150-350 nm, coexisting with LaCo₁₃ phase particles (30-70 nm) and La/La oxide grains (100-200 nm). The observed magnetic properties originate from the high anisotropy field and uniform grain size and shape of the LaCo₅ phase. The demagnetization curve, however, often exhibits a shoulder, which was correlated with the appearance of the magnetically soft 1:13 phase and with the inhomogeneity in the microstructure. All the analyses are



consistent with a nucleation-type LaCo5 magnet, where the coercivity is attributed to the difficulty in the formation of reversed magnetic domains. The observed phases and microstructure are briefly discussed based on a possible nonequilibrium phase transition resulting in chemical inhomogeneity and oxidation.

[61] THE EFFECT OF PARTIAL SUBSTITUTION OF TI WITH ZR, CR OR V IN THE Mg35Ti10Ni55 ELECTRODE ALLOY ON ITS ELECTROCHEMICAL PERFORMANCE

Zhang Y. Chen LX. Lei YQ. Wang QD. - *Electrochimica Acta*. 47(11):1739-1746, 2002

In this paper, Zr, Cr and V were selected as the partial substitution elements for Ti to form Mg35Ti5M5Ni55 quaternary alloys prepared by means of mechanical alloying on the basis of the ternary Mg35Ti10Ni55 electrode alloy previously studied by the authors. It is found that all the three quaternary alloys possess an amorphous main phase and exhibit improved cycling stability than the original ternary Mg35Ti10Ni55. The Zr-substituted alloy has also a higher discharge capacity. X-ray photoelectron spectroscopy (XPS) surface analysis reveals that on the surface of each alloy a multi-component oxide composite film is formed, which is more compact and corrosion resistant than the Mg(OH)₂ film and the (TiO₂)_x(NiO)_y(Mg(OH)₂)_z composite films reported previously. Auger electron spectroscopy (AES) analysis shows that the substituting elements tend to reduce the thickness of the passivation films. The thickness of composite oxide film on the alloys is in the following order: Mg35Ti10Ni55 > Mg35Ti5V5Ni55 > Mg35Ti5Zr5Ni55 > Mg35Ti5Cr5Ni55. The result agrees well with that of the polarization resistances (R_p) values obtained from the electrochemical impedance spectroscopy (EIS) Nyquist diagrams. It demonstrates that the thicker the passivation film is, the higher the R_p is and the more difficultly the charge transfer reaction proceeds. Anodic polarization curves indicate that the addition of Cr and Zr decreases effectively the corrosion current. However, the corrosion of the electrode alloys in the electrolyte is still high and proceeds constantly as the corrosion current remains almost constant during further cycles

[60] CHARACTERIZATION OF NANOCRYSTALLINE PRODUCTS PREPARED BY MECHANOCHEMICAL REDUCTION OF COPPER SULPHIDE

Balaz P. Godocikova E. Boldizarova E. Luxova M. Bastl Z. Jiang J. - *Czechoslovak Journal of Physics*. 52(Suppl A):A65-A68, 2002.

The mechanochemical processing of the copper sulphide with iron in a high-energy mill was studied. The nanosized copper of 10 nm crystallite size and the hexagonal pyrrhotite 1C were identified among products of the reaction by methods of XRD and Mossbauer spectroscopy. In the surface layer of products after the mechanochemical processing copper in its mono- and bivalent forms was identified by XPS method

[59] STUDY OF MAGNETIC RELAXATION IN PARTIALLY OXIDIZED NANOCRYSTALLINE IRON

Kerekes L. Hakl J. Meszaros S. Vad K. Gurin P. Kis-Varga I. Uzonyi I. Szabo S. Beke DL. - *Czechoslovak Journal of Physics*. 52(Suppl A):A89-A92, 2002.

The long term magnetic relaxation is investigated in a nanocrystalline ferromagnet. Samples were prepared by ball milling of microcrystalline iron in Helium atmosphere and vacuum. We have found logarithmic time dependence of the magnetic moment with the relaxation rate independent of the starting magnetic field in the 0-20 mT low field region.

[58] SUPERPARAMAGNETISM AND COLLECTIVE MAGNETIC EXCITATIONS IN NANOSCALE MILLED SPINEL FERRITE

Sepelak V. Mienert D. Baabe D. Petrovic P. Becker KD. - *Czechoslovak Journal of Physics*. 52(Suppl A):A93-A96, 2002.

The changes in magnesium ferrite (MgFe₂O₄) caused by high-energy milling arc investigated by means of Mossbauer spectroscopy. A collapse of the magnetic hyperfine fields far below the Neel temperature of the bulk ferrite is observed in nanoscale milled MgFe₂O₄. The influence of superparamagnetism and collective magnetic excitations on Mossbauer spectra of milled material taken at different temperatures is discussed

[57] MAGNETIC STUDY OF NANOCRYSTALLINE MG-DOPED LITHIUM FERRITE

Pekala M. Berry FJ. Widatallah HM. - *Czechoslovak Journal of Physics*. 52(Suppl A):A101-A104, 2002.

Nanocrystalline magnesium doped ferrites with grain sizes between 60 and 11 nm were prepared by the ball milling. Magnetization of the iron-milled ferrite diminishes monotonically with temperature and vanishes above the relatively high Curie temperature T_C of 900 K. A milling process suppresses T_C down to 830 K, which reveals the exchange interaction diminishing with the reduced grain sizes. The room temperature magnetic moment per formula unit diminishes from 2.15 μ_B for non-milled ferrite to 1.27 μ_B for 11 nm sample. The hyperfine magnetic fields derived from Mossbauer spectra are remarkably suppressed where the grain sizes are reduced below 20 nm

[56] THE INFLUENCE OF MECHANICAL ALLOYING ON THE MAGNETIC PROPERTIES OF U-CO-B SYSTEM.

Timko M. Zentko A. Kovac J. Szlaferek A. Kowalczyk A. - *Czechoslovak Journal of Physics*. 52(Suppl A):A253-A256, 2002.

We have investigated the influence of mechanical milling on structural changes and magnetic properties of the uranium transition-metal boride UCo₄B intermetallic compound. The great decrease of intensity and broadening of diffraction lines have been detected after milling as a consequence of amorphisation of samples. Magnetic measurements have shown that originally prepared polycrystalline sample shows properties of Pauli para-magnet with spin fluctuations. After milling the magnetic ordered state has been observed and high value of high-field susceptibility can be attributed to the itinerant character of investigated samples. The changes of magnetic moment during the milling processes can be attributed to the fact that probability of environment typical to the crystalline state is lowered.

[55] COMPARATIVE ANALYSIS OF THE MECHANISMS AND KINETICS OF MECHANICAL ALLOYING IN THE SYSTEMS Fe(75)X(25) (X = Si, C)



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Mossbauer spectroscopy, X-ray diffraction, and thermomagnetic measurements were used to study in detail the mechanical alloying of iron powders with carbon and silicon in the atomic proportion 75:25. Differences in the sequence and kinetics of solid-state reactions caused by differences in the atomic radii of the components were found. The microscopic mechanisms of deformation-induced atomic mixing are proposed.

[54] CHARACTERISTIC OF MECHANICALLY MILLED TiO₂ POWDERS

Hu JF. Qin HW. Sui ZG. Lu HL. - Materials Letters. 53(6):421-424, 2002

The phase transition of TiO₂ from the anatase to the high-pressure srilankite can be induced by the mechanical milling processes. Accompanying the mechanical milling, the lattice distortion of the milled anatase TiO₂ occurs. The photocatalyst efficiency of the srilankite phase is poorer than that of the anatase phase

[53] PIEZOELECTRIC PROPERTIES OF COLLAGEN-NANOCRYSTALLINE HYDROXYAPATITE COMPOSITES

Silva CC. Pinheiro AG. Figueiro SD. Goes JC. Sasaki JM. Miranda MAR. Sombra ASB. - Journal of Materials Science. 37(10):2061-2070, 2002

