



**RESEAU FRANÇAIS DE
MECANOSYNTHESE**

Lettre N°89

Août 2002

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

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IMPORTANT

**Do Not Forget the Annual Subscription Fees
(i.e. 20 Euros / Year)**

A check will be done during the Summer !!

Bulletin d'adhésion 2002 / Subscription Print

(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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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.

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Congress and School Announcements
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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 \(f\)](mailto: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.

[68] SINTERING BEHAVIOUR OF PIEZOCERAMIC POWDERS PREPARED BY CONVENTIONAL CERAMIC AND MECHANICAL ACTIVATION PROCESS

C Miclea, C Tanasoiu, A Gheorghiu, CF Miclea, V Tanasoiu - 2001 INTERNATIONAL SEMICONDUCTOR CONFERENCE, VOL 1 & 2, PROCEEDINGS, 2001, pp 297-300 - INTERNATIONAL SEMICONDUCTOR CONFERENCE; SINAIA, ROMANIA. OCTOBER 9-13, 2001

Conventional and mechanical activation techniques were used to prepare a soft PZT type piezoceramic material. The resulting powders exhibit micro and nanostructures, respectively as indicated by X-ray and electron microscopy. Average crystallite sizes of 1 μ m and 50 nm respectively were estimated for the powders. The sintered behavior of pressed samples from the two types of powders was investigated within the 800-1300 degreesC temperature range and the densities, structural morphology and dielectric and piezoelectric properties were investigated. Optimum sintering temperature were about 100 degreesC lower for samples made from mechanical activated powder compared to the conventionally ones. Sintered samples from this powder exhibited also enhanced properties compared to those made from the conventionally prepared powder. It is concluded that the mechanical activation technique is a reliable and simple method to prepare high quality PZT type materials.

[67] APPLICATION OF A MECHANOCHEMICAL METHOD FOR RECYCLING WASTE GLASS WITH CO₂ SOLIDIFICATION

LH Liu, T Hashida, S Teramura - MECHANICS AND MATERIAL ENGINEERING FOR SCIENCE AND EXPERIMENTS, 2001, pp 209-213 - INTERNATIONAL SYMPOSIUM OF YOUNG SCHOLARS ON MECHANICS AND MATERIAL ENGINEERING FOR SCIENCE AND EXPERIMENTS; CHANGSHA, PEOPLES R CHINA. AUGUST 11-16, 2001

A mechanochemical method for treatment of powdered (recycled) waste glass and calcium hydroxide is combined with a CO₂ solidification process for strength development of solidified products. Our work has potential for reducing CO₂ emission, which has been identified as a major cause of global warming. Poorly crystalline pre-pulverized waste glass cullet and calcium hydroxide powder was mixed with similar to 70 wt % water and cured at room temperature, prior to CO₂ carbonation, to produce a solidified sample. Solidified samples were heated to examine phase transformations. Sample density, Vickers hardness and flexural strength were determined. Effect of grinding time, Ca/Si ratio and water content on the mechanical properties was investigated. The mechanochemical reaction has a strong influence on the hardening process, due to formation of calcium carbonate. TG-DTA results show that the decomposition temperature of the glass/calcium hydroxide mixture is decreased after mechanochemical treatment, indicating enhanced reactivity of the mixture. Vickers hardness is increased after CO₂ solidification. Our method is effective for the production of various building materials.

[66] THE STUDY OF REGULARITY OF AMORPHOUS FORMATION FOR MULTI-COMPONENT ALLOYS BY MECHANICAL ALLOYING

LL Wang, WQ Huang, XF Li - MECHANICS AND MATERIAL ENGINEERING FOR SCIENCE AND EXPERIMENTS, 2001, pp 581-583 - INTERNATIONAL SYMPOSIUM OF YOUNG SCHOLARS ON MECHANICS AND MATERIAL ENGINEERING FOR SCIENCE AND EXPERIMENTS; CHANGSHA, PEOPLES R CHINA. AUGUST 11-16, 2001

The two parameters, namely enthalpy of formation of alloy A(50)-B-50 $\Delta H(f)$ and the difference of self-diffusion activation energy $\Delta H(s)$ of constituent of alloy, are used to study the regularity of amorphous formation for multi-component alloys, according to the SSAR mechanism of amorphization by mechanical alloying. After analyzing numbers of experiments, a criteria of amorphization of binary alloys, $\Delta H(f) < -0.0024(\Delta H(s))^2 + 0.6000(\Delta H(s)) - 33.0000$ was obtained and used to analyze the 78 alloy systems with available experiments results. The total degrees of accuracy for the amorphous alloys forming or not by mechanical alloying are 87.3%. And for binary and ternary alloys, $\Delta H(f) < -0.0036(\Delta H(s))^2 + 0.7200(\Delta H(s)) - 34.0000$ was obtained and used to analyze the 78 binary alloy and 24 ternary alloy systems with available experimental results. The total degrees of accuracy are 83.7%.

[65] NANOSTRUCTURE FORMATION AND CARBIDES DISSOLUTION IN RAIL STEEL DEFORMED BY HIGH PRESSURE TORSION

YV Ivanisenko, RZ Valiev, W Lojkowski, A Grob, HJ Fecht - ULTRAFINE GRAINED MATERIALS II, 2002, pp 47-54 - 2ND INTERNATIONAL SYMPOSIUM ON ULTRAFINE GRAINED MATERIALS; SEATTLE, WASHINGTON. FEBRUARY 17-21, 2002

The microstructure and the phase composition evolution of a commercial UIC 860V steel during high pressure torsion (HPT) were investigated by means of transmission electron microscopy, thermomagnetic analysis and X-ray diffraction. HPT leads

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Corresp. : <mailto:Eric.Gaffet@utbm.fr>



to a decrease of the grain size to 10 nm and total cementite dissolution. In the nanocrystalline state the steel reaches the hardness of 11 GPa, which is 3 times higher than that of the initial state. The partial cementite dissolution starts already at the strain degree of $\gamma = 92$. The carbon distribution in the iron matrix seems to depend on the deformation degree.

[64] FORMATION OF NANOCRYSTALLINE STRUCTURE IN A NI-20 % CR ALLOY

N Dudova, R Kaibyshev, V Valitov - ULTRAFINE GRAINED MATERIALS II, 2002, pp 75-80 - 2ND INTERNATIONAL SYMPOSIUM ON ULTRAFINE GRAINED MATERIALS; SEATTLE, WASHINGTON. FEBRUARY 17-21, 2002

Microstructural evolution resulting in nanocrystalline structure was examined in a Ni-20%Cr alloy during plastic deformation by high pressure torsion at ambient temperature. It was shown that a gradual evolution of dislocation structure results in the formation of nanoscale grains. This process is accompanied by increasing internal elastic strain and microhardness. In the strain interval of 0-2.5, an extensive accumulation of lattice dislocations takes place. Material volume is subdivided into mutually misorientated areas of uniformly distributed lattice dislocations. The size of such areas tends to decrease and continuous misorientations within initial grains increase with increasing strain. The formation of first nanoscale grains was detected at $\epsilon=2.5$. Following deformation leads to increasing volume fraction of nanoscale grains and decreasing their size. The nanocrystalline structure evolved is characterized by high values of microhardness (4.6GPa) and internal elastic strain.

[63] MICROSTRUCTURE EVOLUTION IN NANOCRYSTAL FORMATION DURING BALL MILLING

ZG Liu, Y Xu, K Tsuchiya, M Umemoto - ULTRAFINE GRAINED MATERIALS II, 2002, pp 105-112 - 2ND INTERNATIONAL SYMPOSIUM ON ULTRAFINE GRAINED MATERIALS; SEATTLE, WASHINGTON. FEBRUARY 17-21, 2002

The microstructure evolution during ball milling to produce nanocrystalline structure has been studied in a Fe-C alloy. Two types of microstructures have been observed. It has been found that structure defects mainly of dislocations, dislocation arrays, dislocation cells are formed in the interior of the milled powders, where weak strain and strain rate are present. However, in the outer surface of the milled powders, localized heavy deformation leads to the formation of layered nanocrystalline structure. The process is suggested to be aided by the temperature rise due to collision, which may cause a dynamic recovery, to transform the high density of dislocations and dislocation cells into grain boundary. A heavily deformed structure between the layered nanocrystals and weakly deformed dislocation cells or grains was observed as well, which is attributed to the localized deformed region without the recovery or recrystallization. The grain refinement of the nanocrystals is believed to be the refinement from nanoscaled layers to equi-axed grains.

[62] FORMATION OF NANOCRYSTALLINE STRUCTURE IN TWO-PHASE TITANIUM ALLOYS BY WARM SEVERE PLASTIC DEFORMATION

GA Salishchev, MA Murzinova, SV Zherebtsov, RM Galejev, OR Valikhmetov - ULTRAFINE GRAINED MATERIALS II, 2002, pp 113-122 - 2ND INTERNATIONAL SYMPOSIUM ON ULTRAFINE GRAINED MATERIALS; SEATTLE, WASHINGTON. FEBRUARY 17-21, 2002

Warm severe plastic deformation realized via multiple forging can be used for formation of fine-grained microstructures with grain size of several hundred nanometers or less in titanium alloys. The smaller the grain size, the better the superplastic properties are resulted from lower temperature deformation. It has been established that the smallest grain size not only depends on the deformation temperature, but also the phase volume fraction, phase particle size and interparticle distance. The application of thermohydrogen treatment improves hot workability of titanium alloys at lower temperatures and permits to combine the deformation with metastable phases decomposition. Additional grain refinement is observed after hydrogen removal during vacuum annealing due to $\beta \rightarrow \alpha$ polymorphic transformation accompanied by recrystallization. The microstructure with a grain size of 25 nm was obtained in two-phase titanium alloy Ti-6.3Al-3.5Mo-1.7Zr (wt.%). This alloy is superplastic at temperature 550degreesC and strain rate $2 \times 10^{-4} s^{-1}$; $m=0.52$, relative elongation achieves 550%.

