

# RESEAU FRANÇAIS DE MECANOSYNTHESE

## Lettre N°67

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**Octobre 2000**

**181** Groupes de Recherche  
(dont 108 à l'étranger / 32 Pays)

**Bureau : E. Gaffet (Président), G. Le Caër (Secrétaire Général), A.R. Yavari (Trésorier)**

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**L. Takacs** - Dpt Physics - Univ. Maryland - USA

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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'99 et quelques éléments mis à jour régulièrement  
concernant les derniers résultats dans ce domaine.

# JRFM'2001

21 et 22 Mai 2001 - Amiens - France

## Thème 2001 :

Influence de la mécanosynthèse sur les propriétés physico - chimiques des matériaux

## Contact :

### Local Committee

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## Mailing Address:

JRFM'2001  
Laboratoire de Réactivité et Chimie des Solides  
Faculté des Sciences  
Université de Picardie Jules Verne  
33 Rue de Saint Leu  
80000 Amiens - France

## Conférences (liste provisoire)

E. Gaffet

"Nanomateriaux : Mécanosynthèse et Activation Mécanique"

CNRS - UMR 5060 "Métallurgies et Cultures" - Groupe "Nanomatériaux"

Université de Technologie de Belfort Montbéliard (UTBM) - 90010 Belfort Cedex

Sophie Galdeano

Etude des corrélations entre les conditions de broyage et la nanostructure dans le composé magnéto-résistif

$\text{Cu}_{80}(\text{Fe}_{0.3}\text{Co}_{0.7})_{20}$

S. Galdeano\*, M-H. Mathon\*, L. Chaffron\*\* et C-H. de Novion\*

\*Laboratoire Léon Brillouin (CEA/CNRS), CEN Saclay, 91191 Gif-sur-Yvette

\*\* DTA/DECM/Section de Recherches de Métallurgie Physique, CEN Saclay, 91191 Gif-sur-Yvette

Sylvie Begin-Colin

"Mécanismes et modélisation de transformations de phase induites par broyage dans  $\text{TiO}_2$ "

T. Giroit, S. Bégin-Colin, G. Le Caër, F. Radjai, X. Devaux

Laboratoire de Science et Génie des Matériaux Métalliques Ecole des Mines

54 042 Nancy Cedex

J.M. Le Breton

"Broyage réactif sous hydrogène d'alliages Nd-Fe-B : destruction et recombinaison de la phase  $\text{Nd}_2\text{Fe}_{14}\text{B}$ "

J.M. Le Breton, G. Khelifati, L. Aymard et J. Teillet

Laboratoire de Magnétisme et Applications - Groupe de Physique des Matériaux

UMR CNRS 6634 Faculté des Sciences de Rouen - 76821 MONT SAINT AIGNAN CEDEX

Luc Aymard

Graphite broyé sous hydrogène ou sous oxygène: propriétés électrochimiques

F. Chevallier, L. Aymard

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**Technical Announcement**

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**M.B.N.srl**  
via Roma, 4  
I-31020 San Vendemiano (TV) - Italie

- **MBN srl**, based in Italy, announces the availability of laboratory (**kg range**) as well as industrial quantities (**ton range**) of materials produced by **mechanosynthesis**.
- More information are available at the Web site: <<http://www.mbn.it/>> .
- **Batches on-demand could be considered**

**Contact** : E-mail: [info@mbn.it](mailto:info@mbn.it)

**Web site**: <http://www.mbn.it>

**Phone Number** : +39 0422 718956

**Fax Number** : +39 0422 718964

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<p><b>VARIO-PLANETARY MILL "pulverisette 4"</b></p> <p>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. Due to the high flexibility available for selecting the grinding parameters, it is possible to achieve results unattainable with any other ball mills.</p> <p>This is 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.</p> <p>When particles &lt; 10 mm are fed in, a final fineness up to 0.1 µm can be achieved. The useful capacity is between 2 x 30 ml in the case of 80 ml grinding bowls and 2 x 125 ml when 250 ml grinding bowl are used.</p> <p><b>Method of operation:</b></p> <p>With standard planetary ball mills the grinding bowls are rotating and mounted eccentrically on a rotating support disc. The rotational speed of the supporting disc can be selected at will; the grinding bowl rotates at a fixed transmission ratio.</p> <p>Due to the overlapping of grinding bowls and supporting disc, the material to be ground and the grinding balls execute movements and trajectories in the grinding bowl, which are defined by the transmission ratio.</p> <p>With the "pulverisette 4" vario-planetary mill the rotational speeds of grinding bowls and supporting disc can be adjusted completely independently of each other. By varying the transmission ratio it is possible to control the movements and trajectories of the grinding balls at will so that the balls strike the inner wall of the bowl vertically (high impact energy), approach each other tangentially (high friction) or just roll down the inner wall of the bowl (centrifugal mills).</p>	<p>All intermediate levels and combinations of frictional and impact pressures can be set as required. By changing the transmission ratio it is therefore possible for the first time to carry out mechanical activation as well as mechanical alloying.</p> <p>Furthermore, it is also possible for the first time to optimally adjust a planetary ball mill to the material to be ground, the size of the grinding bowls and the grinding balls.</p> <p><b>Features of performance:</b></p> <ul style="list-style-type: none"><li>• for the first time, all grinding parameters can be selected at will for optimal preparation of sample</li><li>• Programming of the grinding parameters by PC software as desired</li><li>• RS232 interface for programming and to transfer grinding parameters to the PC</li><li>• Real-time display of the speeds to monitor the grinding process</li><li>• Reversing option (direction of rotation reversed periodically) to improve the grinding results</li><li>• Emulation of various ball mills</li><li>• Variably adjustable pressure on sample (friction and/or impact)</li><li>• Final fineness &lt;&lt; 1 µm</li><li>• Simultaneous grinding in up to 4 small or 2 large grinding bowls</li><li>• Quick, secure fastening of the grinding bowls</li><li>• Ease of cleaning</li></ul> <p><b>contact:</b> Fritsch GmbH (Andrea Köhler- Manufacturers of Laboratory Instruments Industriestrasse 8 D-55743 Idar-Oberstein Phone: ++49/ 67 84/70-46 Fax: ++49/ 67 84/70-11 E-Mail: info@fritsch.de Internet: <a href="http://www.fritsch.de">http://www.fritsch.de</a></p>
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**ANNONCE DE CONGRES ET / OU ECOLES  
CONGRESS AND SCHOOL ANNOUNCEMENTS**

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**J2IM 2000**

**“Joints Intergranulaires et Interphases  
dans les Matériaux“**

Fontainebleau - EDF (Site des Renardières)

4 - 6 Octobre 2000

<http://perso.club-internet.fr/adjr/j2im.html>

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**Journées d'Automne 2000 / SF2M**

Maison de la Chimie à Paris

du 17 au 19 octobre 2000

e\_mail : [sfm@wanadoo.fr](mailto:sfm@wanadoo.fr)

Website : [www.sf2m.asso.fr](http://www.sf2m.asso.fr)

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**Nanomatériaux :  
Vers les Applications Industrielles  
Nanomaterials :  
Towards Engineering Applications**

Colloque : France - Etats Unis - Canada

22 - 25 Octobre 2000 - Montréal, Canada

Contacts : [Champion@glvt-cnrs.fr](mailto:Champion@glvt-cnrs.fr) et/ou [Eric.Gaffet@utbm.fr](mailto:Eric.Gaffet@utbm.fr)

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**PM 2000**

**Powder Metallurgy World  
Congress & Exhibition**

12 - 16 Novembre 2000 Kyoto - Japon

Contact : Fax : 81 - 3 - 3423 - 1600

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**The 1st International Conference on Advanced Materials Processing  
Rotorua, New Zealand, 19-23 November 2000.**

Secretary, ICAMP 2000,

Department of Materials and Process Engineering The University of Waikato

Private Bag 3105, Hamilton, New Zealand

Fax: 64-7-838 4835, e-mail: [d.zhang@waikato.ac.nz](mailto:d.zhang@waikato.ac.nz)

Or visit the conference web site:

<http://mape.waikato.ac.nz/conferences/icamp>

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**"Scattering Studies of Mesoscopic Scale Structure and Dynamics in Soft Matter"**

Messina Italy

22th to 25th of November 2000

(for details see LRFM60)

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**2000 MRS Fall Meeting**

27 Nov - 1 Dec. 2000

Boston - Massachusetts - USA

(for details see LRFM60)

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**International Conference on Trends in Mechanical Alloying Science, Technology and  
Applications (TMA-2001)**

**Janpur, India Feb. 21-23, 2001.**

Secretariat of Int. Conf. TMA-2001

Organizer : Dr. P. R. Soni

Department of Metallurgical Engineering,

Malaviya Regional Engineering College

Jaipur-302017, India

Tel : +91-0141-702042

Fax : +91-0141-702954

E-mail : [psmt@arya.recjai.ernet.in](mailto:psmt@arya.recjai.ernet.in)

Contact Persons : Dr. P. R. Soni - Prof. T. V. Rajan

**Particles 2001**  
24-27 February 2001  
Rosen Center Hotel, Orlando, FL  
<http://www.nanoparticles.org>

**NOUVEAU**

**Co-Sponsored by:**

The Division of Colloid and Surface Chemistry of the American Chemical Society  
The Particle Technology Forum of the American Institute of Chemical Engineers  
The International Fine Particles Research Institute

**Additionally Supported by:**

The Army Research Office  
Marcel Dekker, Inc.

An interdisciplinary international conference focused on particle formation, particle characterization, and particle-based materials synthesis. Application areas span chemical analysis, imaging, printing, pharmaceuticals, coatings, membranes, filters, composites, catalysts, electronics, and optics.

**Plenary Speakers**

Bob Langer (MIT): Smart Particles for Drug Delivery  
Markus Antonietti (MPI Golm): Structured Nanocomposites: The Use of Polymers to Shape, Mold, Handle, and Connect Inorganic Nanostructures  
Arjun Yodh (U Pennsylvania): One, Two, and Three-Dimensional Colloidal Materials

Abstract and Registration Details at <http://www.nanoparticles.org>

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**Science et Technologie des Poudres**

Nancy - France  
3 - 5 Avril 2001

website : <http://www.inpl-nancy.fr/stpoudres3.html>  
e-Mail : [stpoudres@inpl-nancy.fr](mailto:stpoudres@inpl-nancy.fr)

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**PM2 TEC2001**

**2001 International Conference on Powder Metallurgy  
& Particulate Materials**

13 - 17 May 2001 - New Orleans - USA  
Contact : MPIF

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**JRFM'2001**

21 et 22 Mai 2001 - Amiens - France

**Thème :** Influence de la mécanosynthèse sur les propriétés physico - chimiques des matériaux  
Contact : [Luc.Aymardsc.u-picardie.fr](mailto:Luc.Aymardsc.u-picardie.fr)  
ou [Eric.Gaffet@utbm.fr](mailto:Eric.Gaffet@utbm.fr)

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**7th International Symposium on  
Agglomeration**

29, 30, 31 May 2001  
Albi - France

Website : <http://www.univ-inpt.fr/~agglom>  
or <http://www.enstimac.fr>

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**ISMANAM2001**

**University of Michigan, Ann Arbor, Michigan, USA  
on 24-30 June 2001.**

Abstract deadline is March 1st, 2001.

