

# RESEAU FRANÇAIS DE MECANOSYNTHESE

## Lettre N°65

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**Août 2000**

**181 (+1) Groupes de Recherche**  
**(dont 108 (+1) à l'étranger / 32 Pays)**

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

### **International Editorial LRFM Committee**

**P. Balaz** - Institute of Geotechnics - Slovak Academy of Science - Slovakia

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**M. Senna** - Faculty of Science and Technology - Japan

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

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**Books (related to Nanomaterials)**

**Bibliography**

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#### **1 Nouvelle Adhésion**

**V. Berbenni** - Univ. di Pavia - Dpt Chimica Fisica - Pavia - Italie

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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.

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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>

### **VARIO-PLANETARY MILL "pulverisette 4"**

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.

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.

When particles < 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.

#### **Method of operation:**

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.

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.

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).

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.

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.

#### **Features of performance:**

• for the first time, all grinding parameters can be selected at will for optimal preparation of sample

• 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 (direction of rotation reversed periodically) to improve the grinding results

• Emulation of various ball mills

• Variably adjustable pressure on sample (friction and/or impact)

• Final fineness << 1 µm

• Simultaneous grinding in up to 4 small or 2 large grinding bowls

• Quick, secure fastening of the grinding bowls

• Ease of cleaning

#### **contact:**

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](mailto:info@fritsch.de)

Internet: <http://www.fritsch.de>

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**ANNONCE DE CONGRES ET / OU ECOLES  
CONGRESS AND SCHOOL ANNOUNCEMENTS**

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**NCM8  
8<sup>th</sup> International Conference on the Structure of  
Non - Crystalline Solid**

6 - 11 Aout 2000

Website : <http://www.sgt.org>

**XIV<sup>th</sup> International Symposium on the Reactivity of Solids**

Budapest, Hungary through 27-31 August 2000

<http://www.jate.u-szeged.hu/~isrs14>.

**Solid State Chemistry 2000**

Prague, Czech Republic, September 3 - 8,2000

and

**3<sup>rd</sup> INCOME**

**International Conference on Mechanochemistry and Mechanical Alloying**

Prague, Czech Republic, September 4 - 8,2000

Organised by the Institute of Inorganic Chemistry (UACH), Czech Republic

**WebSite** : <http://www.iic.cas.cz/INCOME.htm>

**Inorganic Materials Conference**

University of California Santa Barbara, USA

13 - 16 September 2000 <http://www.elsevier.com/locate/im2000>

**Congrès de la Société Française de Chimie**

Univ. Rennes I

18 - 22 Septembre 2000

**website** : <http://www.sfc.fr>

**MATERIALS WEEK**

**International Congress on Advanced Materials,  
their Processes and Applications**

25 - 28 September 2000

ICM – International Congress Centre Munich in Conjunction with the Exhibition Materialica

website : [www.materialsweek.org](http://www.materialsweek.org)

**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>

**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)

**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)

**PM 2000**

**Powder Metallurgy World  
Congress & Exhibition**

12 - 16 Novembre 2000 Kyoto - Japon

Contact : Fax : 81 - 3 - 3423 - 1600

**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  
Or visit the conference web site:  
<http://mape.waikato.ac.nz/conferences/amp.htm>

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

Messina Italy  
22th to 25th of November 2000  
(for details see LRFM60)

**2000 MRS Fall Meeting**  
27 Nov - 1 Dec. 2000  
Boston - Massachusetts - USA  
(for details see LRFM60)

**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)

**PM2 TEC2001**  
**2001 International Conference on Powder Metallurgy  
& Particulate Materials**  
13 - 17 May 2001 - New Orleans - USA  
Contact : MPIF

**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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**COLLOQUE SUR LES INNOVATIONS  
DANS LES MATERIAUX FRITES**

Poitiers-Futuroscope  
3-4-5 juillet 2001

**NOUVEAU**

Ce colloque, organisé par une commission mixte SF2M/Groupe Français de la Céramique, vise à réunir la communauté industrielle et scientifique du domaine des poudres et matériaux frittés (métalliques et céramiques), afin de faire le point sur les innovations et avancées technologiques de la dernière décennie. Ces innovations portent sur toutes les étapes du procédé, de l'élaboration de la poudre jusqu'à la pièce frittée, ainsi que sur les applications. Elles peuvent être constituées aussi bien de nouvelles connaissances fondamentales que de nouvelles solutions techniques. Le colloque se déroulera sur 3 jours et comprendra une demi-journée consacrée à des visites de laboratoires ou de sites industriels.

Les contributions, sous forme de communications orales ou d'affiches, sont souhaitées dans les quatre thèmes suivants :

- Poudres et Mélanges
- De la poudre au matériau
- De la poudre à la pièce finalisée
- Propriétés et applications des matériaux frittés.

Pour toute information sur ce colloque et recevoir la circulaire de pré-inscription, s'adresser à SF2M. Les Fontanelles, 1 rue de Craïova. 92024 Nanterre Cedex, tél. :01 41 02 03 90 ou 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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**CH. GRAS**

**6 Septembre 2000 - Univ. Dijon**

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

**Jury :**

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

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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 microstructurale 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. Goeriot (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 tungstène 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 BROYAGE 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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**F.CHARLOT**

**Etude et compréhension des réactions auto-entretenuées activées mécaniquement. Elaboration du composé FeAl nanostructuré.**

6 Déc. 1999 - Université de Technologie de Belfort - Montbéliard

Nanomatériaux : Elaboration et Transitions de Phases Hors Equilibre, UPR 806 CNRS, UTBM - Sévenans.

Matériaux à Grains Fins, LRSS UMR 5613 CNRS-Université de Bourgogne.

**Jury** : G. Bertrand, G.LeCaer (Rapp.), F.Thévenot (Rapp.), F.Bernard (Co - Dir.), E.Gaffet (Co - Dir.), J.C.Gachon, M.Bessière, M.Gailhanou

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**Frédéric BERNARD**

25 Novembre 1999 - Amphi de l'ESIREM - Dijon

**De l'introduction de " mécanique " dans l'élaboration de la poudre au massif nanométrique vers la maîtrise des propriétés thermomécaniques.**

**Jury** :D.LOUER, Directeur de Recherches CNRS(Université de Rennes II) rapporteur H. VAN DAMME, Professeur (Université d'Orléans) rapporteur J.C. TEDENAC, Professeur (Université de Montpellier II) Rapporteur J. FOCT, Professeur (Université de Lille) G. LE CAER , Directeur de recherches CNRS (Ecole des Mines, Nancy) G. BERTRAND, Professeur (Université de Bourgogne) A. NONAT, Directeur de Recherches CNRS (Université de Bourgogne) J.C. NIEPCE, Professeur (Université de Bourgogne)

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**Frédérique PERROT-SIPPLE**

17 Novembre 1999 - Université de Bourgogne - Dijon

**Maitrise de la taille de nanograins d'oxydes de structure perovskite pour applications électrocéramiques:**

**- Synthèse par chimie douce,- Broyage par attrition.**

**Jury** : M. J.-M. HAUSSONNE Professeur, Ecole d'Ingénieurs de Cherbourg M. A. ROUSSET Professeur, Université de Toulouse M BEAUGER Alain Ingénieur de Recherche et Développement TPC Saint Apollinaire M. BERTRAND Gilles Professeur, Université de Bourgogne M. CHARTIER Thierry Chargé de Recherche, ENSCI de Limoges M. HUGENTOBLE Denis Directeur Stratégie et Développement ligne céramique, TPC Saint Apollinaire M. MUTIN Jean-Claude Directeur de Recherche, Université de Bourgogne Mme RIEUX Nadine Ingénieur de Recherche Alstom-PERT, Massy **Directeurs de thèse**: M. D. AYMES Maître de Conférences, Université de Bourgogne M. P. PERRIAT Professeur, INSA de Lyon

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**H. SOUHA**

Thèse de Doctorat d'Etat Marocaine

Faculté des Sciences Dahr El Mehraz Fes.