In this paper we did a study of the physicochemical, dielectric and piezoelectric properties of anionic collagen and collagen-hydroxyapatite (HA) composites, considering the development of new biomaterials which have potential applications in support for cellular growth and in systems for bone regeneration. The piezoelectric strain tensor element $d(14)$, the elastic constant $s(55)$, and the dielectric permittivity $\epsilon(11)$ were measured for the anionic collagen and collagen-HA films. For the collagen-HA composite film (Col-HACOM) the main peaks associated to the crystalline HA is present. For the nanocrystalline composite, nanometric HA powder (103 nm particle size) (HAN), obtained by mechanical milling were used. For the composite film (Col-HAN) the HA and CaH(PO₄)₂H₂O phases were detected. One can see that the HA powder (HAN) present the main peaks associated to crystalline HA. The IR spectroscopy measurements on HA-COM and HAN powders, Col-HACOM and Col-HAN composite films and collagen film (Col) presents the main resonances associated to the modes of (PO₄)₃⁻, (CO₃)₂⁻. The IR spectra of Collagen Film (Col) shows the bands associated to amide I (C=O), amide II (N-H) and amide III (C-N) vibrational modes. The scanning electron photomicrography of the Col-HACOM and Col-HAN films, respectively, shows deposits of HA on the surface of collagen. It also shows that HACOM crystals has a dense feature, whereas the HAN crystals has soft porous surface. Energy-dispersive spectroscopy (EDS) analysis showed that the main elements of the hybrid sponge were carbon, oxygen, calcium, and phosphorus. The EDS of HACOM crystal, present in the Col-HACOM composite showed a molar ratio Ca/P = 1.71, whereas the Col-HAN composite the molar ratio of calcium and phosphorus (Ca/P = 2.14) and the amount of carbon were greater. The piezoelectric strain tensor element $d(14)$ obtained for the anionic collagen was around 0.102 pC/N. The collagen composite with nanocrystalline HA crystals (Col-HAN) present a better result ($d(14) = 0.040$ pC/N) compared to the composite with the commercial ceramic ($d(14) = 0.012$ pC/N). This is an indication that the nanometric particles of HA present little disturbance on the organization of the collagen fibers in the composite. In this situation the nanometric HA are the best candidates in future applications of these composites.

[52] PRODUCTION OF FINE POLYMER POWDER UNDER CRYOGENIC CONDITIONS

Liang SB. Hu DP. Zhu C. Yu AB. - Chemical Engineering & Technology. 25(4):401-405, 2002

In order to produce fine polymer powders, a special and unconventional cryogenic grinding system was established using liquid nitrogen, where a jet-vortex mill was used as the grinding mill. The major feature of this grinding process is that heat generation during the grinding period was eliminated. The results suggest that this cryogenic grinding system may be suitable for studying the grinding properties of polymeric materials. It may also be helpful in understanding mechanochemistry, e.g., the t - P - T conditions for different mechanochemical processes under cryogenic conditions (where T is the temperature, and P the pressure of the gas mixture in the grinding chamber). In addition, an Elbow-jet classifier was attached to the jet-vortex mill so that fine, medium and coarse products of polymeric powders could be obtained simultaneously. Chitin, a type of renewable natural polymer, was ground in the system and XRD analysis of ground powders showed they displayed highly activated properties. Unlike a high-energy mechanical milling process, such as a vibratory (bead) mill which requires more milling time t , the final properties of the ground polymer in the cryogenic grinding system were highly dependent on the temperature in the chamber of the jet-vortex mill. The grinding results of chitin also showed that the minimum diameters of the ground polymer products are larger than several tens of micrometers (e.g., 75 μ m). The developed method offers a new choice for the production of materials. polymer modification (e.g., degradation), and recycling of wasted rubber and plastic.

[51] OXYGEN MASS TRANSFER INTO FLOTATION SLURRIES - PART 1: EFFECT OF MILD STEEL CORROSION DURING GRINDING OF QUARTZ

Authors Khan A. Kelebek S. - Transactions of the Institution of Mining & Metallurgy Section C-Mineral Processing & Extractive Metallurgy. 110:C115-C122, 2001

Under ordinary conditions of milling the consumption of oxygen by the grinding media relative to its consumption by a massive sulphide ore is not known. To help to distinguish between the two oxygen mass transfer into flotation slurries has been investigated under various conditions. Aeration studies were conducted on a quartz slurry following its grinding in a mild-steel mill in the absence and presence of sodium chromate as a corrosion inhibitor. During these studies such variables as the oxygen concentration, pH and pulp potential were monitored as a function of time by means of a customized data-acquisition system. A systematic increase in the amount of iron-bearing deposition on quartz particles was observed with increasing grinding time, which was accompanied by a decrease in pulp potential. The oxygen mass-transfer coefficient



showed an exponential decrease from a value of 7.5 s(-1) (in tap water) to about 0.11 s(-1) (in slurry) with increasing fineness of quartz ground in the absence of sodium chromate. This trend appears to be completely reversible through the use of sodium chromate in grinding, the coefficient attaining a value that can be even greater than its original value prior to any grinding. The variations of pH and pulp potentials are discussed in relation to certain electrochemical and hydrolysis reactions relevant to the current system. An Eh-pH diagram constructed using specific experimental conditions indicates formation of an iron chromate as the main product of corrosion inhibition.

[50] SYNTHESIS OF ULTRAFINE WC/CO POWDER BY MECHANOCHEMICAL PROCESS

Ha GH. Kim BK. - Powder Metallurgy. 45(1):29-32, 2002.

A new approach to produce ultrafine WC/Co powder by a mechanochemical process was made to improve the mechanical properties of advanced hardmetals and to cut production costs. For powder preparation, the water soluble salts containing W and Co components were used as starting materials. After synthesis of the precursor powder from an aqueous solution by a spray drying technique, a salt removing heat treatment in air atmosphere was carried out to prepare the oxide powder. The oxide powder was mixed with carbon black by ball milling and this mixture was converted at 800degreesC to the nanophase WC/Co powder in H₂ and N₂ atmospheres. The average size of the WC particle was 100-150 nm. The possibility of achieving high density sintered material with an ultrafine and homogeneous microstructure using grain growth inhibitors, such as tantalum and vanadium carbides, has been shown.

[49] FORMATION OF AMORPHOUS Ti50Cu20Ni24Si4B2 ALLOY WITH A WELL-DEFINED GLASS TRANSITION BY HIGH-ENERGY BALL MILLING [CHINESE]

Zhang LC. Xu J. - Chin Shu Hsueh Pao: Acta Metallurgica Sinica. 38(3):299-302, 2002

Under high-energy ball milling, elemental powder mixtures with a nominal composition of Ti50Cu20Ni24Si4B2 can be amorphized via solid state reaction. A well-defined glass transition before crystallization appears in the obtained amorphous phase during heating in a differential scanning calorimeter. A wide supercooled liquid region, $\Delta T(x)$, is available to be about 57 K. Crystallization of the ball-milled amorphous phase proceeds through one step to form the (Ni, Cu)Ti phase with cubic structure and other unknown phases, which is a eutectic-type

[48] SOLID STATE SYNTHESIS OF STRONTIUM OXOFERRATES FROM THE MECHANICALLY ACTIVATED SYSTEM SRCO3-FE2O3

Berbenni V. Marini A. - Materials Research Bulletin. 37(2):221-234, 2002

The solid state reactions occurring in the mechanically activated System SrCO₃-Fe₂O₃ (composition X-Fe (iron cationic fraction) = 0.5-0.923) have been studied. At X-Fe = 0.5, SrFeO_{2.5+x} (brownmilletite) forms by annealing of the activated mixture at 850degreesC under nitrogen while the non-stoichiometric perovskite (SrFeO_{3-x}) is the equilibrium product when annealing is performed under air. At X-Fe = 0.66 a mixture of SrFe₂O₄ and SrFe₂O₅ is the final product no matter what is the annealing atmosphere. The enthalpies of reaction at these composition have been determined. By increasing iron content (X-Fe = 0.8), the annealing at 850degreesC of the activated mixture leads to SrFe₂O₄, SrFe₂O₅ and SrFe₁₂O₁₉ (indicated in the following with the usual term of strontium hexaferrite). Finally at X-Fe = 0.923, SrFe₁₂O₁₉ is produced from the mechanically activated mixture at temperatures lower (approximate to 800degreesC) than in the case of conventional solid state route. In the case of the last two compositions no reaction enthalpy could be determined as the process is too slow

[47] SYNTHESIS OF LA(MG0.5TI0.5)O-3 CERAMICS FOR MICROWAVE APPLICATIONS

Seabra MP. Ferreira VM. - Materials Research Bulletin. 37(2):255-262, 2002

La(Mg_{0.5}Ti_{0.5})O-3 ceramics were prepared by a non-conventional chemical route, which was based on the Pechini method. For the synthesis of La(Mg_{0.5}Ti_{0.5})O-3 powders, special attention was paid to calcination and milling conditions. Powder morphology and composition were evaluated. Fine La(Mg_{0.5}Ti_{0.5})O-3 powders were obtained at lower temperatures than by conventional methods. Sintering under different conditions was also tested. Dense La(Mg_{0.5}Ti_{0.5})O-3 ceramics were obtained at lower temperatures showing a single phase composition and a homogeneous microstructure. Preliminary dielectric characterization at microwave frequencies was also performed

[46] RAPID FORMATION OF LEAD MAGNESIUM NIOBATE-BASED FERROELECTRIC CERAMICS VIA A HIGH-ENERGY BALL MILLING PROCESS

Kong LB. Ma J. Zhu W. Tan OK. - Materials Research Bulletin. 37(3):459-465, 2002

Pyrochlore-free nano-sized 0.90Pb(Mg_{1/3}Nb_{2/3})O-3(PMN)-0.10PbTiO(3)(PT) and 0.65PMN-0.35PT powders were synthesized from oxides via a high-energy ball milling process. Single perovskite phase PMN-PT were readily formed from the oxide mixture after milling for only 2 h. The grain size calculated from X-ray diffraction (XRD) patterns of all samples is about 20 nm, which is in agreement with the observation from scanning electron microscopy (SEM) (20-50 nm). PMN-PT ceramics were obtained by sintering the milled powders at temperature from 1000 to 1100degreesC for 2 h. The dielectric, ferroelectric properties of the PMN-PT ceramics derived from the synthesized powders were comparable with the reported results in the literature.