Abstracts of 200-400 words should be submitted by e-mail to

**E-Mail :** [ISMANAM-2001@umich.edu](mailto:ISMANAM-2001@umich.edu)

**Web site :** <http://www-ners.engin.umich.edu/ISMANAM2001>.

**NOUVEAU**

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**COLLOQUE SUR LES INNOVATIONS  
DANS LES MATERIAUX FRITTES**

Poitiers-Futuroscope  
3-4-5 juillet 2001

consulter le site [www.sf2m.asso.fr](http://www.sf2m.asso.fr) (rubriques sommaires puis conférences).

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**SOUTENANCES DE THESE**  
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**N. LORRAIN**

**Poudres Nanocomposites Ag - SnO<sub>2</sub> préparées par broyage réactif : mise en oeuvre, frittage et évolution microstructurale**

**30 Octobre 2000 - Université de Grenoble I**

**Jury :** E. Gaffet (Rapp.), F. Thévenot (Rapp.), D. Bouvard, G. Le Caër, C. Carry (Dir. Thèse), L. Chaffron

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**J.Ph. BRAGANTI**

**Synthèse d'alliages amorphes Al-Ni-Zr par broyage mécanique :**  
- étude de la cinétique de cristallisation par calorimétrie,  
- analyse du chemin réactionnel par diffraction des rayons X

**20 octobre 2000 – Université Henri Poincaré – Nancy I – Vandoeuvre lès Nancy**

**Jury :** C. Bergman (Rapp.), J.P. Bros (Rapp.), J.M. Moreau, S. Colin-Bégin, J.C. Gachon, F.A. Kuhnast (Dir)

Le travail de cette thèse est consacré à l'étude de la cristallisation d'alliages amorphes binaires ou ternaires Al-Ni-Zr préparés par broyage mécanique. Ces alliages amorphes sont caractérisés par leur état thermodynamique hors équilibre. Pour envisager des applications, il est nécessaire de connaître leur évolution vers l'état d'équilibre final.

Il est possible d'aborder deux aspects de la cristallisation, cinétique et chemin de cristallisation, grâce aux techniques expérimentales suivantes :

- la microcalorimétrie différentielle à balayage très adaptée à l'étude de la cinétique de cristallisation,
- la diffraction X classique et la diffraction X en temps réel pour l'étude des chemins de cristallisation.

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**M. NAKHL**

**Le broyage énergétique appliqué à :**  
**l'obtention de mélanges composites à base de magnésium utilisables pour le stockage de l'hydrogène,**  
**la modification des propriétés magnétiques d'intermétalliques à base de gadolinium**

**16 OCTOBRE 2000 - UNIVERSITE DE BORDEAUX I - BORDEAUX**

**Jury :** G. Le Caër (Rapp.), E. Gaffet (Rapp.), B. Chevalier, J.-L. Bobet, J. Etourneau

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**CH. GRAS**

**Réactivité et Thermodynamique dans le procédé MASHS**  
**(Mechanically Activated Self - Propagating High - Temperature Synthesis) :**  
**Application aux systèmes Mo / Si et Fe / Si**

**6 Septembre 2000 - Univ. de Bourgogne - Dijon**

**Jury :** Y. Bienvenu (Rapp.), G. Le Caer (Rapp.), G. Bertrand, JP Bonnet, M. Gailhanou, JP Larpin, F. Bernard (Co - directeur) & E. Gaffet (Co - Directeur)

L'objectif principal de ce travail a été de traquer dans toutes les étapes du procédé MASHS (Mechanically Activated Self-propagating High-temperature Synthesis) les différentes modifications induites par l'adjonction d'un pré-broyage au processus de combustion autoentretenue SHS (Self-propagating High-temperature Synthesis).

Dans un premier temps, il a été nécessaire de définir la microstructure des réactifs après cobroyage. La poudre se présente, après traitement mécanique contrôlé, sous la forme d'agrégats microniques (0.2-200 µm) composés de nanocrystallites de métal et de silicium (Mo ou Fe : 50 nm et Si : 30 nm).

Lorsque celle-ci est mise en situation de réaction SHS, une diminution du temps nécessaire à l'ignition comme une accélération de l'onde de combustion liée à la dynamisation de la réaction chimique ont été mises en évidence. D'une manière générale, la multiplication des interfaces Métal/Si créées lors du cobroyage ainsi que l'énergie accumulée conduisent à la diminution de l'énergie seuil d'ignition. En effet, il a été possible d'instaurer une

onde de combustion stable entre des nanocristallites solides de fer et de silicium, et cela malgré les difficultés réactionnelles prédites par le critère semi-empirique d'autopropagation. En outre, la taille des cristallites de fer comme la présence de FeSi et  $\alpha$ -FeSi<sub>2</sub> mécano-induites sont capables de piloter le processus réactionnel tout en définissant la phase majoritairement produite.

En ce qui concerne la formation de MoSi<sub>2</sub>, la diffractions en temps réel (0.033 s) couplée à la thermographie infrarouge (0.0066s) met en évidence une étape de fusion du silicium sans pour autant être capable de déceler quelques intermédiaires de réaction que ce soient. Par contre, le produit final est pur, nanostructuré (60-90 nm) mais très poreux (45 à 50 %)

Par conséquent, le procédé MASHS pêche par son incapacité à produire un matériau dense. A contrario, une mise en température rapide effet joule et une mise sous charge simultanée à chaud, le pré-traitement mécanique s'est révélés comme l'unes des conditions nécessaires à la production de nano-disiliciure de molybdène (80 nm) dense (93%). Les premiers résultats tendent à prouver que ce nouveau matériau présente des modifications importantes de ses propriétés : une dureté à 298 K accrue (+55%) et une meilleure tenue à la corrosion haute température (1273 K)

### Mots clés

*Activation mécanique, Cobroyage à haute énergie, Combustion autoentretenu (SHS), Diffraction des rayons X en temps réel (TRXRD), Thermographie infrarouge, Nanomatériau, Synthèse/densification simultanée, Disiliciure de fer (FeSi<sub>2</sub>) et Disiliciure de molybdène (MoSi<sub>2</sub>)*

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**M. ZOUGGAR**

**"Effets du broyage sur les propriétés structurales et mécaniques de poudres de fer pur et sur l'activation de la nitruration"**

**4 Septembre 2000 - LMP - Poitiers**

**Jury** : A. Fnidiki(Rapp.), E. Gaffet (Rapp), P. Goudeau (Inv.), M. Grosbras (Inv.), A. Straboni , P. Chartier (Co - Dir) & J. Mimault (Co - Dir)

**Résumé** : Ce travail porte sur l'évolution microsturale et mécanique de la poudre de fer élaborée par broyage mécanique, et sur celle subissant par la suite de traitements de recuits sous vide, de nitruration dans l'enceinte d'une presse isostatique à chaude (HIP) ou bien des traitements thermiques en plasma d'ammoniac ••

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**C. GOUJON**

**"Elaboration par cryobroyage et métallurgie des poudres de nanocomposites à matrice d'alliage d'aluminium renforcée par des particules de nitrure d'aluminium"**

**25 Mai 2000 - ENSMSE**

**Jury** : P. Goeuriot (Dir. Thèse), G. Le Caer (Rapp.), D. Michel (Rapp.), F. Bernard, Y. Laurent, M. Suery, F. Thévenot, S. Vicens

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**J. JOARDAR**

**"Synthesis of nanocrystalline aluminides in Al - Ni - Fe system by Mechanical Alloying"**

**Avril 2000**

Thesis Supervisor : B.S. Murty et S.K. Pabi (IIT, Kharagpur)

Thesis Examiners : C.C Koch (North Carolina State University) , P. Ramakrishnan (IIT, Bombay)

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**Christine BARBEAU**

(Laboratoire de Métallurgie Physique - Futuroscope)

**Structure dans les matériaux élaborés sous HIP : cas des alliages à base tungtène par frittage et du carbure de titane par combustion auto-propagée**

**13 mars 2000**

**Thèse de Doctorat de l'Université de Poitiers**

**Jury**: A. TRAVERSE, Directeur de Recherche, LURE Orsay, Rapporteur - F. NARDOU, Professeur, Université de Limoges, Rapporteur, D. VREL, Chargé de Recherche, Université de Villeneuve - Examinateur, M.F. BEAUFORT - Chargé de Recherche CNRS, LMP Poitiers - Examinateur, M. GROSBRAS - Chargé de Recherche CNRS, LMP Poitiers, Examinateur - J. MIMAUT, Professeur, Université de Poitiers, Examinateur et Directeur de Thèse

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**Hugues GUÉRAULT**

**PROPRIÉTÉS STRUCTURALES ET MAGNÉTIQUES DE POUDRES DE FLUORURES NANOSTRUCTURÉES MF<sub>3</sub> (M=Fe, Ga) OBTENUES PAR BROUAGE MÉCANIQUE**

**28 Janvier 2000**

**THÈSE DE DOCTORAT - Université du Maine - Physique des Matériaux et des Surfaces**

**Jury :** Gérard Le CAER, Directeur de Recherche, Ecole des Mines - Nancy (Rapporteur), Marc NOGUES, Chargé de Recherche, Université de Versailles (Rapporteur), Jean-François BÉRAR, Ingénieur de Recherche, CNRS - Grenoble, Frédéric BERNARD, Maître de Conférence, Université de Bourgogne - Dijon, Jean-Yves BUZARÉ, Professeur, Université du Maine - Le Mans, Marc LEBLANC, Professeur, Université du Maine Le Mans, Jean-Marc GRENÈCHE, Directeur de Recherche, Université du Maine (Directeur de thèse)

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**Cyril LENAIN**

**APPLICATION DE LA MECANOCHIMIE A LA PREPARATION D'ALLIAGES HYDRURABLES  
NANOCRISTALLINS AB<sub>5</sub>, MG-NI, AB<sub>2</sub> (M) ET DE COMPOSITES M-C, M-Cu : ETUDE DE LEURS  
PROPRIETES ELECTROCHIMIQUES.**

**THESE DE DOCTORAT** - Specialite: sciences des materiaux presentee a l'Universite de Picardie Jules Verne

**Jury :** M. le Prof. J. Etourneau (Universite Bordeaux) M. D. Fruchart (DR, Lab. Cristallographie, Grenoble) Mme A. Percheron - Guegan (DR, LCMTR, Thiais) M. le Prof. L. Schlapbach (Universite de Fribourg) M. le Prof. J-M. Tarascon (Universite de Picardie) M. L. Aymard (Universite de Picardie)

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**Cooperative Research on Related Areas**  
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**COREE du SUD (19/01/2000)**

From Professor Soon H. Hong  
Dept. of Materials Science and Engineering - Korea Advanced Institute of Science and Technology  
373-1 Kusung-dong, Yusung-gu - Taejon, 305-701, Korea  
E-mail : HYPERLINK mailto:shhong@sorak.kaist.ac.kr / shhong@sorak.kaist.ac.kr  
Fax. : 82-42-869-3310 - Tel. : 82-42-869-3327

We are currently working on the mechanical alloying processes and the characterization of mechanical & thermal properties of nanocrystalline materials and composite materials, such as SiC/Al, WC/Co and W/Cu for structural or thermal management applications. We are very pleased to discuss for international cooperative research on related topics with Members of Mechanosynthese Group.

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

**ESPAGNE (25/09/2000) POSTDOCTORAL POSITION (From M.D. Baro)**

Universitat Autònoma de Barcelona

The Group de Física de Materials II of the Physics Department of the UAB announces the availability of a 18 months full-time Postdoctoral Research position. Applicants should hold a PhD degree in Materials Science, Physics or in a related field. The position requires:

Knowledge of glasses, metastable and nanocrystalline materials.

