

# RESEAU FRANÇAIS DE MECANOSYNTHESE

## Lettre N° 57

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**Décembre 1999**

**166 (+4) Groupes de Recherche**  
(dont 94 (+4) à l'étranger / 35 Pays)

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

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### SOMMAIRE

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#### 5 Nouvelles Adhésions

**Dr. Snezana Boskovic** - Vinca Institute of Nuclear Sciences - Belgrade - Yougoslavie

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**Y. Shi** - National University Singapore - Dpt Materials Science - Singapour

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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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#### Web Sites

- Pour ceux intéressés par le devenir du Rayonnement Synchrotron en France / Europe, un site intéressant et particulièrement actif ces dernières semaines
- People interested by the Synchrotron Radiation Development in France / Europe

**Website : <http://www.Lure.u-psud.fr/actus/avenir>**

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

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**MRS Fall Meeting 99**

29 Novembre - 3 Décembre 1999 - Boston MA - USA  
Website : <http://www.mrs.org>

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**PROCESSING AND CATALYTIC/CHEMICAL PROPERTIES  
OF NANOSTRUCTURED MATERIALS**

January 16-21, 2000  
Maui, Hawaii

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**Sintering 2000**

7th International Conference on Sintering  
Sintering Science and Technology beyond 2000AD  
22 - 25 Février 2000 - New Delhi - Inde  
E-Mail : [gsu@iitk.ac.in](mailto:gsu@iitk.ac.in)

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**Ultrafine Grained Materials**

**Strengthening, Fracture and Creep of Nanostructured Materials Symposia**  
12 - 16 Mars - TMS Annual Meeting - Nashville TN - USA  
Contact : [RSMIOSHRA@ucdavis.edu](mailto:RSMIOSHRA@ucdavis.edu)

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**Nanomatériaux :**

**Vers les Applications Industrielles**

**Nanomaterials :**

**Towards Engineering Applications**

Colloque : France - Etats Unis - Canada  
17-19 Mai 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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**JRFM'2000**

23 - 24 Mai 2000 - Bordeaux France  
Wbesite : <http://www.bls.fr/amatech>

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**III European Conference on Fluidization**

29 - 31 Mai 2000 - Toulouse - France  
E-Mail : [Progep@ensigct.fr](mailto:Progep@ensigct.fr)

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**PM2 TEC2000**

**2000 International Conference on Powder Metallurgy & Particulate Materials**  
31 Mai - 3 Juin 2000 - New York - USA  
Contact : MPIF

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**4th EUROMECH**

26 - 30 Juin 2000 - Metz - France  
E-Mail : [euromech@lpmm.univ-metz.fr](mailto:euromech@lpmm.univ-metz.fr)  
WebSite : <http://www.lpmm.univ-metz.fr/euromech>

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**ISMANAM 2000**

International Symposium on Metastable Mechanically Alloyed and Nanocrystalline Materials  
9 - 14 Juillet 2000 - St Catherines College - Oxford UK

**E-Mail :**

[ismanam2000@materials.ox.ac.uk](mailto:ismanam2000@materials.ox.ac.uk)  
**website :** <http://www.materials.ox.ac.uk/Conferences.ISMANAM2000.html>

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**NCM8**

**8th International Conference on the Structure of  
Non - Crystalline Solid**

6 - 11 Aout 2000  
Website : <http://www.sgt.org>

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**XIVth International Symposium on the Reactivity of Solids**

Budapest, Hungary through 27-31 August 2000  
<http://www.jate.u-szeged.hu/~isrs14>.

