



**RESEAU FRANÇAIS DE  
MECANOSYNTHESE**

**Lettre N°129**

**Décembre 2005**

**189 Groupes de Recherche  
(dont 115 à l'étranger / 34 Pays)**

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**Bulletin d'adhésion au RFM 2005 / 2005 RFM Subscription Print**

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

Eric GAFFET

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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 : Reseau Francais de Mecanosynthèse

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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.

Extrait du site Web de la société Fritsch

(<http://www.fritsch.de>).

Contact : [info@fritsch.de](mailto:info@fritsch.de)

## Fritsch GmbH

Manufacturers of Laboratory Instruments  
Industriestraße 8 - D-55743 Idar-Oberstein - Germany  
Phone: +49/67 84/70-0 / Fax: +49/67 84/70-11



### Vario-Planetary Mill "pulverisette 4"

#### description

A Planetary Mill with infinitely variable grinding conditions and therefore the ideal mill for mechanical activation and alloying. The main applications are in the field of materials research and, of course, wherever a powerful, innovative planetary mill is required.

Typical fields of application:

The "pulverisette 4" vario-planetary mill is capable of emulating ball mills of conventional design, precisely simulating the types of stress entailed and thus reproducing or optimising grinding processes.

Method of operation:

But with the "pulverisette 4" vario-planetary mill **the rotational speeds of grinding bowls and supporting disc can be adjusted completely independently** of each other in contrast to standard planetary mills. By changing the transmission ratio it is therefore possible for the first time to carry out mechanical activation as well as mechanical alloying.

Features of performance:

- free selectable, controlled rotation speed for grinding bowls and supporting disc
- Programming of the grinding parameters by PC software as desired
- RS232 interface for programming and to transfer grinding parameters to the PC
- Real-time display of the speeds to monitor the grinding process
- Reversing option to improve the grinding results
- Emulation of various ball mills
- Variably adjustable pressure on sample (friction and/or impact)
- Simultaneous grinding in up to 4 small or 2 large grinding bowls
- Quick, secure fastening of the grinding bowls "safe lock"

#### technical data

Electrical details:	400 V/3
Frequency:	50-60 Hz
input power:	9000 W
Working Principle:	impact force
Interface:	RS232
Timer:	99 min.
Over All Dimensions (WxDxH):	600x800x1100 mm
Weight net:	320 kg
Weight gross:	380 kg
Max. feed size:	10 mm
Feed quantity:	up to 2 x 225 ml
attainable final fineness:	< 1 µm

#### suitable for the following sample materials

hard:	yes
medium hard:	yes
soft:	yes
brittle:	yes
tough:	yes
fibrous:	yes
temperatur-sensitive:	yes

#### Equipment:

The Vario-Planetary Mill "pulverisette 4" is equipped with 2 bowl fasteners and a "safe lock" clamping system. This mill is delivered without grinding bowls and balls, which are available in 9 different materials and must be ordered as accessories.



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**Congress and School Announcements**  
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**International Symposium on Surface Science and Nanotechnology (ISSS-4),**

November 14-17, 2005

Saitama, Japan

(Contact: WEBSITE: <http://wwwsoc.nii.ac.jp/jps/jps/bbs/iss4.html>)

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**Symposia N, O, P, Q, R, & S on Nano- to Microstructured Materials, Materials Research Society  
Fall Meeting,**

November 28-December 2, 2005

Boston, Massachusetts

(Contact: WEBSITE: [http://www.mrs.org/meetings/future\\_meetings.html](http://www.mrs.org/meetings/future_meetings.html))

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**SPIE Conference on Microelectronics, MEMS, and Nanotechnology,**

December 11-14, 2005

Queensland University of Technology, Brisbane, Australia

(Contact: WEBSITE: <http://spie.org/conferences/calls/05/au/>)

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**2nd International Conference on Nanomaterials and Nanotechnology,**

December 12-15, 2005

London, United Kingdom

(Contact: WEBSITE: <http://whatson.iop.org/events/4589>)

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**SPIE meeting on Microelectronics, MEMS, and Nanotechnology,**

December 14-16, 2005

Queensland University of Technology, Brisbane, Australia

(Contact: WEBSITE: <http://spie.org/conferences/calls/05/au>)

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**European Nano Systems 2005, 1st Workshop on NanoTechnology Transfer in Europe,**

December 14-16, 2005

Hotel Mercure Paris Tour Eiffel Suffren, Paris, France

(Contact: B. Courtois, TIMA Labs, Grenoble, France, EMAIL: [ENS@imag.fr](mailto:ENS@imag.fr), WEBSITE:  
<http://tima.imag.fr/conferences/ENS>)

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**IEEE Conference on Emerging Technologies: Nanoelectronics (NanoSingapore 2006),**

January 10-13, 2006

Meritus Mandarin Hotel, Singapore

(Contact: EMAIL: [cet06@meetmatt.net](mailto:cet06@meetmatt.net), WEBSITE: <http://www.ieeeecet.org/>)

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**MOEMS-MEMS 2006, Micro & Nanofabrication, Photonics West,**

January 21-26, 2006

San Jose Convention Center, San Jose, California

(Contact: WEBSITE: <http://www.spie.org/events/pw>)

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**Nanomaterials Symposia, TMS Annual Meeting,**

March 12-16, 2006

San Antonio, Texas

(Contact: WEBSITE: <http://www.tms.org/Meetings/Annual-06/AnnMtg06Home.html>)

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**6th International Conference on the Scientific and Clinical Applications of Magnetic Carriers,**

May 17-20, 2006

Krems (Vienna Region), Austria

(Contact: WEBSITE:

[http://www.magneticmicrosphere.com/other/MagMeet2006\\_First\\_Announcement.pdf](http://www.magneticmicrosphere.com/other/MagMeet2006_First_Announcement.pdf))

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**European Society for Precision Engineering and Nanotechnology 6th International Conference  
(euspen 2006),**



May 28-June 1, 2006  
Congress Casino in Baden (near Vienna), Austria  
(Contact: WEBSITE: <http://baden2006.euspen.com/>)

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**16th European Conference of Fracture, Failure Analysis of Nano and Engineering Materials and Structures (ECF-16),**

July 3-7, 2006  
Alexandroupolis, Greece  
(Contact: E. Gdoutos, School of Engineering, Democritus University of Thrace, GR-671 00, Xanthi, Greece, FAX: +30-25410-79652, EMAIL: [egdoutos@civil.duth.gr](mailto:egdoutos@civil.duth.gr), WEBSITE: <http://ecf16.civil.duth.gr/>)

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**6th IEEE Conference on Nanotechnology,**

July 16-20, 2006  
Westin Hotel, Cincinnati, Ohio  
(Contact: WEBSITE: <http://www.ececs.uc.edu/~mcahay/Nano2006/index2006.html>)

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**International Conference on Nanoscience and Technology (ICN&T 2006),**

July 30-August 4, 2006  
Convention Center, Basel, Switzerland  
(Contact: WEBSITE: <http://www.icnt2006.ch/>)

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**NANO2006:**

8th International Conference on Nanostructured Materials,  
August 20-26, 2006  
National Science Seminar Complex, Indian Institute of Science, Bangalore, India

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- **INCOME 2006**

<http://www.solid.nsc.ru/INCOME2006/default.htm>



## **Nanotech Jobs And / Or Post Doc Position - Contract Proposal**

**From Ch. Gras (September 2005)**

### **Postdoctoral Researcher in Electron Microscopy of Multifunctional Ceramics Grade RAI A / Salary: £19,460 to £29,128 / Job Ref No DJ05/018**

Applications are invited for a postdoctoral researcher to join the Electron Microscopy Group of Professor David Cockayne FRS to work on high resolution electron microscopy and energy loss studies of SrTiO<sub>3</sub> and BaTiO<sub>3</sub> multifunctional ceramics, with a particular focus on grain boundary structures. This is part of an international collaborative programme ("INCEMS") funded by the EC, and involves research groups from Germany, Ireland, France, Slovenia and the UK. Your role within the project will be to use the unique aberration corrected TEM/STEM, housed in the Department, to investigate the microstructure of interfaces at the highest possible resolution, and to interact with growers and modellers within the collaboration. The characterization will be by a range of techniques including high-resolution TEM (HREM, HAADF) and EELS.

The post is available from October 2005 (or as soon as possible thereafter) for up to 33 months.

Applicants should have a doctorate in materials science, physics or a related discipline and have demonstrated experience in high resolution electron microscopy and in sample preparation of crystalline materials. Experience with EELS would be an advantage. Evidence of ability in the use of computer software, preferably for image simulation, is required. Proven ability to identify research objectives and meet agreed deadlines, self-motivation and flexibility are essential. Excellent written and interpersonal communication skills, the ability to work effectively as part of a team, and evidence of a good publication record are expected. Willingness to travel for short periods to work with collaborators in Europe is required.

Further particulars, including instructions on applying for this post, are available from the web-site: <http://www.materials.ox.ac.uk> or from Mrs K Fewings, Department of Materials, University of Oxford, Parks Road, Oxford OX1 3PH (email: [posts@materials.ox.ac.uk](mailto:posts@materials.ox.ac.uk)), or telephone 01865 273680 (post reference DJ05/018). The closing date for applications is 30 September 2005 with interviews currently planned for the week beginning 17 October 2005.

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

### **Academic Staff**

#### **Professorship of Materials Modelling**

Applications are invited for the above post, tenable from 1 January 2006, or such later date as may be arranged. The University seeks to appoint a person with a record of outstanding international excellence in research in materials modelling who, through leadership and the distinction of his or her contribution to the field, will ensure the pursuit of the highest standards in teaching and research in materials modelling at Oxford and its wide recognition outside. The Professorship of Materials Modelling is held in the Department of Materials. A non-stipendiary fellowship at St. Anne's College is attached to the professorship. Further particulars, including details of how to apply, are available from <http://www.admin.ox.ac.uk/fp/> or from the Registrar, University Offices, Wellington Square, Oxford OX1 2JD (Tel: 01865 270200). The closing date for applications is Monday 3

**From Ch. Gras (March 2005)**

#### **Postdoctoral researcher on novel atomic scale characterisation of advanced thin film structures**

Grade RAI A / Salary: £19,460 to £29,128 / Job Ref No. DJ05/004

Applications are invited for a postdoctoral research post in the Department of Materials, available from 1 June 2005 (or as soon as possible thereafter) for up to three years' duration.