Fundamental understanding of the nucleation and crystal growth theories.

Knowledge of calorimetry and thermostability.

Experience in electron, optical and x-ray based characterisation techniques and practices.

Computer literacy.

Citizenship of EU (except Spain) or Associated states.

Under 35 years old.

Proficient level of English

The position begins with effect from January 2001. The research programme includes a close co-operation with other partners of the Project. Applicants should submit a CV, and a statement describing your interest in the position with two references to:

Professor M.D. Baro,  
Dept. Physics, Edifici Cc,  
08193 Bellaterra,  
Barcelona, Spain;  
Tel: 34 93 5811657.

Electronic applications can be sent to [dolors.baro@uab.es](mailto:dolors.baro@uab.es)"

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**ANGLETERRE (21/09/2000) - From Paul Warren ([paul.warren@materials.oxford.ac.uk](mailto:paul.warren@materials.oxford.ac.uk))**

Job vacancies in a Research Training Network.

Research Training Network on Manufacture and Characterisation of Nanostructured Al alloys

Pre-doctoral/post-doctoral researchers required at 9 institutions across Europe.

The research positions will involve aspects of : materials processing by gas atomization, rapid solidification and mechanical alloying, followed by compaction; microstructural and microchemical characterisation by XRD, DSC, TEM, STEM, APFIM ; thermodynamic / kinetic modelling and molecular dynamic simulation ; mechanical property evaluation by tensile testing, fatigue testing and high strain rate impact testing.

See Network Homepage <http://www.materials.ox.ac.uk/nano-al/> for more details.

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**FRANCE (14 / 02 / 2000)**

**Ph D Thesis Proposal**

A partir de septembre 2000 (Bourse du Ministère)

**"Obtention par mécanosynthèse de mélanges composites à base de magnésium ayant des propriétés d'hydruration optimisées. Caractérisation de leurs propriétés structurales et physiques.**

En résumé, le sujet proposé portera sur les deux points suivants :

- 1 - Etude de mélanges composites Mg (ou Mg<sub>2</sub>Ni) + intermétalliques élaborés par mécanosynthèse (structure, composition chimique, capacités d'absorption d'hydrogène, morphologie, surface, granulométrie, ) ;
- 2 - Réalisation d'électrodes négatives à partir de ces mélanges et études électrochimiques.

Les techniques utilisées au cours de ce travail seront\* :

Préparation : - Broyeur planétaire - Four à lévitation - Four à arc - Banc d'hydruration (construction de courbe PCT)

Caractérisation : - Diffraction des rayons X sur poudres - Microsonde électronique - Microscopie électronique ( à balayage et en transmission) - Mesures de surfaces spécifiques (BET) - Granulométrie (diffraction Laser - "Mesures" électrochimiques - Mesures calorimétriques XPS, EPMA, ...

\* ceci est une liste non exhaustive des différentes techniques que le candidat devra utilisé

**Contact :**

**Jean-Louis BOBET**

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\*\*\*\*\*

**USA (8 / 02 / 2000)**

Rutgers University is seeking a **postdoctoral associate** with demonstrated expertise in powder synthesis and processing (forming and sintering methods) to work on research focused on textured ceramic ferroelectric materials. The candidate must be able to work as part of a multidisciplinary team involving industry and academia focused on making transducer and actuator materials. The candidate should demonstrate the ability to work independently, publish in archival journals and present their work in a public forum. The candidate should send a curriculum vitae, three representative publications (preferably with the candidate as a first author) and the names, addresses, email and phone numbers of three references that can comment on the candidate's capabilities. The position is available immediately at a salary of \$32,000 with health benefits included. The position is available immediately. Placement is preferred prior to August with priority given to a qualified candidate with earlier availability.

**Interested candidates should send correspondence** to: Professor Richard E. Riman, Rutgers University, Department of Ceramic and Materials Engineering, 607 Taylor Road, Piscataway, NJ 08854-8065, riman@alumina.rutgers.edu / 732-445-4946 / 732-445-6264

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**COREE du SUD (10 / 01 / 2000)**

From Professor Soon H. Hong

Dept. of Materials Science and Engineering - Korea Advanced Institute of Science and Technology

373-1 Kusung-dong, Yuseong-gu - Taejon, 305-701, Korea

E-mail : [HYPERLINK mailto:shhong@sorak.kaist.ac.kr](mailto:shhong@sorak.kaist.ac.kr) /shhong@sorak.kaist.ac.kr

Fax. : 82-42-869-3310 - Tel. : 82-42-869-3327

The Composite Materials Laboratory at Korea Advanced Institute of Science and Technology is looking for a postdoctoral position. The postdoctoral contract will be one year on the field of modeling and simulation of mechanical & thermal properties of composite materials or on the field of fabrication process of nano-composite materials. Applicant should be within three years of receipt of Ph.D. degree on related field. For more information, please contact :

## Bibliographie Récente

### Livres ou "Special Issues"

(7/07/2000) - From Victor Rieckansky Publisher

Cambridge International Science Publishing <http://www.demon.co.uk/cambsci/homepage.htm>

#### **MACROMOLECULAR MECHANOCHEMISTRY**

Volume 1: Polymer Mechanochemistry - by Cleopatra Vasiliu OPREA & Florin DAN

Department of Macromolecules, Gh. Asachi, Technical University, 6600 Iasi, Romania

Macromolecular Mechanochemistry presents from theoretical and experimental point of view the main problems of this field, including the results obtained in more than a century of research. It is organised in two volumes: Polymer Mechanochemistry and Polymers with Chemomechanical Functions, respectively. The present volume deals with: Chained Polystage Character of Mechanochemical Process (1), Mechanochemistry of Polymers Deformation (2); Mechanochemistry of Polymer Fracture (including also the Fracture of Composite Materials) (3), and Mechanochemical Processes for Energy Conversion (4). In this frame, the theoretical and experimental material is organised in correlation to the reaction mechanism, the type of mechanical solicitation, and the nature of environmental medium. This book is addressed to professors, students, and researchers involved in the field of polymer science, to engineers from the industry of synthesis and processing of plastic materials, elastomers and fibres, as well as to specialists from all technical domains that exploit polymer-based materials. They will find in the book examination of the theoretical, experimental and applied problems and wide access to the basic literature in this field.

#### Contents

1. Chained polystage mechanism of mechanochemical processes
2. Mechanochemistry of polymers deformation
3. Mechanochemistry of Polymer Fracture
4. Mechanochemical Processes for Energy Conversion

**Volume 1** (ISBN 189832672X) will be published in September 2000, approx. 500 pages, cased, approximate price £80.00; (volume 2 will be published at the end of - 2000)

Send your preliminary order to [orders@cisp.demon.co.uk](mailto:orders@cisp.demon.co.uk)

(9/06/2000)

**"Mechanical Alloying : FABRICATION OF ADVANCED MATERIALS AT ROOM TEMPERATURE"** by M. Sherif El-Eskandarany

(ISBN: 977-299-089-7) Published by DAR AL-FIKR AL-ARABI, Cairo-Egypt.

The price of the book is \$50, and a special discount (20%) is offered to all the RFM member.

#### **Preface**

Mechanical alloying (MA) process using ball-milling and/or rod-milling techniques, has received much attention as a powerful tool for fabrication of several advanced materials, including equilibrium, nonequilibrium (e.g., amorphous, quasicrystals, nanocrystalline, etc.), and composite materials. In addition, it has been employed for reducing some metallic oxides by milling the oxide powders with metallic reducing agents at room temperature. The MA is unique process in that a solid state reaction takes place between the fresh powder surfaces of the reactant materials at room temperature. Consequently, it can be used to produce alloys and compounds that are difficult or impossible to be obtained by the conventional melting and casting techniques.

This book intended primarily to serve as an introduction to the MA process, including general description of the process, starting material requirements, the equipment, characterizations of the milled powders, and consolidation techniques, which used to compact the powder into fully-dense bulk materials.

The book contains several typical examples of selected advanced materials that have been fabricated by MA. This book is aimed at either senior undergraduate/post graduate students or materials scientists/metallurgists. - M. Sherif El-Eskandarany - April 2000 - Cairo - Egypt

#### **Contents**

Introduction - Background - History of Mechanical Alloying - Milling - Factors Affecting the Mechanical Alloying 8  
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(05/05/2000)

### EXTRACTIVE METALLURGY OF ACTIVATED MINERALS

included in series Process Metallurgy, 10

by P. Balaz - Institute of Geotechnics, Slovak Academy of Sciences

ISBN : 0 - 444 - 50206 - 8 / Price USD 144, Euro 124.79)

http : // www.elsevier.nl/inca/publication

Description

Mechanical activation of solids is a part mechanochemistry, the science with a sound theoretical foundation exhibiting a wide range of potential application. Mechanical activation itself is an innovative procedure where an improvement in technological processes can be attained via a combination of new surface area and defects formation in minerals.

Mechanical activation is of exceptional importance in extractive metallurgy and mineral processing and this area forms the topic of this book and is a result of more than twenty years of research and graduate teaching in the field.

In pyrometallurgy, the mechanical activation of minerals makes it possible to reduce their decomposition temperatures or causes such a degree of disordering that the thermal activation may be omitted entirely. The potential mitigation of environmental pollutants is becoming increasingly important in this context.

The lowering of reaction temperatures, the increase of the rate and amount of solubility, preparation of water soluble compounds, the necessity for simpler and less expensive reactors and shorter reaction times are some of the advantages of mechanical activation in hydrometallurgy. The environmental aspects of these processes are particularly attractive.

Several industrial processes are examined and the flowsheets are presented as successful of activation. In these processes, the introduction of a mechanical activation step into the technological cycle significantly modifies the subsequent steps.

The book is designed for researchers, teachers, operators and students in the areas of extractive metallurgy, mineral processing, mineralogy, solid state chemistry and materials science. It will encourage newcomers to the mechanochemistry to do useful research and discover novel applications in this field.