[45] SYNTHESIS OF NANOCRYSTALLINE GAMMA-FE2O3 IN SILICA MATRIX BY MECHANICAL CRYSTALLIZATION FROM PRECURSOR AT ROOM TEMPERATURE

Xue JM. Zhou ZH. Wang J. - Materials Chemistry & Physics. 75(1-3 Special Issue SI):81-85, 2002

gamma-Fe₂O₃ nanocrystallites dispersed in an amorphous silica matrix have been successfully synthesized for the first time by mechanical activation of a chemistry-derived precursor at room temperature. At the initial 10 h of mechanical activation, nanocrystallites of Fe₃O₄ were triggered to form a highly activated matrix. With increasing mechanical activation time, phase transformation from Fe₃O₄ to gamma-Fe₂O₃ occurs. gamma-Fe₂O₃ phase was well established when the precursor



was mechanically activated for 30 h. Further increasing the mechanical activation time to 40 h induced the formation of α -Fe₂O₃. The mechanical activation-grown γ -Fe₂O₃ nanocrystallites are similar to 10-12 nm in size and they are well dispersed in the silica matrix as observed using TEM. They demonstrated superparamagnetic behavior at room temperature when measured using a vibrating sample magnetometer (VSM). In addition, the γ -Fe₂O₃ derived from 30 h of mechanical activation exhibited a saturation magnetization of as high as 62.6 emu g⁻¹).

[44] DOPING EFFECTS OF BIFEO3 IN LAYERED PEROVSKITE SRBI2NB2O9

Gu HS. Xue JM. Gao XS. Wang J. - Materials Chemistry & Physics. 75(1-3 Special Issue SI):105-109, 2002
Nanocrystallites of ferroelectric $x\text{BiFeO}_3(1-x)\text{SrBi}_2\text{Nb}_2\text{O}_9$ (SBFN) with x ranging from 0 to 0.2 were synthesized by mechanical activation of mixed oxides at room temperature. The resulting SBFN exhibit nanocrystalline particles, which consist of layered perovskite crystallites in the range 14-24 nm, depending on the level of BiFeO₃ doping in SBN. Sintered SBFN exhibited a single-phase layered perovskite structure with no detectable secondary phase being present during crystallization. The grain morphology changes from a lamellar structure in SBN to a granular structure at $x = 0.2$. Lattice dimensions of SBN are decreased slightly by doping with BiFeO₃, which also leads to a lower sintering temperature, while the Curie point was shifted upwards with increasing BiFeO₃ doping. The peak impedance of SBFN decreases with frequency and increases with increase level of BiFeO₃ doping in SrBi₂Nb₂O₉. A dielectric enhancement was observed in 0.2BiFeO₃-0.8SrBi₂Nb₂O₉.

[43] HIGH TEMPERATURE PIEZOELECTRIC STRONTIUM BISMUTH TITANATE FROM MECHANICAL ACTIVATION OF MIXED OXIDES

Ng SH. Xue JM. Wang J. - Materials Chemistry & Physics. 75(1-3 Special Issue SI):131-135, 2002
Perovskite strontium bismuth titanate (SrBi₄Ti₄O₁₅, SBIT) of layered structure has been successfully prepared via mechanical activation of mixed oxides of SrO, Bi₂O₃ and TiO₂ for more than 15 h at room temperature. The mechanical activation-derived SBIT can be sintered to a relative density of similar to 98% theoretical at 1175 degreesC which exhibits a maximum dielectric constant of 2770 at the Curie temperature similar to 539 degreesC when measured at 100 kHz. The conductivity of sintered ceramic was studied using an impedance spectroscopy. An activation energy of 0.22 eV is observed at low temperatures and it increases with increasing temperature. A piezoelectric coefficient, $d(33)$, of 24.0 pC/N is measured at room temperature for sintered SBIT.

[42] FERROELECTRIC LEAD SCANDIUM TANTALATE FROM MECHANICAL ACTIVATION OF MIXED OXIDES

Lim J. Xue JM. Wang J. - Materials Chemistry & Physics. 75(1-3 Special Issue SI):157-160, 2002
Conventional mixed oxide preparation of lead scandium tantalate, PbSc_{0.5}Ta_{0.5}O₃ (PST) powder, requires two calcinations steps, the first of which is to form a wolframite precursor and the resulting PST phase is structurally long-range ordered. In this work, we report the synthesis of perovskite PST via a novel one-step mechanical activation route, which leads to a long-range disordered structure as verified by X-ray diffraction. Sintered PSTs from the mechanically activated powders also retain the long-range disordered perovskite structure up to 1250 degreesC, while >95% theoretical density was achieved. They exhibit a spontaneous ferroelectric-relaxor transition at 15 degreesC with a maximum dielectric constant of similar to 13,850 and dielectric loss of <0.04

[41] MECHANICAL ACTIVATION-INDUCED B SITE ORDER-DISORDER TRANSITION IN PEROVSKITE PB(MG1/3NB2/3)O-3-PB(MG1/2W1/2)O-3

Gao XS. Xue JM. Yu T. Shen ZX. Wang J. - Materials Chemistry & Physics. 75(1-3 Special Issue SI):211-215, 2002
Mechanical activation-induced B site order-disorder transition was observed in Pb(Mg_{1/3}Nb_{2/3})O₃-Pb(Mg_{1/2}W_{1/2})O₃ of complex perovskite structure. The disorder triggered by mechanical activation can be steadily recovered by thermal annealing at elevated temperature, as shown by XRD diffraction results. The degree of ordering calculated from Raman spectra demonstrates similar behaviors as those of XRD, except in the low ordering states, i.e. when the ordering domain size in 0.4PMN-0.6PMW is out of the coherence range of XRD. The above order-disorder transition was explained by the competition between the activation-triggered refinement of ordering domains leading to disordering and the thermal activation, which builds up the ordering domains.

[40] NANOCRYSTALLINE PBTIO3 POWDERS FROM AN AMORPHOUS PB-TI-O PRECURSOR BY MECHANICAL ACTIVATION

Yu T. Shen ZX. Xue JM. Wang J. - Materials Chemistry & Physics. 75(1-3 Special Issue SI):216-219, 2002
Mechanical activation is investigated for its efforts in triggering PbTiO₃ nanocrystallites from an amorphous Pb-Ti-O precursor, which was synthesized by co-precipitation. In this study, the amorphous precursor and the compositions that were subjected to mechanical activation for various time periods were investigated using Raman spectroscopy, X-ray diffraction (XRD) and scanning electron microscopy (SEM). A single perovskite PbTiO₃ phase was readily formed in the amorphous Pb-Ti-O precursor by mechanical activation, where a nanocrystalline feature is retained.