**Elaboration par recuit et par réaction de combustion du composé Cu<sub>3</sub>Si à partir d'un mélange de poudres activées mécaniquement. Réactivité du composé Cu<sub>3</sub>Si vis à vis du chlorure cuivreux.**

**Jury** :B. Gillot, G. Bertrand, F. Bernard (Co - Dir.), E. Gaffet (Co - Dir.)

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**O. Held**

**"Etude des réactions de combustions solide-solide ou solide liquide auto-entretenuées pour différents intermétalliques du système Al-Ni-Ti.**

**Elaboration de ces mêmes intermétalliques par broyage mécanique et étude de leur cinétique de cristallisation**

Nancy, Faculté des Sciences, le 5/11/99.

**Jury** : J. L. Jorda, J. M. Moreau, P. Satre, J. C. Gachon, F. A. Kuhnast, F. Bernard, J. F. Bézar, M. Bessière.

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**D. Cracco**

**"Recherche de Nouveaux Alliages Hydrurables de Forte Capacité Massique Utilisable comme Matériaux d'Électrode Négative d'Accumulateur Ni - MH"**

CNRS - Thiais - France - 25 Juin 1999

**Jury** : B. Darriet, L. Schlapbach, B. Knosp, R. Portier, A. Percheron - Guégan

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**A. Gentil - Sagot**

**Amélioration de la tenue au fluage d'un alliage d'argent (AIC) par introduction d'une dispersion d'oxydes.**

**Elaboration par Métallurgie des Poudres**

**Ecole des Mines - Paris - 17 Juin 1999**

**Jury** : M. Grosbras, L. Charrin, S. Kleine, D. Havart, J. - L. Strudel, Y. Bienvenu

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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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**Ph D Position and Post Doc Position  
Requests - Proposals**

**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**

Associate professor - Institut de Chimie de la Matière Condensée de Bordeaux Avenue du Dr A. Schweitzer  
33608 Pessac Cedex FRANCE

Tel : 33-(0)5-56-84-26-53 Fax : 33-(0)5-56-84-24-80 e mail : bobet@icmcb.u-bordeaux.fr

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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, 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

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

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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)

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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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**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)

**[44] MECHANICAL ACTIVATION OF INORGANIC MATERIALS - INTRODUCTION**  
MECHANICAL ACTIVATION OF INORGANIC MATERIALS (Series: SERBIAN ACADEMY OF SCIENCES AND ARTS MONOGRAPHS), 1998, Vol 639, Iss 38, pp 1 -3RD MEETING OF THE DEPARTMENT OF TECHNICAL SCIENCES ON MECHANICAL ACTIVATION OF INORGANIC MATERIALS; BELGRADE, YUGOSLAVIA. , 1998

**[43] METHODS OF MECHANICAL ACTIVATION**  
MECHANICAL ACTIVATION OF INORGANIC MATERIALS (Series: SERBIAN ACADEMY OF SCIENCES AND ARTS MONOGRAPHS), 1998, Vol 639, Iss 38, pp 3-4 - 3RD MEETING OF THE DEPARTMENT OF TECHNICAL SCIENCES ON MECHANICAL ACTIVATION OF INORGANIC MATERIALS; BELGRADE, YUGOSLAVIA. , 1998

**[42] THERMODYNAMICS AND KINETICS OF MECHANICAL ACTIVATION**  
MECHANICAL ACTIVATION OF INORGANIC MATERIALS (Series: SERBIAN ACADEMY OF SCIENCES AND ARTS MONOGRAPHS), 1998, Vol 639, Iss 38, pp 49-55 - 3RD MEETING OF THE DEPARTMENT OF TECHNICAL SCIENCES ON MECHANICAL ACTIVATION OF INORGANIC MATERIALS; BELGRADE, YUGOSLAVIA. , 1998

**[41] MECHANICAL ACTIVATION AND ITS POSSIBILITIES**  
MECHANICAL ACTIVATION OF INORGANIC MATERIALS (Series: SERBIAN ACADEMY OF SCIENCES AND ARTS MONOGRAPHS), 1998, Vol 639, Iss 38, pp 56-61 - 3RD MEETING OF THE DEPARTMENT OF TECHNICAL SCIENCES ON MECHANICAL ACTIVATION OF INORGANIC MATERIALS; BELGRADE, YUGOSLAVIA. , 1998

**[40] REACTIVITY OF CU<sub>3</sub>SI OF DIFFERENT GENESIS TOWARDS COPPER(I) CHLORIDE**

Souha H. Bernard F. Gaffet E. Gillot B. - Thermochimica Acta. 351(1-2):71-77, 2000

A comparative study of the reactivity between copper(I) chloride and three types of Cu<sub>3</sub>Si obtained in a molten medium (Cu<sub>3</sub>Si-Ref) and from mechanical activation following an annealing process (Cu<sub>3</sub>Si-M2AP) or a self-propagating high-temperature synthesis (Cu<sub>3</sub>Si-MASHS) was performed by thermogravimetry under vacuum using non;isothermal and isothermal methods of kinetic measurement. It was established that for the three Cu<sub>3</sub>Si/CuCl systems, the acceleration and decay stages in the temperature range 145-215 degrees C are very closely approximated by an equation of the Prout-Tompkins type where an autocatalytic process was proposed. The lower apparent activation energy obtained for the Cu<sub>3</sub>Si-MASHS/CuCl system (63 kJ mol<sup>-1</sup>) against 68 and 78 kJ mol<sup>-1</sup>) for Cu<sub>3</sub>Si-M2AP and Cu<sub>3</sub>Si-Ref, respectively) has been attributed to a small grain size which induces nanoscale contacts between reactants and impedes CuCl to sublime.

**[39] STRUCTURAL AND SUPERCONDUCTING PROPERTIES OF MECHANICALLY ALLOYED Y-PD1-XTMX-B-C (TM = NI, PT)**

Gumbel A. Eckert J. Handstein A. Schultz L. - Physica B. 284(Part 1):1107-1108, 2000

Among the borocarbides the highest T<sub>c</sub> of about 23K occurs in the system Y-Pd-B-C. Pseudo-quaternary Y(Pd<sub>1-x</sub>Ni<sub>x</sub>)(<sub>2</sub>)B<sub>2</sub>C and Y(Pd<sub>1-x</sub>Pt<sub>x</sub>)(<sub>2</sub>)B<sub>2</sub>C samples were prepared by mechanical alloying followed by a thermal treatment. For Y(Pd<sub>1-x</sub>Ni<sub>x</sub>)(<sub>2</sub>)B<sub>2</sub>C superconductivity was only detected for Ni contents x > 0.3 after annealing. Pt stabilizes the formation of the tetragonal superconducting phase leading to an almost single-phase X-ray diffraction pattern and to T<sub>c</sub> near 15K for Y(Pd<sub>0.8</sub>Pt<sub>0.2</sub>)(<sub>2</sub>)B<sub>2</sub>C after annealing at 1273 K.