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**Solid State Chemistry 2000**

Prague, Czech Republic, September 3 - 8,2000

and  
**3rd 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>

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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/amp.htm>

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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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**SOUTENANCE DE THESE**  
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**F.Charlot**

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

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 (Provisoire) :**

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

L'objectif de ce travail est d'obtenir à partir d'un procédé de synthèse non conventionnel, le procédé MASHS, un matériau nanostructuré et dense. Ce procédé appliqué à la synthèse du composé FeAl, repose sur deux étapes : (i) une première étape dite d'activation mécanique (MA) qui est un prébroyage énergétique de courte durée conduisant à des poudres précurseurs nanostructurées. (ii) la seconde étape consiste à synthétiser des composés ayant une enthalpie de formation négative par une réaction SHS (Self-propagating High-temperature Synthesis. Une partie du travail a été, dans un premier temps, d'étudier l'influence des différents paramètres contrôlant ce procédé MASHS et, dans un second temps, de comprendre les chemins conduisant à ce matériau nanostructuré à partir d'un dispositif original autorisant le suivi in-situ et en temps réel des réactions de combustion en utilisant la diffraction des rayons X (rayonnement synchrotron) couplée à une thermographie infrarouge.

Il a été mis en évidence plusieurs modes d'activation mécanique, le mode de friction et le mode choc direct grâce au contrôle des conditions de broyage appliquées. Il a été clairement démontré que l'utilisation de poudres activées mécaniquement (friction ou mode direct) entraîne une diminution de la température d'amorçage d'au moins 100° C de la réaction de combustion ainsi qu'une vitesse de front de propagation multipliée par 4/5. Ces différentes modifications sont liées à l'existence de la nanostructure induite au cours du broyage. Une des autres conséquences importantes est l'obtention d'échantillons chimiquement homogènes. Les études in-situ en temps réel ont mis en valeur deux phénomènes, le premier est l'existence d'une phase liquide qui implique que la réaction est de type solide-liquide, le second est l'observation d'une phase transitoire, la forme allotropique gamma du fer.

Bien que le procédé MASHS permettent d'obtenir en une seule étape la phase désirée nanostructurée, un des points faibles reste le problème de la densification (densité relative comprise entre 70 et 80 %). Un procédé expérimental a été développé, en collaboration avec Z.A.Munir, permettant de synthétiser, de densifier et d'obtenir une nanostructure en une seule étape. Ces résultats permettent maintenant d'aller vers la compréhension de la granulo-dépendance des propriétés mécaniques.

The purpose of this work is to obtain a densified nanostructured materials with the application of a new process, the MASHS process. This process is applied to elaborate the FeAl compound. This ones involves mainly the combination of two steps; the first step, a mechanical activation, where pure elemental powders were co-milled to obtain precursor nanometric mixture, the second step, a Self-propagating High-temperature Synthesis (SHS) reaction, which uses the exothermicity reaction. A first step is to determine the parameters which control the different steps of MASHS reaction. The second step is to study the reactional mechanisms with an in-situ in real time study using X-ray diffraction (synchrotron beam) coupled with an infrared thermography. We have demonstrated that different kinds of mechanical activation exists (friction mode, direct shock mode), using the mechanically activated powders, the ignition temperature decreases (more than 100° C), the velocity of the combustion front increases (x 4/5) and the product are more homogeneous. The in-situ in a real time investigation shows the existence of a liquid phase, the reaction is solid-liquid, and an intermediate phase, the gamma iron. The MASHS process doesn't allow to obtain a nanodense material. A following work, with the collaboration of Z.A.Munir, allows to synthesize and densify in a one step a nanostructured materials.

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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) Date et lieu :  
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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.**

**Rapporteurs:**

M. J.-M. HAUSSONNE Professeur, Ecole d'Ingénieurs de Cherbourg

M. A. ROUSSET Professeur, Université de Toulouse

**Examineurs**

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. HUGENTOBLER 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

Dans le secteur des céramiques pour l'électronique, les recherches s'orientent vers l'utilisation de poudres de plus en plus fines telles que leur distribution granulométrique et la morphologie des grains soient maîtrisées et contrôlées. Le présent travail portait principalement sur l'obtention de poudres nanométriques d'oxydes mixtes de structure pérovskite de distribution granulométrique bien contrôlée. Pour cela, nous avons développé deux procédés:

· Au cours d'une étude de faisabilité, nous avons montré que le broyage par attrition en milieu aqueux de poudres microniques conduit à des poudres nanométriques. Le procédé le plus efficace consiste à broyer successivement la poudre avec des billes de tailles décroissantes tout en diminuant la fraction volumique de poudre dans la barbotine au cours des différentes étapes.