(3/02/2000)

Two new books on mechanical alloying are now available from Cambridge International Science Publishing (infos fournies par Anne Porter - Publishing Manager - Cambridge International Science Publishing <http://www.demon.co.uk/cambsci/homepage.htm>)

### 1. MECHANICAL ALLOYING - FUNDAMENTALS AND APPLICATIONS

<http://www.demon.co.uk/cambsci/book52.htm> Contents

Introduction (history, benefits of mechanical alloying); Mechanical alloying (alloying mills, mills in practice, improved mills, the process, parameters);

Variations of mechanical alloying (reaction milling, cryomilling, repeated rolling, double mechanical alloying, repeated forging); Process control agents in mechanical alloying; Mechanical alloying mechanisms (ductile-ductile system, ductile-brittle system, brittle-brittle system, metastable phase formation, amorphisation, nanocrystallization, extension of solid solubility, activation of solid state chemical interaction);

Energy transfer and energy maps;

Consolidation of mechanically alloyed powders (consolidation techniques, thermomechanical treatment); Mechanical properties of mechanically alloyed materials (tensile properties, fracture, creep, stress corrosion cracking susceptibility);

Modelling mechanical alloying (mechanistic models, deformation, coalescence and fragmentation, evolution of particle size, milling time, powder heating, powder cooling, atomistic model, thermodynamic and kinetic model)

Joining of mechanically alloyed materials; Rapid solidification and mechanical alloying; Applications (nickel-based superalloys, Al-based materials, supersaturated solutions, magnetic materials, mechanically alloyed powders for spray coatings, superplasticity, tribological materials, composites, amorphous solids, nanocrystalline materials, solid-state chemical reactions, etc). ISBN 1898326568, 160 pages 234 **156 mm, cased**, £45.00, 1999

## **DISPERSION STRENGTHENED ALUMINIUM PREPARED BY MECHANICAL ALLOYING**, by M Besterçi

<http://www.demon.co.uk/cambsci/book51.htm> 1. Characteristics of dispersion-strengthened systems 2. Mechanical alloying (kinetics and mechanism of preparation of the Al-C system by mechanical alloying; compaction of powders and heat treatment of compacts; 3. Microstructure and quantitative evaluation of parameters of dispersion-strengthened materials (definition and properties of interparticle distance; experimental possibilities of determination of structural objects; models of heterogeneous structures and their evaluation; simulation of model structures; analysis of the spatial distribution of particles in the Al-Al4C3 material) 4. Static and dynamic mechanical properties (mechanical properties at elevated temperatures; mechanical properties at 20 °C; effect of interface on the mechanical properties; superplastic properties of the system; thermal stability of the system; creep characteristics; creep-fatigue characteristics)  
References - ISBN 189832655X, 90 pages, 234 **156 mm**, **soft laminated cover**, £25.00, 1999

### **"Mechanical Alloying : Fundamentals and Applications"**

Prof. P.R. Soni (1999) - Cambridge International Science Publishing  
web site : <http://www.demon.co.uk/cambsci/book52.htm>

### **"Non Equilibrium Processing of Materials"**

R.W. Cahn - Elsevier Science - Volume 2 in the Pergamon Materials Series

A large number of technical papers have been published in reviews, monographs and conference proceedings, but have almost always been devoted to a single processing technique. This book, however, covers all the non equilibrium processing methods and their effects in a single volume.

web site : [www.elsevier.nl/locate/isbn/0080426972](http://www.elsevier.nl/locate/isbn/0080426972)

### **Bulk Amorphous Alloys : Preparation and Fundamental Characteristics**

A. Inoue

Materials Science Foundation Vol. 4 - Trans Tech Publications : <http://www.ttp.net>

Interest in bulk amorphous alloys has increased rapidly throughout the world and these materials have now gained a position of great importance in basic science and engineering materials technology. Bulk amorphous alloys based upon the Zr - Al - Ni - Cu, Zr (Ti,Nb) - Al - Ni - Cu and Zr - Ti - Ni - Cu - Be systems have already achieved wide commercial success as components of various technical accessories ranging from sporting goods to optical instruments.

Here is a state of the art reviews on this new group of materials, covering all areas of interest, ranging from the synthesis of these special alloys and their fundamental properties, to their engineering characteristics and applications.

This work will therefore be of equal interest to those who wish to become fully acquainted with the subject, and to those who are already actively engaged in the field.

## **DISPERSION-STRENGTHENED ALUMINIUM PREPARED BY MECHANICAL ALLOYING**

Michal Besterçi, Institute of Materials Research, Slovak Academy of Sciences, Kosice

In the book, the author describes the theoretical and technological fundamentals of mechanical alloying the Al-C system. Special attention is given to material characteristics, the kinetics and mechanism of mechanical alloying, methods of mixture compaction and heat treatment of compacted parts. Models of dispersoid spatial arrangement, dispersoid evaluation and optimisation and experimental possibilities are discussed. The interpretation of the static and dynamic mechanical properties, especially strength and ductility properties at 20 °C, mechanical properties at elevated temperatures are discussed, with emphasis on the effect of interface, superplasticity, creep and creep-fatigue characteristics. Content

Introduction

1. Characteristics of dispersion-strengthened systems

2. Mechanical alloying (kinetics and mechanism of preparation of the Al-C system by mechanical alloying; compaction of powders and heat treatment of compacts;

3. Microstructure and quantitative evaluation of parameters of dispersion-strengthened materials (definition and properties of interparticle distance; experimental possibilities of determination of structural objects; models of heterogeneous structures and their evaluation; simulation of model structures; analysis of the spatial distribution of particles in the Al-Al4C3 material) 4. Static and dynamic mechanical properties (mechanical properties at elevated temperatures; mechanical properties at 20°C; effect of interface on the mechanical properties; superplastic properties of the system; thermal stability of the system; creep characteristics; creep-fatigue characteristics)

Index : ISBN 189832655X, 80 pages, 234 **156 mm**, **soft laminated cover**, £22.00, January 1999

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<http://www.demon.co.uk/cambsci/homepage.htm>

### **"Mechanical Alloying"**

Auteurs : Li Lü & Man On Lai(National University of Singapore)

Kluwer Academic Publishers

**Contents :** Preface - Introduction to Mechanical Alloying - Experimental Set - Up - The Mechanical Alloying Process - Formation of New Materials - Characterization of Powders - Densification - Mechanical Properties - Mechanisms of Mechanical Alloying - Modeling of Mechanical Alloying - Index

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**"Surface-Controlled Nanoscale Materials for High-Added-Value Applications"**

Editors: Kenneth E. Gonsalves, Marie-Isabelle Baraton, Rajiv Singh, Heinrich Hofmann, Jerry X. Chen, and Joseph A. Akkara.

Materials Research Society, Symposium Proceedings Volume 501, 1998

MRS, Warrendale, Pennsylvania, USA (website: <http://www.mrs.org/>)

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**"Nanomatériaux"**

Auteurs : E. Gaffet, S. Begin - Colin, O. Tillement

Editeur : Innovation 128 - 24 Rue du Quatre Septembre - 75002 Paris - France - Fax : 33 1 42 65 47 76

Les dernières années ont vu apparaître dans le monde des matériaux avancés le préfixe "nano" (nanostructuré, nanocristallins, nanophase ou nanométrique) ; les conférences et les forums sur Internet se multiplient où s'échangent des informations sur les avancées scientifiques et technologiques dans ce domaine des matériaux nanostructurés qui se distinguent des matériaux polycristallins conventionnels par la dimension des cristallites les composant ou par la dimension des hétérostructures présentes : ces dimensions sont de quelques dizaines d'angströms, voire de quelques nanomètres. A ces dimensions, les propriétés des matériaux changent radicalement.

Au début des années 90, les japonais ont été les premiers à lancer d'ambitieux programmes de R & D puisque le MITI a consacré aux nanomatériaux près de 200 millions de dollars pour la période 1990 - 2000 et que la Science & Technology Foundation a investi presque la même somme pour co - financer des projets de laboratoires publics et privés. Les Etats Unis puis les pays européens ont investi plus tardivement mais déjà ont obtenu des résultats prometteurs (.....) Certaines applications existent déjà au niveau international, quelque 400 sociétés se partagent aujourd'hui un marché voisin de 1 milliard de dollars mais qui devrait tripler, voire quintupler à l'horizon 2001.(.....)

(...) Pour aider les industriels concernés à imaginer les applications qu'ils pourraient s'approprier et identifier les acteurs internationaux, la présente étude dresse un état de l'art complet des nanomatériaux en décrivant leurs procédés d'élaboration actuels ou envisagés et en détaillant leurs différentes propriétés physico - chimiques et les géométries que l'on peut obtenir.

Enfin l'étude permet de cerner les applications actuelles et potentielles...

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**CHEMISTRY FOR SUSTAINABLE DEVELOPMENT**

**Vol. 6, No. 2-3, MARCH-JUNE 1998**

Proceedings of 2d International Conference on Mechanochemistry

(INCOME-2), which was held in Novosibirsk in 1997.

**Contact :** Prof. • N.Z. Lyakhov, Inst. Sol. State Chem.- Russian Acad Sci. - Kutaleladze, 18 - Novosibirsk - 630128 Russia - The Proceedings will be available by the price 80 USD.

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**Mechanochemistry of Materials**  
**Cambridge International Science Publishing**

Emmanuel Gutman - Materials Eng. Dpt - Ben Gurion University - Beer Sheva - Israel

Considerable advances have been made in mechanochemistry in the last couple of decades. Training of experts in this field with a background in materials science, chemical and mechanical engineering, etc. requires study of the fundamentals of mechanochemistry. There is a need for a textbook in the general and compressed form which would cover many aspects and would be used as a basis for understanding the fundamental principles to control mechanochemical phenomena. This textbook is based on lectures given by Prof. Gutman in a graduate course in the mechanochemistry of materials at the Ben - Gurion University of the Negev. The book contains examples of experimental results to illustrate the mechanochemical phenomena and technologies.

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**BIBLIOGRAPHY ON MECHANICAL ALLOYING AND MILLING**

**Suryanarayana (Inst for Materials and Advanced Processes, University of Idaho, USA )**

The present bibliography covers information on mechanical alloying and milling of materials starting from 1970 (when it was recognized that MA has become a commercial/viable material processing technique instead of just a grinding method) to 1996. All the available references will be presented in a chronological fashion. Under each year, (.....)

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<http://www.demon.co.uk/cambsci/homepage.htm>

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**Proceeding du Congrès "Mechanically Alloyed, Metastable and Nanocrystalline Materials"- Barcelone (1997)**

Editor : M.D. Baro, S. Surinach - Materials Science Forum 269 - 272 (1998)

## PERIODIQUES

(Rubrique réalisée grâce aux moyens de la bibliothèque de  
l'Université de Technologie de Belfort - Montbéliard / UTBM)

### **[43] SEEDING EFFECT IN THE FORMATION OF $Pb(Fe_{2/3}W_{1/3})O_{-3}$ VIA MECHANICAL ACTIVATION OF MIXED OXIDES**

Khim AS. Wang J. Xue JM. - Solid State Ionics. 132(1-2):55-61, 2000

Twenty hours of mechanical activation of  $PbO$ ,  $Fe_2O_3$  and  $WO_3$  at room temperature led to the occurrence of  $PbWO_4$  and  $PbFe_2WO_6.5$ , which are the common transitional phases that are formed during the conventional solid state reaction at elevated temperatures. In addition, the perovskite  $Pb(Fe_{2/3}W_{1/3})O_{-3}$  (PFW) phase cannot be synthesized when  $PbO$  is mechanically activated with pre-reacted  $Fe_2WO_6$ . In contrast, adding 0.4 mole of preformed PFW seeding to the constituent oxides resulted in the suppression of transitional  $PbWO_4$  and  $PbFe_2WO_6.5$  phases during mechanical activation. The PFW seeding also has a strong effect on the types of transitional phases involved when the activated compositions are subjected to an increasing calcination temperature.

### **[42] EFFECT OF HIGH-ENERGY MECHANICAL ACTIVATION ON THE MICROSTRUCTURE AND ELECTRICAL PROPERTIES OF ZNO-BASED VARISTORS**

Fah CP. Wang J. - Solid State Ionics. 132(1-2):107-117, 2000

Zinc oxide-based varistors were prepared by subjecting the mixed oxide powders to high-energy mechanical activation for 5 h and 10 h, respectively. The crystallite sizes of the constituent oxides were refined to 10 similar to 15 nm by 5 h of high-energy mechanical activation. Similarly, the average particle size was refined from 0.45  $\mu m$  to about 0.18  $\mu m$ . The material derived from high-energy mechanical activation exhibit a higher density (> 98% relative density) and more refined microstructure upon sintering at 1100 degrees C than those of the material derived from the powder subjected to no high-energy mechanical activation. They demonstrate much higher  $\sigma$ -values than those of the materials sintered from the oxide powder without high-energy mechanical activation. These enhanced electric properties are due to great structural homogeneity and refined grain size brought about by high-energy mechanical activation.