**[38] AN INVESTIGATION OF THE LOCAL ENVIRONMENTS OF TIN IN TIN-DOPED ALPHA-FE<sub>2</sub>O<sub>3</sub>**

Berry FJ. Bohorquez A. Helgason O. Jiang JZ. McManus J. Moore E. Mortimer M. Mosselmans F. Morup S. - Journal of Physics-Condensed Matter. 12(17):4043-4052, 2000

The extended x-ray absorption fine structure (EXAFS) recorded from tin-doped alpha-Fe<sub>2</sub>O<sub>3</sub>, prepared by the mechanical milling of tin dioxide and alpha-Fe<sub>2</sub>O<sub>3</sub> and by the hydrothermal processing of iron- acid tin-containing precipitates, can be interpreted in terms of a model in which tin occupies both substitutional octahedral sites and the interstitial octahedral sites in the corundum-related alpha-Fe<sub>2</sub>O<sub>3</sub> structure. The EXAFS and Sn-119 Mossbauer spectra suggest that structural models derived from x-ray powder diffraction data do not adequately describe the complexity of the local environment of tin in alpha-Fe<sub>2</sub>O<sub>3</sub>. In particular, the EXAFS and Sn-119 Mossbauer spectra recorded from materials made by mechanical milling show evidence of more disorder. The Sn-119 Mossbauer spectra also indicate that the degree of order in the materials made by both methods is far from perfect and that the microstructural defects are highly sensitive to tin content and the preparative method.

**[37] PHASE FIELDS OF NICKEL SILICIDES OBTAINED BY MECHANICAL ALLOYING IN THE NANOCRYSTALLINE STATE**

Datta MK. Pabi SK. Murty BS. - Journal of Applied Physics. 87(12):8393-8400, 2000

Solid state reactions induced by mechanical alloying (MA) of elemental blends of Ni and Si have been studied over the entire composition range of the Ni-Si system. A monotonous increase of the lattice parameter of the Ni rich solid solution, Ni(Si), is observed with refinement of crystallite size. Nanocrystalline phase/phase mixtures of Ni(Si), Ni(Si)+Ni<sub>31</sub>Si<sub>12</sub>, Ni<sub>31</sub>Si<sub>12</sub>+Ni<sub>2</sub>Si, Ni<sub>2</sub>Si+NiSi and NiSi+Si, have been obtained during MA, over the composition ranges of 0-10, 10-28, 28-33, 33-50, and > 50 at. % Si, respectively. The results clearly suggest that only congruent melting phases, Ni<sub>31</sub>Si<sub>12</sub>, Ni<sub>2</sub>Si, and NiSi form, while the formation of noncongruent melting phases, Ni<sub>3</sub>Si, Ni<sub>3</sub>Si<sub>2</sub>, and NiSi<sub>2</sub>, is bypassed in the nanocrystalline state. The phase formation during MA has been discussed based on thermodynamic arguments. The predicted phase fields obtained from effective free energy calculations are quite consistent with those obtained during MA.

**[36] A THERMAL ANALYSIS INVESTIGATION OF THE HYDRIDING PROPERTIES OF NANOCRYSTALLINE MG-NI BASED ALLOYS PREPARED BY HIGH ENERGY BALL MILLING**

Berlouis LEA. Cabrera E. Hall-Barientos E. Hall PJ. Dodd S. Morris S. Imam MA. - Journal of Alloys & Compounds. 305(1-2):82-89, 2000

A thermal analysis study of the hydrogen loading characteristics of nanocrystalline Mg-Ni alloys (Ni content ranging from 0.1 at% to 10 at%) has been carried out in 3 MPa hydrogen, employing the techniques of differential scanning calorimetry and thermogravimetric analysis (TGA). The measurements confirmed the nonequilibrium state of the samples as prepared by the mechanical alloying technique. An enthalpy associated with the stabilisation of the alloys on first heating in hydrogen was found for all the samples studied. The magnitude of this enthalpy increased with the nickel content of the alloy. All the samples showed rapid uptake of hydrogen at 3 MPa pressure, indicating that the nickel was thus playing a very active role at the alloy surface in dissociating hydrogen and so enabling more rapid hydride formation by the alloy. This catalytic activity of the nickel decreased with temperature cycling over the range 80 degrees C to 500 degrees C. Although TGA analysis, carried out at the end of the cycling period, gave the hydrogen content as 1.1 wt% to 1.7 wt% for the alloys, this is well short of the theoretical amounts expected (7.6 wt% for MgH<sub>2</sub>), indicating that the samples had become deactivated during cycling. No evidence was found of the intermetallic Mg<sub>2</sub>Ni prior to or after hydriding.

**[35] STRUCTURE AND PROPERTIES OF NANOCRYSTALLINE TiC FULL-DENSITY BULK ALLOY CONSOLIDATED FROM MECHANICALLY REACTED POWDERS**

El-Eskandarany MS. - Journal of Alloys & Compounds. 305(1-2):225-238, 2000

High-energy ball milling has been successfully employed for synthesizing nanocrystalline powders of Ti<sub>44</sub>C<sub>56</sub>. The milling procedure involves milling of elemental Ti and C powders at room temperature in an argon gas atmosphere. The progress of the mechanically induced solid state reaction was monitored by means of X-ray diffraction, scanning electron microscopy and transmission electron microscopy and/or high resolution transmission electron microscopy at several stages of the milling time. A single phase of NaCl-type TiC is obtained after 22 ks of milling time. No free Ti and/or C crystals could be detected at this stage of milling. Increasing the milling time leads to a decrease in the grain size of the powders to <50 nm in diameter after 40 ks of milling time. Towards the end of the milling processing time (720 ks) the powders possess excellent morphological characteristics, such as a homogeneous shape (spherical-like morphology) with fine and smooth surface relief and uniform size (<1 μm in diameter). The lattice parameter of this end-product was calculated to be 0.4326 nm. In addition, the powders at this final stage of milling consist of nanocrystalline grains (<5 nm in diameter) with cell-like morphology. In order to determine some physical and mechanical properties of the synthesized TiC material, different samples at different milling times were consolidated into bulk samples, using a plasma activated sintering method. The powders of the final-product (720 ks) has a density of 5.21 g/cm<sup>3</sup>, being nearly the theoretical density of TiC. In addition, this bulk sample maintains its unique structure characteristics with nanocrystalline grains of <60 nm in diameter. On the basis of the results of the present study, the ball-milling technique accompanied with plasma activated sintering can provide powerful tools for the fabrication of nanocrystalline TiC bulk alloys with unique and advanced properties.

**[34] HYDROGEN DESORPTION KINETICS OF A MECHANICALLY MILLED MgH<sub>2</sub>+5at.%V NANOCOMPOSITE**

Liang G. Huot J. Boily S. Schulz R. - Journal of Alloys & Compounds. 305(1-2):239-245, 2000

The hydrogen desorption kinetics of mechanically milled MgH<sub>2</sub> + 5at.%V nanocomposite were determined under various desorption pressures and temperatures. The reaction rate constant was extracted from the time-dependent desorption curves. The relationships of rate constant with pressure and temperature were established. It was found that the hydrogen desorption at high temperature and under high driving force, is controlled by the interface (Mg/MgH<sub>2</sub>) motion. When the driving force is small, the early stage of hydrogen desorption is controlled by nucleation and growth and the later stage is controlled by long range hydrogen diffusion. At temperatures below 523 K, the nucleation and growth process dominates the hydrogen desorption. High temperature annealing (673 K) of the nanocomposite results in slower desorption kinetics and increased activation energy of desorption. At high temperatures, the rate-limiting step changes from interface control (before annealing) to surface control (after annealing), while at low temperatures, the rate-limiting step of desorption does not change after annealing.