[39] SONOCHEMICAL DEPOSITION OF SILVER NANOPARTICLES ON SILICA SPHERES

Pol VG. Srivastava DN. Palchik O. Palchik V. Slifkin MA. Weiss AM. Gedanken A. - Langmuir. 18(8):3352-3357, 2002
Silver nanoparticles with an average size of similar to 5 nm were deposited on the surface of preformed silica submicrospheres with the aid of power ultrasound. Ultrasound irradiation of a slurry of silica submicrospheres, silver nitrate, and ammonia in an aqueous medium for 90 min under an atmosphere of argon to hydrogen (95:5) yielded a silver-silica nanocomposite. By controlling the atmospheric and reaction conditions, we could achieve the deposition of metallic silver on the surface of the silica spheres. The resulting silver-deposited silica submicrosphere samples were characterized with X-ray diffraction, transmission electron microscopy, differential scanning calorimetry, energy-dispersive X-ray analysis, high-



resolution transmission electron microscopy, high-resolution scanning electron microscopy, photoacoustic spectroscopy, and Fourier transform infrared, UV-visible, and X-ray photoelectron spectroscopy

[38] NANOCRYSTALLINE MAGHEMITE (γ -Fe₂O₃) IN SILICA BY MECHANICAL ACTIVATION OF PRECURSORS

Xue JM. Zhou ZH. Wang J. - Journal of the American Ceramic Society. 85(4):807-811, 2002

γ -Fe₂O₃ nanocrystallites dispersed in an amorphous silica matrix have been successfully prepared for the first time by mechanical activation of a chemistry-derived precursor at room temperature. The initial 10 h of mechanical activation triggered the formation of nanocrystallites of Fe₃O₄ in a highly activated matrix. Increasing the mechanical-activation time led to a phase transformation from Fe₃O₄ to γ -Fe₂O₃. The γ -Fe₂O₃ phase was well established after mechanical activation of the precursor for 30 h. Further increasing the mechanical-activation time to 40 h induced the formation of α -Fe₂O₃. The mechanical-activation-grown γ -Fe₂O₃ nanocrystallites were similar to 10-12 nm in size and well dispersed in the silica matrix, as observed using TEM. They demonstrated superparamagnetic behavior at room temperature when measured using a Mossbauer spectrometer and a vibrating sample magnetometer (VSM). In addition, the γ -Fe₂O₃ derived from 30 h of mechanical activation exhibited a value of saturation magnetization as high as 62.6 emu/g

[37] SYNTHESIS OF NANOSTRUCTURED SILICON CARBIDE THROUGH AN INTEGRATED MECHANICAL AND THERMAL ACTIVATION PROCESS

Ren RM. Yang ZG. Shaw LL. - Journal of the American Ceramic Society. 85(4):819-827, 2002

Changes of crystal structures and microstructures of SiO₂ and graphite powder mixtures induced by high-energy milling, the effect of these changes on the reactivity of reactants, and the mechanism of enhanced SiC formation have been studied using a variety of analytical instruments, including X-ray diffractometry, scanning electron microscopy, transmission electron microscopy, solid-state Si-29 nuclear magnetic resonance, and nitrogen adsorption (i.e., the BET method). High-energy milling before carbothermic reduction leads to substantial changes in the structural and energy states of the reactants, which in turn increases the reactivity of the reactants and enhances the formation of nanostructured SiC particles. Furthermore, the structural and energy-state changes contribute to the enhanced SiC formation through the increased reaction kinetics as well as the increased reaction driving force.

[36] B-SITE ORDER-DISORDER TRANSITION IN Pb(Mg_{1/3}Nb_{2/3})O₃-Pb(Mg_{1/2}W_{1/2})O₃ TRIGGERED BY MECHANICAL ACTIVATION

Gao XS. Xue JM. Yu T. Shen ZX. Wang J. - Journal of the American Ceramic Society. 85(4):833-838, 2002

B-site cation order-disorder transition induced by mechanical activation was observed in Pb(Mg_{1/3}Nb_{2/3})O₃-Pb(Mg_{1/2}W_{1/2})O₃ (PMN-PMW) solid solution, which was examined using both XRD diffraction and Raman spectroscopic study. The order-disorder transition is composition dependent. Mechanical activation triggers the B-site disordering, which can be steadily recovered by thermal annealing at elevated temperature, i.e., at temperatures around 600°C. Raman spectroscopy demonstrated that there existed tiny ordered microdomains in 0.4PMN-0.6PMW subjected to up to 20 h of mechanical activation, although they cannot be shown by X-ray diffraction. This is a result of the equilibrium between the mechanical destruction and temperature-facilitated recovering at the collision points during mechanical activation. It is therefore unlikely that a complete disordering can be realized in PMN-PMW by mechanical activation. The disordering in PMN-PMW triggered by mechanical activation occurs simultaneously with the refinement in crystallite size at the initial stage of mechanical activation, suggesting that the fragmentation of crystallites is responsible for the order-disorder transition at least during the initial stage of mechanical activation.

[35] MECHANICAL-ALLOYING-ASSISTED SYNTHESIS OF Ti₃SiC₂ POWDER

Li JF. Matsuki T. Watanabe R. - Journal of the American Ceramic Society. 85(4):1004-1006, 2002

Mechanical alloying (MA) has been used to synthesize Ti₃SiC₂ powder from the elemental Ti, Si, and C powders. The MA formation conditions of Ti₃SiC₂ were strongly affected by the ball size for the conditions used. MA using large balls (20.6 mm in diameter) enhanced the formation of Ti₃SiC₂, probably via an MA-triggered combustion reaction, but the Ti₃SiC₂ phase was not synthesized only by the MA process using small balls (12.7 mm in diameter). Fine powders containing 95.8 vol% Ti₃SiC₂ can be obtained by annealing the mechanically alloyed powder at relatively low temperatures.

[34] MECHANOCHEMICAL SYNTHESIS AND PRESSURELESS SINTERING OF TiB₂-AlN COMPOSITES

Kim HJ. Choi HJ. Lee JG. - Journal of the American Ceramic Society. 85(4):1022-1024, 2002

TiB₂-AlN composites have been fabricated by the pressureless sintering of a mechanochemically processed Ti, Al, and BN powder mixture. TiB₂-AlN powder was obtained from the mixture of Ti, Al, and BN, which had a composition corresponding to 45.7 wt% TiB₂-54.3 wt% AlN, after mechanochemical processing for longer than 24 h. X-ray diffraction and transmission electron microscopy analysis showed that the powder subjected to mechanochemical processing for 60 h consisted of crystallites less than 300 nm in size with a disordered crystal structure. TiB₂-AlN composites with 95% relative density, a flexural strength of 172 MPa, a fracture toughness of 4.6 MPa·m^{1/2}, a hardness of 12.0 GPa, and an electrical resistivity of 1488 $\mu\Omega\cdot\text{cm}$ were obtained by pressureless sintering at 1700°C for 2 h of the powder subjected to mechanochemical processing for 60 h

[33] STRUCTURAL AND MAGNETIC PROPERTIES OF Fe₅₀Co₅₀ SYSTEM

Sorescu M. Grabias A. - Intermetallics. 10(4):317-321, 2002

The Fe₅₀Co₅₀ alloys were prepared by mechanical alloying of the Fe and Co powders using a high-energy ball mill. The Fe₅₀Co₅₀ system was also obtained as a thin film by the pulsed laser deposition method. Both powder and thin film samples were characterized by X-ray diffraction and Mossbauer spectroscopy. The formation of the bcc-FeCo solid solution was



investigated as a function of milling time. Both Fe₅₀Co₅₀ Systems reveal similar magnetic hyperfine fields of about 35 T. Typically, the powders have random spin orientation, while the thin film shows an out-of-plane magnetic texture.

[32] THE FEAL-30% TiC NANOCOMPOSITE PRODUCED BY MECHANICAL ALLOYING AND HOT-PRESSING CONSOLIDATION

Krasnowski M. Witek A. Kulik T. - *Intermetallics*. 10(4):371-376, 2002

Intermetallic matrix composites reinforced with particles such as TiC have attracted a great deal of attention over the past few years. In the present study, the mechanical alloying process followed by hot-pressing consolidation was used to produce FeAl-30%TiC nanocomposite. Since the reduction of grain size to the nanometer scale improves mechanical properties of materials, this composite may be attractive for structural applications. An elemental powder mixture of Al₃₅Fe₃₅Ti₁₅C₁₅ (in at.%) was milled in a high-energy ball mill. The phase transformations in the powder during milling were studied with the use of X-ray diffraction (XRD). Transmission electron microscopy and differential scanning calorimetry were used for examining the microstructure and the thermal stability of the milling product. The results obtained show that high-energy ball milling as performed in this work leads to the formation of a bcc phase identified as the Fe(Al) solid solution and a fee phase identified as TiC, and that both phases are nanocrystalline. Subsequently, the milled powder was sintered at 750 degreesC under pressure of 4 GPa. The XRD investigations of the consolidated pellet revealed that after sintering, the material remained nanocrystalline and that there were no phase changes, except for the ordering of Fe(Al), i.e. formation of FeAl intermetallic compound, during the sintering process. The average hardness of the obtained nanocomposite is 1287 HV0.2 (12.6 GPa) and its density is 98% of the theoretical value

[31] INVESTIGATIONS OF THE FORMATION MECHANISM OF NANOSTRUCTURED NbAl₃ VIA MASHS REACTION

Gauthier V. Bernard F. Gaffet E. Vrel D. Gailhanou M. Larpin JP. - *Intermetallics*. 10(4):377-389, 2002