· **Le second procédé développé est un procédé de chimie douce. Il met en œuvre une émulsion de type eau dans l'huile** entretenue par agitation mécanique au sein de laquelle la réaction de synthèse s'effectue à température modérée. Des poudres de titanate de baryum et/ou de strontium sont ainsi préparées. Ce procédé permet de maîtriser et d'ajuster la taille des particules à l'échelle nanométrique par l'intermédiaire de deux paramètres du milieu réactionnel: la fraction volumique de phase aqueuse et la concentration en précurseurs. Une relation entre la taille des gouttelettes de l'émulsion et la taille des particules des poudres de SrTiO<sub>3</sub> obtenues après précipitation a ainsi été mise en évidence. Elle s'explique à partir d'un modèle basé sur deux hypothèses: l'aire de l'interface eau/huile ne dépend que de l'énergie de cisaillement fournie par l'homogénéiseur et la vitesse de germination est proportionnelle à la concentration en précurseurs.

Les poudres nanométriques de SrTiO<sub>3</sub>, préparées par la technique de l'émulsion entretenue par agitation mécanique, nous ont permis de préciser et d'affiner des résultats obtenus au sein de l'équipe "Matériaux à grains fins". Pour le matériau considéré, le paramètre de maille est indépendant de la taille des particules lorsque celle-ci évolue dans la gamme nanométrique. Seule une évolution de sa composition, comme la présence d'ions OH<sup>-</sup> sous forme de défauts, semble contribuer à l'évolution du paramètre de maille.

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**H. SOUHA**

Thèse de Doctorat d'Etat Marocaine

Lieu : Faculté des Sciences Dahr El Mehrz 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, 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-entretenues 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 (6 May 1999)**

From Professor Soon B. Hong  
Department of Materials Science and Engineering  
Korea Advanced Institute of Science and Technology  
373 - 1 Kusung - dong, Yusung - gu  
Taejon 305 - 701 Korea

One research topic is entitled "Mechanical Behavior and Wear Resistance of Nanocrystalline WC - Co alloy". We are investigating the fabrication process, sintering and mechanical & wear properties of nanocrystalline WC - Co hard materials. The other research topic is titled "Fabrication Process and Mechanical Properties of Mechanically Alloyed Tungsten Heavy Alloys". We are investigating the mechanical alloying process, sintering behavior and mechanical properties of MA tungsten heavy alloys.

We are very pleased to discuss for international cooperative research on above research topics with Member of Mecanosynthese Group  
**E-Mail** : ShHong@Sorak.kaist.ac.kr

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

**NOUVEAU**

**\*\*\* Postdoctoral Position Available for a Ph.D Physicist (13th October 1999)**

The High-Pressure (HP) group of the School of Physics & Astronomy at Tel Aviv University has a one year position available. This position could be extended to two years.

The HP group is known worldwide as one of the leading groups in experimental HP physics and is on the frontier of HP and diamond-anvil-cells based methodology. The main methods used are:

Mössbauer spectroscopy

X-ray diffraction with synchrotron radiation (ESRF, Grenoble) resistivity

Applicants must be between the final stage of dissertation and within three years of receipt of Ph.D diploma.