The post is funded by the EPSRC, under the DTI-LINK ISD (information storage and displays) Initiative. The project is in collaboration with Oxford nanoScience, Hitachi Global Storage and Durham University and will be supervised in Oxford by Professor A Cerezo and Professor A K Petford-Long. The aim of the project as a whole is to provide a scanning atom probe analytical tool complemented by a suitable specimen preparation process to enable improved understanding of advanced materials, and hence to facilitate the development and subsequent reproducible manufacture of new thin film structures for advanced applications such as magnetic recording heads.



Lettre RFM N°129 - Décembre 2005  
Corresp. : <mailto:Eric.Gaffet@utbm.fr>

You will be expected to use advanced three-dimensional atom probe (3DAP) analysis to study the morphology of spin-valve material and media films at near-atomic resolution. Both a conventional atom probe and the scanning atom probe (SAP) instrument being developed under the LINK project will be used. You will also be expected to carry out image processing and data analysis, using the dedicated facilities available within the atom probe facility. Focused ion beam (FIB)-based specimen preparation techniques for the SAP samples will be developed during the project. Some electron microscopy will be required for comparative microstructural analysis. Training in the use of the various instruments will be provided. However, experience with advanced materials characterisation techniques is essential, as is knowledge of either advanced microstructural characterisation, thin film magnetic materials or focussed-ion beam systems. The collaborative nature of this project requires you to have good communications skills and to interact with collaborating partners effectively. You must be self-motivated and able to work both independently and as part of a team. You will be expected to report on your work to the Investigators in Oxford and at joint meetings of the collaborating groups (some of which may be held in the USA), to write reports and scientific publications, and to present results of the work at international conferences.

Further particulars, including instructions on applying for this post, are available from the web-site: <http://www.materials.ox.ac.uk> or from Mrs K Fewings, Department of Materials, University of Oxford, Parks Road, Oxford OX1 3PH (email: [posts@materials.ox.ac.uk](mailto:posts@materials.ox.ac.uk)), or telephone 01865 273680 (post reference DJ05/004). The closing date for applications is 11 March 2005 with interviews currently planned for 21 April 2005.

**PDRA in experimental studies of brittle-ductile transitions in tungsten Grade RAIA / Salary £19,460 to £29,128 / Job Ref No DJ05/005**

Applications are invited for a postdoctoral position available for up to 3 years' duration from 1 July 2005.

This project will investigate brittle-ductile transitions in tungsten, including effects of radiation damage. The work will involve preparation and testing of specimens of single crystal and polycrystalline tungsten, in both the unirradiated and ion-irradiated conditions. Dislocation-based modelling will be used to understand the observed fracture behaviour. The person appointed will work with other researchers performing modelling and experimental studies on related materials, at Oxford and elsewhere.

This research is part of a pan-European integrated project on "materials for extreme environments", in the work-package on materials for fusion reactors . It is funded by the EU and will be carried out under the supervision of Dr S.G. Roberts.

Applicants should have, by the starting date, a PhD, or equivalent, in a relevant physical science subject, together with proven ability in innovative and effective experimental research in mechanical properties of materials. This should preferably include expertise in one or more of: fine-scale mechanical testing; metal single crystals; SEM, TEM and other microscopical methods; dislocations; modelling of mechanical properties (in particular by dislocation dynamics); radiation damage. The post will require proven ability to identify research objectives and complete research tasks within an agreed time-scale. The post holder will need to be flexible in the tasks to achieve the set objectives. A good publication record for stage of career, good communication skills in English, self-motivation and good team work are required, as is ability to travel within the UK and Europe to work with collaborators.

Flexible working may be an option.

Further particulars, including instructions on applying for this post, are available from the web-site: <http://www.materials.ox.ac.uk> or from Mrs K Fewings, Department of Materials, University of Oxford, Parks Road, Oxford OX1 3PH (email: [posts@materials.ox.ac.uk](mailto:posts@materials.ox.ac.uk)), or telephone 01865 273680 (post reference DJ05/005). The closing date for applications is 8 April 2005 with interviews currently planned for the week commencing 9 May 2005

**From E. Gaffet (September 2004)**

**Portail europeen sur la mobilite des chercheurs**

Ce site regroupe plus de 1 500 liens vers des organismes qui proposent des bourses, des primes et des possibilites d'emploi et offrent des opportunités de formation, de mobilite et de developpement de carriere pour les chercheurs de l'ensemble de l'Esace europeen de la recherche. Le portail recense les vacances de postes dans les universites, dans l'industrie, dans les organisations de R&D et les fondations.

<http://europa.eu.int/eracareers>



**From E. Gaffet (August 2004)**

**NANOTECH JOBS:**

Looking for a job in the emerging field of nanotech or have an nanotech opening that you need to fill? Our new site is dedicated to jobs at the intersection of nanotechnology, biotechnology, and information technology. Job seekers can search and apply for jobs, set up job agents, and post their resumes. Employers can post and manage jobs, search resumes, and take advantage of many advertising options on the site. Check out our new jobs site powered by tinytechjobs.com. Hundreds of jobs are posted each day - just visit

<http://forbes.tinytechjobs.com> to register or click on the link below:

<http://forbes.tinytechjobs.com>



## Bibliographie (Ouvrage)

### **Plastic Deformation in Nanocrystalline Materials**

**Auteurs : M. Yu Gutkin and I. A. Ovid'ko**

**Editeur : Springer (2004), 187 pp. ISBN: 3-540-20993-X \$119 / £61.50 / € 79.95**

This book gives an introduction to the mechanics of deformed nanostructures, along with an overview of the current understanding of the structure and deformation behavior of nanocrystalline materials. Theoretical concepts and experimental data are presented and the importance of this field for a range of nanotechnologies is explained.

### **Le procédé SPS (Spark Plasma Sintering )**

**Auteur : E. Gaffet**

**Editeur : Innovation 128 (2004) - ISBN : 2-906024-31-7 - Janvier 2004 - 152 pages**

*(Innovation 128 – 24 rue du Quatre Septembre – 75002 Paris – France – Fax : (+33) 1 42 65 47 76)*

#### **Press Release :**

La mise en oeuvre des courants pulsés de fortes intensités (jusqu'à 10.000 Ampères) sous faibles tensions (inférieure à quelques centaines de Volts) permet d'élargir les capacités de la métallurgie des poudres (consolidation de matériaux hétérogènes & frittage réactif), du soudage / jonction et des traitements de surface.

Le procédé SPS permet de densifier / consolider des matériaux tout en leur conservant leurs caractéristiques initiales (microstructures, nanostructures). Si les durées de mise en oeuvre des procédés de densification classiques s'élèvent plutôt en heures, le procédé SPS permet de consolider des matériaux pour des durées se chiffrant en minutes. Il permet également dans le domaine du frittage de consolider des matériaux poreux (macro et microporosité).

Les domaines d'applications des machines SPS couplant décharge électrique (jusqu'à 8.000 A / 0 - 10 V) et pressions (jusqu'à 500 Tonnes pour les machines industrielles) sont le frittage réactif ou non des nanomatériaux (en conservant l'échelle nanométrique), des composites à matrice métallique et / ou des cermets, des composés intermétalliques, des matériaux thermoélectriques ou la réalisation de matériaux à gradients ou de céramiques renforcées par des fibres ainsi que le frittage de composants aussi différents que polymères /métaux.

Dans le domaine du soudage / jonction, ce type de procédés permet de lier des matériaux réputés difficilement soudables. Ces matériaux peuvent se présenter sous forme de poudres et/ou de feuillards.

Dans le domaine des traitements de surface, ils permettent par exemple de modifier la densité superficielle de revêtements et /ou la porosité de surface.

Au début 2003, on comptait déjà 150 machines commercialisées au Japon mais seulement deux en Europe (Stockholm et Dresde) et une seule aux Etats-Unis pour des applications dans les domaines tels que les outils de coupe (WC avec ou sans Co), l'industrie automobile (densification de pièces à bas d'Al), l'aéronautique (mise en forme d'alliages spéciaux), les composites renforcés, l'optique (densification de céramiques transparentes), la thermique (matériaux à gradients) et la biologique (biocéramiques). Il aura fallu attendre ce printemps 2004 pour qu'une première machine soit installée en France.

Un état de l'art exhaustif est établi et une analyse de près d'une centaine de brevets vient compléter ce rapport. Cette étude très documentée est rédigée par l'un des meilleurs spécialistes européens des nanomatériaux et de la mécanosynthèse qui a été personnellement amené à mettre en oeuvre le procédé SPS. En contact étroit avec les acteurs internationaux du domaine SPS, l'auteur livre des informations qui vous permettront d'évaluer sans retard les avantages de cette nouvelle technologie dans vos applications.



**Bibliographie  
(Ouvrage)**

**Les nanosciences  
Nanotechnologies et nanophysique**

**Les nanosciences  
Nanotechnologies et nanophysique**

Sous la direction de :

*Lahmani (Marcel) / Dupas (Claire) / Houdy (Philippe)*

A la croisée de multiples disciplines (physique, chimie, biologie, mécanique, etc.), les nanotechnologies et les nanosciences s'imposent aujourd'hui comme l'un des champs majeurs de recherche des années à venir. En effet, la maîtrise de l'échelle nanométrique aura des implications dans des domaines aussi variés que le magnétisme, le stockage de l'information, la biologie, ou encore l'électronique, avec le développement de composants totalement nouveaux.

Des techniques de champ proche aux procédés de lithographie, des fullerènes à l'électronique de spin, ce livre, destiné à la fois aux étudiants et aux chercheurs, dresse un panorama complet et actuel, aussi bien théorique que technique, des nanosciences.

*Marcel Lahmani a été ingénieur à IBM et professeur associé à l'université d'Evry. Il est depuis 1989 vice président du Club Nanotechnologie.*

*Claire Dupas est professeure des universités et directrice de l'ENS de Cachan.*

*Philippe Houdy est professeur à l'université d'Evry et directeur du laboratoire d'étude des milieux nanométriques.*

© 2004, 17 x 24 cm, 720 pages, code Belin 003627, ISBN 2-7011-3627-X

**Prix : 47,5 Euros**

Les personnes intéressées peuvent commander le livre auprès de :

Edition Belin

8 rue Férou

75278 Paris Cedex 06

<http://www.editions-belin.com/csl/master.asp>

**Pour plus d'information :** <http://www.editions-belin.com/belin.asp?ad=o003627>



## Bibliographie

### Mechanosynthesis references

### Références NanoMatériaux / Mécanosynthèse

#### Commencer semaine 47

#### [48] PROPERTIES OF A FIREPROOFING FORMULATION BASED ON LIQUID GLASS AND MECHANICALLY ACTIVATED ALUMINUM OXIDE

Eremina-NV; Avvakumov-EG; Zelinskii-VY - RUSSIAN-JOURNAL-OF-APPLIED-CHEMISTRY. JUL 2005; 78 (7) : 1043-1047

Moistening of filler particles with binder, curing, and high-temperature synthesis were studied in a liquid-glass formulation based on aluminum oxide. Selected kinetic parameters of these processes were determined. The influence exerted by the phase composition and mechanical activation on the adhesion, yield of the synthesis product, and basic properties of the formulation were analyzed.