### **[41] SYNTHESIS OF POLYCUPROORGANOSILOXANES WITH MECHANOCHEMICAL ACTIVATION**

Kapustina AA. Shapkin NP. Gavrilova NI. Kalugina MY. Bessonova VI. - Russian Journal of General Chemistry. 70(2):241-243, 2000

The feasibility was examined for preparing polycuprodiphenylsiloxanes from copper acetylacetonate and diphenylsilanediol under conditions of mechanochemical activation in an oscillatory activator at a frequency of 2.5 Hz and a load-to-packing weight ratio of 1.8. Under these conditions the components react only partially. The fractional composition of the resulting product is nonuniform and depends on the time of mechanochemical treatment. The compounds were characterized by elemental analysis, IR spectroscopy, and gel chromatography.

### **[40] PECULIARITIES OF THE SOLID-PHASE FORMATION OF STRONTIUM PENTAHYDROALUMINATE FROM BINARY HYDRIDES BY MECHANOCHEMICAL ACTIVATION AND MODELING OF THE PROCESS ON THE BASIS OF THERMOGASOVOLUMETRY DATA**

Dymova TN. Konoplev VN. Sizareva AS. Aleksandrov DP. - Russian Journal of Coordination Chemistry. 26(8):531-537, 2000

Crystals of aluminium and strontium hydrides taken in the 1 : 1 molar ratio and subjected to mechanochemical activation (MCA) first give  $Sr(AlH_4)_2$ , which then exothermally adds the remaining  $SrH_2$  to form  $SrAlH_5$  (similar to 80% yield). When a reactor is cooled (by reducing the power load), the synthesis terminates at the stage of formation of unstable  $Sr(AlH_4)_2$ , which undergoes mechanolysis with the in situ detachment of  $AlH_3$  and formation of a mixture of  $SrAlH_5$  and the reactive metallic aluminium from  $AlH_5$ . Schemes for the  $SrAlH_5$  formation under "rigid" conditions (without heat removal) and "mild" conditions (with the air-cooled reactor) have been suggested. A diagram of the mechanolysis of the hydride phases (a dependence of the quantitative transformations of these phases on the MCA time) is presented. Upon heating,  $SrAlH_5$  exothermally disproportionates at a high rate with the in situ detachment of  $AlH_3$  and the formation of  $Sr_3(AlH_6)_2$ .

### **[39] SOLID-STATE ALLOYING IN NANOSTRUCTURED BINARY SYSTEMS WITH POSITIVE HEAT OF MIXING**

Ma E. Sheng HW. He JH. Schilling PJ. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 286(1):48-57, 2000

While many binary systems exhibit a positive heat mixing that precludes intermixing in conventional bulk diffusion couples, it is possible to alloy in solid state some of these bulk immiscible elements in nanostructures. Molecular dynamics simulations demonstrate that in low-dimensional systems such as surfaces and in sub-nanometer layered superlattice structures, excess enthalpic and entropic energy contributions can provide a driving force for spontaneous intermixing to form substitutional solid solution alloys. Such driving forces diminish, however, in coarser nanophase binary mixtures when domain sizes reach beyond approximate to 1 nm. In this case, true alloying on the atomic level can be achieved by employing an external forcing mechanism such as severe mechanical deformation. In addition to single-phase alloys, we demonstrate, using X-ray absorption near-edge structure (XANES) analysis, a novel two-phase coexistence controlled by kinetically imposed polymorphic constraints. Using a phenomenological model, possible mechanisms responsible for driven alloying are discussed with reference to several previous proposals in the literature.

### **[38] NANOSTRUCTURED TIN POWDER PREPARED VIA AN INTEGRATED MECHANICAL AND THERMAL ACTIVATION**

Ren RM. Yang ZG. Shaw LL. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 286(1):65-71, 2000

A new process for large-scale production of nanostructured TiN powder is described in this paper. In this process  $TiO_2$  and graphite are used as the starting materials and the carbothermic reduction and the simultaneous nitriding reaction are enhanced by high-energy milling prior to the reactions. The product from this process is nanostructured

TiN powder. It is found that under the same nitridation condition, 100% TiN is formed for TiO<sub>2</sub> and graphite powder mixtures with high-energy milling, whereas no TiN is obtained for powder mixtures without milling. Furthermore, more TiN is formed as the time of high-energy milling increases. The enhanced carbothermic reduction and nitridation process have been related to the reduced crystallite size, large grain boundary area, large specific surface area; presence of amorphous phases and the increased defect concentration in the powder mixture introduced by high-energy milling.

**[37] NANOCRYSTALLINE 0.54PZN-0.36PMN-0.1PT OF PEROVSKITE STRUCTURE BY MECHANICAL ACTIVATION**

Wan DM. Xue JM. Wang J. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 286(1):96-100, 2000

Nanocrystallites of relaxer ferroelectric composition in the system of 0.54Pb(Zn<sub>1/3</sub>Nb<sub>2/3</sub>)O-3-0.36Pb(Mg<sub>1.3</sub>Nb<sub>2/3</sub>)O-3-0.1PbTiO<sub>3</sub> (PZN-PMN-PT) were triggered to form in mixed oxides of PbO, ZnO, MgO, Nb<sub>2</sub>O<sub>5</sub> and TiO<sub>2</sub> by an increasing degree of mechanical activation at room temperature. They exhibited a nanocrystalline perovskite structure, as indicated by the phase analysis using X-ray diffraction. Transitional pyrochlore and PT phases, which have commonly been observed in the solid state reaction of constituent oxides at high temperatures, were not observed during mechanical activation prior to formation of the perovskite phase. This suggests that the one or more interfacial diffusions and reactions that occur in the conventional solid state reaction are not responsible for the phase formation triggered by mechanical activation at room temperature. The activation-derived PZN-PMN-PT powder was highly sintering-reactive and it was sintered to > 99% theoretical density at 1100 degrees C for 1 h. Sintered PZN-PMN-PT demonstrated a maximum dielectric constant of similar to 19 000 and a dielectric loss of 0.015 at the Curie temperature of 103 degrees C when measured at 1 kHz.

**[36] THE EFFECT OF MG CONTENT ON MICROSTRUCTURE AND HYDROGEN ABSORPTION PROPERTIES OF MECHANICAL ALLOYED MMNI(3.5)(COALMN)(1.5)-MG**

Zhu M. Zhu WH. Gao Y. Che XZ. Ahn JH. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 286(1):130-134, 2000

In this work high energy ball milling was used to prepare nanophase composite hydrogen storage alloys in MmNi(3.5)(CoAlMn)(1.5)-Mg system. Particle size distribution analyzer, X-ray diffractometer, SEM and TEM were used to characterize microstructure of the composite. Hydrogen absorption PCT and kinetics of the composite were measured and compared with that of melted pure MmNi(3.5)(CoAlMn)(1.5) alloys. The effect of Mg on the microstructure and hydrogen absorption properties was obtained by investigating the composites of different Mg contents. The result showed that nanophase composites were obtained. The hydrogen absorption properties measurement proved that forming nanophase composite is an effective method for improving hydrogen absorption properties of alloys.

**[35] PROCESSING OF CU-AL<sub>2</sub>O<sub>3</sub> METAL MATRIX NANOCOMPOSITE MATERIALS BY USING HIGH ENERGY BALL MILLING**

Ying DY. Zhang DL. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 286(1):152-156, 2000

A Cu-20vol.%Al<sub>2</sub>O<sub>3</sub> nanocomposite material has been successfully produced by using a new approach. This approach involves mechanical alloying of Cu and Al powders to produce Cu-Al alloy powder, mechanical milling of the Cu-Al powder together with CuO powder, and consolidation of the composite powder produced by milling. It has been observed that mechanical alloying of Cu and Al powders with Cu/Al atomic ratio of six leads to formation of a Cu(Al) solid solution. When the Cu(Al) solid solution powder is milled with CuO powder, reaction between Cu(Al) and CuO occurs, extracting Al solute from the Cu(Al) solution and turning it back to pure Cu. It has been identified that the Al<sub>2</sub>O<sub>3</sub> particles in the consolidated composite material have a size smaller than 200 nm in diameter.

**[34] INTENSE MILLING NANOCRYSTALLINE FE<sub>73.5</sub>CU<sub>1</sub>NB<sub>3</sub>SI<sub>13.5</sub>B<sub>9</sub>: A SOFT MAGNETIC MATERIAL IN POWDERED FORM**

Xu H. He KY. Qiu YQ. Wang ZJ. Feng W. Dong YD. Xiao XS. Wang Q. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 286(1):197-200, 2000

The change of structure by intense milling on the nanocrystalline Fe<sub>73.5</sub>Cu<sub>1</sub>Nb<sub>3</sub>Si<sub>13.5</sub>B<sub>9</sub> alloy was investigated. The magnetic properties of nanocrystalline Fe<sub>73.5</sub>Cu<sub>1</sub>Nb<sub>3</sub>Si<sub>13.5</sub>B<sub>9</sub> dust cores (core of compacted powder) were studied. It was found that the nanostructured ribbons obtained from the crystallization of amorphous state by a proper annealing treatment could be changed into amorphous powder via short time milling. By increasing the milling time, the milled powder return to crystallization. It was also found that the permeability of the nanocrystalline dust cores had nearly not any changes in the frequency range from 1 to 100 kHz. The quality factor Q of the nanocrystalline dust cores increased gradually with increasing frequency. The quality factor Q of nanocrystalline dust cores became higher at the frequency over 50 similar to 70 kHz in comparison with that of the permalloy dust core.

**[33] EFFECT OF BALL MILLING ON SEMICRYSTALLINE BISPHENOL A POLYCARBONATE**

Font J. Muntasell J. - Materials Research Bulletin. 35(5):681-687, 2000

The effect of ball milling on semicrystalline bisphenol A polycarbonate (PC) has been studied. The degree of crystallinity obtained in the recrystallization process of the samples amorphized by milling has been related to the crystallinity of PC prior to the grinding. Some differences have been found between PC amorphized by milling and by quenching from the liquid state.

**[32] HYDROTHERMAL SYNTHESIS OF SUBMICROMETER CRYSTALS OF BOEHMITE**

Tsuchida T. - Journal of the European Ceramic Society. 20(11):1759-1764, 2000

The effect of solvents, mechanically ground gibbsite and amorphous hydrated alumina gels on the formation of well-crystallized and submicrometer-size crystals of boehmite was studied. Nearly monodispersed boehmite particles of 0.2-0.3 μm with a parallelogram shape were hydrothermally synthesized from ball-milled gibbsite in water at 200 degrees C for 6 h. The 100 nm-sized crystals of boehmite were prepared from amorphous hydrated alumina gels at 255 degrees C for 24 h.

**[31] ROOM TEMPERATURE MAGNETIC HARDENING IN MECHANICALLY MILLED FERROMAGNETIC-ANTIFERROMAGNETIC COMPOSITES**

Sort J. Noguez J. Amils X. Surinnach S. Munnoz JS. Baro MD. - Journal of Magnetism & Magnetic Materials. 219(1):53-57, 2000

Enhancements in coercivity, squareness and energy product have been obtained at room-temperature by mechanically milling mixtures of ferromagnetic (FM) (Co) and antiferromagnetic (AFM) (NiO) powders. All these effects decrease with increasing temperatures, up to  $T = 600$  K, just above the Neel temperature of the antiferromagnet. Due to the competition between coercivity enhancement and reduction of saturation magnetization in AFM/FM mixtures, although the maximum coercivity enhancement has been found for (AFM)1:1(FM) weight ratio, the maximum energy product is obtained for composites with larger FM content, i.e. (AFM)2:3(FM).