For more information, please contact:

Dr. Moshe P. Pasternak

School of Physics and Astronomy

Tel Aviv University

69978 Tel Aviv, ISRAEL

email: hh136@ccsg.tau.ac.il

**\*\*\* Looking for Job Position (6th July 1999)**

I have a Ph.D. in Physics (Mechanical Alloying by Ball Milling) from the Australian National University (Canberra) and extensive experience (more than 17 years and more than 40 publications) in the area of Materials Sciences, Mechanical Engineering and Electronics. The topic of my Ph.D. was Production of Hard Materials by Mechanical Alloying (under the guidance of Dr. A. Calka). I have worked on the production of hard compounds by ball milling such as nitrides, carbides and special alloys. I have completed a post-doctoral fellowship program in Japan (Tokyo University of Technology and The Photon Factory at Tsukuba) and I am currently working as a X-ray Diffraction Officer at CSIRO in Melbourne (Australia). My contract expires and I am looking for a new position.

For more information please contact: ph./fax +61-3-95433002 or email: Jonian.Nikolov@Minerals.CSIRO.au

**\*\*\*\* Proposals**

**FRANCE (2/07/99)**

Joindre Pascal Viel , tél 01 69 08 41 47 CEA SRSIM Bt 461, 91191 Gif sur yvette

Proposition de post doc qui débuterait idéalement en septembre 99. Le financement du post doc est acquis (1 an) Le lieu de travail est le centre de Saclay (DSM/DRECAM/SRSIM) Le sujet concerne une étude très appliquée sur la dépollution des eaux de rejets industriels : Mise au point et étude d'un procédé d'élimination des métaux lourds basé sur la fabrication d'un filtre actif (complexation-décomplexation) utilisant la modification de surfaces métalliques par des films polymères électrographés en couches minces.

Conditions: avoir sa thèse depuis moins d'un an, ne pas avoir été salarié depuis sauf pour un an de postdoc à l'étranger et avoir moins de 30 ans

**FRANCE (6/06/99)**

P. Bracconi (Univ. Dijon) propose une position de Post Doc en Métallurgie des Poudres, de nationalité autre que française (Poste Fléché CNRS)

Contact : pbrac@u-bourgogne.fr

### **GRECE (11/06/99)**

The PEML (Photonics and Electronics Materials Lab) at FORTH, Herklion Crete, Greece, offers two positions available for European Post and Pre Docs, to work within the framework of two TMR networks (HAFAM and MICROSYNC) concerning microfabrication and microassembling"

Contact : Prof. G. Kiriakidis : Kiriakid@iesl.forth.gr

### **ISRAËL (14/5/99)**

A postdoctoral position is available at the High-Pressure group of the School of Physics & Astronomy, Tel Aviv University. This position is available starting with the 1999/2000 academic year for one year, with a possible extension for two years. For additional information, please contact Moshe P. Pasternak by email to hh136@post.tau.ac.il or visit the MEDC web site (www.unca.edu/medc).

### **Denmark (22/02/99)**

One Ph.D. position will be available in the department of Physics at the Technical University of Denmark from 1st April 1999. The candidate will work in the area of Crystallization Kinetics in Bulk Metallic Glasses, which is associated with a Talent Project supported by the Danish Research Council. The position is for three years, and monthly salary is about 20,000 DKr (3300 USD). Applications including a CV, publication list, and names of three references should be sent as soon as possible to:

Assoc. Prof. Jianzhong Jiang  
Department of Physics, Building 307  
Technical University of Denmark  
DK-2800 Lyngby, Denmark  
e-mail jiang@fysik.dtu.dk  
fax. +45 45 93 23 99  
tel +45 45 25 31 65

### **Québec (CANADA) ( 22/01/99)**

Institut National de la Recherche Scientifique

DÉpartement Énergie et Matériaux

POST-DOCTORAL POSITION IN Ni-MH BATTERY TECHNOLOGY

Candidates will be interested in developing a research project focused on the study of new materials for use as negative electrode in nickel-metal hydride (Ni-MH) batteries. Mg-based compounds as electrode material and high-energy ball milling as synthesis method will be privileged. Particular efforts will be performed in order to clarify the correlation between the structure, the composition and the morphology of the alloy and its electrochemical performances.

Experience in electrochemistry and materials science is essential, a working knowledge of Ni-MH battery is an advantage.