**[33] HYDROGEN DESORPTION PROPERTIES OF MECHANICALLY ALLOYED MgH<sub>2</sub> COMPOSITE MATERIALS**

Reule H. Hirscher M. Weisshardt A. Kronmüller H. - Journal of Alloys & Compounds. 305(1-2):246-252, 2000

Composite H storage materials were produced by mechanical alloying of MgH<sub>2</sub> with metallic additives La(Ni<sub>0.7</sub>Fe<sub>0.3</sub>)<sub>5</sub>, Pd<sub>3</sub>Fe and (Fe<sub>0.8</sub>Mn<sub>0.2</sub>)Ti and with the non-metallic additive Si. The H desorption properties of these alloys were investigated by thermal desorption spectroscopy. The desorption spectra yielded maximum H desorption rates at about 500 K for MgH<sub>2</sub> + La(Ni<sub>0.7</sub>Fe<sub>0.3</sub>)<sub>5</sub>, between 510 K and 560 K for MgH<sub>2</sub> + Pd<sub>3</sub>Fe and at about 570 K for MgH<sub>2</sub> + (Fe<sub>0.8</sub>Mn<sub>0.2</sub>)Ti, compared to a maximum at about 720 K for pure MgH<sub>2</sub> powder. These results showed that composite materials with metallic additives reveal enhanced desorption kinetics. In contrast, no shift of the desorption maximum to lower temperatures was obtained by mechanically alloying MgH<sub>2</sub> + Si. Scanning electron microscopy investigations showed similar microstructures for all composite materials, with MgH<sub>2</sub> covering the additive particles like a thin film of irregular thickness. Owing to these results, a model for the H desorption process was developed with a catalytic process at the MgH<sub>2</sub>-additive interface playing a major role.

**[32] INFLUENCE OF CYCLING ON THE THERMODYNAMIC AND STRUCTURE PROPERTIES OF NANOCRYSTALLINE MAGNESIUM BASED HYDRIDE**

Dehouche Z. Djaozandry R. Huot J. Boily S. Goyette J. Bose TK. Schulz R. - Journal of Alloys & Compounds. 305(1-2):264-271, 2000

We have investigated the effect of prolonged cycling on the hydriding/dehydriding properties and on the structure of nanocrystalline MgH<sub>2</sub>-V composite produced by high-energy ball milling. The hydrogen charge and discharge kinetics of the nanocomposite hydride were tested at 300 degrees C using up to 2000 cycles. Pressure composition isotherms at 300 degrees C were also carried out. The nanocomposite exhibits good reversibility in its hydrogenation/dehydrogenation curves after 2000 cycles. The results show some improvements in hydrogen capacity during cycling; this enhanced H-solubility is believed to be the result of structural relaxation. The sample resistance to

hydrogen decrepitation was also evaluated via additional experiments involving SEM, BET specific surface area and X-ray crystal structure characterisations. These observations indicate that the nanostructured Mg-based composite does not decrepitate much upon cycling. However, a slight deterioration in the discharge rate of the nanocrystalline magnesium hydride is observed, apparently related to the crystal growth during cycling.

**[31] CHARACTERISTICS OF Mg<sub>2</sub>Ni<sub>0.75</sub>Co<sub>0.25</sub> ALLOY AFTER SURFACE TREATMENT**

Yang HB. Yuan HT. Zhou ZX. Wang GS. Zhang YS. - Journal of Alloys & Compounds. 305(1-2):282-289, 2000  
Mg<sub>2</sub>Ni<sub>0.75</sub>Co<sub>0.25</sub> alloy has been successfully synthesized by the Ball milling diffusion method. A part of the product was treated by different concentrations of a NH<sub>4</sub>F solution. The results showed that the alloy treated by a 0.06 M NH<sub>4</sub>F solution had the best performance. During treatment, NH<sub>3</sub>, H<sub>2</sub>O, one of the byproducts could be used to indicate the end of etching process. A study of the surface state of the alloy showed that beneath the honeycomb MgF<sub>2</sub> layer, a metallic Ni-rich layer was formed. This layer was believed to have a high activity, the concentration of Ni approaching 70 at.%, which was much higher than 25 at.% of that in the bulk. The hydrogen desorption capacity reached the highest value after only 2 adsorption/desorption cycles. The capacity wets 372 ml(S.T.P.)H<sub>2</sub>/g.alloy (250 degrees C) (H/M=1.18). The desorption properties of treated and untreated alloys showed that the desorption plateau could not be changed even after surface treatment. The specific surface was 4.1 m<sup>2</sup>/g after surface treatment. All of the above results revealed quite different properties from that of the untreated alloy. It is possible that the existence of the honeycomb MgF<sub>2</sub> layer and the metallic Ni-rich layer was the main reason of the improvement of the hydriding/dehydriding properties.

**[30] NEAR ROOM TEMPERATURE SYNTHESIS OF CERIA-ZIRCONIA SOLID SOLUTION BY SOLID PHASE REACTION**

Suda A. Kandori T. Ukyo Y. Sobukawa H. Sugiura M. - Nippon Seramikkusu Kyokai Gakujutsu Ronbunshi-Journal of the Ceramic Society of Japan. 108(5):473-477, 2000

The surprisingly low temperature (similar to 323 K) synthesis of the ceria-zirconia solid solution upon milling ceria powder with zirconia mill and zirconia balls (in ethanol and/or water) was studied. Solid solutions up to 60 mol%ZrO<sub>2</sub> were obtained, whose formation was proved to result from solid phase reaction between ceria and zirconia powders, enhanced by contact stress (either shear or compressive one). Furthermore, the occurrence of large plastic deformation, breaking and mutual combining was found on the ceria powder before and after solid solution. The grain size of ceria-zirconia solid solution was less than or equal to 20 nm, which would cause on easy rearrangement of low diffusion atoms of cerium and zirconium to form the solid solution at such a low temperature. In addition, the possible existence of a solid solution for which the migration of the constituents is much faster (or the stability of the solid at the composition is much higher) than that of other composition, was suggested around 50 mol%CeO<sub>2</sub>-50 mol%ZrO<sub>2</sub>.

**[29] REDUCTION AND SINTERING OF ALUMINA/TUNGSTEN NANOCOMPOSITES - POWDER PROCESSING, REDUCTION BEHAVIOR AND MICROSTRUCTURAL CHARACTERIZATION**

Sekino T. Yu JH. Choa YH. Lee JS. Niihara K. - Nippon Seramikkusu Kyokai Gakujutsu Ronbunshi-Journal of the Ceramic Society of Japan. 108(6):541-547, 2000

Reduction behavior of tungsten oxide mixed with alumina powder and its sintering processes were investigated to fabricate Al<sub>2</sub>O<sub>3</sub>/W nanocomposites, Submicron-sized WO<sub>3</sub> powder could be obtained by a conventional ball-milling technique. Hygrometry analysis of Al<sub>2</sub>O<sub>3</sub>/WO<sub>3</sub> mixture revealed that WO<sub>3</sub> powder was reduced in two steps at 873 and 973 K, X-ray diffraction and transmission electron microscopy (TEM) analysis confirmed that Magneli phases, such as WO<sub>2</sub> and WO<sub>x</sub> (n=2.89 to 2.92), were obtained by the reduction of WO<sub>3</sub> at 873 K. However, WO<sub>3</sub> was completely reduced to metallic W when the reduction temperature was higher than 1173 K. A thermodynamic analysis qualitatively agreed with the experimental result that WO<sub>2</sub> and WO, coexisted at intermediate temperatures. Reduction followed by hot-press sintering of Al<sub>2</sub>O<sub>3</sub>/WO<sub>3</sub> mixtures was carried out to obtain Al<sub>2</sub>O<sub>3</sub>/W nanocomposites. Agglomeration and/or incomplete reduction was found at high W content; however, dense Al<sub>2</sub>O<sub>3</sub>/W composites with >98% of their theoretical density were obtained at low W content (i.e., < 10 vol%). A microstructural investigation of the dense composite revealed that it consisted of both micro- and nano-sized W dispersions. Furthermore, a large number of W particles of around 40 nm were found to be homogeneously dispersed in the Al<sub>2</sub>O<sub>3</sub> matrix. These particles were much finer than the WO<sub>3</sub> particles obtained by ball-milling.