**[30] MOSSBAUER SPECTROSCOPY, CRYSTALLOGRAPHIC, MAGNETIC AND ELECTRONIC STRUCTURE OF ZNFE<sub>3</sub>N AND INFE<sub>3</sub>N**

Kuhnen CA. de Figueiredo RS. dos Santos AV. - Journal of Magnetism & Magnetic Materials. 219(1):58-68, 2000

In this work, mechanical alloying is used to obtain the  $\gamma$ -Zn<sub>0.6</sub>Fe<sub>3.4</sub>N perovskite nitrides. A single-phase  $\gamma$ -ZnFe<sub>3</sub>N was not obtained but indium substitution leads to a single-phase compound not fully ordered. X-ray diffraction and Mossbauer measurements were performed on these compounds. We found that InFe<sub>3</sub>N is paramagnetic while magnetic measurements made at low temperature give 4.65  $\mu$ (B) as the magnetic moment per unit formula for zinc substitution. Self-consistent LMTO calculations were performed for  $\gamma$ -ZnFe<sub>3</sub>N and  $\gamma$ -InFe<sub>3</sub>N fully ordered compounds. Ferromagnetic calculations for  $\gamma$ -InFe<sub>3</sub>N give null magnetic moment, that is, paramagnetic is the stable phase. For  $\gamma$ -ZnFe<sub>3</sub>N, the calculated magnetic moment was 3.60  $\mu$ (B), per unit formula. Also, theoretical results, in the zinc case, were compared with Mossbauer results in terms of isomer shift and magnetic hyperfine field.

**[29] MECHANOCHEMICAL SYNTHESIS AND SINTERING OF (ZRO<sub>2</sub>)(0.97)(Y<sub>2</sub>O<sub>3</sub>)(0.03)**

Morozova LV. Panova TI. Lapshin AE. Glushkova VB. - Inorganic Materials. 36(8):835-838, 2000

**[28] MG<sub>2</sub>SN AS A NEW LITHIUM STORAGE INTERMETALLIC COMPOUND**

Sakaguchi H. Maeta H. Kubota M. Honda H. Esaka T. - Electrochemistry. 68(8):632-635, 2000

To obtain a new anode material of lithium battery, electrochemical properties were investigated on Mg<sub>2</sub>Sn intermetallic compound synthesized by mechanical alloying (MA) method. As a result, we found that the reversible insertion-extraction of a large amount of lithium can electrochemically occur in mechanically alloyed Mg<sub>2</sub>Sn. The Mg<sub>2</sub>Sn changed its own crystal structure from cubic to orthorhombic with increasing MA time. The compound exhibiting good performances as an anode had a mixture of cubic and orthorhombic phases. The discharge capacity of the Mg<sub>2</sub>Sn electrode was still maintained at 250-300 mA h g<sup>-1</sup> even after the 20 times charge-discharge cycling. The reaction proceeded without any phase separation and/or decomposition of the compound. Mg<sub>2</sub>Sn would be a promising anode material for lithium secondary batteries.

**[27] FORMATION OF OXIDE PHASES IN THE SYSTEM EU<sub>2</sub>O<sub>3</sub>-FE<sub>2</sub>O<sub>3</sub>**

Ristic M. Nowik I. Popovic S. Music S. - Croatica Chemica Acta. 73(2):525-540, 2000

Evolution of oxide phases in the Eu<sub>2</sub>O<sub>3</sub>-Fe<sub>2</sub>O<sub>3</sub> system was investigated by X-ray powder diffraction, Fe-57 and Eu-151 Mossbauer spectroscopy and Fourier transform infrared spectroscopy. Samples were prepared by the solid state reaction of the corresponding oxides for two molar ratios, Eu<sub>2</sub>O<sub>3</sub> : Fe<sub>2</sub>O<sub>3</sub> = 1 : 1 and 3 : 5. After heating the mixed oxide powder with molar ratio Eu<sub>2</sub>O<sub>3</sub> : Fe<sub>2</sub>O<sub>3</sub> = 1:1 up to 900 degrees C, EuFeO<sub>3</sub> and traces of Eu<sub>2</sub>O<sub>3</sub> were detected by XRD, while after additional heating up to 1100 degrees C traces of Eu<sub>3</sub>Fe<sub>5</sub>O<sub>12</sub> (EuG) were also detected. Fe-57 and Eu-151 Mossbauer spectroscopy showed the presence of EuFeO<sub>3</sub>. For the molar ratio Eu<sub>2</sub>O<sub>3</sub> : Fe<sub>2</sub>O<sub>3</sub> = 3 : 5, EuG was formed between 1100 and 1300 degrees C. In the sample produced at 1300 degrees C, the measured hyperfine fields at the iron sites, at room temperature, were H-a = 495 and H-d = 402 kOe, and the hyperfine fields at the europium sites, at 90 K, were H-I = 631 kOe and H-II = 572 kOe. Europium orthoferrite was the intermediate phase in the garnet formation. Assignations of IR bands corresponding to EuFeO<sub>3</sub> and EuG are discussed. Mechanical activation of the mixed oxide powder was important for the formation of polycrystalline EuG, as a single phase.

**[26] THE FULLERENE CROSS-DIMER C-130: SYNTHESIS AND PROPERTIES**

Komatsu K. Fujiwara K. Murata Y. - Chemical Communications. (17):1583-1584, 2000

The fullerene C-60-C-70 cross-dimer C-130 was synthesized by the mechanochemical solid-state reaction using a high-speed vibration milling technique, and was characterized by UV-vis and C-13 NMR spectroscopy as well as electrochemical methods.

**[25] RAPID SOLID-STATE TRANSFORMATION OF TETRAHEDRAL [ALH<sub>4</sub>]<sup>(-)</sup> INTO OCTAHEDRAL [ALH<sub>6</sub>]<sup>(3-)</sup> IN LITHIUM ALUMINOHYDRIDE**

Balema VP. Dennis KW. Pecharsky VK. - Chemical Communications. (17):1665-1666, 2000

Polycrystalline LiAlH<sub>4</sub> transforms into polycrystalline Li<sub>3</sub>AlH<sub>6</sub> and Al during short time (5 min) ball-milling with 3 mol% TiCl<sub>4</sub> at room temperature; solid-state rearrangement of tetrahedral [AlH<sub>4</sub>]<sup>(-)</sup> into octahedral [AlH<sub>6</sub>]<sup>(3-)</sup> at ambient conditions was previously unknown; this transformation does not occur during ball-milling of pure LiAlH<sub>4</sub> or in the presence of 3 mol% TiCl<sub>4</sub> without mechanochemical treatment.

**[24] DESCRIPTION IN A LOCAL MODEL OF THE MAGNETIC FIELD DISTRIBUTIONS OF MECHANICALLY ALLOYED FE-SI (3.5 WT%)**

Sthepa HS. Restrepo J. Fajardo M. Alcazar GAP. - Physica Status Solidi B-Basic Research. 220(1):347-349, 2000

A local model based on the idea that the atoms are distributed within a crystalline lattice, following a binomial probability method for describing the hyperfine field distribution (HFD) obtained from room temperature Mossbauer spectra is presented. It is applied to disordered, 3.5 wt%, Fe-Si mechanically alloyed during 15, 30, 50 and 75 h. This model also permits, through a fitting parameter, to evaluate the influence of the alloy milling time. The theoretical curves have fitted quite well with the experimental ones and the fitting parameters show that, in agreement with the experimental data, when each silicon atom substitutes an iron atom in the nearest neighborhood, an approximately 3

T diminishment in the hyperfine field is presented. This model also allows us to conclude that the presence of silicon atoms in the next nearest neighborhood have practically no influence in the hyperfine field, because its representative parameter fits for these alloys with a zero value.

**[23] MAGNETIC AND STRUCTURAL BEHAVIOUR OF MECHANICALLY ALLOYED FEXMN<sub>0.912</sub>-XAL<sub>0.075</sub>CO<sub>0.01</sub>CU<sub>0.003</sub> ALLOYS WITH 0.50 ≤ X ≤ 0.80**

Cruz B. Alcazar GAP. Aguilar Y. - *Physica Status Solidi B-Basic Research*. 220(1):381-383, 2000

An experimental analysis of structural and magnetic properties for the system of Fe<sub>x</sub>Mn<sub>0.912-x</sub>Al<sub>0.075</sub>Co<sub>0.01</sub>Cu<sub>0.003</sub> alloys with 0.50 less than or equal to x less than or equal to 0.80 is presented. These alloys were obtained by mechanical alloying in a planetary milling machine using 22 h of milling time. By X-ray diffractions (XRD) it is shown that alloys with a percentage between 50 and 75 at% of Fe exhibit a f.c.c. phase, while for alloys with 80 at% Fe there is a coexistence of f.c.c. and b.c.c. phases. It is also shown that the grain size does not change noticeably with Fe content increase. Using Mossbauer spectroscopy an antiferromagnetic behaviour is found, which is typical of f.c.c. phase and it tends to a paramagnetic one with Fe content increase. For the 80 at% Fe alloy, the coexistence of ferromagnetic (typical of b.c.c. phase) and antiferromagnetic phases is obtained. An increase of the isomer shifts (IS) parameter with an increase of the Fe concentration was obtained.

**[23] ROOM TEMPERATURE MAGNETIC PROPERTIES OF THE MECHANICALLY ALLOYED FE<sub>0.8</sub>-XMNXAL<sub>0.2</sub> SYSTEM**

Restrepo J. Alcazar GAP. Gonzalez JM. - *Physica Status Solidi B-Basic Research*. 220(1):429-434, 2000

Samples of nominal composition Fe<sub>0.8-x</sub>Mn<sub>x</sub>Al<sub>0.2</sub> (0 less than or equal to x less than or equal to 0.3) were prepared by mechanical alloying from high-purity powder elements. Milling times of 24, 72, 144 and 308 h and a sample-balls weight ratio of 1:12 were considered in the present study. The room temperature magnetic properties were studied by using Fe-57 Mossbauer spectroscopy and vibrating sample magnetometry. Thus, the Mn content dependences of the average hyperfine field, the specific magnetization and the coercive force were obtained. Results revealed the occurrence of a ferro-paramagnetic Mn driven phase transition attributed to the increase of competitive interactions. The Mn concentration for which this transition occurs, was influenced by the milling time and the structure. The phase distribution of the samples was determined through X-ray diffraction from which structural phase changes of the type b.c.c. to f.c.c., were detected. Such changes were also strongly influenced by the milling time and the Mn concentration. Hence, the manganese and the milling time appeared as stabilizing factors of the f.c.c. phase inducing a paramagnetic behavior.