Applicants must have obtained their Ph-D between July first, 1996 and January first, 2000.

The work will start between June 1st, 1999 and May 31, 2000.

Initial appointment is for one year, renewable for one year. Salary is \$28,000/year, which could be increased with qualifications and experience.

Applicants should send a CV including a list of publications before March 1st, 1999 to:

Pr. Lionel ROUE  
INRS- Énergie et Matériaux  
1650, bd. Lionel Boulet  
Varenes, Québec, CANADA  
J3X 1S2  
E-Mail: HYPERLINK mailto:roue@inrs-ener.quebec.ca / roue@inrs-ener.quebec.ca

## Bibliographie Récente

### Livres ou "Special Issues"

**NOUVEAU (15/11/99)**

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

Prof. P.R. Soni (1999) - Cambridge International Science Publishing

web site : <http://www.demon.co.uk/cambsi/book52.htm>

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#### "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)

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#### 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.

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

Michal Besterci, 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-Al<sub>4</sub>C<sub>3</sub> 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

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

Cambridge International Science Publishing 7 Meadow Walk, Great Abington, Cambridge CB1 6AZ, England Fax

+44 1223 894539; Tel +44 1223 893295 Email: [cisp@cisp.demon.co.uk](mailto:cisp@cisp.demon.co.uk)

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

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#### "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"

Lettre RFM N° 57 - Décembre 1999

Corresp. : [Eric.Gaffet@utbm.fr](mailto:Eric.Gaffet@utbm.fr)

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

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Les dernières années ont vu apparaître dans le monde des matériaux avancés le préfixe "nano" (nanostructuré, nanocristallins, nanophase ou nanométrie) ; 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 lancé 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...

### 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.

### 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.

### 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)

### [55] MAGNETIC INTERACTIONS IN HEMATITE SMALL PARTICLES OBTAINED BY BALL MILLING

Borzi RA. Stewart SJ. Punte G. Mercader RC. Vasquez-Mansilla M. Zysler RD. Cabanillas ED. - Journal of Magnetism & Magnetic Materials. 205(2-3):234-240, 1999

Pure hematite particles of an average size of 14 nm were obtained after ball milling alpha-Fe<sub>2</sub>O<sub>3</sub> in a vertical vibrating mixer mill for 10 h. Mossbauer and magnetization measurements indicate the existence of strong interparticle interactions that can be varied by the dispersion of the particles in different ways.

NiFe<sub>2</sub>O<sub>4</sub> ultrafine particles prepared by co-precipitation/mechanical alloying

Shi Y. Ding J. Liu X. Wang J. - Journal of Magnetism & Magnetic Materials. 205(2-3):249-254, 1999

Nickel ferrite ultrafine particles have been prepared by the combination of co-precipitation and mechanical alloying. Sodium chloride was added in order to avoid agglomeration. This work has shown that nickel ferrite phase can be formed directly during mechanical milling of hydroxide precursor. Ultrafine NiFe<sub>2</sub>O<sub>4</sub> particles were obtained after a simple washing process. The XRD study showed that sodium chloride can be efficiently removed during the washing with deionized water. These ultrafine particles had a fairly uniform structure and a mean particle size of 10 nm from the TEM measurement. The ultrafine powder possessed good soft magnetic properties and superparamagnetic behavior.

### [54] MAGNETIC PROPERTIES OF BA HEXAFERRITE AND FE COMPOUNDS PRODUCED BY MILLING AND ANNEALING IN AIR

Bercoff PG. Bertorello HR. - Journal of Magnetism & Magnetic Materials. 205(2-3):261-269, 1999