The nanostructured NbAl₃ intermetallic compound was synthesized using the mechanically-activated self-propagating high-temperature synthesis (MASHS) technique. This process results from the combination of two steps: a short duration ball-milling of a pure elemental Nb + 3Al powder mixture followed by a self-propagating high-temperature synthesis (SHS) reaction induced by the Nb + 3Al reaction exothermicity. Synchrotron time-resolved XRD coupled with a 2D infrared camera were used to investigate the structural and thermal evolutions during the SHS reaction, and to study in situ the mechanism of NbAl₃ formation. The influence of the incoming heat flux and the mechanical activation effect on the phase transformation kinetics induced by the SHS process were studied. Owing to the temporal resolution of 100 and 200 ms between two consecutive diffraction patterns and IR images, respectively, it was shown that solid niobium reacts with liquid aluminium via a heterogeneous nucleation reaction, to form the NbAl₃ compound by successive combustion fronts

[30] DISORDERED MAGNETIC PHASE IN PARTIALLY OXIDIZED BULK NANOCRYSTALLINE IRON

Hakl J. Kerekes L. Meszaros S. Vad K. Gurin P. Kis-Varga M. Uzonyi I. Szabo S. Beke DL. - *Czechoslovak Journal of Physics*. 52(2):151-154, 2002

Magnetically frozen partially disordered state was observed in nanocrystalline bulk iron prepared by ball-milling of microcrystalline Fe. Role of nanostructure and oxidized fraction is pointed out. Although we have found a small constituent which is proportional to 1/T in the ZFC curve, the magnetization scaling with T^{-3/2} at high fields and the hysteretic behavior in FC-ZFC curves signalizes the presence of frozen ferromagnet at low temperatures

[29] SR₃Ti₂O₇ RUDDLESDEN-POPPER PHASE SYNTHESIS BY MILLING ROUTES

Hungria T. Lisoni JG. Castro A. - *Chemistry of Materials*. 14(4):1747-1754, 2002

This paper presents a new method of synthesis, mechanochemical activation, for the preparation of the Sr₃Ti₂O₇ Ruddlesden-Popper (RP) phase. The results obtained from different starting reagents (SrO, SrCO₃, or Sr(OH)₂·8H₂O and TiO₂ anatase) and mechanical activation systems (vibrating and planetary mills) are compared. An optimized protocol is established that permits one to avoid the high temperatures and long reaction times needed in the classical solid-state reaction method; as a byproduct, very pure phases with controlled particle sizes are isolated as well. The products were studied by X-ray diffraction at room temperature and above, thermal analysis techniques, infrared spectroscopy, and scanning electron microscopy. The insight obtained in this work can be extrapolated to other Sr-2[Sr_n-1Ti_nO_{3n+1}] Ruddlesden-Popper materials that have not been isolated, until now, as single phases,

[28] MAGNETIC ENHANCEMENT OF GAMMA-Fe₂O₃ NANOPARTICLES BY SONOCHEMICAL COATING

Shafi KVPM. Ulman A. Dyal A. Yan XZ. Yang NL. Estournes C. Fournes L. Wattiaux A. White H. Rafailovich M. - *Chemistry of Materials*. 14(4):1778-1787, 2002

We show that sonochemistry is an efficient and facile route for quantitative coating of gamma-Fe₂O₃ nanoparticles with octadecyltrihydrosilane (OTHS, CH₃(CH₂)₁₇SiH₃). The presence of C-H stretching (2950-2850 cm⁻¹) and bending (1475-1375 cm⁻¹) bands, and the absence of peaks at 2150 and 925 cm⁻¹ (Si-H stretching and bending respectively) confirm the presence of grafted hydrocarbon chains in the irradiated sample, hence the reaction of OTHS with gamma-Fe₂O₃ nanoparticles. The coated nanoparticles show increased magnetization compared to the uncoated ones. X-ray diffraction, magnetization studies, Mossbauer spectra, and electron paramagnetic resonance spectra reveal that the magnetic ordering is more prominent in the OTHS-coated gamma-Fe₂O₃ sample because of improved crystallinity.

[27] IMPROVED SUPERCONDUCTING PROPERTIES IN NANOCRYSTALLINE BULK MgB₂

Gumbel A. Eckert J. Fuchs G. Nenkov K. Muller KH. Schultz L. - *Applied Physics Letters*. 80(15):2725-2727, 2002

High density nanocrystalline MgB₂ bulk superconductors with distinctly improved pinning were prepared by mechanical alloying of Mg and B powders at ambient temperatures followed by hot pressing. The nanocrystalline samples reveal high j(c)=10(5) A/cm(2) at 20 K and 1 T together with an irreversibility line strongly shifted towards higher fields resulting in H-



irr(T) similar to 0.8 H-c₂(T), whereas typically H-irr(T) similar to 0.5 H-c₂(T) is observed for untextured bulk samples. These values exceed those of all other reported bulk samples and are in the range of values for thin films. The improved pinning of this material, which mainly consists of spherical grains about 40-100 nm in size, is attributed to the large number of grain boundaries in the nanocrystalline state.

[26] TRANSLUCENT PMN AND PMN-PT CERAMICS FROM HIGH-ENERGY BALL MILLING DERIVED POWDERS

Kong LB. Ma J. Zhu W. Tan OK. - Materials Research Bulletin. 37(1):23-32, 2002

Translucent PMN and PMN-PT ceramics were prepared from nano-sized powders produced by a high-energy ball milling process. A two-step process was used to achieve the translucent PMN and PMN-PT ceramics, in which samples were sintered at 950°C for 1 h at first and then annealed at 1000°C for 6-24 h. Transmittances at 600 nm wavelength of the PMN, PMN-0.10PT and PMN-0.35PT ceramics annealed for 12 h were 21, 16 and 11%, respectively. The formation mechanism of translucent PMN and PMN-PT ceramics in the present work is similar to that of transparent PLZT ceramics.

[25] FAST AMORPHIZATION AND CRYSTALLIZATION IN AL-Fe BINARY SYSTEM BY HIGH-ENERGY BALL MILLING

Zou Y. Saji S. Kusabiraki K. - Materials Research Bulletin. 37(1):123-131, 2002

Large amount of amorphous phase of Al-Fe binary system was obtained by MA of elemental powders using a high-energy ball mill at milling intensity of 150G (G is the gravitational acceleration). XRD, HRTEM and DSC were used to analyze the process of amorphization and crystallization. The time required achieving almost complete amorphous state is only 4.2 ks for Al-25 at.%Fe system and 3 ks for Al-30 at.%Fe system, respectively. The time of amorphous formation is very shorter than that of previous reports on Al-Fe binary system. Further milling causes rapid crystallization of the amorphous phase. By analysis of S(Q), the presence of a strong Al-Fe chemical short-range order in the amorphous matrix is suggested. Moreover, the superstructure of these Al-Fe clusters in the amorphous matrix is similar to the solid structure of Al₅Fe₂, and the clusters transform into the nucleus of Al₅Fe₂ intermetallic compound under the action of milling energy

[24] THE INFLUENCE OF MECHANICAL MILLING AND SUBSEQUENT CALCINATION ON THE FORMATION OF LITHIUM FERRITES

Widatallah HM. Berry FJ. - Journal of Solid State Chemistry. 164(2):230-236, 2002

The influence of ball milling and subsequent calcination of a 2.5:1 molar mixture of alpha-Fe₂O₃ and Li₂CO₃ on the formation of lithium ferrites has been investigated. Premilling was found to considerably lower the temperature at which the lithium ferrites LiFeO₂ and LiFe₅O₈ are formed. A beta-to-alpha-phase transition in LiFe₅O₈ was found to take place on cooling from ca. 1000°C depending on the milling history and cooling regime

[23] MECHANOCHEMICAL COPOLYMERIZATION OF METHYL METHACRYLATE AND STYRENE INITIATED BY THE GRINDING OF QUARTZ

Hasegawa M. Kimata M. Kobayashi SI. - Journal of Applied Polymer Science. 84(11):2011-2017, 2002

The copolymerization of methyl methacrylate and styrene mechanochemically initiated by the grinding of quartz in the monomer mixture was investigated by using a vibrating ball mill. The effect of the grinding of quartz on the copolymerization was investigated by characterizing both the polymer formed and the ground quartz. The copolymerization proceeds apparently from the quartz grindings and was closely related to the total surface area of the ground quartz. The molecular weight distributions of the copolymers formed were unimodal as found in a homopolymer of only methyl methacrylate. It was clear from the composition analysis of the copolymer that the mechanochemical copolymerization of methyl methacrylate and styrene proceeded with a radical polymerization mechanism because of radicals on the quartz surface produced by the grinding. The monomer reactivity ratios obtained by the Fineman-Ross method suggested that the copolymers formed were alternating copolymers