[61] HETEROGENEOUS MICROSTRUCTURAL EVOLUTION AND REACTIONS DURING REPEATED INTENSE DEFORMATION

RJ Hebert, JH Perepezko - ULTRAFINE GRAINED MATERIALS II, 2002, pp 141-150 - 2ND INTERNATIONAL SYMPOSIUM ON ULTRAFINE GRAINED MATERIALS; SEATTLE, WASHINGTON. FEBRUARY 17-21, 2002

With the recent improvements in severe plastic deformation and cold rolling, deformation processing has become an alternative to rapid-solidification processing for the synthesis of bulk metastable structures. The systematic study of the deformation patterns that develop during repeated rolling of multilayer based on a stacked elemental crystalline array including the analysis of interface areas and the evolution of interface roughening, offers an effective approach to the examination of the mechanism involved in deformation alloying, nanostructure synthesis and amorphization. A key-factor for deformation-induced phase transformations is the occurrence of atomic scale mixing effects at interfaces. The results from cold rolling experiments on Al-Pt and Al-Hf alloys demonstrate that the overall multilayer refinement represents an essential component for the prediction of amorphization by cold rolling in addition to the heat of mixing and a deformation enhanced mixing kinetics at individual interfaces. The effect of cold rolling on the devitrification behavior of Al-based glasses has also been addressed in a systematic study. For marginal glass formers, such as melt-spun Al88Y7Fe5, cold rolling induced a complete crystallization without annealing while for melt-spun Al85Ni10Ce5, a crystalline phase has not been observed under the same deformation conditions. The structural changes are observed to develop heterogeneously throughout the sample over several deformation cycles due to the distribution of microstructural size scales. These developments highlight the importance of the localized micro structural response to the overall sample deformation.

[60] HARDNESS AND MICROSTRUCTURE CHANGES IN SEVERELY DEFORMED AND RECRYSTALLIZED TANTALUM



KT Hartwig, SN Mathaudhu, HJ Maier, I Karaman - ULTRAFINE GRAINED MATERIALS II, 2002, pp 151-160 - 2ND INTERNATIONAL SYMPOSIUM ON ULTRAFINE GRAINED MATERIALS; SEATTLE, WASHINGTON. FEBRUARY 17-21, 2002

Vacuum arc remelted (VAR) pure tantalum was deformed at room temperature to a strain of 4.6 by multipass equal channel angular extrusion through a tool containing an abrupt 90 degree angle. The as-worked microstructure shows a progression from dense dislocation tangles containing primitive dislocation cells at a strain of 1.2, to the development of well defined sub-grains with dimensions on the order of hundreds of nanometers at a strain of 4.6. The recrystallization temperature decreases from about 1100degreesC to 900degreesC as the strain increases from 1.2 to 4.6. The recrystallized grain size for the material strained to 4.6 is under 15 microns. The Hall-Petch relationship is found to reasonably predict subgrain size from microhardness measurements in the most severely deformed material.

[59] GRAIN REFINEMENT AND PHASE TRANSFORMATIONS IN AL AND FE BASED ALLOYS DURING SEVERE PLASTIC DEFORMATION

Authors SV Dobatkin - ULTRAFINE GRAINED MATERIALS II, 2002, pp 183-192 - ND INTERNATIONAL SYMPOSIUM ON ULTRAFINE GRAINED MATERIALS; SEATTLE, WASHINGTON. FEBRUARY 17-21, 2002

The formation of ultrafine grained structure in metals and alloys during severe plastic deformation (SPD) at low temperature is well established. However, this deformation does not always lead to a nanocrystalline structure. The present work shows the possibility for additional refinement of microstructure down to nanocrystalline range (grain size less than 100 nm) in Al and Fe based alloys due to phase transformations during SPD by torsion under high pressure.

[58] METASTABLE NANOSTRUCTURED ALLOYS PROCESSED BY SEVERE PLASTIC DEFORMATION

V Stolyarov, R Valiev - ULTRAFINE GRAINED MATERIALS II, 2002, pp 209-218 - 2ND INTERNATIONAL SYMPOSIUM ON ULTRAFINE GRAINED MATERIALS; SEATTLE, WASHINGTON. FEBRUARY 17-21, 2002

The paper is devoted to comparative studies of microstructure and proper-ties of several multiphase alloys processed by two severe plastic deformation (SPD) methods - high pressure torsion (HPT) and equal channel angular pressing (ECAP). The microstructures processed at maximal strains possible presently for each of these methods are examined. Three alloys are considered here: (i) shape memory TiNi alloy; (ii) immiscible Al-Fe - alloy; (iii) multiphase hard magnetic Pr2Fe14B - alloy. It is shown that SPD of these alloys can lead both to strong microstructure refinement and also a formation of supersaturated solid solutions and amorphization of intermetallic phases, leading to narrostructured metastable states. During further heating the aging effects take place and the processed alloys demonstrate enhanced mechanical and physical properties. The efficiency of the applied SPD techniques for fabrication of metastable nanostructured states is considered and discussed.

[57] SYNTHESIS OF ND2Ti2O7/AL2O3 NANOCOMPOSITES BY SPARK-PLASMA-SINTERING AND HIGH-ENERGY BALL-MILLING

GD Zhan, J Kuntz, JL Wan, J Garay, AK Mukherjee - ULTRAFINE GRAINED MATERIALS II, 2002, pp 219-224 - 2ND INTERNATIONAL SYMPOSIUM ON ULTRAFINE GRAINED MATERIALS; SEATTLE, WASHINGTON. FEBRUARY 17-21, 2002

Spark-plasma-sintering (SPS) is a new process by which ceramics can be consolidated very rapidly to full density. Al2O3/9vol.-%-Nd2Ti2O7 nanocomposites with different crystal structure of starting alumina nanopowders have been successfully consolidated to > 98% of theoretical density by SPS at relatively low temperatures. High-energy ball milling can decrease the agglomeration of nanoscaled powders and lead to the gamma-Al2O3 to alpha-Al2O3 phase transformation during milling and then enhance the densification at lower temperatures. The Al2O3/9vol.-%-Nd2Ti2O7 nanocomposite through high-energy ball-milling of gamma-Al2O3 nanopowder could be consolidated by SPS at a temperature as low as 1050 degreesC and the resultant microstructure consists of ultrafine equiaxed grains with an average grain size of similar to 200 nm. However, the sintering temperature for Al2O3/9vol.-%-Nd2Ti2O7 nanocomposite without high-energy ball milling of alpha-Al2O3 nanopowder was 50 degreesC higher than that for composite by high-energy ball-milling and grain size was up to 281 nm. These results suggest that the combination of spark-plasma-sintering and high-energy ball milling could result in ultrafine matrix composites.

[56] PROPERTIES AND MICROSTRUCTURE OF ALUMINA-NIOBIUM NANOCOMPOSITES MADE BY NOVEL PROCESSING METHODS

JD Kuntz, JL Wan, GD Zhan, AK Mukherjee - ULTRAFINE GRAINED MATERIALS II, 2002, pp 225-233 - 2ND INTERNATIONAL SYMPOSIUM ON ULTRAFINE GRAINED MATERIALS; SEATTLE, WASHINGTON. FEBRUARY 17-21, 2002

Alumina-niobium nanocomposites have been fabricated using high-energy ball milling and electric field assisted sintering (EFAS) or high pressure sintering (HPS). The 10 volume percent niobium nanocomposites have fracture toughnesses greater than 6 MPa rootm with only a marginal decrease in hardness. This is nearly twice as tough as a pressureless sintered composite of the same composition reported in work by Garcia et al [1]. This increase in toughness can be attributed to the novel microstructure in the nanocomposites. The present study shows a metallic phase distribution of similar to 20 nm particles along with a continuous 3-4 nm layer at boundaries between alumina grains. This microstructure should lead to toughening by increasing ductility at the crack tip instead of the traditional ligament bridging in the crack wake which is typical of micron-scaled metallic-phase toughened ceramics.

[55] THE USE OF SPD FOR FABRICATION OF BULK NANOSTRUCTURED MATERIALS FROM BALL-MILLED POWDERS



GI Raab, NA Krasilnikov, E Thiele, R Klemm - ULTRAFINE GRAINED MATERIALS II, 2002, pp 245-251 - 2ND INTERNATIONAL SYMPOSIUM ON ULTRAFINE GRAINED MATERIALS; SEATTLE, WASHINGTON. FEBRUARY 17-21, 2002

The possibility of obtaining bulk billets with ultra fine grain structure by means of intensive plastic deformation (high pressure torsion, equal channel angular pressing, multiple-forging) from nanostructured ball-milled powders of copper, iron and nickel was investigated. Bulk billets with grain size less than 100 nm, density close to theoretical limit and high thermal stability and hardness were obtained.

[54] RECENT DEVELOPMENTS OF SPD PROCESSING FOR FABRICATION OF BULK NANOSTRUCTURED MATERIALS

R Valiev - ULTRAFINE GRAINED MATERIALS II, 2002, pp 313-322 - 2ND INTERNATIONAL SYMPOSIUM ON ULTRAFINE GRAINED MATERIALS; SEATTLE, WASHINGTON. FEBRUARY 17-21, 2002

During the last decade severe plastic deformation (SPD) techniques have been successfully used for fabrication of nanostructures in various bulk metallic materials. This paper presents several results from recent investigations of SPD materials focussing on two main objectives: circle modelling and experimental works on SPD techniques, aiming to produce homogeneous nanostructures in bulk large-size billets and to process hard-to-deform and low-ductile materials; circle establishing the influence of SPD (strain amount, temperature, applied pressure etc) and microstructure (types of grain boundaries, defect structures) parameters on enhancement of properties in as-processed materials.

[53] DEFORMATION MECHANISMS AT DIFFERENT GRAIN SIZES IN A CRYOGENICALLY BALL-MILLED AL-MG ALLOY

XZ Liao, JY Huang, YT Zhu, F Zhou, EJ Lavernia - ULTRAFINE GRAINED MATERIALS II, 2002, pp 323-330 - 2ND INTERNATIONAL SYMPOSIUM ON ULTRAFINE GRAINED MATERIALS; SEATTLE, WASHINGTON. FEBRUARY 17-21, 2002

An Al-7.5 wt. % Mg alloy was ball-milled in liquid N-2 for eight hours and its microstructures were investigated using transmission electron microscopy. Electron diffraction confirmed that the resulting powder is a supersaturated Al-Mg solid solution with a face-centered cubic structure. Three nanostructures with different grain size ranges and shapes were observed and the deformation mechanisms in these structures were found to be different. The reasons for the different deformation mechanisms were discussed.