#### [47] STRUCTURAL-PHASE COMPOSITION AND CORROSION BEHAVIOR OF FINELY DISPERSED FE-C POWDERS IN NEUTRAL MEDIA

Lomayeva-SF; Bokhonov-BB; Syugaev-AV; Elsukov-EP; Reshetnikov-SM - PROTECTION-OF-METALS. SEP-OCT 2005; 41 (5) : 465-471

Effects of the structural-phase state and the structure of the surface layer on the corrosion behavior of mechanically activated finely dispersed Fe-C powders were studied. The powders prepared by mechanical milling of iron in graphite or heptane were mixtures of phases (nanocrystalline alpha-Fe + amorphous Fe-C phase (AP), alpha-Fe + Fe<sub>3</sub>C, and alpha-Fe + graphite) in different ratios. Corrosion tests were carried out in a model medium (0.85% NaCl) at 37 degrees C. The oxide surface layer of powders containing a high content of AP was found to exhibit protective properties. The corrosion behavior of the powders is insensitive to such changes in the structural-phase state as the formation of carbides, graphite encapsulation, grain size alterations, and hydrogenation.

#### [46] ENHANCED J(C) IN AIR-PROCESSED GDBA2CU3O7-DELTA SUPERCONDUCTORS

Xu-CX; Hu-AM; Sakai-N; Izumi-M; Hirabayashi-I - PHYSICA-C-SUPERCONDUCTIVITY-AND-ITS-APPLICATIONS. OCT 1 2005; 426 Part 1 : 613-617

Superconducting properties of the melt-growth bulk GdBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub>-delta (Gd123) were studied with the addition of BaO<sub>2</sub>, fine Gd<sub>2</sub>BaCuO<sub>5</sub> (Gd211) and Gd<sub>2</sub>Ba<sub>4</sub>MoCuO<sub>z</sub> (Gd2411) from the viewpoint of improvement of the critical current density (J(c)) by growth process in air. The addition of BaO<sub>2</sub> with 0.1 in a molar ratio to Gd123 effectively enhanced J(c). This is coming from the suppression of the solid solution growth in Gd123 with Ba-rich phase. The addition of the ball-milled fine Gd211 particles enhanced J(c) to 90,000 A/cm(2) at 77 K under self-field. Single phase Gd2411 was successfully synthesized in air and added into Gd123 system with substitution in part by Gd211 as the second phase. The J(c) of the samples mixed with Gd2411 reached 84,500 A/cm(2) at 77 K under self-field. We conclude that the present additive materials with a proper amount are very effective to improve the J(c) value of Gd123 bulk superconducting magnets.

#### [45] DEVELOPMENT OF TECHNOLOGY FOR A RELIABLE FABRICATION OF LRE-123 MATERIALS FOR LEVITATION APPLICATIONS ABOVE 80 K

Muralidhar-M; Sakai-N; Jirsa-M; Murakami-M; Koshizuka-N; Hirabayashi-I - PHYSICA-C-SUPERCONDUCTIVITY-AND-ITS-APPLICATIONS. OCT 1 2005; 426 Part 1 : 777-782

Nanoscale secondary phase Gd<sub>2</sub>BaCuO<sub>5</sub> "Gd-211" particles less than 60 nm in size, added to mixed ternary LRE-Ba<sub>2</sub>Cu<sub>3</sub>O<sub>y</sub> [LRE = (Nd<sub>0.33</sub>Eu<sub>0.33</sub>Gd<sub>0.33</sub>), (Sm<sub>0.33</sub>Eu<sub>0.33</sub>Gd<sub>0.33</sub>) (Nd<sub>0.33</sub>SM<sub>0.33</sub>Gd<sub>0.33</sub>)] "NEG-123, SEG-123, NSG-123" oxide powders and melt-processed in Ar-1%pO(2) gas mixture, significantly improve electromagnetic performance at low magnetic fields. As confirmed by transmission electron microscopy and dynamic force microscopy, the nanoparticles in the LRE-123 matrix not only survive the melt-texturing process but, thanks to their contamination by Zr coming from ball milling process, these particles in majority transform into a new type of even smaller defects. Further improvement in flux pinning was reached when both 123 and 211 powders were thoroughly ball milled before the melt-growth process to the similar small size. As a result, J(c) values of 260 kA/cm(2) and 55 kA/cm(2) were observed in remnant state at 77 K and 90 K, respectively.

#### [44] DYNAMIC DEFORMATION AND FRACTURE BEHAVIOR OF ULTRAFINE-GRAINED ALUMINUM ALLOY



Lettre RFM N°129 - Décembre 2005  
Corresp. : <mailto:Eric.Gaffet@utbm.fr>

#### **FABRICATES BY EQUAL-CHANNEL ANGULAR PRESSING**

Kim-YG; Hwang-B; Lee-S; Kim-WG; Shin-DH - METALLURGICAL-AND-MATERIALS-TRANSACTIONS-A-PHYSICAL-METALLURGY-AND-MATERIALS-SCIENCE. NOV 2005; 36A (11) : 2947-2955

In the present study, ultrafine-grained microstructures of a conventional 5083 aluminum alloy were fabricated by equal-channel angular pressing, and their dynamic deformation and fracture behavior were investigated. Dynamic torsional tests were conducted on four aluminum alloy specimens using a torsional Kolsky bar, and then the test data were analyzed in relation to microstructures, tensile properties, and adiabatic shear-banding behavior. The equal-channel angular-pressed (ECAP) specimens consisted of ultrafine grains and contained a considerable amount of second-phase particles, which were refined and distributed homogeneously in the matrix as the equal-channel angular pressing pass number increased. The dynamic torsional test results indicated that the maximum shear stress increased, while the fracture shear strain remained constant, with increasing equal-channel angular pressing pass number. Observation of the deformed area beneath the dynamically fractured surface showed that a number of voids initiated mainly at second-phase particle/matrix interfaces and that the number of voids increased with increasing pass number. Adiabatic shear bands of 200 to <300  $\mu\text{m}$  in width were formed in the as-extruded and 1-pass ECAP specimens having coarser particles, whereas they were hardly formed in the four-pass and eight-pass ECAP specimens having finer particles. The possibility of adiabatic shear-band formation was explained by concepts of absorbed deformation energy and void initiation.

#### **[43] STRUCTURAL EVOLUTION IN NANOCRYSTALLINE FE-10 WT PCT CR ALLOY POWDER PRODUCED BY MECHANICAL ALLOYING-AN ATOMIC-FORCE MICROSCOPY STUDY**

Rao-MA; Bhargava-S; Deva-D - METALLURGICAL-AND-MATERIALS-TRANSACTIONS-A-PHYSICAL-METALLURGY-AND-MATERIALS-SCIENCE. NOV 2005; 36A (11) : 3195-3204

A mechanical alloying (MA) method was used to synthesize Fe-10 wt pct Cr alloy powder. The formation of an Fe-Cr solid solution during milling was studied using atomic-force microscopy (AFM), with the help of an atomic-force microscope in acoustic AC (AAC) mode. The AFM amplitude images indicated that the interlamellar spacing in the structure decreased with an increase in the milling time, and finally gave way to a nonlamellar structure. For structures obtained by milling up to 40 hours, AFM phase-contrast images showed regions of inhomogeneity. Surface-topography images of the granular milled powder showed that the powder surfaces were not smooth, but consisted of a typical hills-and-valley structure. The mean height of the hills decreased with an increase in the milling time. Powders milled up to 20 hours showed a structure that contained grains and subgrains. However, as the interlamellar spacing in granules was reduced, the clear definition of the grains disappeared. Only subgrains were observed in powders milled for time intervals  $\geq 40$  hours. With the milling time  $\geq 40$  hours, the subgrains not only got more and more refined, they also got elongated in the direction of granular flow. The subgrains in the powder milled for 100 hours were found to have an aspect ratio of 2.5 to 3.0; their smaller dimensions varied from 5 to 30 nm.

#### **[42] EFFECT OF TUNGSTEN CARBIDE ADDITIONS ON THE MICROSTRUCTURE AND PROPERTIES OF HOT-PRESSED ALUMINA**

Acchar-W; Cairos-CA; Segadaes-AM - MATERIALS-SCIENCE-AND-ENGINEERING-A-STRUCTURAL-MATERIALS-PROPERTIES-MICROSTRUCTURE-AND-PROCESSING. OCT 15 2005; 406 (1-2) : 74-77

Studies published in the literature have shown that alumina reinforced with refractory carbides is a good alternative to cemented carbides, for cutting tools and wear-resistant applications. In this work, alumina reinforced with tungsten carbide was homogenized and mixed in a planetary ball mill and subsequently hot-pressed at 1450 degrees C under 20 MPa in flowing argon. Hot-pressing resulted in a material with a hardness and fracture toughness of 19 GPa and 7.1 MPa  $\text{m}^{1/2}$ , respectively. X-ray diffraction analysis showed the presence of alumina and WC. No other new crystalline phase was identified. The sintered material showed a very homogeneous distribution of small dark grains of WC in an alumina matrix, which appears to have very large grains with very clean grain boundaries. The WC grains tend to lie on grain boundaries but can also be seen at triple points. The material also shows large alumina grains containing WC inclusions, which points to the possible occurrence of abnormal grain growth.

#### **[41] DEFORMATION MODE AND PLASTIC FLOW IN ULTRA FINE GRAINED METALS**

Segal-VM - MATERIALS-SCIENCE-AND-ENGINEERING-A-STRUCTURAL-MATERIALS-PROPERTIES-MICROSTRUCTURE-AND-PROCESSING. OCT 15 2005; 406 (1-2) : 205-216

Mechanical behavior of ultra fine grained (UFG) metals fabricated by severe plastic deformation (SPD) is considered in the paper. The mechanisms of a crystallographic glide during a continuous micro flow and shear band (SB) localization/fragment rotation during a discontinuous micro flow are analyzed by simple models. It is shown that localized flow and the transition to localization are sensitive to deformation mode and conditions of processing or subsequent loading. Experimental data on texture evolution and tensile properties of ultra fine and fine grained aluminum alloy Al-0.5Cu as well as dynamic recrystallization of high purity aluminum Al5N5 are presented for pure shear and simple shear deformation modes. These results comply with theoretical models. Tensile tests of ultra fine grained structures reveal two stages of localization, into a



sample neck and inside a planar material layer. In contrast to ordinary materials, the second stage modifies tensile loading and leads to different fracture mechanisms.