[22] FORMATION OF NICKEL ALUMINIDES BY MECHANICAL ALLOYING AND THERMODYNAMICS OF INTERACTION

Portnoy VK. Blinov AM. Tomilin IA. Kuznetsov VN. Kulik T. - Journal of Alloys & Compounds. 336(1-2):196-201, 2002

Structure and temperature stability of Ni-Al alloys obtained by mechanical alloying (MA) were studied using X-ray diffraction and differential scanning calorimetry (DSC). MA of Ni-Al powders produces the B₂ phase for the composition range 40-61 at.% Ni, and a nanocrystalline supersaturated solid solution Ni(Al) in the range 65-85 at.% Ni. NIA of Ni_{62.5}Al_{37.5} leads to a nanocrystalline phase, probably with L1(0) structure. The NiAl (B₂) phase is formed through direct rapid exothermic solid state reaction without formation of intermediate solid solutions. Formation of the ordered tetragonal Ni₅Al₃ phase occurs after prolonged deformation of the nonstoichiometric B₂ phase with 62.5 at.% Ni. The results of MA were compared with calculated Gibbs energies of principal phases namely FCC, L1(2) and B₂ using the results of computer assessment of the system. The final product of MA is always single phase which has minimal Gibbs energy of the competing phases at alloy composition. This indicates a leading role of the thermodynamic factors on phase formation by MA

[21] COMPARATIVE STUDY OF MECHANICAL ALLOYING OF (Mg PLUS Al) AND (Mg PLUS Al PLUS Ni) MIXTURES FOR HYDROGEN STORAGE

Bououdina M. Guo ZX. - Journal of Alloys & Compounds. 336(1-2):222-231, 2002

Mg is a desirable hydrogen-storage material of high capacity, but suffers from low kinetics and is difficult to activate. Addition of appropriate alloying elements, such as Al and Ni, and a fine microstructure can ease the problems. In order to increase the solubility of Al into Mg and to refine the powder structure, a (Mg+10 at.% Al) mixture was investigated by mechanical alloying. It was noted that the amount of elemental Al decreases with increasing milling time. After 20 h of



milling, an h.c.p. (Mg, Al) solid solution was obtained with a volume contraction of 1.2%. An energy dispersive X-ray (EDX) analysis confirmed its formation and showed its chemical composition as (Mg+12 at.% Al). When 10 at.% Ni was further added to the (Mg, Al) solid solution as a catalytic element, a new Mg₁₂Al₁₇ phase was formed, in addition to a modified (Mg, Al, Ni) solution. When the (Mg+10 at.% Al) mixture milled for 2 h was annealed at 400degreesC for 2 h, all the Al dissolved into Mg and formed a single (Mg, Al) solid solution. When the (Mg+10 at.% Al+10 at.% Ni) mixture was sintered at the same condition, a new AlNi phase was formed, in addition to the presence of a (Mg, Al, Ni) solid solution

[20] PREPARATION OF PMN-PT CERAMICS VIA A HIGH-ENERGY BALL MILLING PROCESS

Kong LB. Ma J. Zhu W. Tan OK. - Journal of Alloys & Compounds. 336(1-2):242-246, 2002

Pyrochlore-free nano-sized (1-x)PMN-xPT (x=0, 0.10, 0.35) (PMN-PT) powders were synthesized from a mixture of the commercial oxides PbO, MgO, TiO₂ and Nb₂O₅ by a high-energy ball milling process. The grain size estimated from XRD patterns for the three samples was about 10-30 nm. The high sinterability of the synthesized PMN and PMN-PT powders was evidenced by the fact that almost fully dense ceramics for the three systems could be readily achieved at temperatures as low as 950degreesC. The relationship between microstructure, electrical properties of the PMN and PMN-PT ceramics and sintering temperature was investigated. The dielectric properties of 0.90PMN-0.10PT are comparable to those of the traditional ceramic samples, while 0.65PMN-0.35PT has a better higher dielectric constant than those of traditional ceramics after sintering at 1100degreesC for 1 h.

[19] NANOCRYSTALLINE TITANIUM-TYPE METAL HYDRIDE ELECTRODES PREPARED BY MECHANICAL ALLOYING

Jurczyk M. Jankowska E. Nowak M. Jakubowicz J. - Journal of Alloys & Compounds. 336(1-2):265-269, 2002

Mechanical alloying (MA) was employed to produce nanocrystalline TiFe_{1-x}Ni_x alloys (x=0, 0.25, 0.5, 0.75 and 1.0). XRD analysis showed that, after 25 h of milling, the starting mixture of the elements had decomposed into an amorphous phase. Following annealing in high purity argon at 750degreesC for 0.5 h, XRD confirmed the formation of the CsCl-type structures with crystallite sizes of about 50 nm. These materials were used as negative electrodes for a Ni-MHx battery. With increasing nickel content in TiFe_{1-x}Ni_x, the material shows an increase in discharge capacity which passes through a maximum for x=0.75. In the nanocrystalline TiFe_{0.25}Ni_{0.75} powder, discharge capacities of up to 155 mA h g⁽⁻¹⁾ (at 40 mA g⁽⁻¹⁾ discharge current) were measured. The titanium-based hydrogen storage alloys are attractive for secondary batteries, because of inexpensive raw materials.

[18] HYDROGEN SORPTION OF MG-BASED MIXTURES ELABORATED BY REACTIVE MECHANICAL GRINDING

Bobet JL. Chevalier B. Song MY. Darriet B. Etourneau J. - Journal of Alloys & Compounds. 336(1-2):292-296, 2002

The use of mechanical grinding (MG) under H₂ of magnesium powder improves the hydrogen sorption properties. The hydrogenation of Mg starts in situ during the milling process that allows suppressing the activation procedure generally requested for Mg. The effects of the addition of various elements or compounds have been studied. The hydriding is a two-step process: nucleation and diffusion. A direct relationship exists between the nucleation duration and the specific surface. A critical milling time exists below which the diffusion process is improved and above which no further improvement is observed (the maximum internal stress in the powder is also reached at this critical time). The diffusion is controlled by the number of crystallites per particle that can be reduced by increasing the milling time up to 10 h. The addition of Co (catalyst), YNi (hydrogen pump) or oxides (abrasive element and nucleation centre) leads to an improvement of the hydrogen sorption properties (but a strong dependence upon the milling time is reported). Finally, the sorption properties of our mixtures are comparable with thus reported for MgH₂-metal mixtures.

[17] PREPARATION OF THE SOLID SOLUTION Sn0.5Ti0.5O2 FROM AN OXIDE MIXTURE VIA A MECHANOCHEMICAL PROCESS

Kong LB. Ma J. Huang H. - Journal of Alloys & Compounds. 336(1-2):315-319, 2002

The solid solution Sn_{0.5}Ti_{0.5}O₂ was prepared by a high-energy ball milling process from the oxide mixture of SnO₂ and TiO₂. After milling for 5 h, the sample was still a mixture of TiO₂ and SnO₂ although the grain size was greatly reduced. Ten-hour milling led to single phase Sn_{0.5}Ti_{0.5}O₂. The effect of calcination on the phase formation and stability was investigated. The Sn_{0.5}Ti_{0.5}O₂ was stable up to 900degreesC and separated into a Sn-rich phase (Sn_{0.77}Ti_{0.23}O₂) and a Ti-rich phase (Sn_{0.17}Ti_{0.83}O₂) at temperature above 1000degreesC.

[16] REACTION SINTERING WITH NEGATIVE VOLUME CHANGES

Authors Gilev VG. - Inorganic Materials. 38(3):296-301, 2002

The preparation of high-porosity sialon and SiC materials by reaction sintering accompanied by weight loss and shrinkage is considered. beta-Sialon ceramics with a 60% porosity, pore size of 0.7 μm, and gas permeability K = 0.04 μm² are obtained via carbothermal reduction and simultaneous nitriding of kaolin in kaolin + graphite green compacts. The SiC materials prepared from mixtures of SiO₂ and graphite have a porosity of 83% and a pore size of 1 μm. The most promising technique is elemental synthesis. In the SiC materials prepared by milling silicon + graphite mixtures to a specific surface area of 80 m²/g, pressing at 100-200 MPa, and sintering in argon at 1600degreesC for 15 min, the pore size is as small as 0.2-0.3 μm.