[52] PROPERTIES AND NANOSTRUCTURES OF MATERIALS PROCESSED BY SPD TECHNIQUES

YT Zhu, JY Huang - ULTRAFINE GRAINED MATERIALS II, 2002, pp 331-340

Metallic materials usually exhibit higher strength but lower ductility after being plastically deformed by conventional techniques such as rolling, drawing and extrusion. In contrast, nanostructured metals and alloys processed by severe plastic deformation (SPD) have demonstrated both high strength and high ductility. This extraordinary mechanical behavior is attributed to the unique nanostructures generated by SPD processing. It demonstrates the possibility of tailoring the microstructures of metals and alloys by SPD to obtain superior mechanical properties. The SPD-generated nanostructures have many features related to deformation, including high dislocation densities, and high- and low-angle grain boundaries in equilibrium or non-equilibrium states. This paper reviews the mechanical properties and the defect structures of SPD-processed nanostructured materials.

[51] THE MICROSTRUCTURES AND COMPRESSIVE DEFORMATION BEHAVIORS OF NANOCRYSTALLINE Al-5 AT.% Ti COMPACTS PREPARED BY UHP-HP

KB Moon, KS Lee - ULTRAFINE GRAINED MATERIALS II, 2002, pp 439-446 - 2ND INTERNATIONAL SYMPOSIUM ON ULTRAFINE GRAINED MATERIALS; SEATTLE, WASHINGTON. FEBRUARY 17-21, 2002

Two types of bulk nanocrystalline Al-5 at.% Ti alloys have been prepared by ultra high pressure hot pressing (UHP-HP) method of mechanical alloyed powders and their mechanical properties have been investigated through compression tests at room temperature and high temperatures (300, 400 and 500degreesC). TiH₂ and Al₃Ti acted as effective dispersoids to prevent grain growth during the consolidation process. A full density was reached within 250 s at 120degreesC under 4.8 GPa, in specimen A120, and its microstructure was nanocomposite type with grain size less than 50 nm. The consolidation temperature of 120degreesC is about 300-400degreesC lower than the conventional temperatures. Abnormal grain growth was observed and some Al grains grew up over 500 nm in specimen A300 prepared at 300degreesC. Specimen A300 had a microstructure consisting of the large Al grains and nano-sized Al₃Ti dispersoids. The mechanical properties and the deformation behaviors of specimens A120 and A300 were very different in the compression tests. The compressive stress of specimen A120 was 1010 MPa and that of specimens A300 was 467 MPa at room temperature. The strength of specimen A120 decreased greatly with increasing ductilities at 300-500degreesC. Specimen A300 showed very small change in ductility and strength with temperature.

[50] STRUCTURE AND MECHANICAL BEHAVIOR OF THE AMG6 ALUMINUM ALLOY AFTER SEVERE PLASTIC DEFORMATION AND ANNEALING

MV Markushev, MY Murashkin - ULTRAFINE GRAINED MATERIALS II, 2002, pp 521-530 - 2ND INTERNATIONAL SYMPOSIUM ON ULTRAFINE GRAINED MATERIALS; SEATTLE, WASHINGTON. FEBRUARY 17-21, 2002

The microstructure of the AMg6 aluminum alloy subjected to severe plastic deformation (SPD) using equal-channel angular extrusion and subsequent annealing was studied by optical and transmission electron microscopy and X-ray diffraction. The features of transformation the deformation submicrocrystalline (SMC) structure into submicro- and microcrystalline (MC) grain structures upon annealing are considered. The phenomenology and the nature of recrystallization processes are



discussed. Mechanical behavior of the severely deformed and annealed alloy upon tension and bending at room temperature is investigated, The influence of SMC and MC structure on the alloy serrated flow, strength, hardness, ductility, toughness and crack resistance (crack formation and crack growth) is shown. The reasons for improved strength, low ductility and crack resistance of the alloy after SPD are analyzed.

[49] DEFECT CHARACTERIZATION OF EQUAL CHANNEL ANGULAR PRESSED CU BY SELECTIVE ANNEALING TREATMENT

E Schafler, A Dubravina, Z Kovacs - ULTRAFINE GRAINED MATERIALS II, 2002, pp 605-613- 2ND INTERNATIONAL SYMPOSIUM ON ULTRAFINE GRAINED MATERIALS; SEATTLE, WASHINGTON. FEBRUARY 17-21, 2002

Cu rods with a diameter of 20mm were deformed by Equal Channel Angular Pressing (ECA) by applying two different deformation paths, with (route B) and without (route A) rotation by 90degrees of the sample around its length axis between single ECA passes. Residual electrical resistivity (RER) measurements as well as X-ray Bragg Profile Analysis (XPA) have been performed, in addition to mechanical properties investigated. After the initial measurements of the as-deformed state, an isochronal annealing treatment has been carried out, and the three investigation methods were performed intermittently after certain temperatures of the annealing program, in order to differentiate between various deformation induced lattice defects and/or their arrangements. The results have been analyzed in terms of annealing of deformation induced dislocations and vacancy agglomerates. Compared to conventional cold work procedures, deformation by ECA achieves a strongly enhanced concentration of vacancy type defects as well as a markedly higher thermal stability of macroscopic strength.

[48] SIZE AND SHAPE OF NANO-GRAINS IN POLYCRYSTALS SUBJECTED TO SPD

KJ Kurzydowski - ULTRAFINE GRAINED MATERIALS II, 2002, pp 615-622 - 2ND INTERNATIONAL SYMPOSIUM ON ULTRAFINE GRAINED MATERIALS; SEATTLE, WASHINGTON. FEBRUARY 17-21, 2002

Severe plastic deformation (SPD) can be used to produce nano-polycrystalline materials. Grain size and shape in such polycrystals are far different from those observed in conventionally processed materials. This paper describes advanced methods of grain size quantification, which are based on principles of stereology, and computer aided image analysis. In particular the question of grain size homogeneity is discussed. Image analysis can also be used to quantify the shape of nano-grains. To this end, shape factors can be used which describe among other their elongation. Examples are given how the advanced quantitative methods can be used to characterize and model processes taking place during annealing of SPD processed materials.

[47] X-RAY ANALYSIS OF SPD NANOSTRUCTURED MATERIALS

V Alexandrov, AR Kilmametov, NA Enikeev, AA Dubravina, R Valiev - ULTRAFINE GRAINED MATERIALS II, 2002, pp 623-632 - 2ND INTERNATIONAL SYMPOSIUM ON ULTRAFINE GRAINED MATERIALS; SEATTLE, WASHINGTON. FEBRUARY 17-21, 2002

Recent results of the development and application of X-ray structural analysis for investigation of materials subjected to severe plastic deformation (SPD) by the techniques of high pressure torsion (HPT) and equal channel angular (ECA) pressing are presented in this paper. The microstructure evolution during SPD is analyzed using Cu subjected to the HPT as an example. Special emphasis is laid on the new data obtained when analyzing the microstructure of SPD-processed BCC (W) and HCP (Ti) metals in comparison with previously investigated FCC metals. The interpretation of the X-ray data and the complex defect structure of SPD Cu by computer modeling considering various assemblies of extrinsic grain boundary dislocations are also presented in the paper. The interrelation between the increased (especially large in grain-boundary areas) atomic displacements, the decreased Debye temperature and the observed increase in the grain boundary diffusion coefficient of Cu in nanostructured Ni processed by SPD is analyzed.

[46] MICROSTRUCTURAL EVOLUTION OF CRYOMILLED NANOCRYSTALLINE AL-TI-CU ALLOY

Z Lee, R Rodriguez, EJ Lavernia, SR Nutt - ULTRAFINE GRAINED MATERIALS II, 2002, pp 653-659 - 2ND INTERNATIONAL SYMPOSIUM ON ULTRAFINE GRAINED MATERIALS; SEATTLE, WASHINGTON. FEBRUARY 17-21, 2002

The microstructural evolution during processing of a nanocrystalline Al-Ti-Cu alloy was investigated using transmission and scanning electron microscopy. Grain refinement was achieved by cryomilling of elemental powders, and powders were consolidated by hot isostatic pressing (HIP) followed by extrusion to produce bulk nanocrystalline Al-Ti-Cu alloys. In an effort to enhance ductility and toughness, multi-scale structures were produced, which included nanocrystalline grains, elongated coarse-grains of pure Al, and intermediate grains. Pure aluminum grains were elongated along the extrusion direction and formed coarse-grain bands comprised of sub-grains. Nanocrystalline second phases were distributed in the intermediate grains and nanocrystalline regions. The distribution and identity of these phases were determined by analytical and high-resolution microscopy. Examination of bulk tensile fracture specimens revealed unusual failure mechanisms and interactions between ductile coarse-grains and nanocrystalline regions.

[45] MICROSTRUCTURE AND MECHANICAL PROPERTIES OF NON-HEAT TREATABLE ALUMINUM ALLOYS PRODUCED BY ACCUMULATIVE ROLL BONDING PROCESS

SB Kang, HW Kim, CY Lim, ZP Xing - ULTRAFINE GRAINED MATERIALS II, 2002, pp 661-667 - 2ND INTERNATIONAL SYMPOSIUM ON ULTRAFINE GRAINED MATERIALS; SEATTLE, WASHINGTON. FEBRUARY 17-21, 2002

Non-heat treatable aluminum alloys were subjected to Accumulative Roll Bonding(ARB) process for grain refinement to submicrometer levels. Microstructures after ARB and subsequent annealing treatments were observed using transmission



electron microscope. Microhardness and tensile tests were conducted on ARBed alloys to examine the strength and elongation at ambient temperature. The microhardness and strengths of ARBed alloys are considerably higher than those of commercially available respective alloys. Meanwhile, elongation is greatly decreased after the first cycle and shows no further decrease with increasing the number of cycles. In the range of 2 to 5 cycles in ARB process, AA1100 alloys exhibited strain hardening behavior, but AA8011 alloy demonstrated to be transferred to strain softening behavior. The former is attributed to the dominant contribution of strain hardening, but the latter to the precipitation of Si and recovery during ARB process.