**[40] GAO<sub>2</sub>H, ALPHA-GA<sub>2</sub>O<sub>3</sub> AND BETA-GA<sub>2</sub>O<sub>3</sub> POWDERS SYNTHESIZED FROM BALL-MILLED GAN POWDERS**

Xiao-HD; Ma-HL; Liang-W; Xue-CS; Zhuang-HZ; Ma-J; Hu-WR - MATERIALS-CHEMISTRY-AND-PHYSICS. DEC 15 2005; 94 (2-3) : 261-265

GaO<sub>2</sub>H, alpha-Ga<sub>2</sub>O<sub>3</sub> and beta-Ga<sub>2</sub>O<sub>3</sub> powders were synthesized from mechanically ground GaN powders with thermal annealing in a nitrogen atmosphere. The structural properties of GaO<sub>2</sub>H, alpha-Ga<sub>2</sub>O<sub>3</sub> and beta-Ga<sub>2</sub>O<sub>3</sub> powders were investigated by X-ray powder diffraction (XRD), Fourier transform infrared (FT-IR) and scanning electron microscopy (SEM). The studies revealed that the samples obtained by ball-milled GaN for 4 h are orthorhombic crystalline GaO<sub>2</sub>H phase. However, when GaO<sub>2</sub>H were annealed in a nitrogen atmosphere at 550 and 950 degrees C, alpha-Ga<sub>2</sub>O<sub>3</sub> and beta-Ga<sub>2</sub>O<sub>3</sub> powders were obtained, respectively. SEM images indicated that the morphologies of GaO<sub>2</sub>H, alpha-Ga<sub>2</sub>O<sub>3</sub> and beta-Ga<sub>2</sub>O<sub>3</sub> are ruleless, and their sizes are in the range of about 300-70, 150-70, and 150-70 nm, respectively.

**[39] HYDROGEN CYCLING OF NIOBIUM AND VANADIUM CATALYZED NANOSTRUCTURED MAGNESIUM**

Schimmel-HG; Huot-J; Chapon-LC; Tichelaar-FD; Mulder-FM - JOURNAL-OF-THE-AMERICAN-CHEMICAL-SOCIETY. OCT 19 2005; 127 (41) : 14348-14354

The reaction of hydrogen gas with magnesium metal, which is important for hydrogen storage purposes, is enhanced significantly by the addition of catalysts such as Nb and V and by using nanostructured powders. In situ neutron diffraction on MgNb<sub>0.05</sub> and MgV<sub>0.05</sub> powders give a detailed insight on the magnesium and catalyst phases that exist during the various stages of hydrogen cycling. During the early stage of hydriding (and deuteriding), a MgH<sub>1-x</sub> phase is observed, which does not occur in bulk MgH<sub>2</sub> and, thus, appears characteristic for the small particles. The abundant H vacancies will cause this phase to have a much larger hydrogen diffusion coefficient, partly explaining the enhanced kinetics of nanostructured magnesium. It is shown that under relevant experimental conditions, the niobium catalyst is present as NbH<sub>1</sub>. Second, a hitherto unknown Mg-Nb perovskite phase could be identified that has to result from mechanical alloying of Nb and the MgO layer of the particles. Vanadium is not visible in the diffraction patterns, but electron micrographs show that the V particle size becomes very small, 2-20 nm. Nanostructuring and catalyzing the Mg enhance the adsorption speed that much that now temperature variations effectively limit the absorption speed and not, as for bulk, the slow kinetics through bulk MgH<sub>2</sub> layers.

**[38] EFFECT OF TEMPERATURE ON SYNTHESIS AND PROPERTIES OF ALUMINUM-MAGNESIUM MECHANICAL ALLOYS**

Urnbrajkar-SM; Schoenitz-M; Jones-SR; Dreizin-EL - JOURNAL-OF-ALLOYS-AND-COMPOUNDS. OCT 27 2005; 402 (1-2) : 70-77

The syntheses of an Al<sub>0.7</sub>Mg<sub>0.3</sub> mechanical alloy was studied using a planetary mill. Several distinct temperature regimes of mechanical alloying were achieved using milling jars equipped with finned heat sinks and an external air conditioner installed to cool the entire milling chamber. Wireless temperature sensors were attached to the milling jars to monitor the process temperature. Intermediate and final products were collected and were analyzed by electron microscopy and X-ray diffraction. The temperature history of the milling jars exhibited two peaks during mechanical alloying. The first peak occurred when particles of the starting powders deformed to produce flakes. The second peak was observed when the flakes agglomerated and re-fragmented forming layered composites that served as precursors for the mechanical alloy. The temperature of milling affected the magnesium solubility of the produced Al-Mg mechanical alloys. Decreasing the milling temperature from similar to 70-80 degrees C to 20-30 degrees C resulted in an increase of the dissolved Mg concentration in Al from 2-3 at.% to similar to 25 at.% for the Al<sub>0.7</sub>Mg<sub>0.3</sub> composition. The formation of intermetallic phases was favored at higher milling temperatures, where high solubilities cannot be achieved.

**[37] STRUCTURAL TRANSFORMATIONS IN GRAPHITE INDUCED BY MAGNETO-MECHANICAL-MILLING IN HYDROGEN ATMOSPHERE**

Smolira-A; Szymanska-M; Jartych-E; Calka-A; Michalak-L - JOURNAL-OF-ALLOYS-AND-COMPOUNDS. OCT 27 2005; 402 (1-2) : 256-262

Laser desorption time-of-flight mass spectrometry, X-ray diffraction and Mossbauer spectroscopy methods were used for characterization of phase transformations induced in graphite during controlled reactive ball milling in hydrogen atmosphere. During milling, the crystalline structure of the graphite transformed to nanostructure as proved by X-ray diffraction studies. The hydrogen storage capacity of the nanostructured graphite depends strongly on the energy of milling process and reached 2.718 wt.% in this study. The mass spectrometry method revealed in the milling products a variety of bare carbon clusters as well as hydrogenated carbon clusters which may have from one to four hydrogen atoms. Complementary measurements performed using Mossbauer spectroscopy allowed it to recognize the crystalline phases of iron compounds observed in the X-ray diffraction patterns.



**[36] MR EFFECTS IN HALF-METALLIC CrO<sub>2</sub>/MOO<sub>2</sub> MIXTURES**

Kimishima-Y; Satoh-Y; Yamada-W; Uehara-M; Sasaki-T - IEEE-TRANSACTIONS-ON-MAGNETICS. OCT 2005; 41 (10) : 2748-2750

Mixtures of half metallic CrO<sub>2</sub> and metallic MoO<sub>2</sub> were prepared by ball milling. Specific magnetization sigma and magneto-resistance (MR) were measured for (CrO<sub>2</sub>)(1-x)(MoO<sub>2</sub>)(x) for x = 0 to 1. Above x = 0.1, sigma and MR-ratio (MRR) were monotonously decreased with increasing MoO<sub>2</sub> content. At x = 0.05, enhancements of saturated magnetization sigma(s), remanent magnetization sigma(r), and coercive force H-c were observed. Slightly increasing MRR and spin polarization coefficient P were also found at x = 0.05. These anomalous behaviors were possibly due to the difference of energy band structure of half-metallic CrO<sub>2</sub> and Pauli paramagnetic MoO<sub>2</sub> in the powder mixture system.

**[35] DYNAMIC MAGNETOELASTIC PROPERTIES OF EPOXY-BONDED TERFENOL-D PARTICULATE COMPOSITE WITH A PREFERRED [112] CRYSTALLOGRAPHIC ORIENTATION**

Or-SW; Carman-GP - IEEE-TRANSACTIONS-ON-MAGNETICS. OCT 2005; 41 (10) : 2790-2792

The dynamic magnetoelastic properties of a [112]-oriented magnetostrictive composite comprising 0.49 volume fraction of needle-shaped, [112]-oriented Terfenol-D particles and Spurr epoxy were investigated as a function of magnetic bias field. These properties included elastic moduli (E-3(H) and E-3(B)), dynamic strain coefficient (d(33)), and magnetoelastic coupling coefficient (k(33)). A randomly oriented composite with 0.51 volume fraction of irregular-shaped, ball-milled Terfenol-D particles was also prepared and characterized for comparison. It was found that the [112] preferential particulate orientation increases non-180 degrees domain-wall motion in the [112]-oriented composite, resulting in maximum negative-Delta E, d(33), and k(33) values of about 10%, 5.5 nm/A, and 0.34, respectively. These are the largest property values reported as yet for magnetostrictive particulate composites, being up to 67% larger than the randomly oriented composite and approach to 65% of the monolithic Terfenol-D.

**[34] INTERGRANULAR TUNNELING MAGNETORESISTANCE OF MECHANICALLY ALLOYED (CR-,M)O-2 POWDER COMPACTS**

Tsunoda-M; Sato-T; Zhang-Q; Jeyadevan-B; Takahashi-M - IEEE-TRANSACTIONS-ON-MAGNETICS. OCT 2005; 41 (10) : 3400-3402

The (Cr-M)O-2 (M = V, Mn, Fe, Co) powders were synthesized by the mechanochemical method and the magnetoresistance (MR) effect of their powder compacts was investigated as a function of the type and content of the dopant. The findings are summarized as follows. 1) Substituting limit of M for Cr in CrO<sub>2</sub> was 30 at% for V, Mn, and Co, and 5 at% for Fe. 2) Doping with V effectively decreases the T-C, while Fe significantly elevates the same (420 K with 5 at% Fe). Furthermore, the changes observed in the cases of Co and Mn was marginal. 3) MR ratio was significantly enhanced with the doping of 5 at% of Fe, however, decreased in the cases of V and Co doping. 4) The reason for the MR enhancement in (Cr-Fe)O-2 powder compacts is due to the change in the barrier characteristics.