**[28] CHARACTERIZATION OF FINE-GRAINED BISMUTH VANADATE CERAMICS OBTAINED USING NANOSIZED POWDERS**

Shantha K. Varma KBR. - Journal of the American Ceramic Society. 83(5):1122-1128, 2000

Nanocrystalline bismuth vanadate (n-BiV) powders with a crystallite size of <50 nm have been prepared, at room temperature, by subjecting a stoichiometric mixture of bismuth oxide and vanadium pentoxide to mechanical activation. The n-BiV powders show enhanced sinterability, in comparison to the conventionally prepared micrometer-sized bismuth vanadate (m-BiV) powders and yield ceramics with a uniform microstructure, High-density (similar to 98%) of the theoretical value), fine-grained (average grain size of similar to 2 μm) ceramics, obtained using n-BiV have a high dielectric constant and a high pyroelectric coefficient and are associated with low dielectric loss, both at room temperature and at the transition temperature. These fine-grained ceramics show diffused phase transition and relaxer behavior, which are attributed to the irregular distribution of defects and/or compositional inhomogeneities in these ceramics. The fine-grained ceramics exhibit ferroelectric hysteresis loops with higher remanent polarization and lower coercive field values than the coarse-grained ceramics.

**[27] FORMATION OF NANOMETRIC TiB<sub>2</sub> FROM TiO<sub>2</sub>**

Welham NJ. - Journal of the American Ceramic Society. 83(5):1290-1292, 2000

Titanium diboride can be produced by ball-milling a mixture of TiO<sub>2</sub>, B<sub>2</sub>O<sub>3</sub>, and Mg metal for between 10 and 15 h. The reaction was found to be completed during the milling with no evidence of residual Mg. The unwanted phase, MgO, was readily removed by leaching in acid. The leached powder obtained after 15 h milling had a particle size of <200 nm and was highly faceted. The particle size decreased to similar to 50 nm after 100 h milling and seemed to be relatively monodisperse. Scherrer calculation of the crystallite size showed that the product particles were probably single crystal.

**[26] INNOVATIVE NANOSIZE LITHIUM STORAGE ALLOYS WITH SILICA AS ACTIVE CENTRE**

Wang GX. Sun L. Bradhurst DH. Zhong S. Dou SX. Liu HK. - Journal of Power Sources. 88(2):278-281, 2000  
Two types of nanosize intermetallic alloy powders. NiSi and FeSi, are prepared by high-energy ball-milling. The alloys are used as electrode materials in lithium test cells. During lithium insertion into the alloy electrodes, Si acts as active centres, which react with Li to form  $\text{Li}_x\text{Si}$  alloys. A high lithium storage capacity of 1180 mA h  $\text{g}^{-1}$  is observed for the NiSi electrode, with some reversibility. A mechanism for the reaction of NiSi and FeSi with  $\text{Li}^+$  is proposed.

**[25] TEMPERATURE MAGNETIC AND MOSSBAUER STUDIES OF IRON-TIN ALLOYS PRODUCED BY MILLING [RUSSIAN]**

Elsukov EP. Konygin GN. Voronina EV. Korolev AV. Ulyanov AI. Godovikov SK. Zaiganov AV. - Fizika Metallov i Metallovedenie. 88(5):42-49, 1999

**[24] TURBIDIMETRIC EVALUATION OF THE DISPERSION PROPERTIES OF DISPERSE DYES**

Chung YS. - Textile Research Journal. 70(6):550-554, 2000

The dispersion of disperse dyes is examined by two turbidity measurements. Each dispersion of C.I. Disperse Blue 56 and C.I. Disperse Yellow 54 is prepared by a milling process. The complex refractive index can describe the turbidity of dye dispersion by applying Mie's theory. The turbidity ratio-the ratio of the turbidity at the maximum absorption wavelength to that at 850 nm-is theoretically calculated and experimentally measured. Dye particle size gradually decreases with milling time. The turbidity ratio of the dispersion of disperse dyes increases with decreasing geometric mean radius, and shows more noticeable dependence on the mean radius of the particles than the turbidity. Dye dispersion properties are evaluated in terms of the geometric mean radius by the turbidity ratio.

**[23] MECHANICAL ACTIVATION IN TECHNOLOGY OF METALS EXTRACTION**

Balac P. - Metall. 54(4):190-195, 2000.

The paper deals with the results of the utilization of mechanical activation in metals extraction from sulfide minerals. The definition of mechanical activation is given as well as the description of mills suitable for its application. The enhancement of the leaching processes in hydrometallurgy is illustrated. The description of the established technologies (Lurgi-Mitterberg, Irigetmet, Metprotech, Activax, Melt) applying mechanical activation for metals extraction is given in the last part of the paper.

**[22] ANNEALING PROCESS OF FINE GRAINED CO-STABILISED (Y,MG)-PSZ/MGAL2O4 CERAMICS**

Ma YL. Sun XB. Zheng JP. - Materials Science & Technology. 16(5):480-482, 2000

Co-stabilised (Y,Mg) partially stabilised zirconia (PSZ) ceramics with  $\text{MgAl}_2\text{O}_4$  spinel additions were produced, with industrial zirconia as the main starting material. Powders were prepared using a mechanical milling-mixing process. The effect of the annealing process on the mechanical properties, phase compositions, and microstructure of fine grained PSZ ceramics was investigated. MST/4493.

**[21] THE EFFECTS OF MIXING ON SCALE-UP - HOW CRYSTALLIZATION AND PRECIPITATION REACT**

Genck WJ. - Chemical Processing. 63(5):47-+, 2000

Mixing can have a dramatic effect on scale-up, Crystal size distribution, purity, shape, surface area and polymorphs, as well as downstream filtration, drying and milling, all can be changed by the type of mixing used in atl application.

**[20] MECHANOCHEMICAL SYNTHESIS OF NANO-SIZED CEO2**

Gopalan S. Singhal SC. - Scripta Materialia. 42(10):993-996, 2000

**[19] SOFT MAGNETIC PROPERTIES OF NANOCRYSTALLINE Ni3Fe AND Fe75Al12.5Ge12.5**

Fraser HN. Shull RD. Hong LB. Stephens TA. Gao ZQ. Fultz B. - Nanostructured Materials. 11(8):987-993, 1999  
Magnetization curves were measured on Ni3Fe and Fe75Al12.5Ge12.5, nanocrystals of different grain sizes. These materials were prepared by high-energy ball milling, followed by annealing at various temperatures. The alloy compositions were chosen because they have low magnetostriction in bulk form, implying that strain in the samples should have little effect on their magnetic properties. The M-H magnetization curves were used to obtain the coercivity, the maximum permeability, and the saturation magnetization. Differences in these magnetic properties were related to changes in grain size and internal RMS strain. In spite of the low hulk magnetostriction of these materials, the internal stress controlled the coercivity. The changes in permeability, however, were not as expected from the trend in grain size. We suggest that the powder morphology, plays an important role in determining the soft magnetic properties of these nanocrystalline alloys.

**[18] THE EFFECT OF NI ON THE CRYOGENIC ATTRITOR MILLING OF METGLAS Fe78B13Si9**

Huang B. Jiang HG. Perez RJ. Nutt SR. Lavernia EJ. - Nanostructured Materials. 11(8):1009-1016, 1999

The addition of 17 at.% of elemental Ni powders to the cryogenic attritor milling of Metglas Fe78B13Si9 slowed the mechanical crystallization of the alpha-Fe and Fe2B phases. Transmission electron microscopy (TEM) observation and energy dispersive spectroscopy (EDS) analysis indicated that no more than 3.60 at.% of Ni dissolved into the Metglas, which was well within the equilibrium solubility limit of Ni in alpha-Fe. It is proposed that the addition of Ni impede mechanical crystallization during attritor milling by inhibiting bending and wear-like processes which could otherwise cause crystallization.