The structure and magnetic properties of different magnetic compounds are studied. The compounds are produced by first milling 80% Ba hexaferrite and 20% Fe for 40 h (giving severely deformed hexaferrite plus Fe), and for 60 h (giving magnetite plus Ba carbonate). These two precursors are annealed at 850, 900 and 1000 degrees C in air and characterized by their structure and magnetic properties. When the precursor consists of deformed hexaferrite and Fe, the thermal treatment promotes the elimination of defects in the hexaferrite and the oxidation of Fe to hematite and some magnetite. In the other case, that is, when the precursor is magnetite and Ba carbonate the annealing produces the re-transformation to the original hexaferrite and the excess of magnetite transforms to hematite. In this situation the annealing at 1000 degrees C produces a compound with a coercivity of 4.4 kOe and a maximum magnetization at 15 kOe of over 51 emu/g, values which are comparable to the ones in the literature for this material considering that the fraction of hexaferrite in the samples is 80%.

### [53] NANO-MECHANO-ELECTROCHEMISTRY OF THE IRON (100) SURFACE IN SOLUTION

Seo M. Chiba M. Suzuki K. - Journal of Electroanalytical Chemistry. 473(1-2):49-53, 1999

An in-situ nanoindentation test using a Berkovich pyramidal diamond indenter was performed to examine the mechano-electrochemical properties of the single crystal iron (100) surface passivated at a constant potential for 1 h in pH 8.4 berate solution. The load-depth curve measured at a maximum load, W-max = 50 mu N and a load speed of 50 mu N s(-1) showed an evidence of plasticity irrespective of the potential at which the passive film was formed. The average hardness of the iron surface obtained from the unloading curve ranged from 2.0 to 2.5 GPa and it increased with increasing potential in the passive region higher than 0.25 V versus SHE, indicating that the passive film contributes to the increase in hardness of the iron surface. Moreover, the indentation at a maximum load, W-max = 200 mu N and a load speed of 20 mu N s(-1) was performed in order to observe the time variation of the shape of the indent with AFM after the indentation. The indent shape for the iron surface cathodically reduced did not change significantly with time after the indentation. In contrast, the indent shape for the iron surface passivated at 1.0 V spread and became obscure with time, which was ascribed to the active dissolution of iron near and under the indenter, followed by the repassivation.

### [52] LEAD ZIRCONATE TITANATE-BARIUM TITANATE BY MECHANICAL ACTIVATION OF MIXED OXIDES

Gan BK. Xue JM. Wan DM. Wang J. - Applied Physics a (Materials Science Processing). 69(4):433-436, 1999

For the first time, the perovskite phase in 0.9Pb(Zr<sub>0.52</sub>Ti<sub>0.48</sub>)O<sub>3</sub>-0.1BaTiO<sub>3</sub>(0.9PZT-0.1BT) is triggered to form in an oxide matrix consisting of PbO, ZrO<sub>2</sub>, BaO, and TiO<sub>2</sub>. Unlike in the solid-state reaction activated by calcination at an intermediate temperature, intermediate phases, such as PbTiO<sub>3</sub>, were not observed with increasing degree of mechanical activation prior to the formation of perovskite 0.9Pb(Zr<sub>0.52</sub>Ti<sub>0.48</sub>)O<sub>3</sub>-0.1BaTiO<sub>3</sub> phase. This suggests that the nucleation and subsequent growth of perovskite crystallites in the activated oxide composition are not a result of the interfacial reactions that normally occur in the temperature-driven solid-state reaction. The mechanical activation-derived powders can be sintered to a relative density of > 97% theoretical at 1100 degrees C. Sintered 0.9Pb(Zr<sub>0.52</sub>Ti<sub>0.48</sub>)O<sub>3</sub>-0.1BaTiO<sub>3</sub> derived from mechanical activation of constituent oxides exhibits a maximum dielectric constant of 775 and a minimum dielectric loss of < 2% at room temperature when measured at 1.0 kHz.