[44] SYNTHESIS OF FE-CU NANOPARTICLES BY MECHANOCHEMICAL PROCESSING USING A BALL MILL

Todaka Y. McCormick PG. Tsuchiya K. Umemoto M. - Materials Transactions. 43(4):667-673, 2002

Fe-Cu nanoparticles were synthesized by mechanochemical processing, which utilizes the reaction of FeCl₃ and CuCl₂ with Na during ball milling. Morphologies, structures and magnetic properties of the synthesized nanoparticles were investigated. The crystallite size and mean particle size of the washed nanoparticles after 84 h milling were approximately 9 nm and 50 nm, respectively. During ball milling, the crystallite size remained constant at approximately 14 nm, while the particle size increased. The elemental mapping images of Fe and Cu by imaging filter revealed that the synthesized nanoparticles were a solid solution of the Fe-Cu system. It was seen that numerous hexagonal plates of Fe(OH)₂ exist in the washed nanoparticles. The plate., had a few nm in thickness and a few hundred, nm in diameter. The coercivity of the synthesized Fe-Cu nanoparticles after 48 h milling was as high as 33.5 kA/m, This is because the particle is close to the critical size for a single magnetic domain of alpha-Fe

[43] STRUCTURE AND TRANSPORT PROPERTIES OF (Bi_{1-x}Sb_x)₂Te₃ THERMOELECTRIC MATERIALS PREPARED BY MECHANICAL ALLOYING AND PULSE DISCHARGE SINTERING

Xue-Dong L. Park YH. - Materials Transactions. 43(4):681-687, 2002

Mechanical alloying followed by pulse discharge sintering (MA-PDS) has been employed to develop the bulk (Bi_{1-x}Sb_x)₂Te₃ thermoelectric materials with various Sb alloying contents. Substitutional solid solutions of (Bi_{1-x}Sb_x)₂Te₃ are formed in the whole Sb content range by MA-PDS process. The sintered compacts are dense and have refined microstructures. Systematic investigations on the electrical, thermal and thermoelectric properties reveal that the transport properties of the obtained (Bi_{1-x}Sb_x)₂Te₃ samples are quite sensitive to the Sb alloying content. At room temperature, the samples with $x < 0.57$ exhibit n-type semi-conduction. However, at $x > 0.57$, the samples become p-type. The pure constituents of Bi₂Te₃ and Sb₂Te₃ as well as the Sb-poor. n-type samples exhibit the room-temperature figure of merit of the order of 1.0×10^{-3} K⁻¹. High values of figure of merit have been obtained in the Sb-rich, p-type samples. The maximum value of 3.35×10^{-3} K⁻¹ is attained at $x = 0.80$, which corresponds to the carrier concentration and Hall mobility of 1.95×10^{19} cm⁻³ and 207 cm²/Vs, respectively.

[42] NEW V₄₅Zr₂₀Ni₂₀Cu₁₀Al_{2.5}Pd_{2.5} GLASSY ALLOY POWDER WITH WIDE SUPERCOOLED LIQUID REGION

El-Eskandarany MS. Inoue A. - Materials Transactions. 43(4):770-772, 2002

New multicomponent V-based glassy alloy powder has been synthesized by mechanical alloying a mixture of elemental V₄₅Zr₂₀Ni₂₀Cu₁₀Al_{2.5}Pd_{2.5} powder at room temperature, using a low energy ball mill. The glassy powder of the end-product (720ks) in which its glass transition temperature (T_g) lies at a rather high temperature (745 K). Crystallized through two sharp exothermic reactions at 843K and 919K, respectively. The total enthalpy change of crystallization ($\Delta H(x)$) is -1.78 kJ/mol. The supercooled liquid region before crystallization, $\Delta T(x)$ of the synthesized glassy powder shows a high value (98 K) for a metallic glassy system. The reduced glass transition temperature (ratio between T_g and liquidus temperatures. T_l (T_g/T_l)) was found to be 0.52.

[41] MECHANICAL ALLOYING AND MICROSTRUCTURE OF A Nb-20% V-15% Al ALLOY

Dymek S. Lorent A. Wrobel M. Dollar A. - Materials Characterization. 47(5):375-381, 2001

A niobium-based alloy with 20% V-15% Al (at.%) was synthesized by mechanical alloying of elemental powders. During milling, the splitting of Nb X-ray peaks into two components was observed. Each component was found to correspond to a niobium solid solution (Nb-I and Nb-II) with a different lattice parameter. The intensities of Nb-I peaks on X-ray diffraction patterns decreased with the milling time and disappeared completely after 180 h of milling while the intensities of Nb-II peaks gradually increased. The powders were hot pressed and micro structural and phase analyses of the consolidated material were carried out. The microstructure consisted of Nb solid solution, Nb₃Al-base intermetallic with the A15 crystal structure and dispersoid Al₂O₃. Also an unexpected, detrimental, Nb₂Al-base sigma phase was found. The volume fraction of the sigma phase depended on the temperature of consolidation.

[40] THE DIFFICULTY OF ISOLATING GRAIN BOUNDARY COMPONENTS IN THE MOSSBAUER SPECTRA OF BALL-MILLED MATERIALS: IRON AND SILVER-IRON ALLOYS

Rixecker G. - Solid State Communications. 122(6):299-302, 2002.

Although Fe-57 Mossbauer spectroscopy is well known to be a particularly suitable method for the characterization of nanostructured solids, attempts to isolate spectral features that can be associated with grain boundaries encounter unexpected difficulties in the case of ball-milled or mechanically alloyed metallic materials. In this paper, the examples of ball-milled alpha-Fe and Ag(Fe) alloys prepared by mechanical alloying are discussed in order to show what kinds of structural information can still be extracted from the hyperfine parameters, in spite of the presence of large concentrations of lattice defects and long-range magnetic interactions.

539] STRUCTURAL CHANGES IN CELLULOSE-CONTAINING MATERIALS IN THE COURSE OF MECHANICAL ACTIVATION



Altunina LK. Gossen LP. Tikhonova LD. Yarmukhametova EG. - Russian Journal of Applied Chemistry. 75(1):166-167, 2002

[38] STRUCTURE AND HARD MAGNETIC PROPERTIES OF BARIUM HEXAFERRITE WITH AND WITHOUT LA2O3 PREPARED BY BALL MILLING

Babu V. Padaikathan P. - Journal of Magnetism & Magnetic Materials. 241(1):85-88, 2002

The barium hexaferrites have been prepared by ball milling of a BaO₂ and Fe₂O₃ mixture followed by thermal heat treatments. The structure and magnetic properties were investigated using X-ray diffraction, scanning electron microscopy and vibrating sample magnetometer techniques. The effect of grain refiner was also studied and it was found that the hard magnetic properties were improved significantly. The sintered product of barium hexaferrite powders prepared from ball milling has higher coercive force than that of other barium hexaferrite made from oxide/carbonate

[37] NANOCRYSTALLINE SM-12.5(CO,ZR)(87.5) MAGNETS: SYNTHESIS AND MAGNETIC PROPERTIES

Tang H. Liu Y. Sellmyer DJ. - Journal of Magnetism & Magnetic Materials. 241(2-3):345-356, 2002

Isotropic nanostructured Sm_{12.5}Co_{87.5-x}Zr_x (0 less than or equal to x less than or equal to 5) magnets with the TbCu₇ structure have been synthesized by mechanical milling and subsequent annealing. The remanence is found much enhanced in the nanostructured Sm(Co,Zr)₇, magnets with a grain size of 10-20 nm. A suitable amount of Zr addition (x = 1, 2) is helpful to improve hard magnetic properties, including high remanence ratio of 0.71, coercivities up to 21 kOe, and energy products up to 13.0 MGOe, and hard magnetic properties are sensitive to the milling time and the annealing temperature. The dependence of hard magnetic properties on milling time may be correlated to the refinement of grain size and the intergranular structure induced during the milling and the subsequent annealing. The enhancement of remanence due to a small amount of Zr in this series of Sm_{12.5}Co_{87.5}Zr_x powders results from the enhanced exchange coupling between the nanosized grains. The hard magnetic properties are discussed with correlation to the milling process and in terms of the nanostructure developed in these materials

[36] SYNTHESIS OF AMORPHOUS AND QUASICRYSTAL PHASES BY MECHANICAL ALLOYING OF Ti45ZR38Ni17 POWDER MIXTURES, AND THEIR HYDROGENATION

Takasaki A. Han CH. Furuya Y. Kelton KF. - Philosophical Magazine Letters. 82(6):353-361, 2002

Mechanical alloying of Ti₄₅Zr₃₈Ni₁₇ powder mixture forms an amorphous phase, but subsequent annealing causes the formation of an icosahedral (i) phase. The maximum hydrogen concentration that can be loaded at 573 K at a hydrogen pressure of 3.8 MPa is the same ([H]/[M] approximate to 1.5) for the amorphous and i-phase powders. With hydrogenation, the i-phase is almost stable, forming no hydrides, whereas the amorphous phase transforms to a fcc hydride. The activation energy for hydrogen desorption for the i-phase is about 127 kJ mol⁻¹, which is lower than that for the amorphous phase, suggesting that the i-phase powder may have better properties for hydrogen-storage applications.