**[33] MICROSTRUCTURE OF NANOCOMPOSITE R-FE-B DIE-UPSET MAGNETS (R=PR, ND) PRODUCED FROM MECHANICALLY MILLED POWDERS**

Zhang-Y; Gabay-AM; Hadjipanayis-GC - IEEE-TRANSACTIONS-ON-MAGNETICS. OCT 2005; 41 (10) : 3883-3885

Microstructure and magnetic properties of die-upset R-Fe-B magnets (R = Nd, Pr) produced from high-energy ball milled powders have been studied. The Nd-Fe-B series alloys had nominal compositions Nd<sub>x</sub>Fe<sub>93.5-x</sub>Ga<sub>0.5</sub>B<sub>6</sub> (x = 14 - 16), and the Pr-Fe-B series alloys were nominally the stoichiometric Pr<sub>12</sub>Fe<sub>81</sub>Ga<sub>1</sub>B<sub>6</sub> and Pr<sub>12</sub>Fe<sub>80</sub>Cu<sub>1</sub>Ga<sub>1</sub>B<sub>6</sub> alloys. Results show that a relatively small difference in the rare earth content results in dramatic differences in both the magnetic properties and microstructure of Nd<sub>x</sub>Fe<sub>93.5-x</sub>Ga<sub>0.5</sub>B<sub>6</sub> magnets. Small amount of Cu addition in Pr-Fe-B alloys promotes the formation of textured microstructure, leading to the increase in magnetic properties.

**[32] OBSERVATION OF EXTENDED COPPER PASSIVITY IN CARBONATE SOLUTIONS AND ITS FUTURE APPLICATION IN COPPER CMP**

Ein-Eli-Y; Abelev-E; Auinat-M; Starosvetsky-D - ELECTROCHEMICAL-AND-SOLID-STATE-LETTERS. 2005; 8 (12) : B69-B71

In the present work we report on carbonate-based solutions, which can provide copper passivity in a wide potential range with the capability for producing protective characteristics similar to Al and W. This report focuses mainly on the identification of copper passivity parameters, such as potential range, anodic current limits, current characteristics, and rates of passivation and repassivation subsequent to mechanical damage of copper passivity in carbonate-based solutions. Copper is fully passivated in sulfate solutions containing potassium carbonate. The observed passivity is more pronounced in solutions containing higher carbonate content. On the contrary, increasing the sulfate concentration has the opposite effect on copper passivity than carbonate does. Friction of the passive film by repeated surface abrading resulted in a rapid repassivation of the mechanically activated surface sites. Thus, the use of a carbonate anion as a passivating component in a future chemical mechanical planarization (CMP) slurry design should be considered.



**[31] MG ALLOY MATRIX COMPOSITE REINFORCED WITH TiNi CONTINUOUS FIBER PREPARED BY BALL-MILLING/HOT-PRESSING**

Yan-B; Li-G - COMPOSITES-PART-A-APPLIED-SCIENCE-AND-MANUFACTURING. 2005; 36 (11) : 1590-1594

Mg alloy matrix composite reinforced with TiNi continuous fiber was successfully prepared by ball-milling/hot-pressing process. No oxide was revealed from the X-ray diffraction of the ball-milled Mg alloy powders with an average crystalline size of 39 nm. A nearly full-dense matrix was achieved and the optical microscopy showed fine and homogenous matrix with undamaged TiNi continuous fibers in the composite. Remarkable interfacial reaction product or layer was not observed using a scanning electron microscope and the microhardness in the interfacial zone was higher than that of the matrix, indicating an adequate bonding was formed between the matrix and fiber. The ball-milling/hot-pressing process established in this study is reliable and easy to operate.

**[30] MACROKINETICS OF MECHANOSYNTHESIS IN SOLID-GAS SYSTEMS. I. MATHEMATICAL SIMULATION**

Smolyakov-VK; Lapshin-OV; Maksimov-YM - COMBUSTION-EXPLOSION-AND-SHOCK-WAVES. SEP-OCT 2005; 41 (5) : 554-565

A mathematical model for mechanosynthesis in solid-gas systems is constructed which includes the equations of mechanical-reactor heat balance, grinding, the excess-energy dynamics in the condensed substances, which determines the effect of mechanical activation on the chemical-reaction rate, and the reaction kinetics. A brief analysis of the model is presented. A description is given of two simplified models of independent significance derived from the general model, which can be studied by analytical methods. Synthesis in a preactivated system is studied in detail. Relations determining the characteristics of the synthesis are obtained. The accuracy of the analytical estimates is verified by numerical simulation.

**[29] MACROKINETICS OF MECHANOSYNTHESIS IN SOLID-GAS SYSTEMS. II. EXPERIMENTAL STUDIES. ANALYSIS OF RESULTS**

Smolyakov-VK; Itin-VI; Golobokov-NN; Kasatskii-NG; Lapshin-OV; Maksimov-YM; Terekhova-OG; Shkoda-OA - COMBUSTION-EXPLOSION-AND-SHOCK-WAVES. SEP-OCT 2005; 41 (5) : 566-572

Results from experimental studies of the nonisothermal mechanochemical reaction in the titanium-nitrogen system. Experimental data are compared with simulation results. The effective kinetic parameters of mechanical activation of the reactant and the chemical reaction are determined from analytical relations. It is shown that the developed mathematical model is suitable for the analysis of the macrokinetics of nonisothermal chemical reactions in solid reactant-reactive gas systems.

**[28] PREPARATION AND PROPERTIES OF Mg<sub>1.75</sub>Al<sub>0.25</sub>Ni<sub>1-x</sub>Cr<sub>x</sub>(0 ≤ x ≤ 0.2) ALLOYS**

Wang-MH; Zhang-LZ; Sun-LX; Zhang-Y; Xu-F; Tan-ZC; Yuan-HT; Zhang-T - CHEMICAL-JOURNAL-OF-CHINESE-UNIVERSITIES-CHINESE. OCT 10 2005; 26 (10) : 1877-1880

Mg<sub>1.75</sub>Al<sub>0.25</sub>Ni<sub>1-x</sub>Cr<sub>x</sub>(0 ≤ x ≤ 0.2) alloys were synthesized by diffusing method(DM) on the basis of the ternary Mg<sub>1.75</sub>Al<sub>0.25</sub>Ni alloy. The X-ray diffraction showed that the structure of the Cr-partially-substituted alloy did not differ from that of Mg<sub>1.75</sub>Al<sub>0.25</sub>Ni alloy but there was a change in the lattice parameter. It was found that the Cr-substituted alloys showed enhanced discharge capacities. The cycle life of the alloys was improved when x (Cr) reached 0.1. The anti-corrosion order was Mg<sub>1.75</sub>Al<sub>0.25</sub>Ni<sub>0.9</sub>Cr<sub>0.1</sub> > Mg<sub>1.75</sub>Al<sub>0.25</sub>Ni > Mg<sub>1.75</sub>Al<sub>0.25</sub>Ni<sub>0.8</sub>Cr<sub>0.2</sub> > Mg<sub>2</sub>Ni. The result of the charge-discharge experiment also supported the conclusion. The structure and the component of alloy phase were changed after ball milling but the discharge capacity of the Mg<sub>1.75</sub>Al<sub>0.25</sub>Ni<sub>0.9</sub>Cr<sub>0.1</sub> alloy was not enhanced.

**[27] ELECTRON MICROSCOPY CHARACTERIZATION OF MECHANICALLY ALLOYED AND HOT CONSOLIDATEDS CU-CR<sub>3</sub>C<sub>2</sub> PARTICLES**

Lopez-M; Camurri-C; Vergara-V; Jimenez-JA - REVISTA-DE-METALURGIA. JUL-AUG 2005; 41 (4) : 308-312

Mechanically alloyed copper-ceramic composites have been obtained with the purpose of studying their use as copper-based material for electrical equipment. For high-temperature applications, dispersion-strengthened copper alloys are attractive due to their excellent combination of thermal and electrical conductivity, mechanical strength retention and microstructural stability. In this work, powder mixtures of pure copper with 2 vol % Cr<sub>3</sub>C<sub>2</sub>, milled during 4, 6, 10, 12 and 15 h in a high-energy planetary balls mill under argon atmosphere, were consolidated by hot isostatic pressing, applying a pressure of 100 MPa at 1073 K for two hours, to obtain materials with a fine microstructure. The Cu-Cr<sub>3</sub>C<sub>2</sub> alloys were studied by scanning electron microscopy (SEM), electron microprobe (EPMA) and transmission electron microscopy (TEM). Mechanical properties and electrical conductivity were also studied. The average tensile strength and electrical conductivity were found to be 500 MPa and 50 % IACS, respectively. The Cr<sub>3</sub>C<sub>2</sub> ceramics show good stability during hot consolidation. Contributing to a further strengthening of the alloy during the hot consolidation, uniformly-distributed Fe-carbide particles of nanometric size precipitated in the copper matrix. Fe-Cr oxycarbides formed in the interphase between Cr<sub>3</sub>C<sub>2</sub> particles and the copper matrix cause the low ductility of Cu-Cr<sub>3</sub>C<sub>2</sub> alloys. Said particles are attributed to impurities/contamination generated from the milling process.



**[26] DEVELOPMENT AND CHARACTERIZATION OF COPPER BASE COMPOSITE MATERIALS STRENGTHENED WITH TiB<sub>2</sub> PARTICLES**

Lopez-M; Camurri-C; Corredor-D; Jimenez-JA - REVISTA-DE-METALURGIA. JUL-AUG 2005; 41 (4) : 313-318

In this work, mechanical and electrical properties of copper base composites alloys strengthened by dispersed TiB<sub>2</sub> particles are characterized. Powders of copper and TiB<sub>2</sub> were mechanically ball milled in a planetary mill during 12 and 36 h under a protective atmosphere of argon. Resulting powders were compacted uniaxially at 923 K under 90 MPa during 2 h. Part of these compacts were hot rolled at temperatures ranging between 1073 and 1023 K with 20 % reduction. Comparing the properties of materials after this thermomechanical process with that of as-consolidated, it was observed an electrical conductivity increase, and a decrease on hardness and ductility.

**[25] MICROSTRUCTURE AND MECHANICAL PROPERTIES OF NANOCRYSTALLINE TITANIUM AND Ti-TA-Nb ALLOY MANUFACTURED USING VARIOUS DEFORMATION METHODS**

Dutkiewicz-J; Kusnierz-J; Maziarz-W; Lejkowska-M; Garbacz-H; Lewandowska-M; Dobromyslov-AV; Kurzydowski-KJ - PHYSICA-STATUS-SOLIDI-A-APPLICATIONS-AND-MATERIALS-SCIENCE. SEP 2005; 202 (12) : 2309-2320

Mechanical properties and TEM microstructure studies have been carried out of nanocrystalline titanium, Ti<sub>10</sub>Nb<sub>10</sub>Ta and Ti<sub>10</sub>Nb obtained by various technological routes, including: powder metallurgy (ball milling and hot pressing), Equal Channel Angular Pressing (ECAP), hydroextrusion (HE) and high pressure torsion (HPT). The HE processed material in the form of 20 mm rods was extruded at a strain rate of  $2.5 \times 10^2 \text{ s}^{-1}$  to a diameter of 3 mm, which corresponds to the true strain of 3.8. Resulting Yield Strength (YS) at the crystal size below 80 nm exceeded 1000 MPa, i.e. attained a value of 3 times more than the initial material. Equal-Channel Angular Pressing (ECAP) at 723 K was applied to produce nanostructured titanium. Grain refinement was observed already after one pass (considerable number of grains with  $d < 100 \text{ nm}$  was noted). It was accompanied by a growth of strength and slight decrease in the elongation. ECAP processing up to 4 passes resulted in further slight growth of strength and further slight loss of elongation. The titanium powder prepared by ball milling in a high energy mill decreased its crystal size down to 10 nm and reached microhardness HV<sub>20</sub> = 1000. The additions of Nb and Ta resulted in a similar grain refinement but lower hardness. Uniaxial hot pressing at 650 degrees C, followed by vacuum annealing resulted in similar microhardness as for powders. TEM studies performed using quantitative metallography allowed to estimate mean grain size at 150 nm. HPT technique at the pressure of 5 GPa resulted in finest grain size as compared to other preparation techniques leading to nanoscale grain refinement in Ti samples. The mean crystal size was estimated at about 30 nm.