### [51] CHARACTERIZATION OF MECHANICALLY ALLOYED TERNARY FE-TI-AL POWDERS

Zhu SM. Iwasaki K. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 270(2):170-177, 1999

The effects of Ti-substitution for Fe in the Fe<sub>3</sub>Al system on the mechanical alloying process were investigated. For this purpose, blended elemental powders with the following nominal compositions (at.%): Fe<sub>75</sub>Al<sub>25</sub>, Fe<sub>70</sub>Ti<sub>5</sub>Al<sub>25</sub>, Fe<sub>65</sub>Ti<sub>10</sub>Al<sub>25</sub>, Fe<sub>60</sub>Ti<sub>15</sub>Al<sub>25</sub>, were mechanically alloyed in a high energy attritor-type ball milling system for up to 100 h. The structural evolution in these powders was characterized by scanning electron microscopy, differential thermal analysis and X-ray diffraction techniques. It was found that elemental powders were progressively transformed into nanocrystalline solid solutions during mechanical alloying. The addition of Ti in the powders shortened the milling time for solid solution formation. With increasing Ti content, the grain size of the solid solutions decreased, but the lattice parameter increased. Upon heating, the milled powders were transformed into ordered (Fe,Ti)<sub>3</sub>Al intermetallic compounds in an extended range of temperature (from 350 to 500 degrees C). Ti

addition enhanced the occurrence of DO, ordering in heated powders.

**[50] IMPROVED AG-SNO<sub>2</sub> ELECTRICAL CONTACT MATERIAL PRODUCED BY MECHANICAL ALLOYING**

Zoz H. Ren H. Spath N. - Metall. 53(7-8):423-428, 1999

Very pure electrical contact material Ag-SnO<sub>2</sub> composite powders have been produced by mechanical alloying (MA). The starting powders - the silver tin alloy Ag<sub>3</sub>Sn and silver oxide Ag<sub>2</sub>O - were mixed in a specific concentration ratio and then milled by the use of a 'Simoloyer' (horizontal rotary ball mill). During the milling process a chemical reaction takes place and simultaneously a mechanical alloying is carried out which leads to a high dispersed phase distribution of nanoscaled SnO<sub>2</sub>-partides in a silver matrix. The influence of milling parameters on the milling process such as temperature, atmosphere, rotary velocity and ball/powder ratio were studied to optimize the process. It has been found that the temperature is the most significant factor to vary the milling time. The as-milled powders have been characterized by scanning electron microscope (SEM), transmission electron microscope TEM and X-ray diffraction (XRD).

**[49] THERMAL AND MECHANICAL PROPERTIES OF RARE EARTH-DOPED CERIA CERAMICS**

Sameshima S. Ichikawa T. Kawaminami M. Hirata Y. - Materials Chemistry & Physics. 61(1):31-35, 1999

Oxalate coprecipitation method was applied to prepare rare earth-doped ceria powders with a composition of Ce<sub>0.8</sub>R<sub>0.2</sub>O<sub>1.9</sub> (R=Sm, La). The precipitate produced was decomposed at 600 degrees C in air to the oxide solid solution. After milling with alpha-alumina ball, the powders were densified to 98-99% calculated density at 1600 degrees C. No influence of doped rare earth element on the thermal expansion coefficient of CeO<sub>2</sub> was measured (11.4 x 10<sup>-6</sup>-11.8 x 10<sup>-6</sup> K<sup>-1</sup>). The Ce<sub>0.8</sub>Sm<sub>0.2</sub>O<sub>1.9</sub> formed by colloidal processing and with 86-98% relative density offered 53-81 MPa of four-point flexural strength and 9.1 of Weibull modulus. Large flaws of 200-300 μm sizes produced near surface were fracture origins. The Young's modulus and Vicker's hardness were also measured for Ce<sub>0.8</sub>Sm<sub>0.2</sub>O<sub>1.9</sub>. These measured thermal and mechanical data were compared with those of component materials of solid oxide fuel cell.