**[22] EFFECT OF MILLING TIME AND COMPOSITION ON THE MAGNETIC AND STRUCTURAL PROPERTIES OF THE SYSTEM FEXMN<sub>0.95</sub>-XAL<sub>0.05</sub> (0.40 ≤ X ≤ 0.50)**

Rodriguez R. Alcazar GAP. - *Physica Status Solidi B-Basic Research*. 220(1):441-444, 2000

The magnetic and structural properties of the Fe<sub>x</sub>Mn<sub>0.95-x</sub>Al<sub>0.05</sub> (0.40 less than or equal to x less than or equal to 0.50) alloy system prepared by mechanical alloying during 12, 24, 36 and 48 hours, were studied by Mossbauer spectroscopy and X-ray diffraction. By X-ray it was proved that the lattice parameter is nearly constant (approximate to 3.64 Angstrom) with the milling time and composition. All the diffractograms exhibit the f.c.c. phase characteristic peaks, except for the 12 hours milling samples which present additional Fe, Mn and Al peaks. Then for this time the alloys were not consolidated. Also, it was proved that the mean grain size increases between 12 and 24 hours milling and then decreases for 36 and 48 hours. This decrease is attributed to the breaking of the grains. The Mossbauer spectra of the 12 hours milling samples were fitted with a hyperfine field distribution and a sextet, corresponding to the f.c.c. alloy and the Fe, respectively. From the fits of the spectra it was obtained that the mean hyperfine field decreases from 2.7 to 2.2 T when the Fe content increases from 40 to 50% for the 12 hours milling samples and it remains nearly constant with the Fe content (approximate to 2.9T) for 24, 36 and 48 hours milling samples.

**[21] MAGNETIC AND STRUCTURAL STUDY OF MECHANICALLY ALLOYED FE<sub>0.7</sub>-XMNXAL<sub>0.3</sub>**

Rico MM. Zamora LE. Alcazar GAP. Gonzalez JM. Hernando A. - *Physica Status Solidi B-Basic Research*. 220(1):445-448, 2000

In this work the magnetic and structural properties of the Fe<sub>0.7-x</sub>Mn<sub>x</sub>Al<sub>0.3</sub> (0.00 less than or equal to x less than or equal to 0.30) alloy system, prepared by mechanical alloying are reported. The study was carried out, using X-ray diffraction (XRD), Mossbauer spectroscopy (MS) and vibrating sample magnetometry (VSM), in samples prepared by milling for 24, 48, 72 and 84 hours pure elements powders. X-ray data showed that, after milling, all the samples had a b.c.c. structure. The exception are those with x = 0.30, milled for 72 and 84 hours, which presented a mixture of b.c.c. and f.c.c. phases. The lattice parameters were, for each composition, nearly independent of the milling time and increase with x. The mean hyperfine fields of the different alloys, calculated from the fits of the Mossbauer spectra to a hyperfine field distribution (HFD), decreased monotonically with the increase of the Mn content and increased with the milling time. Similar results were obtained for the saturation magnetization dependence on the Mn content and milling times. These results allow to conclude that the increase in the Mn content produces a small lattice expansion and a weakening of the ferromagnetic character of the system. In contrast with this, the induction of disorder with the milling time enhances the ferromagnetic character of the samples.

**[20] USE OF EMANATION THERMAL ANALYSIS IN CHARACTERISATION OF NANOSIZED HEMATITE PREPARED BY DRY GRINDING OF GOETHITE**

Perez-Maqueda LA. Subrt J. Balek V. Criado JM. Real C. - *Journal of Thermal Analysis*. 60(3):997-1007, 2000.

Hematite nanoparticles of narrow size distribution were prepared by grinding of goethite. Intermediate and final products of grinding were characterised by different techniques, including the less-common emanation thermal analysis (ETA). ETA was shown to be a useful technique for characterising processes of surface annealing, initial sintering and growth of hematite particles under in situ conditions of thermal treatment. A good agreement was found between results of ETA, TG, XRD, IR spectrometry, transmission electron microscopy and scanning electron microscopy, used for characterisation of thermal behaviour of the goethite samples ground for varying time (0-70 h).

**[19] FLOTATION BEHAVIOR OF SULFIDES ON MECHANICAL ACTIVATION**

Kirillova YA. Yusupov TS. Shumskaya LG. Asanov IP. - *Journal of Mining Science*. 36(1):87-90, 2000

Mechanical activation during grinding affects the flotability of pyrite, arsenopyrite, and galena. It is established that the S, Fe, and As atoms at the surfaces of the initial materials are mainly in the oxidized state. The transformations occurring on the surfaces of mechanically activated sulfide minerals are the cause of the changes in flotability.

**[18] SYNTHESIS OF Nb/Nb5Si3 IN-SITU COMPOSITES BY MECHANICAL MILLING AND REACTIVE SPARK PLASMA SINTERING**

Ma CL. Kasama A. Tan Y. Tanaka H. Tanaka R. Mishima Y. Hanada S. -Materials Transactions Jim. 41(6):719-726, 2000

Nb-ss/Nb5Si3 in-situ composites (Nb-16 mol%Si) for high-temperature structural applications were synthesized by reactive spark plasma sintering (RSPS), mechanically milled (MM) Nb-Si powders. Microstructures were characterized by OM, SEM, XRD and EPMA, and the mechanical properties of the composites were investigated by microhardness and compression tests. This consolidation process can produce the compacts of Nb-ss/Nb5Si3 in-situ composites having equiaxed grain microstructures and a nearly full density when RSPS is carried out above the melting point of silicon. The RSPS in-situ composites exhibit large compressive ductility at room temperature and considerably high strength at elevated temperatures.

**[17] GIBBS-THOMSON EFFECT IN NANOCRYSTALLINE FE-GE**

Sarkar S. Bansal C. Chatterjee A. - Physical Review B. 62(5):3218-3222, 2000

We studied the phase transformation behavior of chemically disordered bcc (alpha) phase Fe<sub>1-x</sub>Ge<sub>x</sub> alloys near the Fe<sub>3</sub>Ge stoichiometry synthesized in the nanocrystalline state by mechanical alloying of the elemental constituents. The evolution of the equilibrium L1(2) ordered (epsilon') phase was seen to occur via a metastable DO<sub>3</sub>-ordered (alpha(1)) phase, but a significant alpha(1)->epsilon' phase transformation took place only after the growth of the grains. This behavior is understood with the help of a capillary effect or the Gibbs-Thomson effect wherein the grain boundary energy of the nanosize grains raises the Gibbs free energy of the epsilon' phase relative to the alpha phase for small sizes and the epsilon' phase grows only after a certain grain size is reached.

**[15] EFFECT OF HEBM ON THE CATION DISTRIBUTION OF MN-FERRITE**

Mahmoud MH. Hamdeh HH. Abdel-Mageed AI. Abdallah AM. Fayek MK. - Physica B. 291(1-2):49-53, 2000

The effect of high-energy ball milling (HEBM) on the cation distribution of manganese ferrite (MnFe<sub>2</sub>O<sub>4</sub>) was investigated by means of Mossbauer spectroscopy in the presence of high external magnetic field. The results show that the Fe<sup>3+</sup> ion occupancy to A site increases with increasing the milling time. Particle-size investigation shows that this increase of the inversion parameter could be attributed to a particle-size reduction in the ball-milled samples

**[14] INFLUENCE OF SiC PARTICULATE SIZE ON THE MICROSTRUCTURAL EVOLUTION AND MECHANICAL PROPERTIES OF AL-6Ti-6Nb MATRIX COMPOSITES**

Jia DC. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 289(1-2):83-90, 2000

Three kinds of SiC particulates (SiCp), whose diameters range from millimeter through micrometer to nanometer (designated as m-SiCp, mu-SiCp and n-SiCp, respectively), were separately incorporated into an Al-6wt.%Ti-6wt.%Nb alloy by high energy ball milling. The as-milled composite particles are appreciably refined particularly when finer SiC particulate was added, and so do the in situ formed Al<sub>3</sub>Ti, AlNb<sub>2</sub> dispersoids as well as the Nb particles. The room temperature strength and ductility of the composites, except for the stiffness, deteriorate unexpectedly. Nonetheless, elevated temperature strength and thermal stability of the composites are improved and greater strength over that of the Al-6Ti-6Nb matrix alloy is exhibited above 240 degrees C. The changes of the mechanical properties from room to elevated temperature are correlated with the microstructure and fractographs.

**[13] EVOLUTION OF MICROSTRUCTURES AND NITROGEN SORPTION DURING HIGH-ENERGY MILLING OF SILICON IN AMMONIA**

Yang ZG. Ren RM. Shaw LL. - Journal of the American Ceramic Society. 83(8):1897-1904, 2000

The structural transformation, microstructural changes, and nitrogen sorption of crystalline Si induced by high-energy ball milling in ammonia have been investigated. It is found that significant refinement of particles and crystallites occurs at the early stage of milling (within similar to 10 h of milling), after which the average particle size and crystallite size decrease gradually and approach a lower limit of similar to 100 and 6.0 nm, respectively. It is proposed that refinement of particle sizes is dominated by the fragmentation process, whereas refinement of crystallite sizes is dictated by plastic deformation. Substantial amorphization does not occur until most of the crystallites have reached the average size of 6.5-6.0 nm. The crystallite-refinement-induced amorphization has been identified as the major mechanism for the nanocrystalline to-amorphous transformation. A substantial amount of nitrogen is sorbed by Si powders and primarily dissolved in the amorphous Si phase.

**[12] MECHANOCHEMICAL REACTION OF POLYMERIC OXOVANADIUM(IV) COMPLEXES WITH SCHIFF BASE LIGANDS DERIVED FROM 5-NITROSALICYLALDEHYDE AND DIAMINES**

Tsuchimoto M. Hoshina G. Yoshioka N. Inoue H. Nakajima K. Kamishima M. Kojima M. Ohba S. - Journal of Solid State Chemistry. 153(1):9-15, 2000

Light brown [VO(5-NO(2)salen)] . 2H(2)O (1; H-2(5-NO<sub>2</sub> salen) = N, N'-di-5-nitrosalicylidene-1,2-ethanediamine) and orange [VO(5-NO(2)sal-meso-stien)] . 0.5H(2)O (2; H-2(5-NO(2)sal-meso-stien) = N,N'-di-5-nitrosalicylidene-(R,S)(S,R)-1,2-diphenyl-1,2-ethanediamine) which have linear chain structures in the solid state turn dark brown and yellow-brown, respectively, when ground thoroughly in mortars. The IR spectra of 1 and 2 in Nujol mulls show strong V = O stretching bands at 875 and 905 cm<sup>-1</sup>, respectively. On the other hand, the spectra of the finely ground complexes in Nujol mulls show very weak V = O stretching bands. X-ray powder diffraction patterns of the ground complexes scarcely show diffraction peaks. The spectroscopic and magnetic data suggest that the linear chain structures in the crystals of 1 and 2 are destroyed on grinding to yield the fragments of the polymeric chains.

**[11] STUDIES OF X-RAY AMORPHOUS PHASE IN MECHANOCHEMICAL SYNTHESIS OF IRON SILICIDES FROM ELEMENTS**

Golubkova GV. Lomovsky OI. Vlasov AA. Davlitova LS. Belyaev EY. Malakhov VV. - Journal of Alloys & Compounds. 307:131-136, 2000

Iron disilicide synthesis was developed by means of mechanochemical activation under intensive mechanical treatment. The X-ray amorphous phases formed during mechanical treatment of initial iron and silicon were investigated. The methods of the differential dissolution, differential thermal analysis and X-ray phase analysis were used. The formation of supersaturated amorphous solid solution of silicon in iron with limited concentration 17 at.% Si takes place during the initial period of activation. Primary phase obtained in this system is iron monosilicide formed in the decomposition of amorphous supersaturated solution.