**[24] MECHANICAL PROPERTIES OF SEVERELY DEFORMED ZA-27 ALLOY USING EQUAL CHANNEL ANGULAR EXTRUSION**

Purcek-G; Altan-BS; Miskioglu-I; Patil-A - MATERIALS-SCIENCE-AND-TECHNOLOGY. SEP 2005; 21 (9) : 1044-1048

As cast ZA-27 alloy was subjected to equal channel angular extrusion (ECAE) with up to four passes using three different processing routes, and its mechanical properties (strength, hardness, ductility and extrusion load) were evaluated. The changes in the microstructure were also investigated. The ECAE was found to be quite effective in enhancing the mechanical properties of ZA-27 alloy. The strength and hardness of the alloys increased after the first ECAE pass followed by a gradual decrease with further passes for all processing routes. The elongation to failure, however, exceptionally increased with increase in the number of passes for all processing routes. Combined high strength and good ductility were obtained in the alloy after the first pass. The strength and maximum extrusion load showed similar trends with the number of passes for all processing routes.

**[23] SINTERING AND ELECTRICAL CONDUCTIVITY IN FAST OXIDE ION CONDUCTORS La<sub>2-x</sub>R<sub>x</sub>Mo<sub>2-y</sub>W<sub>y</sub>O<sub>9</sub> (R : ND, GD, Y)**

Georges-S; Goutenoire-F; Lacorre-P; Steil-MC - JOURNAL-OF-THE-EUROPEAN-CERAMIC-SOCIETY. NOV 2005; 25 (16) : 3619-3627

Attrition and ball milling are used as mechanical means to reduce grain size of optimized fast oxide-ion conductors La<sub>2-x</sub>R<sub>x</sub>Mo<sub>2-y</sub>W<sub>y</sub>O<sub>9</sub> (R: rare earths). Dilatometry is used to determine the optimal sintering conditions in order to obtain high density samples (greater than 96% of relative density) with help of scanning electron microscopy to characterize their microstructure. The optimal sintering temperatures are highly dependent on the chemical composition, and therefore identical annealing temperatures do not warrant similar relative densities. Complex impedance spectroscopy show that above the transition temperature of La<sub>2</sub>Mo<sub>2</sub>O<sub>9</sub> at 580 degrees C, the conductivity of all the studied compounds is lower than that of the parent compound, whereas just below the transition, in most cases the stabilization of the cubic phase increases conductivity. An interesting result is that tungsten substitution, which stabilizes La<sub>2</sub>Mo<sub>2</sub>O<sub>9</sub> against reduction, does not affect significantly the oxide ion conduction.

**[22] SOLID-STATE SYNTHESIS OF NEW GLASSY CO<sub>65</sub>TI<sub>20</sub>W<sub>15</sub> ALLOY POWDERS AND SUBSEQUENT**



Lettre RFM N°129 - Décembre 2005  
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#### DENSIFICATION INTO A FULLY DENSE BULK GLASS

El-Eskandarany-MS; Omori-A; Inoue-A - JOURNAL-OF-MATERIALS-RESEARCH. OCT 2005; 20 (10) : 2845-2853

The mechanical alloying method was used to synthesize a single glassy phase of Co<sub>65</sub>Ti<sub>20</sub>W<sub>15</sub> alloy powders, using a high-energy ball mill. The glass transition temperature of the end-product, which was obtained after 173 ks of milling time, lies at 786 K, whereas the crystallization takes place at 878 K through a single sharp exothermic peak with an enthalpy change of crystallization of -4.37 kJ/mol. The reduced glass transition temperature was found to be 0.51. This glassy alloy powders exhibit a very large supercooled liquid region (92 K) for a ternary metallic system. The spark plasma sintering method was used to consolidate the glassy powders under an argon gas atmosphere at 843 K with a pressure of 19.6-38.2 MPa. The sample that was consolidated within 180 s maintains its chemically homogeneous glassy structure with a relative density of above 99.6%. Neither the supercooled liquid region nor crystallization temperature was affected by such a rapid consolidation procedure. Thus, the thermal stability of the bulk glassy sample is almost identical with the original glassy powders. The Vickers microhardness of the bulk glassy Co<sub>65</sub>Ti<sub>20</sub>W<sub>15</sub> reveals high values, ranging between 8.69 and 8.83 GPa. The fabricated bulk glassy alloy shows high compressive strength of 2.44 GPa with a Young's modulus of 176.81 GPa. Neither yielding stress, nor plastic strain could be detected for this glassy alloy, which its elastic strain is 1.33%.

#### [21] ELECTROCHEMICAL PROPERTIES OF SI/Ni ALLOY-GRAPHITE COMPOSITE AS AN ANODE MATERIAL FOR LI-ION BATTERIES

Park-MS; Lee-YJ; Rajendran-S; Song-MS; Kim-HS; Lee-JY - ELECTROCHIMICA-ACTA. SEP 30 2005; 50 (28) : 5561-5567

Si/Ni alloy and graphite composites were synthesized using arc-melting followed by high energy mechanical milling (HEMM). Alloy particles comprising of NiSi<sub>2</sub>, NiSi and Si phases were distributed finely and uniformly on the surface of graphite in the composites obtained after HEMM. The composite containing 60 wt.% of Si/Ni alloy exhibited a stable capacity of similar to 780 mAh/g. Fourier transform infrared spectroscopy (FTIR) analysis confirmed that some bonds were formed between alloy and graphite after HEMM, which appeared to retain the electrical connection between alloy and graphite during cycling. X-ray diffraction (XRD) analysis indicated that NiSi<sub>2</sub> and NiSi phases, which acted as an inactive alloy matrix remained invariant during charge and discharge. In addition to NiSi<sub>2</sub> and NiSi phases, disordered graphite layers also played the role of media for the accommodation of large volume change of Si during cycling. The large reversible capacity and good cycleability showed that Si/Ni alloy and graphite composite could be an alternative to conventional graphite-based anode materials for lithium-ion secondary batteries.

#### [20] MECHANOCHEMICAL ACTIVATION OF ALUMINUM: 3. KINETICS OF INTERACTION BETWEEN ALUMINUM AND WATER

Streletskii-AN; Kolbanev IV; Borunova-AB; Butyagin-PY - COLLOID-JOURNAL. SEP-OCT 2005; 67 (5) : 631-637

Two regimes of oxidation by water are revealed for nanocrystalline aluminum prepared by the mechanical activation of its mixture with graphite and distributed in the matrix of amorphous carbon. At the temperatures 50 degrees C < T < 90 degrees C, nanosized aluminum particles interact with water under quasi-isothermal conditions. The main products are hydrogen and pseudoboehmite AlOOH; a low content of bayerite Al(OH)<sub>3</sub> is also formed. After the induction period, the kinetics of interaction can be satisfactorily described by the law of a diminishing sphere. The effective activation energy of the reaction is equal to 61 +/- 10 kJ/mol and is identical for the samples of submicron aluminum prepared by different procedures. At temperatures above 90-95 degrees C, the oxidation of mechanically activated aluminum by water is transformed into a thermally self-accelerated explosion process. Under these conditions, the oxidation of aluminum to alpha-Al<sub>2</sub>O<sub>3</sub> is accompanied by an exothermal reaction between the metal and the carbon matrix during which aluminum carbide Al<sub>4</sub>C<sub>3</sub> is formed.

#### [19] CALORIMETRIC INVESTIGATION ON MECHANICALLY ACTIVATED STORAGE ENERGY MECHANISM OF SPHALERITE AND PYRITE

Xiao-ZL; Chen-QY; Yin-ZL; Hu-HP; Wu-DX - THERMOCHIMICA-ACTA. OCT 1 2005; 436 (1-2) : 10-14

The structural changes of mechanically activated sphalerite and pyrite under different grinding conditions were determined by X-ray powder diffraction (XRD), laser particle size analyzer and elemental analysis. The storage energy of mechanically activated sphalerite and pyrite was measured by a calorimetric method. A thermochemical cycle was designed so that mechanically activated and non-activated minerals reached the same final state when dissolved in the same oxidizing solvent. The results show that the storage energy of mechanically activated sphalerite and pyrite rises with increased in grinding time, and reaches a maximum after a certain grinding period. The storage energy of mechanically activated pyrite decreases when heated under inert atmosphere. The storage energy of mechanically activated sphalerite and pyrite remains constant when treated below 573 K under inert atmosphere. The percentage of the storage energy caused by surface area increase during mechanical activation decreases with increasing grinding time. These results support our opinion that the mechanically activated storage energy of sphalerite is closely related to lattice distortions, and the mechanically activated storage energy of pyrite is mainly caused by the formation of reactive sites on the surface.