[35] MICROSTRUCTURE AND MAGNETIC PROPERTIES OF TWO-PHASE EXCHANGE-COUPLED SMC05/SM-2(CO, M)(17) (M=FE, ZR, CU) NANOCOMPOSITES

Yan A. Bollero A. Gutfleisch O. Muller KH. - Journal of Physics D-Applied Physics. 35(9):835-838, 2002

The microstructure and magnetic properties of nanocomposite SmCo₅/Sm₂Co₁₇, Sm-2(CoFe)(17), Sm-2(CoFeZr)(17), Sm-2(CoFeCuZr)(17) powders prepared by intensive milling and subsequent annealing have been studied systematically. A small amount of Fe substitution for Co was found to slightly increase the saturation magnetization and maximum energy product of SmCo₅/Sm₂Co₁₇ powders. The coercivity is enhanced by Zr and especially Cu. The highest maximum energy product of 11.5 MGOe and coercivity of 2.4 T were obtained in 40% SmCo₅ + 60% Sm-2(Co_{0.9}Fe_{0.1})(17) and 40% SmCo₅ + 60% Sm-2(Co_{0.74}Fe_{0.1}Cu_{0.12}Zr_{0.04})(17) powders, respectively. All the hysteresis loops showed a smooth demagnetization curve. A few percent of Zr substitution proved to be effective in refining, the microstructure and Cu increased the volume fraction of SmCo₅ phase in the mixture of two phase powders. Measurements of delta M and recoil curves indicated that intergranular exchange coupling and exchange spring magnet behaviour were observed in the above samples

[34] MECHANOCHEMICAL TREATMENT OF HIGH SILICA BAUXITE WITH LIME

McCormick PG. Picaro T. Smith PAI. - Minerals Engineering. 15(4):211-214, 2002

The mechanochemical processing of bauxite with lime has been investigated. The milling of bauxite and lime mixtures in an attritor mill resulted in the formation of an iron rich hydrogarnet phase in bauxite/lime slurries containing greater than 9% CaO. Up to 90% of the quartz contained in the bauxite was found to have been removed by the hydrogarnet reaction. The hydrogarnet compound was stable during the high temperature alumina extraction step, resulting in a 30% drop in caustic soda consumption. Alumina extraction was not influenced by the mechanochemical processing.

[33] INVESTIGATION OF THE EFFECTS OF CHROME BALL CHARGE ON SLURRY RHEOLOGY AND MILLING PERFORMANCE

Shi FN. - Minerals Engineering. 15(4):297-299, 2002

Ten surveys of the ball milling circuit at the Mt Isa Mines (MIM) Copper Concentrator were conducted aiming to identify any changes in slurry rheology caused by the use of chrome balls charge, and the associated effect on grinding performance. Slurry rheology was measured using an on-line viscometer. The data were mass balanced and analysed with statistical tools. Comparison of the rheogram demonstrated that slurry density and fines content affected slurry rheology significantly, while the effect of the chrome ball charge being negligible. Statistical analysis showed the effects of mill throughput and cyclone efficiency on the Grinding Index (a term describing the overall breakage). There was no difference in the Grinding Index between using the chrome ball charge and the ordinary steel ball charge

[32] AUTOGENOUS MILLING - EFFECTS ON FLOTATION OF MT. LYELL COPPER ORE BROUYAGE AUTOGENE



Clarke NC. Henley KJ. Wu T. le Page M. - CIM Bulletin. 95(1060):75-81, 2002

In 1994, Gold Mines of Australia acquired the rights to the historic Mt. Lyell copper mine, and commenced a feasibility study into expanding production. The ore was shown in laboratory tests to be amenable to single-stage, fully autogenous grinding (FAG) of underground primary crushed ore, and this route offered the maximum cost savings. Flotation performance was expected to be enhanced, However, batch flotation tests done on pilot-scale autogenously ground ore showed an unexplainable recovery loss of between 1% and 3% Cu. A second pilot plant trial was set up, with the ground product feeding banks of continuous flotation cells, Single-stage FA G, FAG/pebble and FAG/ball circuits were tested. Results confirmed a loss of about 3% recovery with single-stage FAG milling. The FAG/ball circuit eliminated the loss. Analyses showed that autogenous milling probably produced more chalcopyrite/pyrite composites and that recovery of these composites was lower than with ball milling. In size fractions below about 53 microns, where chalcopyrite is well liberated, copper recovery was less affected by milling method and flotation selectivity. It was concluded that for the Mt. Lyell situation, a FAG/ball circuit was preferable to single-stage FAG

[31] MICROSTRUCTURE AND EVOLUTION OF MECHANICALLY-INDUCED ULTRAFINE GRAIN IN SURFACE LAYER OF AL-ALLOY SUBJECTED TO USSP

Wu X. Tao N. Hong Y. Xu B. Lu J. Lu K. - Acta Materialia. 50(8):2075-2084, 2002

Experiments were conducted to investigate the ultrafine-grained (UFG) microstructures in the surface layer of an aluminum alloy 7075 heavily worked by ultrasonic shot peening. Conventional and high-resolution electron microscopy was performed at various depth, of the deformed layer. Results showed that UFG structures were introduced into the surface layer of 62 μm thick. With increasing strain, the various microstructural features, e.g., the dislocation emission source, elongated microbands, dislocation cells, dislocation cell blocks, equiaxed submicro-, and nano-crystal grains etc.. were successively produced. The grain subdivision into the subgrains was found to be the main mechanism responsible for grain refinement. The simultaneous evolution of high boundary misorientations was ascribed to the subgrain boundary rotation for accommodating further strains. Formed inicrostructures were highly nonequilibrium.

[30] MECHANICALLY-AMPLIFIED PLASMA PROCESSING FOR DRUG ENGINEERING

Kuzuya M. Sasai Y. Mouri M. Kondo S.- Thin Solid Films. 407(1-2):144-150, 2002

We report here special features of mechanochemical reaction of plasma-irradiated polyethylene (PE), low density PE (LDPE) and high density PE (HDPE). A dangling bond site (DBS) of three component radicals formed on a PE surface by argon plasma-irradiation disappears rapidly by mechanical vibration with a Teflon twin-shell blender, causing successively solid state radical recombination reaction. When mechanical vibration of plasma-irradiated PE is similarly conducted together with a powdered drug, the sustained release powders are obtained due to trapping of drugs into the powder matrix formed by mechanochemical solid state recombination of plasma-induced PE surface radicals.

[29] FORMATION OF NANOCRYSTALLINE STRUCTURE IN STEELS BY BALL DROP TEST

Umamoto M. Huang B. Tsuchiya K. Suzuki N. - Scripta Materialia. 46(5):383-388, 2002

Formation of nanocrystalline structure, in a eutectoid steel by severe plastic deformation has been studied by a 'ball drop test'. The microstructures and hardness similar to those of nanocrystalline structures produced by ball milling have been obtained near the surface of specimens. The high strain rate of around $10(4) \text{ s}^{-1}$ is proposed to be an essential condition to produce nanostructure by deformation.

[28] FORMATION OF NICKEL ALUMINIDES UPON MECHANICAL ALLOYING

Portnoi VK. Blinov AM. Tomilin IA. Kuznetsov VN. Kulik T. - Physics of Metals & Metallography (English Translation of Fizika Metallov i Metallovedenie). 93(4):331-337, 2002

X-ray diffraction (XRD) and differential scanning calorimetry (DSC) were used to study the structure and thermal stability of Ni-Al alloys produced by mechanical alloying. In the concentration range of 40-61 at. % Ni, the following three phases were shown to form: at 40-61 at. % Ni, a B2 phase (CsCl type); at 65-85 at. % Ni, a nanocrystalline solid solution Ni(Al); and at a composition of Ni_{62.5}Al_{37.5}, a nanocrystalline phase that could be interpreted as an ordered tetragonal phase L1(0). As a final product of mechanical alloying, singlephase structures with a minimum Gibbs energy of the competing phases are always formed, which indicates the key role of thermodynamic factor for phase formation upon mechanosynthesis

[27] IMPROVING THE ENERGY PRODUCT OF HARD MAGNETIC MATERIALS

Sort J. Surinach S. Munoz JS. Baro MD. Nogues J. Chouteau G. Skumryev V. Hadjipanayis GC. - Physical Review B. 6517(17):4420, 2002

A route toward enhancing the energy product (BH)(max) of permanent magnetic materials, at room temperature, based on ferromagnetic-(FM-) antiferromagnetic (AFM) exchange interactions has been developed. The exchange coupling, which is induced by ball milling hard magnetic SmCo₅ with AFM NiO powders, results in an enhancement of coercivity H-C and squareness ratio M-R/M-S (remnant-saturation magnetizations), which depends on the FM:AFM ratio and the processing conditions. However, the presence of the AFM in the composite results also in a competing effect, i.e., reduction of the overall saturation magnetization, which decreases (BH)(max). Nevertheless, it has been found that after an optimization of the FM:AFM ratio and the milling conditions it is possible to achieve an improvement of (BH)(max).

[26] COMPARISON OF DIFFERENT ALUMINA POWDERS FOR THE AQUEOUS PROCESSING AND PRESSURELESS SINTERING OF AL₂O₃-SiC NANOCOMPOSITES

Baron B. Kumar CS. Le Gonidec G. Hampshire S. - Journal of the European Ceramic Society. 22(9-10):1543-1552, 2002

Four different alumina powders, from European and Japanese sources having similar particle size (350-700 nm) were used for the fabrication of nanocomposites. They were compared in terms of green properties, sintering behaviour, microstructure



and mechanical properties. The processing route used (attrition milling and freeze-drying) leads to a reduction in green density of the processed aluminas and composites compared to the as-received alumina. All powders had similar green properties except one, which contained a binder from the manufacturer. The presence of this binder led to the formation of hard agglomerates. In this case the pressing did not eliminate, totally, the inter-agglomerate pores, leading to an incomplete sintering. Calcining the powder to remove the binder resulted in similar pressing and sintering behaviour to the other powders and densities > 99% were achieved at 1750 degreesC by pressureless sintering. All the composites exhibited similar microstructures (matrix grain size similar to 3 µm) and elastic properties, hardness and fracture toughness. A finer matrix microstructure could be obtained with one of the European powders which achieved similar to 99% density at 1700 degreesC. The presence of 5 vol.% SiC resulted in a mean grain size of similar to 2 µm for the alumina matrix compared with 13.9 µm for a monolithic alumina prepared under identical conditions

[25] INFLUENCE OF THE DEAGGLOMERATION PROCEDURE ON AQUEOUS DISPERSION, SLIP CASTING AND SINTERING OF Si3N4-BASED CERAMICS