**[17] NANOMETRIC GRAIN FORMATION IN DUCTILE POWDERS BY LOW-ENERGY BALL MILLING**

Guerrero-Paz J. Jaramillo-Vigueras D. - Nanostructured Materials. 11(8):1123-1132, 1999

Based on microstructural observations by TEM and in particle size distribution done by sedimentation-photometry, a new grain size refinement mechanism for ductile powders in mechanical alloying is proposed. A 90-95%, of the particle population was of submicrometric fragmented particles. These were detected from the beginning of the milling process up to 90 ks. It seems that the fragmentation of the original particles occur-red under dynamic conditions to generate those submicrometric ones. Under these conditions the original grain size (100 nm to 350 nm) was preserved and a low level of dislocations was observed at these submicrometric particles. Once these submicrometric particles were deformed, grains smaller than 20 nm were observed. It seems from TEM results that the submicrometric fragmented particles were also deformed under dynamic conditions. This could be a new grain size refinement mechanism present in ductile metallic powder systems where the fragmentation is the dominant stage

from the beginning of the milling up to some intermediate milling time. In the Cu-20at%Ni, Cu and Ni systems where the particle coalescence process was the dominant stage during all the milling process, a derivation of the mechanism proposed by Hellstern [3] was identified. In our case, powders were mainly deformed by slip and not by shear. It recognizes that the way to refine the grain size in milled powders is influenced at least by the metallic system used as well as by the equipment and the process conditions employed.

**[16] COMPARISON OF GRAIN SIZE DISTRIBUTIONS OBTAINED BY XRD AND TEM IN MILLED FCC POWDERS**

Guerrero-Paz J. Jaramillo-Vigueras D. - *Nanostructured Materials*. 11(8):1195-1204, 1999

Measurements of grain size in powders of the Cu-15at%Al, Cu-20at%Ni, Cu and Ni systems, milled for different times were conducted. X-ray diffraction (XRD) (Warren-Averbach method) and transmission electron microscopy were used for that purpose: From both techniques, distributions of grain diameter (length) were obtained, which permitted to compare both results. Such results were interpreted by considering previous studies of particle size evolution and microstructural evolution. A better comprehension of phenomena that occur in the mechanical alloying, as the grain refinement and the solid solution formation, is attained. The grain size results obtained by the two techniques were coincident for the Cu-15at%Al system. This system did not present adherence of the powders to the milling media. In the case of the Cu-20at%Ni, Cu and Ni systems, that presented adherence, the results were coincident solely in the powder milled for 864 ks. This is explained due to the microstructural homogenization, reached until that time. The grain size as a function of the milling time of the two classes of FCC systems, being referred to the adherence phenomenon, presented opposed trends. For example, for the Cu-at15at%Al system, the 70% of the population of grains had a grain size smaller than 7.5, 12 and 20 nm for the milling times of 180, 360 and 864 ks respectively. In the case of the Cu-20at%Ni, Cu and Ni systems, the 70% of the population of grains had a grain size smaller than 35, 22 and 16 nm for the milling times of 180, 360 and 864 ks respectively. These contrary trends reflect different mechanisms of grain size refinement. A maximum value of grain size of 20 nm required to form the solid solution in the Cu-at15%Al and Cu-20%atNi systems was found.

**[15] MECHANICAL ALLOYING OF THE TI-AL SYSTEM IN ATMOSPHERE OF HYDROGEN AND ARGON**

Takasaki A. Furuya Y. - *Nanostructured Materials*. 11(8):1205-1217, 1999

Three kinds of Ti-Al powders, Ti72Al28, Ti57Al43 and Ti48Al52, were mechanically alloyed by a planetary ball mill in atmosphere of argon or hydrogen, oases (0.1 MPa with alloying times up to 30 h. The mechanical alloying (MA) process as well as the phase variations of each powder after subsequent heating at 1173 K were investigated. About 5000 wppm hydrogen, which could be easily removed by a heat treatment at 800 K (heating rate was 20 K/min). was occluded in all powders during MA in the hydrogen atmosphere: whereas the mechanically alloyed powders in the argon atmosphere occluded about 1000 wppm hydrogen. In the hydrogen atmosphere, the titanium powder easily crumbled into finer particles, assisting the diffusion of aluminum into titanium (solid-solid reaction) at an early stage of the MA process and accelerating the formation of an amorphous-like phase at a longer MA process. The phase formation after heat treatment of MA powders at 1173 K could be estimated by the Ti-Al binary phase diagram without the effect of the gas atmosphere.

**[14] LOCAL ICOSAHEDRAL SYMMETRIES IN AL-MO ALLOYS PREPARED BY BALL MILLING**

Monagheddu M. Delogu F. Schiffini L. Fratini R. Enzo S. - *Nanostructured Materials*. 11(8):1253-1261, 1999

Annealing products of mechanically treated Al75Mo25 powders have been studied by X-ray powder diffraction with the Rietveld method. The differential scanning calorimetry traces of as-milled powders showed transformation features whose temperature decreased as a function of mechanical treatment time. Different phases were identified across the calorimetric events, which were also reported in the high-temperature phase diagram of the Al-Mo system, such as Al12Mo, Al5Mo, Al4Mo, Al3Mo and Al22Mo5. Nevertheless, the solid state transformation processes activated in the calorimeter at ambient pressure are of different nature with respect to those observed by means of neutron diffraction during in situ annealing under vacuum. In the specimen mechanically alloyed for 32 hours the Al12Mo phase was observed after annealing with both processes. In the crystal geometry of Al12Mo, which is regarded as a first order approximation of the quasi-crystalline phase, the molybdenum atoms are arranged in a BCC fashion and are surrounded icosahedrally by twelve aluminum atoms.

**[13] ALUMINUM TITANATE FORMATION BY SOLD-STATE REACTION OF ALUMINA AND TITANIA [SPANISH]**

Uribe R. Baudin C. - *Boletín de la Sociedad Española de Cerámica y Vidrio*. 39(2):221-228, 2000

In this work, the formation of A(2)TiO(5) from equimolar mixtures of high purity and fine grained ( $d(50) < 50 \mu m$ ) Al2O3 and TiO2 powders and the influence of the microstructure of the obtained compacts on the milling efficiency have been studied. Different thermal treatments with maximum temperatures between 1350 and 1650 degrees C have been considered and their influence in the degree of reaction and the cell size of Al2TiO5 have been established. Green compacts have been obtained by isostatic pressing. Some compacts have been thermally treated between 1350 and 1650 degrees C using a single thermal cycle. The other compacts have been thermally treated in two stages using 1600 degrees C as maximum temperature, with an intermediate process of grinding, milling and pressing after the initial low temperature (1350-1500 degrees C) treatment. The evolution of the reaction has been studied by X-ray diffraction, optical microscopy and scanning electron microscopy. The efficiency of the milling processes has been followed using the evolution of the particle size distribution. The results showed that the degree of reaction changes from practically no reaction at 1350 degrees C to the complete reaction at 1650 degrees C, except for the presence of some isolated alumina particles. The thermal treatment conditions used to obtain the materials determine the obtained microstructures of Al2TiO5 and hence affect the grinding conditions.