**[18] EFFECTS OF THE SYNTHESIS PROCEDURE, DOPING AND NON-STOICHIOMETRY ON THE ORDER-DISORDER TRANSFORMATION IN  $\text{Ln}_2\text{Ti}_2\text{O}_7$  (Ln=Tm-Lu) OXYGEN-ION CONDUCTORS**

Shlyakhtina-AV; Knotko-AV; Boguslavskii-MV; Stefanovich-SY; Peryshkov-DV; Kolbanev IV; Shcherbakova-LG - SOLID-STATE-IONICS. SEP 2005; 176 (29-30) : 2297-2304

The ionic conductivity of the  $\text{Ln}_2\text{Ti}_2\text{O}_7$  (Ln=Tm-Lu) pyrochlores is found to correlate with the degree of anti-site cation disordering in these materials. The highest ionic conductivity is exhibited by  $\text{Ln}_2\text{Ti}_2\text{O}_7$  containing 3.53-4.5% Ln(Ti)+Ti-Ln anti-structure pairs. This degree of cation disordering can be achieved by heat treatment just below the melting point of the material, by mechanical activation with subsequent high-temperature firing and by doping with small amounts of cations of different sizes (as, e.g., in the case of  $(\text{Yb}_{0.91}\text{Sc}_{0.09})_2\text{Ti}_2\text{O}_7$ ). The Ln(Ti)+Ti-Ln anti-structure pairs seem to interact with oxygen vacancies. The defect structure of  $\text{Lu}_{2+x}\text{Ti}_{2-x}\text{O}_{7-x/2}$  (34.5-44 mol%  $\text{Lu}_2\text{O}_3$ ) oxygen-ion conductors is investigated. The materials with  $x=0.052$  (34.5 mol%  $\text{Lu}_2\text{O}_3$ ); 0.096 (35.5 mol%  $\text{Lu}_2\text{O}_3$ ); 0.286 (40 mol%  $\text{Lu}_2\text{O}_3$ ) contain the whole one type of anti-site defect, Lu-Ti, and possess an ionic conductivity of about  $10^{-3}$  S/cm at 740 degrees C. The conductivity of the materials containing Lu-Ti defects is lower than that of the stoichiometric materials containing Lu-Ti + Ti-Ln anti-structure pairs. A strong correlation is found between the variable positional parameter X of O(2) (position 480 in the structure of  $\text{Lu}_{2+x}\text{Ti}_{2-x}\text{O}_{7-x/2}$ , Ti-2(-), O $_{7-x/2}$  ( $x=0, 0.052, 0.096, 0.286$ ) nanoceramics and the I-311/I-222 and I-331/I-222 intensity ratios in their X-ray diffraction patterns.

**[17] HIGH-STRENGTH PM ALUMINIUM BY MILLING IN AMMONIA GAS AND SINTERING**

Cintas-J; Cuevas-FG; Montes-JM; Herrera-EJ - SCRIPTA-MATERIALIA. NOV 2005; 53 (10) : 1165-1170

High-strength Al has been produced by attrition milling in ammonia gas atmosphere and powder consolidation by cold pressing and sintering. Consolidated compacts reach full densification, have an almost nanocrystalline grain (size 200 nm), and abundant nanocrystalline dispersoids. The bulk material has a high tensile strength (515 MPa) and its high-temperature behaviour is outstanding.

**[16] MECHANOCHEMICAL ACTIVATION OF STRONTIUM BISMUTH TANTALATE SYNTHESIS**

Chew-CL; Srinivas-A; Sritharan-T; Boey-FYC - SCRIPTA-MATERIALIA. NOV 2005; 53 (10) : 1197-1199

Strontium bismuth tantalate is usually synthesized by solid state reaction of component oxide mixtures at temperatures above 1000 degrees C. This report shows that prior mechanochemical activation in a high energy ball mill reduces the synthesis temperature to about 650 degrees C. During milling, the minor constituent raw materials appear to dissolve in the major constituent  $\text{Bi}_2\text{O}_3$

**[15] LARGE ENHANCEMENT IN MECHANICAL PROPERTIES OF THE 6061 AL ALLOYS AFTER A SINGLE PRESSING BY ECAP**

Kim-JK; Kim-HK; Park-JW; Kim-WJ - SCRIPTA-MATERIALIA. NOV 2005; 53 (10) : 1207-1211

The equal-channel angular pressed (ECAPed) 6061 Al alloys processed with various pre- and post-ECAP heat treatments were compared in terms of the balance between strength and tensile ductility, and it was concluded that the post-ECAP low-temperature aging treatment was most effective in improving the strength whilst retaining a moderate level of tensile ductility.

**[14] STRUCTURAL AND PHASE TRANSFORMATIONS DURING ISOTHERMAL ANNEALING OF MECHANICALLY ALLOYED IRON-AMORPHOUS FE-C PHASE NANOCOMPOSITE: FORMATION OF CEMENTITE**

Elsukov-EP; Fomin-VM; Vytovtov-DA; Dorofeev-GA; Zagainov-AV; Arsent'eva-NB; Lomaeva-SF - PHYSICS-OF-METALS-AND-METALLOGRAPHY. SEP 2005; 100 (3) : 251-269

X-ray diffraction, Mossbauer spectroscopy, atomic force microscopy, differential thermal analysis, and magnetic measurements have been used to study structural phase transformations during isothermal annealing at 250,400, and 500 degrees C of an iron-amorphous-Fe-C-phase nanocomposite obtained by mechanically alloying a mixture of Fe and C powders taken in a an atomic proportion of 85:15. Three stages of transformations have been established: structural relaxation and an increase in the concentration of C in the amorphous phase to 25 at. %, crystallization of the amorphous phase with the formation of distorted cementite  $(\text{Fe}_3\text{C})(d)$ , and the formation of an undistorted cementite  $\text{Fe}_3\text{C}$ . The specific features of the local atomic structure of the two modifications of cementite are discussed in some detail.

**[13] ROLE OF STRAIN-INDUCED ANISOTROPY ON MAGNETIC ENHANCEMENT IN MECHANICALLY ALLOYED  $\text{Co}_0.2\text{Zn}_0.8\text{Fe}_2\text{O}_4$  NANOPARTICLE**

Bhowmik-RN; Ranganathan-R; Nagarajan-R; Ghosh-B; Kumar-S - PHYSICAL-REVIEW-B. SEP 2005; 7209 (9) : NIL\_398-NIL\_407

Mechanically alloyed  $\text{Co}_0.2\text{Zn}_0.8\text{Fe}_2\text{O}_4$  nanoparticle has shown the enhancement of both magnetization and ferrimagnetic order which seems to be unusual according to the existing core-shell model of ferrimagnetic nanoparticle. Many concurrent effects such as site exchange of cations among A and B sites, and reduction of B site spin canting have been identified for



such unusual magnetic enhancement. We do observe the usual superparamagnetic relaxation effect for the smaller particles. The annealing of the alloyed nanoparticle results in grain growth kinetics and the appearance of mixed magnetic state, similar to the bulk material, for larger particles. In addition, large strain induced anisotropy of as-alloyed nanoparticle sharply decreases during thermal activated grain growth process and anisotropy remains almost constant for larger particles. The present work is focused on the effect of strain-induced anisotropy to control grain boundary magnetic contributions and subsequently, over all magnetization in mechanically alloyed  $\text{Co}_{0.2}\text{Zn}_{0.8}\text{Fe}_2\text{O}_4$  nanoparticles.

#### [12] ARRANGEMENTS OF MAGNETIC MOMENTS IN NANOCRYSTALLINE $\text{Fe}_{48}\text{Al}_{52}$

Szymanski-K; Satula-D; Dobrzynski-L; Voronina-E; Yelsukov-EP; Miyanaga-T - PHYSICAL-REVIEW-B. SEP 2005; 7210 (10) : NIL\_355-NIL\_366

Combined techniques, EXAFS, magnetization, and Mossbauer polarimetry are used to investigate the orientation of Fe magnetic moments in a  $\text{Fe}_{48}\text{Al}_{52}$  disordered alloy prepared by mechanical grinding. Local Fe magnetic moments and their contributions to the net magnetization at selected external fields and temperatures were estimated. It was found that components of the Fe magnetic moments parallel to the net magnetizations reduce their values much faster with an increasing number of neighboring Al atoms than the total iron moments. Data analysis indicates that magnetic moments of Fe atoms surrounded by (7Al+1Fe) in the first coordination shell and by (1Al+5Fe) in the second coordination shell possess nonzero magnetic moments that form a noncolinear structure.

#### [11] EFFECT OF FABRICATION METHOD ON MICROSTRUCTURE AND PROPERTIES OF $\text{Al}_2\text{O}_3$ -TiC COMPOSITES

Zhang-YF; Wang-LJ; Jiang-W; Bai-GZ; Chen-LD - MATERIALS-TRANSACTIONS. SEP 2005; 46 (9) : 2015-2019

$\text{Al}_2\text{O}_3$ -TiC composite powders were prepared by high-energy ball milling (HEBM) and wet-planetary ball milling (WPBM) from different starting materials, and subsequently sintered by Spark Plasma Sintering (SPS). The effect of the fabrication method on the microstructure and properties of  $\text{Al}_2\text{O}_3$ -TiC composites was investigated. The results showed that  $\text{Al}_2\text{O}_3$ -TiC composites with fine and homogeneous microstructures were prepared by SPS from high-energy ball milled reactants, which had excellent comprehensive properties: bending strength of 944 +/- 21 MPa, Vickers hardness of 21.0 +/- 0.3 GPa, fracture toughness of 3.87 +/- 0.20  $\text{MPa}\cdot\text{m}^{1/2}$ , and electrical conductivity of  $1.2787 \times 10^5 \text{ S}\cdot\text{m}^{-1}$ . The relationship between microstructure and properties and the strengthening and toughening mechanisms was discussed. In comparison with the traditional WPBM route, HEBM was a favorable technique used to produce a high homogeneous microstructure and promising mechanical and electrical conduction properties

#### [10] PREDICTION AND EXPERIMENTAL TESTING OF SPHERICAL MILLING MEDIA WEAR RATE

Zhong-LY; Wu-BL; Zhang-LM; Fang-F; He-XY - MATERIALS-TRANSACTIONS. SEP 2005; 46 (9) : 2036-2040

This paper presents a conceptually simple derivation of grinding media mass wear rate model. An experiment of milling one media in ball mill was designed to reveal the relation between media mass wear rate and the contacting points of the media with the other media and/or mill wall and the pressure acting on the point. It was found that mass wear loss of spherical grinding media is proportion to the media contacting points with other media and/or mill wall and the pressure acted on it. A physical model:  $f = Am(-1/3)t(-1) + b$  was established by theory derivation and experiment testing. Two kinds of ceramic ball grinding media, 97 porcelain media and 75 porcelain media, were prepared. The mass wear rate of the two kinds of media was determined by intergrinding with ordinary corundum milling media in ball mill. The experimental results were equated using least square method. The mass wear rate equating results of both kinds of ceramic grinding media was almost the same as the experimental results indicating the accuracy of the model. It is found that the higher the media performance was, the more accuracy the media wear rate as predicted by the model. The possibility of using this model in practical operations was also discussed in this paper.