Oliveira MILL. Chen KX. Ferreira JMF. - Journal of the European Ceramic Society. 22(9-10):1601-1607, 2002

The influence of the deagglomeration procedure on the rheological behaviour of Si3N4-based aqueous suspensions, the slip casting performance and the final properties after sintering were investigated. Ball milling and planetary milling performed deagglomeration of powders. The experimental results showed that the time required to obtain the same degree of deagglomeration was considerably shorter in the case of planetary milling. The decrease in viscosity during the milling procedure enabled well dispersed and relatively high-concentrated (55-vol.%), suspensions to be obtained by adding successive 5-vol.% increments of solids to an initial 45-vol.% suspension. The time required to achieve complete deagglomeration of the starting suspension or after adding each 5-vol.% solids' increments was of 4 h for planetary mill and varied from 24 to 48 h for ball mill, with increasing solids loading, reaching total deagglomeration times of 12 and 96 h, respectively. The results have shown that, for a given solids volume fraction, both the degree of deagglomeration and the time required to achieve it, i.e. the duration of the contact between the powders and the dispersing aqueous solution, are key factors for achieving dense and homogeneous green microstructures, and for improving the densification behaviour and final properties of sintered bodies

[24] REMARKABLE IMPROVEMENT OF COERCIVITY IN NANOSTRUCTURED TbMn6Sn6 POWDERS PRODUCED BY MECHANICAL MILLING

Zhao P. Zhang J. Zhang SY. Zhang HW. Sun ZG. Shen BG. - Journal of Applied Physics. 91(10 Part 3):7860-7862, 2002
Nanostructured TbMn6Sn6 powders have been obtained by mechanical milling cast alloys and subsequent annealing at different temperatures. Powders were synthesized by milling for 1, 3, 5, and 8 h, and annealed from 573 to 773 K for 20 min. For the TbMn6Sn6 powders milled for 3 h and annealed at 723 K for 20 min, the highest coercivity, about 12 kOe is achieved at room temperature. The powder's coercivities increase with decreasing temperature. The average grain size of the optimum powders is about 14 nm according to the x-ray diffraction patterns by using the Sherrer's formula. The smooth and nearly square demagnetization curve suggests a very fine and uniform grain size, and the enhanced M-r/M-s (similar to 0.6) ratio indicates the existence of intergrain exchange interactions among the TbMn6Sn6 grains. The observed remarkable improvement of magnetic hardening in powders is believed to arise from the single-domain size in the powders

[23] PHASE EVOLUTION, STRUCTURE, AND MAGNETIC PROPERTIES OF Nd8.4Fe86Mo1.1B4.5 NANOCOMPOSITE MAGNETS

Cui BZ. Sui YC. Sun XK. Xiong LY. O'Shea MJ. Zhang ZD. - Journal of Applied Physics. 91(10 Part 3):7881-7883, 2002

The relationship between the phase evolution, nanostructure, exchange coupling, and magnetic properties of Nd8.4Fe86Mo1.1B4.5 magnets prepared by melt spinning (MS) and mechanical milling (MM) are studied by x-ray diffraction, Mossbauer spectroscopy, positron lifetime measurements, high resolution transmission electron microscopy observations, and magnetization measurements. The maximum magnetic energy product (BH)(max) for MM magnets annealed at 650 degreesC or below are notably higher than those of the corresponding MS samples due to the larger amount of the Nd(Fe,Mo)(7) present in the MS samples.

[22] HIGH-PERFORMANCE NANOCRYSTALLINE PRFeB-BASED MAGNETS PRODUCED BY INTENSIVE MILLING

Bollero A. Gutfleisch O. Muller KH. Schultz L. Drazic G. - Journal of Applied Physics. 91(10 Part 3):8159-8161, 2002

The intensive milling technique has been used to produce nanocrystalline isotropic PrFeB-based powders. Highly coercive powders were obtained using the base composition R16T76B8 (R: rare earth, T: transition metal) with Dy and Zr additions achieving a high value of $\mu(0)H(c)=2.66$ T for Pr15Dy1Fe75.9B8Zr0.1. The influence of Dy and Zr, and the substitution of Pr by Nd, on the microstructural and magnetic properties have been studied. Reduction of the R content, increase of the T content, and the presence of Co in the starting composition gave rise to an improved performance with values of $J(r)=0.92$ T, $\mu(0)H(c)=1.25$ T, and $(BH)(max)=140$ kJ/m³ for Pr9Nd3Dy1Fe72Co8B6.9Zr0.1, originating from a very fine microstructure with a mean grain size of 20 nm. Furthermore, intensive milling has been shown to be a very versatile technique to produce high-performance nanocomposite magnets by blending this latter alloy with different fractions of soft magnetic α -Fe (x=5-35 wt % Fe). A 25 wt % Fe addition resulted in an optimum combination of magnetic properties with a very high $(BH)(max)$ value of 178 kJ/m³ due to an effective exchange coupling between the hard- and soft-magnetic phases. A Curie temperature of about 370 degreesC was observed for this magnet. Demagnetization recoil loops of both single-phase and nanocomposite magnets showed clear differences with relatively open minor loops in the case of the latter



due to the exchange-spring mechanism present. ΔJ plots for the nanocomposite magnets showed a negative deviation of the demagnetizing remanence from the Wohlfarth model, indicative of exchange-coupling interactions being dominant

[21] MECHANICALLY MILLED NANOSTRUCTURED (SM,PR)(12.5)CO85.5ZR2 MAGNETS WITH TBCU7 STRUCTURE

Tang H. Zhou J. Sellmyer DJ. - Journal of Applied Physics. 91(10 Part 3):8162-8164, 2002

Nanostructured (Sm,Pr)(Co,Zr) magnets with the TbCu7-type structure have been synthesized by mechanically milling (Sm_{1-x}Pr_x)(12.5)Co_{85.5}Zr₂ alloys (0 less than or equal to x less than or equal to 0.8) followed by appropriate annealing. Magnetic properties, structure, and microstructure have been investigated. It is found that single-phase (Sm,Pr)(Co,Zr)₇ magnets with the TbCu7 structure and with nanoscale grain size (14-19 nm) form in the whole composition range. Intrinsic coercivity H_{ci} decreases from 20.7 to 5.6 kOe with increasing Pr content from 0 to 0.8, while energy products (BH)_{max} shows an optimum value of 12.6 MGOe (H_{ci} of 17.9 kOe) at x=0.2 due to increase in remanent magnetization. Remanence ratio is observed in the range of 0.64-0.69, with an optimum value of similar to 0.69 at x=0.3. The high coercivity is ascribed to the formation of nanocrystalline (Sm,Pr)(Co,Zr)₇ phase of the TbCu7-type structure with large anisotropy, and the remanence enhancement may result from the exchange-coupling interactions between nanosized (Sm,Pr)(Co,Zr)₇ grains. The magnetic properties can be understood in term of the anisotropy of the magnetically hard phase and the interactions between the nanosized grains

[20] PHASE TRANSITION AND MAGNETOTRANSPORT PROPERTIES OF BALL-MILLED HALF-METALLIC CrO2

Wang KY. Spinu L. He J. Zhou W. Wang W. Tang J. - Journal of Applied Physics. 91(10 Part 3):8204-8206, 2002

Small CrO₂ particles with mean diameters ranging from 11 to 25 nm have been prepared by ball milling. X-ray diffraction studies show a continuous lattice expansion with increasing milling time. A phase transition to Cr₂O₃ also occurs with a sudden increase in the amount of Cr₂O₃ found between 5 and 8 h of milling. The decreases of low-field magnetoresistance with increasing milling time is correlated to the expansion of the lattice parameters of CrO₂, which probably leads to the reduction in its spin polarization. High-field magnetoresistance increases with the milling possibly due to the enhanced mixed valence of the chromium, which supports the double exchange model.

[19] MAGNETIC NANOGRANULARITY AND SPIN-GLASS BEHAVIOR IN MECHANICALLY ALLOYED FE35AL50B15

De Toro JA. de la Torre MAL. Riveiro JM. Bland J. Goff JP. Thomas MF. - Journal of Applied Physics. 91(10 Part 3):8396-8398, 2002

The combination of Mossbauer spectroscopy and magnetization measurements is demonstrated to be efficient in the determination of the phases responsible for the glassy magnetic dynamics of the highly disordered alloy Fe₃₅Al₅₀B₁₅ produced by mechanical alloying. Its spin-glass behavior is apparent from the FC-ZFC magnetization irreversibility, the thermoremanent magnetization, and the slightly frequency-dependent peak in the temperature dependence of the ac susceptibility. Furthermore, the maximum at T(p) approximate to 22 K in the real component of the ac susceptibility is accompanied by a sudden onset of the imaginary component. The fit of the frequency dependence of T-p to the critical slowing down law yielded a critical exponent $\nu = 8 \pm 1$. However, Mossbauer spectroscopy uncovered the existence of two different Fe environments, of which only the minor one freezes at low temperatures into a distribution of magnetic sextets. This result, combined with the superparamagnetic behavior detected at moderate fields, allowed us to ascribe the spin glass behavior to the presence of interacting, very fine, ferromagnetic clusters (estimated size similar to 1 nm). The origin of this nanoscale compositional heterogeneity is argued to be related to regions of partially unalloyed Fe. The conclusions are contrasted with other mechanically alloyed systems with reported spin glass behavior or compositional inhomogeneity.