**[10] CONCERNING THE TRANSFORMATIONS OF Ti3Ir ALLOY DURING HIGH-ENERGY BALL-MILLING**

Balema VP. Pecharsky AO. Pecharsky VK. - Journal of Alloys & Compounds. 307:184-190, 2000

Thermodynamically stable Ti3Ir with the Cr3Si (A15)-type crystal structure can gradually be transformed into a metastable Ti3Ir alloy with a bcc crystal structure by high-energy ball-milling for 12.5 h. The long-term ball-milling for up to 112 h, however, is accompanied by a solid-state reaction between Ti3Ir and the vial material leading to the formation of a nano-crystalline or amorphous MgZn2-type phase with the Ti(Fe1.85Ir0.15) stoichiometry and a micro-crystalline thermodynamically stable CsCl-type phase. Both the CsCl- and MgZn2-type phases were obtained in the crystalline form after annealing ball-milled Ti3Ir. Properties of the A15 Ti3Ir alloy changed considerably after ball-milling. The superconducting transition observed in the original A15 alloy at similar to 4.3 K no longer exists in the ball-milled material. The electrochemically determined hydrogen storage capacity of the ball-milled alloy also decreases in comparison to the A15 material from 269 to 104 mAh/g. On the contrary, the electrochemical discharge capacity of Ti3Ir increases from 45 to 104 mAh/g after ball-milling.

**[9] EFFECT OF CARBON-CONTAINING COMPOUNDS ON THE HYDRIDING BEHAVIOR OF NANOCRYSTALLINE Mg2Ni**

Bouaricha S. Dodelet JP. Guay D. Huot J. Boily S. Schulz R. - Journal of Alloys & Compounds. 307:226-233, 2000

Nanocrystalline Mg2Ni was prepared by high-energy ball milling during 40 h and then further milled during 30 min with 3, 5 and 10 wt.% of carbonaceous compounds. Three carbonaceous compounds were used, namely fullerene (C-60), graphite and a highly conducting carbon support (Vulcan XC72). The structural and hydriding characteristics of the resulting materials were compared with those of the primitive material. The specific surface area of nanocrystalline Mg2Ni increases from 1.06 m(2) g(-1) to 3.45 m(2) g(-1) or 6.00 m(2) g(-1) when it is milled with 5 or 10 wt.% C-60. The dissolution of C-60 in toluene yields a further increase of the specific surface area. The specific surface area of Mg2Ni milled with 5 wt.% graphite is 3.61 m(2) g(-1), but almost no change in the specific surface area of Mg2Ni is observed when it is milled with Vulcan. No change in the crystallographic structure of Mg2Ni could be evidenced by X-ray diffraction as a result of the milling step with either carbonaceous compound. When normalized for the total amount of hydride-forming material, Mg2Ni/C-60, Mg2Ni/graphite and Mg2Ni/Vulcan have the same hydrogen absorption capacity as that of nanocrystalline Mg2Ni. On the contrary an irreversible loss of hydrogen absorption capacity is observed following the leaching of C-60 from the material. This most probably results from a slight oxidation of magnesium during the leaching process. At 275 degrees C, there is no noticeable change in the kinetics of absorption between the various compounds, but the time needed to complete desorption of Mg2Ni under vacuum is drastically reduced by a factor of 2-3 for Mg2Ni/C-60, Mg2Ni/graphite and Mg2Ni/Vulcan.

**[8] FORMATION OF TITANIUM NITRIDES VIA WET REACTION BALL MILLING**

Zhang F. Kaczmarek W. Lu L. Lai MO. - Journal of Alloys & Compounds. 307:249-253, 2000

A new mechanochemical route to synthesize TiN has been developed by ball milling elemental titanium powders with the organic compound pyrazine in a benzene solution for periods of up to 336 h. The structures of the milled powders were examined by means of X-ray diffractometry. Titanium nitrides were formed directly during the milling. Unlike the dry ball-milling process, the present wet milling resulted in the formation of an intermediate titanium nitride Ti2N which has never been found in previous studies using dry milling. With increasing milling time, Ti2N was observed to gradually transform into the stoichiometric compound TiN. Upon heating, the Ti2N formed during milling was completely transformed to TiN. The intrinsic mechanisms of the solid-liquid reactions are discussed.

**[7] SYNTHESIS AND CHARACTERISTICS OF CONSOLIDATED NANOCRYSTALLINE TWO-PHASE Ag50Ni50 ALLOY BY HOT PRESSING**

Zhao ZL. Zhao Y. Niu Y. Wang CL. Wu WT. - Journal of Alloys & Compounds. 307:254-258, 2000

Silver and nickel are mutually only very slightly soluble and have a positive heat of mixing. Ag50Ni50 powder was prepared by mechanical alloying (MA) for 200 h and subsequent hot pressing (HP) at 620 degrees C under 60 MPa. XRD results indicated that ball milling produced a two-phase mixture of supersaturated, nanocrystalline Ag-rich (alpha-phase) and Ni-rich (beta-phase) solid solutions with grain sizes of 5 and 3 nm, respectively. The grain sizes increased to about 40 and 3-6 nm after hot pressing. The HP Ag50Ni50 had a density very close to the theoretical value. The HP alloy consisted of an interconnected network of the alpha-phase and islands of a two-nanophase mixture rich in the beta-phase. The stability of the microstructure of the alloy and the effects of mechanical alloying on the supersaturation of the two phases and on the formation of the Ag-rich network are also discussed.

**[6] HYDROGENATION OF TiFe BY HIGH-ENERGY BALL MILLING**

Chiang CH. Chin ZH. Perng TP. - Journal of Alloys & Compounds. 307:259-265, 2000

The hydrogenation properties of TiFe, TiFe2 and pure Ti during high-energy ball milling in hydrogen atmosphere were studied. By ball milling, TiFe could absorb hydrogen without activation treatment. For Ti powder, a single phase TiH1.924 was formed. In addition, TiFe2 could also be hydrided by ball milling in hydrogen, which was ascribed to decomposition to form TiFeHx, TiH1.924 and Fe. Based on the hydrogenation properties of Ti, TiFe, and TiFe2 during dynamic ball milling and the thermal stability of the milled powders, it is proposed that the reactions in the milling of TiFe in hydrogen involve four steps: (1) fresh surfaces created by collision with the balls; (2) absorption of hydrogen by the powder; (3) oversaturation of hydrogen in the powder; (4) decomposition of TiFeHx to TiH1.924 and Fe.

**[5] PREPARATION AND HYDROGENATION OF MULTICOMPONENT AB(2)-TYPE Zr-Mn-V-CO-NI AMORPHOUS ALLOY**

Lai LC. Lee CL. Perng TP. - Journal of Alloys & Compounds. 307:266-271, 2000

Amorphous powder of the multicomponent alloy  $ZrMn_{0.6}V_{0.1}Co_{0.2}Ni_{1.2}$  was prepared by ball milling of the intermetallic compound. The amorphous phase was formed after milling for 80 h. Hydrogenation of the amorphous powder was conducted and a comparison was made with that of the crystalline alloy. The absorption rate increased as the temperature increased, but the rate was slower than that of the crystalline phase. There was no plateau region in the pressure-composition-temperature curve. The maximum hydrogenation capacity was less, and a large amount of hydrogen was trapped at lower pressures.

#### **[4] HYDROGEN STORAGE PROPERTIES OF AMORPHOUS AND NANOCRYSTALLINE $MmNi_{4.2}Al_{0.8}$ ALLOYS**

Jurczyk M. - Journal of Alloys & Compounds. 307:279-282, 2000

The hydrogen storage properties of amorphous and nanocrystalline  $MmNi_{4.2}Al_{0.8}$  alloys were studied. These materials were prepared by mechanical alloying (MA). The amorphous phase forms directly from the starting mixture of the elements, without other phase formation. Heating the MA powders at 750 degrees C for 0.5 h resulted in the formation of the hexagonal CaCu<sub>5</sub>-type structure. Gas-phase hydrogen sorption in an amorphous as well as a nanocrystalline  $MmNi_{4.2}Al_{0.8}$  alloy was also investigated. The nanocrystalline powders prepared by MA have a lower hydrogen storage capacity than powders from arc-melted ingots. The difference is mainly due to iron impurities trapped in the mechanically alloyed powders from erosion of the milling media.

#### **[3] HYDROGEN STORAGE CHARACTERISTICS OF MAGNESIUM MECHANICALLY ALLOYED WITH $YNi_{5-x}Al_x$ (X=0, 1 AND 3) INTERMETALLICS**

Khrussanova M. Bobet JL. Terzieva M. Chevalier B. Radev D. Peshev P. - Darriet B. - Journal of Alloys & Compounds. 307:283-289, 2000

The hydrogen sorption properties of the composite materials Mg+10 wt%  $YNi_{5-x}Al_x$  (x=0, 1 and 3) prepared by ball milling were studied. In the case of  $YNi_2Al_3$  addition, mixtures with a nominal content of 1 and 50 wt% of the intermetallic compound were also investigated. The crystal structure of the  $YNi_4Al$  phase was determined (CaCu<sub>5</sub> type structure, a=0.4940 nm, c=0.4036 nm). It was established that the presence of  $YNi_4Al$  or  $YNi_2Al_3$  in the composites significantly improved the kinetics of hydrogen absorption and desorption, whereas  $YNi_5$  did not affect the sorption process. An explanation of the results obtained, based on the catalytic effect of the  $YNi_4Al$  and  $YNi_2Al_3$  intermetallics is proposed. It is shown that a higher intermetallic content of the composites is accompanied by: (i) an increase of the initial rate of their hydriding, (ii) a decrease of the maximum hydrogen capacity, and (iii) an increasing of the latent period of dehydriding.

#### **[2] THE ELECTRONIC AND ELECTROCHEMICAL PROPERTIES OF THE $LaNi_5$ , $LaNi_4Al$ AND $LaNi_3AlCo$ SYSTEMS**

Szajek A. Jurczyk M. Rajewski W. - Journal of Alloys & Compounds. 307:290-296, 2000

Nanocrystalline  $LaNi_5$ -type materials have been prepared by mechanical alloying (MA) followed by annealing. The amorphous phase forms directly from the starting mixture of elements, without formation of other phases. The  $LaNi_5$ ,  $LaNi_4Al$  and  $LaNi_3AlCo$  systems crystallize in the hexagonal CaCu<sub>5</sub> type structure (P6/mmm). The electrochemical properties of these type of materials, in which there has been a partial substitution of Ni by Al and Al-Co, have been investigated and discharge capacity enhancement has been observed. The electronic structure has been studied by the tight binding version of the linear muffin-tin orbital method in the atomic sphere approximation (TB-LMTO ASA). Total energy calculations showed that the Al and Co impurities prefer 3g positions.

#### **[1] MATERIALS PREPARATION BY BALL MILLING**

Dunlap RA. Small DA. MacKay GR. O'Brien JW. Dahn JR. Cheng ZH. - Canadian Journal of Physics. 78(3):211-229, 2000

Ball milling is an important method for the preparation of materials both for the investigation of fundamental physical properties and for a number of possible commercial applications. In the present report, the basic principles of ball milling are discussed. This includes a brief overview of experimental techniques as well as an introduction to modeling methods used to analyze milling processes. This will be followed with a discussion of some of the experiments involving ball milling that are being conducted at Dalhousie University. Specifically, a summary of results on mechanically alloyed Fe-Al and experimental studies of the formation of metal hydrides by ball milling will be presented.

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