**[12] EFFECTS OF NITROGENATION AND COMPOSITION ON PHASE TRANSFORMATION AND MAGNETIC PROPERTIES OF ND10FE82B8-XMOX (X=0 TO 8) ALLOYS**

Cui BZ. Sun X. Liu W. You CY. Geng DY. Zhang ZD.- *Zeitschrift für Metallkunde*. 91(4):302-305, 2000

The effects of nitrogenation on the phase transformation and the magnetic properties of Nd10Fe82B8-xMox (x = 0 to 8) alloys prepared by mechanical alloying have been studied. For the alloys annealed at 850 degrees C for 20 min. Nd2Fe14B/ $\alpha$ -Fe-type nanocomposite magnets are formed at x = 0 to 2. Nd2Fe14B and Nd(Fe,Mo)(12) coexist in

the alloys at  $x = 3$  to 5, and the tetragonal Nd(Fe,Mo)(12) with ThMn<sub>12</sub> type structure is formed as main phase in the alloys at  $x = 6$  to 8. With increasing Mo content  $I$ , the coercivity  $\mu(0)H(c)$  and the maximum magnetic energy product  $(BH)_{max}$  of the annealed alloys first increase and reach maxima at  $x = 2$ . Upon nitrogenation, the Nd<sub>2</sub>Fe<sub>14</sub>B compound is completely decomposed to form an N-containing amorphous matrix and nanostructured  $\alpha$ -Fe as well as an Nd(Fe,Mo)(12)N- $\delta$  compound. With increasing Mo content  $x$ ,  $\mu(0)H(c)$  and  $(BH)_{max}$  of the nitrided alloys increase sharply at  $x = 6$  due to the formation of a great amount of nearly single-phase Nd(Fe,Mo)(12)N- $\delta$ .

**[11] PHYSICO-CHEMICAL ASPECTS OF POLYETHYLENE PROCESSING IN AN OPEN MIXER 6. DISCUSSION OF HYDROPEROXIDE FORMATION AND DECOMPOSITION**

Gugumus F. - Polymer Degradation & Stability. 68(3):337-352, 2000

The work concerns different mechanistic and kinetic aspects of hydroperoxide formation and decomposition. It has been shown previously that initiation in mixers cannot be attributed to the free radicals formed on hydroperoxide decomposition but must result essentially from primary initiation. The discussion in this work showed that this primary initiation should result from mechanical breakdown of the polymer in the mixer. The absence of any significant temperature effect on the initial rate of hydroperoxide formation with PE-LD leads to additional results concerning the initiation and termination reactions. The best explanation involves a model based on shear induced free radical formation depending on polymer melt viscosity. Formation of chain carrying radicals by diffusion out of the cage is also governed by melt viscosity. The oxidation chains initiated this way are terminated essentially by geminate recombination following chemical migration of the free valence according to the "relay race" mechanism. The initial rate of hydroperoxide formation on polyethylene processing has been related to the polymer melt flow by an empirical equation. This equation shows the contrary effects of melt viscosity on mechanically induced chain breaking (enhanced with increasing viscosity) and on diffusion of the free radicals out of the cage (reduced with increasing viscosity). The ratio of the initial rate of hydroperoxide formation to the maximum of the hydroperoxide concentration reached gives access to the rate constant for hydroperoxide decomposition. The activation energies determined this way do neither correspond to conventional monomolecular nor to conventional bimolecular hydroperoxide decomposition. Hence, these conventional reactions do not determine the course of the oxidation of polyethylene in open mixers. The discussion shows that many ideas on mechanisms and kinetics in polymer melts need to be revised. The same should be valid even more so for polymers in the solid phase.

**[10] HIGH-ENERGY MECHANICAL MILLING OF POLY(METHYL METHACRYLATE), POLYISOPRENE AND POLY(ETHYLENE-ALT-PROPYLENE)**

Smith AP. Shay JS. Spontak RJ. Balik CM. Ade H. Smith SD. Koch CC. - Polymer. 41(16):6271-6283, 2000

High-energy mechanical milling has been performed on poly(methyl methacrylate) (PMMA) at ambient and cryogenic temperatures, as well as on polyisoprene (PI) and poly(ethylene-alt-propylene) (PEP) at cryogenic conditions only. Milling conducted at ambient temperature has a substantially greater impact on the molecular characteristics of PMMA than milling at cryogenic temperatures. An increase in the milling time is accompanied by substantial reductions in PMMA molecular weight and, hence, glass transition temperature and impact strength under both sets of experimental conditions. An unexpected trend identified here is that the PMMA molecular weight distribution initially broadens and subsequently narrows with increasing milling time. Solid-state mechanical milling promotes comparable decreases in molecular weight and glass transition temperature in PEP (at a slower rate relative to PMMA), but induces chemical crosslinking in PI, as confirmed by FTIR spectroscopy. Charlesby-Pinner analysis yields not only the degree of PI crosslinking, but also the relative crosslinking and scission rates of PI, during cryogenic milling.

**[9] ANGULAR DEPENDENCE OF COERCIVITY IN SM<sub>2</sub>FE<sub>17</sub>CX MAGNETS**

Hu JF. Wang YZ. Hu BP. Wang ZX. Zhou SZ. - Physica Status Solidi A-Applied Research. 178(2):779-781, 2000

In the present paper, the angular dependence of coercivity for the Sm<sub>2</sub>Fe<sub>17</sub>C<sub>x</sub> magnets has been investigated in detail. Our results indicate that the angular dependence of the coercivity depends strongly on the coercivity value of the sample. For the sample of low coercivity with short ball milling time, the coercivity increases at first, develops a peak and then decreases with increasing angle between the easy axis and the direction of the external field. In contrast, minimum coercivity behavior has been observed in the sample of larger coercivity with longer ball milling time.

**[8] SYNTHESIS OF QUASICRYSTALLINE PHASE BY MECHANICAL ALLOYING OF Al<sub>70</sub>Cu<sub>20</sub>Fe<sub>10</sub>**

Barua P. Srinivas V. Murty BS. - Philosophical Magazine A-Physics of Condensed Matter Defects & Mechanical Properties. 80(5):1207-1217, 2000

An icosahedral phase (i-phase) has been synthesized by mechanical alloying of an elemental blend of Al<sub>70</sub>Cu<sub>20</sub>Fe<sub>10</sub>. Mechanical alloying of Al and Fe in the stoichiometric ratio of 3:1 and continued milling after the addition of Al and Cu to yield the nominal composition of Al<sub>70</sub>Cu<sub>20</sub>Fe<sub>10</sub> does not result in the i-phase formation which requires subsequent annealing at 600 degrees C for 4 h for its formation. A single i-phase cannot be obtained even after annealing. In all cases, an Al(Cu, Fe) solid solution (beta phase) has been found along with the i-phase. The i-phase appears to form by a peritectoid type of reaction between the beta phase and Al<sub>2</sub>Cu. The electrical resistivity of the nanocrystalline i-phase obtained in the present study was found to be about 2-3 orders higher than that found in thin films and it shows semiconducting behaviour.

**[7] ULTRAFINE FERRITE PARTICLES PREPARED BY COPRECIPITATION/MECHANICAL MILLING**

Ding J. Liu XY. Wang J. Shi Y. - Materials Letters. 44(1):19-22, 2000

Hard- and soft-magnetic ultrafine ferrite powders (BaFe<sub>12</sub>O<sub>19</sub>, CoFe<sub>2</sub>O<sub>4</sub> or NiFe<sub>2</sub>O<sub>4</sub>) have been successfully synthesized using a combination of coprecipitation and mechanical milling. These ultrafine powders possess excellent magnetic and structural properties. This new preparation method shows significant potential for commercial production of high-quality magnetic ferrite particles, as well as for other nonmagnetic ultrafine powders.