[18] MAGNETIC PROPERTIES OF THE LOW-TEMPERATURE PHASE OF MNBI

Saha S. Obermyer RT. Zande BJ. Chandhok VK. Simizu S. Sankar SG. Horton JA. - Journal of Applied Physics. 91(10 Part 3):8525-8527, 2002

MnBi forms peritectically at similar to 450 degreesC. Preparation of MnBi employing conventional techniques such as arc melting and induction melting results in the segregation of manganese. In order to avoid this segregation, we followed the procedure recommended by Guo [X. Guo, A. Zaluska, Z. Altounian, and J. O. Strom-Olsen, J. Mater. Res. 5, 2646 (1990)] and prepared a low-temperature phase of MnBi by melt spinning, followed by heat treatment. Fine powder of MnBi was prepared by ball milling the melt-spun ribbons for various lengths of time. Magnetic properties of these powders were determined. In particular, the temperature dependent coercivity was studied from room temperature to 360 degreesC for the powders ball milled for 2 and 10 h. The coercivity is found to increase with the increase in temperature reaching a maximum of 25.8 kOe at 280 degreesC and then decrease as the temperature is increased further. We also found that a peak in coercivity is observed for the samples milled for 10 h. MnBi shows a first-order transition to a paramagnetic phase at 360 degreesC. In an attempt to increase this transition temperature, an alloy of composition Mn_{0.75}Ni_{0.25}Bi_{0.5}Sb_{0.5} was made by induction melting. The transition temperature increases from 360 degreesC for MnBi to 400 degreesC for Mn_{0.75}Ni_{0.25}Bi_{0.5}Sb_{0.5}

[17] MAGNETIC AND MECHANICAL PROPERTIES OF (FE, CO)-PT BULK ALLOYS PREPARED THROUGH VARIOUS PROCESSING TECHNIQUES

Saha S. Thong CJ. Huang MQ. Obermyer RT. Zande BJ. Chandhok VK. Simizu S. Sankar SG. - Journal of Applied Physics. 91(10 Part 3):8810-8812, 2002

Magnetic and mechanical properties of Fe₆₀Pt₄₀, Fe_{60.5}Pt_{39.5} and (Fe_{1-x}Co_x)(60.5)Pt-39.5 bulk alloys prepared by a number of processing techniques have been examined. Processing techniques include induction melting, mechanical milling (at similar to 77 K), hot and cold work, and melt extraction. Magnetic properties were determined in the temperature range from 300 to 1100 K using a vibrating sample magnetometer. Melt extracted Fe_{60.5}Pt_{39.5} sample appeared to be fully dense



and the magnetic properties found to be $4\pi M(s)$ (at 1.5 T) similar to 1.08 T, $H(c)$ similar to 270.6 kA/m, and $(BH)_{max}$ similar to 55.7 kJ/m³. Freezer milled Fe_{60.5}Pt_{39.5} sample (loose powder) showed a saturation induction of 1.33 T, and coercivity of 270.6 kA/m at room temperature. Curie temperature for this sample is found to be 450 degreesC. For the Fe_{45.37}Co_{15.13}Pt_{39.5} (loose powder) sample, coercivity increases to 318 kA/m and the Curie temperature increases to 540 degreesC. Tensile strength was measured for selected samples. It is found that Fe-Pt and (Fe,Co)-Pt magnets are about 5-10 times mechanically stronger than the rare earth based permanent magnets. Preliminary examination of the structural and magnetic properties of these alloys indicates that the (Fe,Co)-Pt bulk alloys are an excellent system to explore exchange coupling mechanism in permanent magnets.

[16] TEXTURAL CHARACTERISATION OF IRON-PROMOTED RANEY NICKEL CATALYSTS SYNTHESISED BY MECHANICAL ALLOYING

Salmones J. Zeifert B. Cabanas-Moreno JG. Aguilar-Rios G. Rojas F. Ramirez-Cuesta A. - Adsorption Science & Technology. 19(10):871-885, 2001.

Mesoporous binary Al_xNi_y and ternary Al_xNi_yFe_z Raney-type catalysts were synthesised by a two-step procedure involving two main processes, i.e. (i) mechanical metal alloying and (ii) alkaline aluminium leaching. Pure metallic powders of Al, Ni and Fe (if required) were first mechanically alloyed in an attrition mill and then subjected to KOH leaching to selectively remove part of the aluminium atoms. After a slow drying process, a fine, nanostructured slit-shaped material was obtained. Substrate characterisation involved studies by atomic absorption (AA), X-ray diffraction (XRD), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDXS) in the SEM and nitrogen physisorption. An intermetallic beta- or B₂-(AlNi) phase with a metastable bcc crystalline structure was formed as a non-equilibrium phase after the metal alloying process. Because of aluminium removal, the beta-(AlNi) phase was transformed into the more stable nickel fcc structure. In this work, some important physicochemical properties of binary (Al-Ni) and ternary (Al-Ni-Fe) catalysts, with especial attention to textural properties adduced from nitrogen physisorption, are presented and discussed.

[15] ACCELERATION OF PARTICLE BREAKAGE RATES IN WET BATCH BALL MILLING

Tangsathikulchai C. - Powder Technology. 124(1-2):67-75, 2002

Batch wet grinding of 20 x 30 mesh quartz for slurry concentrations up to 56 vol.% solid showed an increase in the specific breakage rate of the top size feed as fines built up in the mill. The degree of this acceleration effect was represented by a parameter termed the acceleration factor, which depended both on the slurry concentration and the fineness of grinding. An empirical equation was developed to correlate the acceleration factor with slurry apparent viscosity, a characteristic size distribution and slurry concentration. The mechanism of rate acceleration was hypothesized to result from the ability of the top size particles to adhere on the grinding surfaces by means of liquid surface tension and pulp consistency and further influenced by the degree of flow turbulence prevailing in the charge. The overall effect gave rise to the classification of particles so that some remained in suspension and some resided on the grinding surfaces. (

[14] THE EQUIVALENCE BETWEEN DIFFERENT RESIDENCE TIME DISTRIBUTION MODELS IN BALL MILLING

Cho HC. Austin LG. - Powder Technology. 124(1-2):112-118, 2002

A number of different models have been used to describe the residence time distributions (RTDs) measured for dry and wet ball milling by various investigators in various countries. It is useful to be able to compare the results between different workers and this requires a knowledge of the equivalence of the models. A method is presented here that permits this comparison for the usual two parameter residence time distribution models, where one parameter is the mean residence time and the other is a characteristic parameter that defines the shape of the residence time distribution. The method defines the best correlation as that which produces the closest agreement of the predicted mill product size distributions over a wide range of values of mean residence time. The correlations are given in graphical form and in equation form. In some circumstances simulation results are not sensitive to the RTD model chosen, provided that the correct match of characteristic parameters is used

[13] EFFECT OF BALL MILLING CONDITIONS ON THE MICROSTRUCTURE AND THE TRANSFORMATION BEHAVIOR OF TI-NI AND TI-NI-CU SHAPE MEMORY ALLOY POWDERS

Nam TH. Kang SH. - Metals & Materials International. 8(2):145-150, 2002

Ti-50Ni and Ti-40Ni-10Cu (at.%) shape memory alloy powders have been fabricated by the ball milling method. Their alloying behaviour and transformation behaviour were investigated by means of optical microscopy, electron microscopy, X-ray diffractometry and differential scanning calorimetry. As-milled Ti-Ni powders fabricated with a milling time of less than 20 hrs were a mixture of pure elemental Ti and Ni, and therefore we were unable to obtain alloy powders because the combustion reaction between Ti and Ni occurred during heat treatment. Since those fabricated with a milling time of more than 20 hrs were a mixture of Ti-rich and Ni-rich Ti-Ni solid solution, it was possible to obtain alloy powders without a combustion reaction during heat treatment. Clear exothermic and endothermic peaks appeared in the cooling and heating curves, respectively, in DSC curves of 20 hr and 30 hr milled Ti-Ni powders. On the other hand, in DSC curves of 1 hr, 10 hrs, 50 hrs and 100 hrs, the thermal peaks were almost discernible. The optimum ball milling time for fabricating Ti-Ni alloy powders was 30 hrs. Ti-40Ni-10Cu alloy powders were fabricated successfully by the optimum ball milling conditions deduced from Ti-50Ni alloy powders.

[12] EFFECT OF MECHANICAL ALLOYING ON THE FORMATION OF SM₂FE₁₇NX COMPOUND

Lee CH. Kwon YS. - Metals & Materials International. 8(2):151-154, 2002

Elemental powders of iron and samarium were mechanically alloyed in the concentration range of Sm_xFe_{100-x} (x = 11, 13, 15, 17). A two-phase mixture of amorphous Sm-Fe and alpha-Fe phases was formed in all compositions studied. The effect



of the starting composition on the formation of $\text{Sm}_2\text{Fe}_{17}$ intermetallic compound was investigated by annealing mechanically alloyed powders. When the Sm content was 15 at.%, the annealed powders consisted of nearly a $\text{Sm}_2\text{Fe}_{17}$ single phase. For the preparation of a hard magnetic $\text{Sm}_2\text{Fe}_{17}\text{N}_x$ compound, additional nitriding treatment was performed under an N_2 gas flow at 450°C for various time intervals. It was found that nitrogenation for 3 hours was just enough to allow the formation of the $\text{Sm}_2\text{Fe}_{17}\text{N}_x$ compound. The coercivity increased when the nitrogenation time increased up to 3 hours and then tended to decrease with further nitrogenation

[11] PROPERTIES OF CO-DOPED N-TYPE FeSi_2 PROCESSED BY MECHANICAL ALLOYING

Ur SC. Kim IH. Lee JI. - *Metals & Materials International*. 8(2):169-175, 2002

Iron-silicide was produced with a mechanical alloying process and consolidated through vacuum hot pressing. The as-milled powders were of metastable state and fully transformed into the $\beta\text{-FeSi}_2$ phase through subsequent isothermal annealing. The as-consolidated iron silicides consisted of an untransformed mixture of $\alpha\text{-Fe}_2\text{Si}_5$ and $\epsilon\text{-FeSi}$ phases and a partially transformed $\beta\text{-FeSi}_2$ phase was found in the low density compact. Isothermal annealing was carried out to induce transformation into a thermoelectric semiconducting $\beta\text{-FeSi}_2$ phase. The transformation behavior of the $\beta\text{-FeSi}_2$ was investigated utilizing DTA, SEM, and XRD analyses. Isothermal annealing at 830°C in vacuum led to a thermoelectric semiconducting $\beta\text{-FeSi}_2$ phase transformation, but some residual metallic α and ϵ phases were unavoidable even after 96 hours of annealing. The iron silicide microstructures were investigated using SEM and TEM. The mechanical and thermoelectric properties of the $\beta\text{-FeSi}_2$ materials before and after isothermal annealing are characterized in this study

[10] MICROSTRUCTURE CHARACTERIZATION OF THE NiAl INTERMETALLIC COMPOUND WITH FE, GA AND MO ADDITIONS OBTAINED BY MECHANICAL ALLOYING

Albiter A. Bedolla E. Perez R. - *Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing*. 328(1-2):80-86, 2002

The microstructure and chemical characteristics of nanocrystalline NiAl intermetallic phase with a B2 crystalline structure are studied. Nanophase NiAl powder with the addition of minor elements (Fe, Ga and Mo) was prepared by mechanical alloying under argon atmosphere. Structural characterization based on X-ray diffraction (XRD) patterns and transmission electron microscopy (TEM) observations were carried out on the NiAl compounds. The effects of the different microalloyed elements on the microstructure are explored.