#### [9] OXIDATION RESISTANCE OF BOILER STEELS WITH $\text{Al}_2\text{O}_3$ - $\text{Y}_2\text{O}_3$ NANO- AND MICRO-COMPOSITE COATINGS PRODUCED BY SOL-GEL PROCESS

Yao-MM; He-YD; Zhang-W; Gao-W - MATERIALS-TRANSACTIONS. SEP 2005; 46 (9) : 2089-2092

$\text{Al}_2\text{O}_3$ - $\text{Y}_2\text{O}_3$  nano- and micro-composite coatings have been deposited on boiler steel substrates using sol-gel composite coating technology. The processes include dipping samples in a sol-gel solution dispersed with fine ceramic powders, which is prepared by high-energy ball milling. Scanning electron microscopy (SEM) and XRD analyses show that the one-layer coating (i.e. dipping for one time) has a thickness of more than 2  $\mu\text{m}$ . The coating is mainly composed of  $\alpha$ - $\text{Al}_2\text{O}_3$  and  $\gamma$ - $\text{Al}_2\text{O}_3$ , and is relatively dense without cracking after drying and sintering treatments. The oxidation tests performed in air at 600 degrees C show that the coatings possess much improved resistance to high temperature oxidation and scale spallation. It is believed that the nano-structured composite particles and reactive elements are integrated into the coatings, which played an important role in improving their oxidation resistance.

#### [8] ON IRON CONTAMINATION IN MECHANICALLY ALLOYED CR-SI POWDERS

Fernandes-BB; Rodrigues-G; Coelho-GC; Ramos-AS - MATERIALS-SCIENCE-AND-ENGINEERING-A-STRUCTURAL-



MATERIALS-PROPERTIES-MICROSTRUCTURE-AND-PROCESSING. SEP 25 2005; 405 (1-2) : 135-139

The present work reports on iron contamination and phase transformation during high-energy ball milling from high-purity elemental powder Cr-25Si, Cr-37.5Si, Cr-50Si, and Cr-66Si mixtures (at%) and their subsequent heat treatment. Samples were characterized in the as-milled state as well as after heat treatment by X-ray diffraction (XRD), scanning electron microscopy (SEM), and microanalysis via energy dispersive spectrometry (EDS). Only Cr peaks were observed in Cr-25Si and Cr-37.5Si powders after milling for 200 h, suggesting that amorphous phases can be formed. In Cr-50Si and Cr-66Si powders, the CrSi and CrSi<sub>2</sub> phases were formed during ball milling, respectively. In Cr-25Si and Cr-50Si milled powders, heat-treated at 1200 degrees C for 4h, the formation of the Cr<sub>3</sub>Si and CrSi phases dissolving up to 15 and 16.9 at%Fe, respectively, was noted. In addition, the ternary Cr<sub>3</sub>Fe<sub>2</sub>Si(2) phase was also formed in heat-treated Cr-25Si powders. A small amount of Cr<sub>5</sub>Si<sub>3</sub> and CrSi<sub>2</sub> was formed in heat-treated Cr-37.5% and Cr-66Si powders, respectively.

#### [7] ELECTROCHEMICAL PROPERTIES OF COSB<sub>3</sub>(-MCMB) ANODES FOR LITHIUM-ION BATTERIES

Zhang-LJ; Zhao-XB; Xia-DG - MATERIALS-LETTERS. NOV 2005; 59 (27) : 3448-3451

Cobalt antimonide, CoSb<sub>3</sub>, has been prepared by vacuum melting and ball-milling. The electrochemical cycling behaviors of CoSb<sub>3</sub> were evaluated using lithium anode model cell Li vertical bar LiPF<sub>6</sub> (EC+DMC) vertical bar CoSb<sub>3</sub>. The reversible capacities of CoSb<sub>3</sub> intermetallic electrode reached 570 and 290 mAh g<sup>-1</sup>, respectively for the first cycle and the twentieth cycle. It was demonstrated that cycle stability of CoSb<sub>3</sub> could be largely improved by milling after mixing with mesocarbon microbeads. There were 50.9% and 77.5% retention of the initial capacity at the twentieth cycle, respectively for CoSb<sub>3</sub> and CoSb<sub>3</sub>-MCMB hybrid.

#### [6] FULLERENE-LIKE STRUCTURES AS INTERACTION SITES BETWEEN CARBON BLACK AND RUBBER

Cataldo-F - MACROMOLECULAR-SYMPOSIA. AUG 2005; 228 : 91-98

Ball-milling of N660 carbon black and graphite causes a deep activation of its surface activity which can be measured by a significant increase in the bound rubber level and in the amount of grafted rubber in comparison to the pristine untreated samples. The bound rubber measurement has been done also on a natural rubber masterbatch filled with extracted fullerene carbon black (EFCB). Also in this case extremely high levels of rubber grafting have been achieved in comparison to pure untreated graphite. It is discussed and demonstrated that the fullerene-like nanostructures in carbon blacks play a key role in the formation of bound rubber phenomenon and in grafting natural rubber on carbon black surface.

#### [5] SOLID STATE SYNTHESIS AND THERMOELECTRIC PROPERTIES OF MG-SI-GE SYSTEM

Song-RB; Liu-YZ; Aizawa-T - JOURNAL-OF-MATERIALS-SCIENCE-AND-TECHNOLOGY. SEP 2005; 21 (5) : 618-622

Thermoelectric materials, Mg<sub>2</sub>Si<sub>1-x</sub>Ge<sub>x</sub> (x=0, 0.2, 0.4, 0.6, 0.8, 1), have been prepared by bulk mechanical alloying (13MA) and hot pressing (HP). The electrical conductivity, Seebeck coefficient and thermal conductivity were measured from room temperature up to about 700 K. The electrical conductivity of all the samples increases with increasing temperature, while the Seebeck coefficient and thermal conductivity decrease with increasing temperature. Mg<sub>2</sub>Si and Mg<sub>2</sub>Si<sub>0.8</sub>Ge<sub>0.2</sub> possess negative type of conductivity, while for other compounds it is positive. At the same time, the effect of hot processing condition on thermoelectric properties was also investigated. The maximum figure of merit of Mg<sub>2</sub>Si Ge<sub>0.6</sub>(0.4) was obtained with the processing parameter of BMA at 600 cycles and hot pressing at 773 k and 1 GPa for 1 h.

#### [4] RECYCLING PROCESS OF WC-CO CERMETS BY HYDROTHERMAL TREATMENT

Kojima-T; Shimizu-T; Sasai-R; Itoh-H - JOURNAL-OF-MATERIALS-SCIENCE. OCT 2005; 40 (19) : 5167-5172

Hydrothermal extraction process of Co binder phase from WC-Co cermet was investigated in order to establish a novel recycling system of WC-Co cermet scraps. When the cermet chips were hydrothermal-treated in hydrochloric acid above 110 degrees C, Co binding phase was efficiently extracted and the cermet chips were disintegrated into relatively large fragments. After hydrothermal treatment, WC sintered body become very brittle and pulverized easily by ball milling and the mean particle size of thus obtained WC particle became similar to that of virginal WC particle. The recycled WC powder was a little easier to undergo oxidation than the virginal WC powder, so that the mechanical properties of recycled WC-Co cermets were degraded. However, the degradation of mechanical properties was prevented only by drying the WC powder more carefully. This hydrothermal process will be one of the recycling systems for WC-Co cermets

#### [3] DOPPLER BROADENING MEASUREMENTS OF THE ELECTRON-POSITRON ANNIHILATION RADIATION IN NANOCRYSTALLINE ZrO<sub>2</sub>

Chakrabarti-M; Bhowmick-D; Sarkar-A; Chattopadhyay-S; Dechoudhury-S; Sanyal-D; Chakrabarti-A - JOURNAL-OF-MATERIALS-SCIENCE. OCT 2005; 40 (19) : 5265-5268

ZrO<sub>2</sub> powders have been ground by ball mill grinder to achieve the particle size down to 10 nm. Typical defects introduced during ball mill grinding have been studied by positron annihilation lifetime measurement technique and coincidence Doppler broadened positron annihilation radiation spectroscopic technique. Coincidence Doppler broadened positron annihilation spectra for ball mill ground and unground ZrO<sub>2</sub> samples have been analyzed by constructing ratio curve with defects free Al



single crystal. Results indicate an increase of cation defects in ZrO<sub>2</sub> samples due to the reduction of particle size by the ball mill grinding process

#### [2] IN SITU FORMATION OF BN NANOTUBES DURING NITRIDING REACTIONS

Yu-J; Chen-Y; Wuhler-R; Liu-ZW; Ringer-SP - CHEMISTRY-OF-MATERIALS. OCT 4 2005; 17 (20) : 5172-5176

High-yield multiwalled boron nitride (BN) nanotubes have been produced using a ball milling-annealing method. The BN nanotubes with a diameter less than 10 nm and a well-crystallized multiwalled structure were formed via an in situ nitriding reaction. The systematic investigation of the formation process at different annealing temperatures and for different times suggested that the formation of the unique multiwalled structure was attributed by a two-dimensional growth of the BN phase and a nonmetal catalytic growth.

#### [1] DRY GRINDING KINETICS OF BINARY MIXTURES OF CERAMIC RAW MATERIALS BY BOND MILLING

Ipek-H; Ucbas-Y; Yekeler-M; Hosten-C - CERAMICS-INTERNATIONAL. 2005; 31 (8) : 1065-1071

The kinetics of batch dry grinding of binary mixtures of ceramic raw materials, namely quartz-kaolin, quartz-potassium feldspar and kaolin-potassium feldspar, from the feed sizes of -3.350 + 2.360, -2.000 + 1.400, -0.850 + 0.600, -0.500 + 0.355 and -0.300 + 0.212 mm have been determined using a Bond mill with a mixture of ball sizes of 38.10, 31.75, 25.40, 19.05 and 12.70 mm diameter and total mass of 22.648 kg. The Bond mill used was a size of 30.5 cm diameter, 30.5 cm length, with a total volume of 22,272 cm<sup>3</sup>. The fractional ball filling was 22% of mill volume and the mill speed was 70 rpm. The breakage parameters were obtained for those mineral mixtures to predict the product size distributions. As the feed sized given above, which were ground in the mill, increase, the specific rate of breakage (S-i) values also increase, which means faster breakage with higher Si value occurs in the order of quartz-kaolin, quartz-potassium feldspar and kaolin-potassium feldspar mixtures when comparing the characteristic alpha values (slope of S-i versus size relationship with higher alpha value). The cumulative breakage distribution function (B-ij) values obtained for these mineral mixtures were slightly different in terms of the fineness factor, gamma. This means that quartz-potassium feldspar mixture produced less fines with higher gamma value, while kaolin-potassium feldspar gave more fines with lower gamma value. The simulations of the product size distribution for these mixing were very close to the experimental data. Finally, slowing down effect, treated with false time concept, started earlier than the expected for these binary mixtures. There were some correlations found between the simulated time (theta) and experimental time (t).