[15] INFLUENCE OF COLLOIDAL ABRASIVE SIZE ON MATERIAL REMOVAL RATE AND SURFACE FINISH IN SiO2 CHEMICAL MECHANICAL POLISHING

Zhou CH. Shan L. Hight JR. Danyluk S. Ng SH. Paszkowski AJ. - Tribology Transactions. 45(2):232-238, 2002

This paper addresses the influence of nano-scale abrasive particle size in the polishing of thermally-grown silicon dioxide on 100 mm diameter, p-type, (100), single crystal silicon wafers. The abrasive particles are incorporated in a chemical slurry,



which is used in chemical mechanical polishing (CMP). Polishing (material removal) rate was measured with six (6) slurries, each with a different mean abrasive particle diameter of 10, 20, 50, 80, 110 and 140 nm. The experimental results indicate that the material removal rate (MRR) is related to the particle size, Results confirm that there exists an optimum abrasive size (80 nm) with respect to material removal rate and surface finish, for a given set of experimental conditions. The variation of the MRR vs. particle size (077 a log-log plot) varies as $d^{3.4}$. In addition, the surface polished with 10 nm and 20 nm abrasives was stained by the slurries. For a pad surface roughness of 5.2 μm (Ra), the slurry containing the 80 nm particles resulted in the highest material removal rate and best surface finish. The effects of polishing conditions and abrasive concentrations are also investigated in this paper. The nano-film polishing model that relates the particle size to the roughness of the pad can explain the polishing mechanism

[14] THREE-DIMENSIONAL ATOM PROBING OF SUPERSATURATED MECHANICALLY ALLOYED CU-20AT.% CO

Czubayko U. Wanderka N. Naundorf V. Ivchenko VA. Yermakov AY. Uimin MA. Wollenberger H. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 327(1 Special Issue SI):54-58, 2002

Cu-20at.% Co was mechanically alloyed from pure elements by ball milling for 28 h. The powder was compacted into pellets at room temperature by applying a pressure of 0.5 GPa. Microstructural investigations were carried out by three-dimensional atom probing (3DAP) after annealing at 400 degreesC for 2 h in Ar atmosphere. The annealed Cu 20at% Co exhibits a heterogeneous microstructure. Cu-rich and Co-rich particles with 8.7 at% Co and 1.3 at% Cu, respectively, and highly supersaturated regions with average Co concentrations between 25 and 35 at.% were detected. A new, sensitive statistical analysis of the 3DAP data obtained from a supersaturated region revealed concentration fluctuations of 24 at% Co with an average wave length of about 2 nm indicating spinodal decomposition

[13] PREPARATION AND CORROSION BEHAVIOR OF NANOCRYSTALLINE IRON GRADIENT MATERIALS PRODUCED BY POWDER PROCESSING

Elkedi O. Cao HS. Guay D. - Journal of Materials Processing Technology. 121(2-3):383-389, 2002

A powder processing technique is developed for the fabrication of a functionally gradient material obtained by different mixtures of nanocrystalline iron and as-received microcrystalline iron. The corrosive behavior of hot compacted nanocomposite iron in 0.5 M sulfuric acid media has been investigated by potentiodynamic test method. The obtained results revealed that an increase of nanocrystalline iron fraction improves the corrosion resistance in the iron nanocomposites

[12] INTENSIVE IMPROVEMENT OF REDUCTION RATE OF HEMATITE-GRAPHITE MIXTURE BY MECHANICAL MILLING

Khaki JV. Kashiwaya Y. Ishii K. Suzuki H. - ISIJ International. 42(1):13-22, 2002.

The effect of ball milling of raw materials on the reaction behavior of composite mixture of hematite and graphite have been studied. Hematite was mixed with 19.84 wt% graphite (C/O ratio was 1.1 in composite mixture) and subjected to ball milling. The milling time was changed from 6 to 100 hr with the hematite-graphite mixture. On the other hand, graphite or hematite was milled alone and then mixed with non-milled hematite or graphite, respectively. The effect of milling time on reduction process in an Ar atmosphere was studied by TG-DTA. The samples were heated by a constant heating rate of 10degreesC/min from room temperature up to 1 100degreesC and maintained for 30 min at this temperature. The rate of reaction (RTG) was obtained by the differentiation of weight loss curve. It was found that the RTG curve consisted of three reaction curves which were hematite-magnetite (HM), magnetite-wustite (MW) and wustite-metallic iron (WF) reductions. The curve corresponding to the HM reduction located in low temperature range and stood alone from other two reduction curves (MW and WF reductions), The curves of MW and WF reductions were overlapped. The pulse-like reduction curve corresponding to WF reduction was observed in the longer milling time, which meant extremely high rate of reduction. The temperatures decreased and the reaction degrees at each peaks increased with increasing milling time. The kinetic analysis applying the single and consecutive reaction was carried out. The calculated reaction curves were in good agreement with the observations which showed that the reaction mainly occurred in this system was the solid oxide-solid carbon reaction. The variation of parameters of rate constant presented the different mechanism of reaction between shorter and longer milling time at the border of 24 hr

[11] THE EFFECT OF MICROSTRUCTURE ON DUCTILITY OF NiAl

Dollar A. Dymek S. Dollar M. - Archives of Metallurgy. 47(1):3-15, 2002.

NiAl, an intermetallic compound with the B2 ordered structure, exhibits potential as a viable high temperature structural material. However, before this material (and other intermetallics) can be of practical use a number of technical problems must be overcome, including lack of ductility at the room temperature. In an attempt to address the brittleness of NiAl, and based on a premise that grain refinement may lead to significant ductility improvements mechanical alloying and nanocrystalline synthesis were used to produce fine-grained NiAl. The mechanically alloyed NiAl with an average grain size of about 0.5 μm , unlike its coarse grained, cast counterpart, exhibits a notable room temperature compressive ductility due to two contributing microstructural factors: i) the development of the <110> texture during hot extrusion leading to the activation of additional slip systems, and ii) the predominance of low angle grain boundaries. In the nanocrystalline form, NiAl with an average grain size in the range from 2 to 10 nm, exhibits measurable room temperature ductility in biaxial disc bend tests, unlike its coarse-grained counterpart. This observation can be explained assuming that diffusional, rather than dislocation, mechanisms control plastic deformation of the nanocrystalline NiAl. The emphasis of the present paper is on rationalizing the improved room temperature ductility in mechanically alloyed and nanocrystalline NiAl. The most significant conclusion of the present discussion - contrary to widespread beliefs - is that the grain size plays only an indirect role in controlling ductility. [10] ON THE WAY TO THE OPTIMIZATION OF LI3FE2(PO4)3 POSITIVE ELECTRODE MATERIALS

Morcrette M. Wurm C. Masquelier C. - Solid State Sciences. 4(2):239-246, 2002



The electrochemical performances of the two polymorphs of $\text{Li}_3\text{Fe}_2(\text{PO}_4)_3$ are reported. We show that reversible capacities as high as 110 mA h g^{-1} at C/20 regime with an excellent capacity retention are obtained by playing with particle sizes and surface. Tailored made particles were done chemically, by developing a new low temperature synthetic route. In contrast, the modification of the particle surface was achieved by ball-milling of carbon/ $\text{Li}_3\text{Fe}_2(\text{PO}_4)_3$ mixtures, confirming again the positive attribute of such process regarding the electrochemical performances of transition metal phosphates

[9] ANALYSIS OF MECHANISMS LIMITING THE DISPERSITY OF POWDERS PRODUCED BY BALL MILLING

Vasil'ev LS. Lomaeva SF. - Physics of Metals & Metallography (English Translation of Fizika Metallov i Metallovedenie). 93(2):166-174, 2002

Mechanisms limiting the dispersity of powders produced by mechanical disintegration are discussed. The main mechanism is shown to be the passage of nanocrystalline powder particles into the superplastic state after which the disintegration stops and all further treatments only lead to changes in the shape of particles. By introducing the notion of macrodislocation, i.e., a linear defect representing a distortion of the regular packing of grains in a nanocrystal, both the transition into the superplastic state and the process of fracture were described. The limits of disintegration of metallic powders have been estimated. The results agree well with experimental data obtained by atomic force microscopy of iron powders disintegrated with additions of surfactants. A scheme of the formation and development of the nanostructure and phase composition of powder particles disintegrated in ball mills in liquid organic media is suggested which takes into account the processing features related to multiple high-strain-rate hardening: high-temperature polygonization, and recrystallization and multiple quenching in the liquid medium.