[9] A STUDY ON PREPARATION OF FUNCTIONALIZED ULTRA-FINE POLYPROPYLENE POWDER THROUGH A MECHANOCHEMICAL METHOD

Liu Y. Wang Q. - *Polymer Journal*. 34(3):132-137, 2002.

In this paper, a mechanochemical method was established to prepare functionalized ultra-fine polypropylene powder. Through pan-milling by a self-designed pan-type mechanochemical reactor, polypropylene (PP) was finely pulverized, during the process, inorganic peroxide solution was sprayed onto the PP powder surface to realize its rapid functionalization taking advantages of the mechanochemical effects caused by milling. Analysis results of electron spectroscopic chemical analysis (ESCA) show the PP ultra-fine powder prepared by this method can obtain over 0.1 oxygen content (O/C). Some factors influencing functionalization degree, including concentration, spraying amount of inorganic peroxide solution and milling temperature, were discussed. Besides, the degradation of PP during this process was investigated

[8] MICROSTRUCTURE CHARACTERIZATION OF THE NiAl INTERMETALLIC COMPOUND WITH FE, GA AND MO ADDITIONS OBTAINED BY MECHANICAL ALLOYING

Albiter A. Bedolla E. Perez R. - *Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing*. 328(1-2):80-86, 2002

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[7] PREPARATION OF TiB₂ POWDERS BY MECHANICAL ALLOYING

Hwang Y. Lee JK. - *Materials Letters*. 54(1):1-7, 2002

TiB₂ powders with submicrometer size were synthesized by mechanical alloying (MA) the mixture of elemental Ti and B powders in argon atmosphere using a steel jar. The effect of heat of formation and the atomic size factor on MA as well as the phase change during MA were investigated. The amorphization reaction, a common process during MA, had not been observed and the starting raw materials directly converted into final stable phase. When Zr of which atomic radius was larger than that of Ti was substituted for Ti, the alloying time was greatly reduced. On the contrary, substitution of Ta for Ti prolonged the alloying time because of the less negative heat of formation of TaB₂ than that of TiB₂.

[6] ALL-SOLID-STATE LITHIUM BATTERY WITH $\text{LiCo}_0.3\text{Ni}_0.7\text{O}_2$ FINE POWDER AS CATHODE MATERIALS WITH AN AMORPHOUS SULFIDE ELECTROLYTE

Machida N. Maeda H. Peng H. Shigematsu T. - *Journal of the Electrochemical Society*. 149(6):A688-A693, 2002

An all-solid-state battery was fabricated with $\text{LiCo}_0.3\text{Ni}_0.7\text{O}_2$ fine powder as cathode materials. The $\text{LiCo}_0.3\text{Ni}_0.7\text{O}_2$ fine powder was synthesized with oxalate decomposition methods and the average particle size of the powder was 2 μm diam. In the all-solid-state battery, $\text{a-60Li}_2\text{S} \cdot 40\text{SiS}_2$ powder, which was prepared by a mechanical milling process, was used as the solid electrolyte. The anode was metallic indium foil 0.127 mm thick. The charge-discharge cycling test of the battery was carried out under a constant current density of 64 $\mu\text{A cm}^{-2}$ with a 3.8 and 2.0 V charge-discharge criteria. The



battery showed a rechargeable specific capacity of 98 mAh g⁻¹ based on the cathode materials. The charge-discharge cycle efficiency of the battery was almost unity after the second cycle.

[5] MAGNETIC AND STRUCTURAL STUDY OF MECHANOCHEMICAL REACTIONS IN THE AL-Fe₃O₄ SYSTEM

Botta PM. Bercoff PG. Aglietti EF. Bertorello HR. Lopez JMP. - Journal of Materials Science. 37(12):2563-2568, 2002

The solid state reaction between Al and Fe₃O₄ (magnetite) using mechanochemical activation of powder mixtures under Ar atmosphere is studied. The phase evolution during the reaction is analyzed by X-ray diffraction (XRD), vibrating sample magnetometry (VSM), differential thermal analysis (DTA) and scanning electron microscopy (SEM). At 37 minutes of high-energy ball-milling the disappearance of reactive phases and the production of alpha-Fe, FeAl₂O₄ and alpha-Al₂O₃ is observed, together with significant changes in the magnetic behavior of the system. The composition and properties of samples heated up to 1200°C are also investigated. The behavior of the saturation magnetization M_s is interpreted on the basis of the formation of a variable composition spinel phase Fe [Al_{1-x} Fe_{2-x}] O₄ with 0 less than or equal to x less than or equal to 2 and a canting effect due to the presence of Al³⁺ ions in the spinel structure

[4] DRASTIC COERCIVITY RELAXATION IN AMORPHOUS Fe₇₄Al₅P₁₁C₆B₄ AND ITS DEPENDENCE ON THE PREPARATION METHOD

Schlorke-de Boer N. Schafer R. Eckert J. Schultz L. - Journal of Applied Physics. 91(10 Part 1):6601-6610, 2002

Amorphous Fe_{74-x}Al₅P₁₁C₆B₄Gax (x=0 or 2) powders with a wide supercooled liquid region were prepared by high energy ball milling of rapidly quenched ribbons and subsequently hot pressed in the viscous state to receive bulk glasses. Almost complete coercivity relaxation of the ball milled powders was achieved by annealing. The coercivity H_c of as-milled ribbons drops drastically from more than 2200 Am⁻¹ after 1 h of milling to 14.6 Am⁻¹ after optimum annealing. The coercivity of this powder and that of the as-quenched ribbons (7 Am⁻¹) differs only by the different contributions of surface irregularities H_c(surf) to the total coercivity H_c(tot). Drastic coercivity relaxation was also achieved by consolidating the as-milled powders. A minimum coercivity H_c of 30 Am⁻¹ was found for the bulk sample after compaction of 1 h ball milled powders. It is of the same order of magnitude as H_c of cast amorphous rods. The milling-induced coercivity increase as well as the coercivity relaxation are discussed in terms of the change of magnetic anisotropy and the creation and elimination of stress and shear bands in the amorphous phase. (

[3] SYNTHESIS OF NANOSIZED (Li_{0.5x}Fe_{0.5x}Zn_{1-x})Fe₂O₄ PARTICLES AND MAGNETIC PROPERTIES

Gee SH. Hong YK. Park MH. Erickson DW. Lamb PJ. Sur JC. - Journal of Applied Physics. 91(10 Part 2):7586-7588, 2002

In an attempt to synthesize nanosized (Li_{0.5x}Fe_{0.5x}Zn_{1-x})Fe₂O₄ (0 less than or equal to x less than or equal to 1) particles with high magnetic saturation and low coercivity, the energetic ball milling technique was employed. LiCO₃, alpha-Fe₂O₃, and ZnO powders were used as starting materials. The ball milled, partially crystallized lithium zinc ferrite starts to crystallize at about 600 degreesC. This is much lower than the temperature of 1000 degreesC, which is used in conventional methods. Particle size of lithium zinc ferrite was in the range of 20 to 50 nm. Regardless of the annealing temperature, the saturation magnetization increases with increasing x and reaches the maximum (about 80 emu/g) at x=0.7 [(Li_{0.35}Fe_{0.35}Zn_{0.3})Fe₂O₄], followed by a decrease to 60 emu/g for x=1 [(Li_{0.5}Fe_{0.5})Fe₂O₄]. On the other hand, the coercivity of x=0.7 composition decreases with increasing annealing temperatures. Saturation magnetization and low coercivity for x=0.7 annealed at various temperatures are discussed in terms of site occupation

[2] STRUCTURE AND PROPERTIES OF MECHANICALLY ACTIVATED BARIUM PEROXIDE

Massalimov IA. Kireeva MS. Sangalov YA. - Inorganic Materials. 38(4):363-366, 2002

Data are presented on the structural properties, water solubility, and chemical reactivity of mechanically activated BaO₂. The lattice parameters of BaO₂ are found to decrease markedly after mechanical activation for a certain time, which is accompanied by an appreciable increase in solubility and reactivity.

[1] KINETICS OF THE INITIAL STAGE OF MECHANICAL ALLOYING IN THE Fe(80)/Zr(20) SYSTEM

Povstugar IV. Butyagin PY. Dorofeev GA. Elsukov EP. - Colloid Journal. 64(2):178-185, 2002

The kinetics of the initial stage of mechanical alloying in the Fe/Zr system (80 : 20 at. %) was studied using X-ray diffraction, Mossbauer spectroscopy, and magnetic measurements. The stage of structural disordering of the mixture is shown to proceed quickly and end by a dose of 9 kJ g⁻¹. Further, intense formation of the amorphous phase takes place; as the dose of 16 kJ g⁻¹ is reached, all zirconium contained in the initial mixture passes into this phase, and its amount does not grow any more. The composition of the amorphous phase corresponds to Fe₇₃Zr₂₇ and remains constant throughout the mechanical processing up to a dose of 25 kJ g⁻¹. Annealing of the reaction mixture at 700°C results in decomposition of the amorphous phase and formation of an intermetallic with the composition and structure corresponding to Fe₂₃Zr₆. No contamination of the mixture by the material of the balls and vessel during mechanical treatment was detected



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