**[6] STUDY OF THE REACTION OF LITHIUM WITH ISOSTRUCTURAL A(2)B AND VARIOUS AL<sub>x</sub>B ALLOYS**

Larcher D. Beaulieu LY. Mao O. George AE. Dahn JR. - Journal of the Electrochemical Society. 147(5):1703-

1708, 2000

The electrochemical alloying reaction of Li with isostructural A(2)B acid Al-based alloys has been investigated. The binary A(2)B alloys we selected (Sb<sub>2</sub>Ti, Sb<sub>2</sub>V, Sn<sub>2</sub>Co, Sn<sub>2</sub>Mn, Sn<sub>2</sub>Fe, Al<sub>2</sub>Cu, and Ge<sub>2</sub>Fe) are isostructural (Al<sub>2</sub>Cu type) and comprise an active element (A) that alloys with lithium, and an inactive one (B) that does not. These compounds were prepared by mechanical alloying and have small grain size (10-20 nm). With the exception of Al<sub>2</sub>Cu, we observed a full reaction of A with lithium  $A(2)B + 2xLi \rightarrow B + 2Li(x)A$ , where the theoretical values of x are 1 for Al, 3 for Sb, and 4.4 for Si, Ge, and Sn). Extremely slow electrochemical cycling at 55 degrees C and potentiostatic tests at lithium potential proved the total inactivity of the Al<sub>2</sub>Cu vs. lithium. However, thermodynamic considerations predict that the reaction of Al<sub>2</sub>Cu with Li should occur and that the formation of LiAl should be observed. Other Ri-transition metal intermetallics were studied and were also found to be inert toward Li, suggesting that the Al-transition metal bond has unique features.

**[5] EFFECT OF CO AND W ADDITIONS ON THE STRUCTURE AND MAGNETIC PROPERTIES OF ND<sub>2</sub>FE<sub>14</sub>B/ $\alpha$ -FE NANOCOMPOSITE MAGNETS**

You CY. Sun XK. Liu W. Cui BZ. Zhao XG. Zhang ZD. - Journal of Physics D-Applied Physics. 33(8):926-931, 2000

The effect of Co addition combined with W on the microstructure and magnetic properties of nanocomposite magnets with composition of (1 - x wt%)Nd<sub>8.55</sub>Fe<sub>84.49</sub>W<sub>0.60</sub>B<sub>0.636</sub> + x wt% CO, prepared by mechanical alloying, has been investigated. The addition of W can increase the unit cell volume of soft and hard phases in nanocomposite magnets, and cause the anisotropy field of the hard phase component to increase. Therefore, the addition of W can improve coercivity. On the other hand, addition of Co leads to a decrease of the grain size of the  $\alpha$ -Fe soft phase component in nanocomposite magnets annealed at temperatures lower than 670 degrees C. It leads further to a decrease of the optimal annealing temperatures, an increase of the remanence and maximum magnetic energy product, and a slight decrease of the coercivity. With increasing the content of Co, the dependence of remanence J(r) on annealing temperature T-a changes from a convex shape, giving a maximum, to a monotonically decreasing one. These phenomena have been explained in terms of the variation of grain growth and exchange coupling between the grains, due to the addition of Co and W.

**[4] LOW-ENERGY BALL-MILLING: TRANSFORMATIONS OF BORON NITRIDE POWDERS. CRYSTALLOGRAPHIC AND CHEMICAL CHARACTERIZATIONS**

Gasgnier M. Szwarc H. Ronez A. - Journal of Materials Science. 35(12):3003-3009, 2000

BN powders (with and without water) which have undergone low-energy ball-milling have been studied through X-ray diffraction, transmission electron microscopy, granulometry, electron energy loss (EELS) and IR spectroscopies. The main result is the formation of the orthorhombic and cubic high pressure phases of BN. A "turbostratic" behaviour of hexagonal BN has also been observed. As expected, the grain sizes decrease markedly. It is to be noticed that the presence of water induces some chemical reactions which are revealed through ammonia-like and nitride compounds scents. However, according to EELS measurements, the main transformations are structural ones.

**[3] SYNTHESIS OF NANOCRYSTALLINE CUBIC SUBSTOICHIOMETRIC WC<sub>1-z</sub> POWDERS BY MECHANOCHEMICAL TECHNOLOGY**

Tan GL. Wu XJ. Zhao MH. Zhang HF. - Journal of Materials Science. 35(12):3151-3154, 2000

Nanocrystalline cubic substoichiometric WC<sub>1-z</sub> powders were synthesized by a mechanochemical method at room temperature, a process of highly reactive energetic ball milling of WO<sub>3</sub> + 3Mg mixtures containing excessive graphite as a source of carbon for W phase. The excessive graphite plays a very important role in the formation of cubic phase, otherwise the h.c.p. phase is formed. The entropy evaluation shows that the reduction reaction is a self-propagating process. After milling for 90 hours and excluding MgO from the product by using HCl solution, the cubic substoichiometric WC<sub>1-z</sub> powders containing a little excessive graphite with the grain size in the range of 4 to 20 nm were obtained.

**[2] MAGNETIC IRREVERSIBILITY IN ULTRAFINE ZNFE<sub>20</sub>4 PARTICLES**

Goya GF. Rechenberg HR. Chen M. Yelon WB. - Journal of Applied Physics. 87(11):8005-8007, 2000

Pure ultrafine ZnFe<sub>20</sub>4 particles have been obtained from mechanochemical synthesis of the ZnO and Fe<sub>2</sub>O<sub>3</sub> oxides. The average grain diameter was estimated from x-ray diffraction to be  $\langle d \rangle = 36(6)$  nm. Refinement of neutron diffraction data showed that the resulting cubic spinel structure is oxygen deficient, with similar to 7% of Fe<sup>3+</sup> ions occupying the tetrahedral A sites. Magnetization curves taken at 4.2 K showed the absence of saturation in fields up to H=9 T, associated with a spin-canting produced by the milling process. Field-cooled (FC) and zero-field cooled (ZFC) curves showed irreversible behavior extending well above room temperature, which is associated with spin disorder. Annealing samples at 300 degrees C yields an average grain size  $\langle d \rangle = 50(6)$  nm, and similar to 16% of Fe<sup>3+</sup> ions at A sites. Partial oxygen recovery is also deduced from neutron data refinement in annealed samples. Concurrently, decrease of magnetic irreversibility is noticed, and assigned to partial recovery of the collinear spin structure. Complex Mossbauer spectra were observed at room temperature and 80 K, with broad hyperfine field distributions spanning from similar to 10 to similar to 40 T. At T=4.2 K, hyperfine field distributions indicate high disorder in Fe local environments. The above data suggest the existence of Fe-rich clusters, yielding strong superexchange interactions between Fe ions at A and B sites of the spinel structure.

**[1] DEFORMATION PARAMETERS GOVERNING TENSILE ELONGATION FOR A MECHANICALLY MILLED AL-1.1AT.%MG-1.2AT.%CU ALLOY TESTED IN TENSION AT CONSTANT TRUE STRAIN RATES**

Hasegawa T. Takahashi T. Okazaki K. - Acta Materialia. 48(8):1789-1796, 2000 For a mechanically milled Al-1.1at.%Mg-1.2at.%Cu alloy, tensile deformation was carried out at temperatures of 523-823 K and at true strain rates of  $1 \times 10^{-3}$ - $5 \times 10^0$ /s. The analysis of true stress-true strain curves from a dislocation dynamics viewpoint showed that a large value of re-mobilization probability of unlocked immobile dislocations Omega is mainly responsible for an enormous elongation (1.3 in true strain) observed at an intermediate strain rate (100/s) and temperature (748 K). Discussion on thermally activated process for Omega and dislocation kinetics indicated that the suppression of plastic instability leading to superplastic elongation is controlled by liberating immobile dislocations from a solute

atmosphere. The strain rate sensitivity  $m$  was formulated on a basis of dislocation dynamics, and a relation of  $\Omega$  proportional to  $m(2)$  was found to field between them.

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