[8] STRUCTURAL OBSERVATION OF ENERGY STORAGE MATERIALS PREPARED BY MA

Fukunaga T. Itoh K. Orimo S. Fujii H. - Physica B. 311(1-2):95-101, 2002

Mechanical alloying (MA) was employed to synthesize hydrogen storage materials and cell materials as anodes of secondary lithium-ion batteries. Amorphous MgNi alloy was prepared by MA. The structures of dilutely and fully deuterated MgNi alloys ($\text{MgNiD}_{0.1}$, $\text{MgNiD}_{1.6}$) were observed using a combination of X-ray and neutron diffractions. The radial distribution functions $\text{RDF}(r)$ of the amorphous MgNiD_x alloys provided us information that deuterium atom occupies the tetrahedral site composed of nearly $2\text{Mg}(2)\text{Ni}$. Hexagonal graphite was found out to absorb deuterium during the MA process, It changed into the nano-crystalline one after milling. Two types of the location of deuterium atoms for $\text{CD}_{0.661}$ powder were found in $\text{RDF}(r)$ observed by neutron diffraction. One is the C-D covalent bond and the other is due to deuterium located between layers of the graphite. Lithium-graphite intercalation compound was obtained from graphite and lithium mixed powders by MA. The short-range structure around a Li atom was observed by using the advantage of neutron diffraction. The coordination number of the C-Li correlation indicates the formation of $\text{LiC}_{3.1}$ intercalation compound

[7] RAPID SYNTHESIS OF MESOPOROUS TiO_2 WITH HIGH PHOTOCATALYTIC ACTIVITY BY ULTRASOUND-INDUCED AGGLOMERATION

JC. Zhang LZ. Yu JG. - New Journal of Chemistry. 26(4):416-420, 2002.

Three-dimensional and thermally stable mesoporous TiO_2 was synthesized without the use of any surfactants via treatment with high intensity ultrasound irradiation for a short period of time. Monodispersed TiO_2 sol particles were formed initially by ultrasound-assisted hydrolysis of acetic acid-modified titanium isopropoxide. Then, the mesoporous spherical or globular particles, which have a narrow pore size distribution, were produced by controlled condensation and agglomeration of these sol nanoparticles under high intensity ultrasound irradiation. The mesoporous TiO_2 was characterized by XRD, TEM, nitrogen adsorption, TGA/DTA and FTIR. Low-angle XRD and TEM show the mesoporous TiO_2 has a wormhole-like structure and a lack of long-range order. Nitrogen adsorption results indicate that the mesoporous TiO_2 retains mesoporosity with a narrow pore size distribution and high surface area to at least 673 K. The thermal stability of mesoporous TiO_2 is attributed to its thick inorganic walls, consisting of TiO_2 nanoparticles. TGA study shows that this synthetic method is environmentally friendly. The photocatalytic activity of mesoporous TiO_2 for the oxidation of acetone in air was measured. As-prepared mesoporous TiO_2 has negligible activity due to its amorphous structure. Calcined mesoporous TiO_2 shows better activity than commercial photocatalyst P25. The reasons for the high activity of mesoporous TiO_2 are discussed.

[6] A NEW APPROACH TO PREPARING TIPS FOR ATOM PROBE FIELD ION MICROSCOPY FROM POWDER MATERIALS

Wu F. Bellon P. Lau ML. Lavernia EJ. Lusby TA. Melmed AJ. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 327(1 Special Issue SI):20-23, 2002

A new approach to preparing tips for atom probe field ion microscopy from powder materials is described, It is especially useful for particle sizes exceeding $> 0.050 \text{ mm}$. Particles resulting from mechanical alloying of Cu and Ag, and from cryomilling of conventional Ni powders have been prepared this way for field ion imaging and atom probe composition analysis. In both cases, useful information complementing results from transmission electron microscopy and X-ray diffraction was obtained.

[5] THREE-DIMENSIONAL ATOM PROBING OF SUPERSATURATED MECHANICALLY ALLOYED Cu-20at.\% Co

Czubayko U. Wanderka N. Naundorf V. Ivchenko VA. Yermakov AY. Uimin MA. Wollenberger H. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 327(1 Special Issue SI):54-58, 2002

Cu-20at.\% Co was mechanically alloyed from pure elements by ball milling for 28 h. The powder was compacted into pellets at room temperature by applying a pressure of 0.5 GPa. Microstructural investigations were carried out by three-dimensional atom probing (3DAP) after annealing at 400 degreesC for 2 h in Ar atmosphere. The annealed Cu 20at.\% Co exhibits a heterogeneous microstructure. Cu-rich and Co-rich particles with 8.7 at% Co and 1.3 at% Cu, respectively, and highly supersaturated regions with average Co concentrations between 25 and 35 at.% were detected. A new, sensitive statistical analysis of the 3DAP data obtained from a supersaturated region revealed concentration fluctuations of 24 at% Co with an average wave length of about 2 nm indicating spinodal decomposition.

[4] ANNEALING STUDY OF Fe_2O_3 NANOPARTICLES: MAGNETIC SIZE EFFECTS AND PHASE TRANSFORMATIONS

Xu XN. Wolfus Y. Shaulov A. Yeshurun Y. Felner I. Nowik I. Koltypin Y. Gedanken A. - Journal of Applied Physics. 91(7):4611-4616, 2002

Sonochemically synthesized Fe_2O_3 nanoparticles were annealed in air or in vacuum while their magnetization was continuously recorded. Annealing in vacuum at temperatures T_a between 240 and 450 degreesC produced nanophases of



gamma-Fe₂O₃ with average particle size ranging from 4 to 14 nm, depending on T-a. Phase transformation into alpha-Fe₂O₃ occurred directly by annealing in air, or via an intermediate Fe₃O₄ phase by annealing in vacuum at temperatures higher than 450 degreesC. Mapping the correlation between the magnetic properties and the annealing conditions, enables control of the annealing process to obtain nanocrystals of gamma-Fe₂O₃, alpha-Fe₂O₃, or Fe₃O₄ with different particle size and magnetic properties.

[3] NANOSTRUCTURED ICOSAHEDRAL PHASE FORMATION IN AL70CU20FE10 BY MECHANICAL ALLOYING: COMPREHENSIVE STUDY

Barua P. Murty BS. Mathur BK. Srinivas V. - Journal of Applied Physics. 91(8):5353-5359, 2002

A systematic study on the formation and the stability of Al₇₀Cu₂₀Fe₁₀ quasicrystalline alloy has been carried out through mechanical alloying. The influence of the milling parameters, sequence of additive elements, and heat treatment on the formation of icosahedral phase has been investigated through x-ray diffraction analysis and transmission electron microscopy. A combination of milling and heat treatment of Al₇₀Cu₂₀Fe₁₀ composition resulted in the formation of a single icosahedral phase (i-phase) quasicrystal with face-centered structure. We present evidence that suggests the i phase is of a nanoquasicrystalline nature. From the present investigations, we propose an i-phase formation mechanism in Al-Cu-Fe system.

[2] CYCLIC PHASE TRANSFORMATIONS OF MECHANICALLY ALLOYED CO75TI25 POWDERS

El-Eskandarany MS. Aoki K. Sumiyama K. Suzuki K. - Acta Materialia. 50(5):1113-1123, 2002

Cyclic crystalline-amorphous-crystalline phase transformations have been investigated during high-energy ball milling of a mixture of elemental Co₇₅Ti₂₅ powder under an argon gas atmosphere. The results have shown that the single amorphous phase, which is obtained after 11 ks of mechanical alloying time, transforms into a new metastable phase of nanocrystalline bcc-Co₃Ti upon milling for 86 ks. However, it transforms to the same amorphous phase upon further milling (360 ks). The present work shows the effects of the milling time and the milling speed on the structure of the mechanically alloyed powders

[1] EVOLUTION OF RECRYSTALLISATION TEXTURE AND MICROSTRUCTURE IN LOW ALLOYED TITANIUM SHEETS

Wagner F. Bozzolo N. Van Landuyt O. Grosdidier T. - Acta Materialia. 50(5):1245-1259, 2002

The evolution of microstructure and crystallographic texture in low alloyed titanium sheets, initially deformed by 80% cold rolling, are investigated at different stages of the recrystallisation process. Optical and transmission electron microscopies, as well as X-ray diffraction and EBSD are used to provide information about recrystallisation mechanisms and kinetics. Orientation Density Function (ODF) differences are used to quantitatively compare recrystallised and deformed states. The main texture features of the deformed state evolve only slightly during the primary recrystallisation. The major changes in texture result from secondary recrystallisation or grain growth. Primary recrystallisation can be roughly separated into two stages. The first one is very fast and corresponds to the appearance of new grains in about 80% of the material volume. The second stage is more sluggish. It corresponds to the disappearance of the so-called "white grains", which did not twin during deformation due to their stable orientation near { $\phi(1) = 0^\circ$, $\phi = 45^\circ$, $\phi(2) = 0^\circ$ }. Recovery is an important mechanism throughout the process and deformation heterogeneities must be taken into account for a good understanding of the recrystallisation in titanium.



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