**[48] A MEASURE OF ENHANCED DIFFUSION KINETICS IN MECHANICAL ALLOYING OF CU-18 AT.% AL BY PLANETARY BALL MILLING**

Das D. Chatterjee PP. Manna I. Pabi SK. - Scripta Materialia. 41(8):861-866, 1999

**[47] MECHANOCHEMISTRY: AN OVERVIEW**

Fernandez-Bertran JF. - Pure & Applied Chemistry. 71(4):581-586, 1999

The field of mechanochemistry is reviewed. A large number of mechanochemical reactions are described as well as industrial applications

**[46] INTERCALATION OF COPPER INTO SOME LAYERED AND TUNNEL CHALCOGENIDES BY MECHANICAL ALLOYING**

Ohtani T. Tsubota A. Ohshima K. - Materials Research Bulletin. 34(7):1143-1152, 1999

Cu atoms were intercalated into the layered chalcogenides 2H(a)-Nb<sub>1.05</sub>Se<sub>2</sub> and 1T-TiS<sub>2</sub> by milling mixtures of Cu and chalcogenides in a planetary ball mill. The intercalation compounds 2H(a)-Cu<sub>x</sub>Nb<sub>1.05</sub>Se<sub>2</sub> (0 < x less than or equal to 0.4), 2H(b)Cu(1.0)Nb(1.05)Se(2), and 1T-Cu<sub>x</sub>TiS<sub>2</sub> (0 < x less than or equal to 0.5) were obtained by milling for 30-80 min. Among them, 1T-Cu<sub>x</sub>TiS<sub>2</sub> (0.3 less than or equal to x less than or equal to 0.5) is a metastable phase compound. Cu atoms were intercalated into the tunnels in Nb<sub>3</sub>Te<sub>4</sub> by milling mixtures of Cu and Nb<sub>3</sub>Te<sub>4</sub> for 60 min. Cu<sub>x</sub>Nb<sub>3</sub>Te<sub>4</sub> was obtained in the range of 0 less than or equal to x 1.0.

**[45] WET MILLING OF AL-CONTAINING POWDER MIXTURES AS PRECURSOR MATERIALS FOR REACTION BONDING OF ALUMINA (RBAO) AND REACTION SINTERING OF ALUMINA-ALUMINIDE ALLOYS (3A)**

Essl F. Bruhn J. Janssen R. Claussen N. - Materials Chemistry & Physics. 61(1):69-77, 1999

Wet milling of powders containing metallic and ceramic particles in different organic milling media has been investigated. These powders are used as precursors for the reaction bonding of aluminum oxide (RBAO) and for the reaction sintering of alumina-aluminide alloys (3A). An attempt is made to explain effects on the milling efficiency in terms of interactions between the organic medium and the particle surfaces. The effects of oxide particle and crystal size have also been examined. The milling efficiency can be optimized by choosing proper milling medium and a suitable particle and crystal size of the abrasive components. Highest milling efficiency was obtained when the powder mixture was milled in a nonpolar organic medium (cyclohexane). The abrasion contribution of the oxide particles is more significant in slurries of higher stability, i.e., in acetone; The area of new Al surfaces formed per milling time is discussed in terms of an in situ determination of the water consumption, where an ethanol-water mixture was used as milling medium, and in terms of the fraction of Al oxidized per time of milling.

**[44] MECHANOLYSIS OF GLUCOSE-BASED POLYSACCHARIDES AS STUDIED BY ELECTRON SPIN RESONANCE**

Kuzuya M. Yamauchi Y. Kondo S. - Journal of Physical Chemistry B. 103(38):8051-8059, 1999

We report the detailed study of mechanically induced free radical (mechanoradical) formation of glucose-based polysaccharides such as cellulose and amylose based on electron spin resonance (ESR) on its comparison with plasma-induced radicals of polysaccharides. The observed ESR spectra of mechanically fractured samples by ball milling at room temperature have shown the multicomponent spectra, which differ in pattern from those of plasma-irradiated cellulose but are similar to those of plasma-irradiated amylose. The systematic computer simulations disclosed that the observed spectra of cellulose consist of three kinds of spectral components, an isotropic doublet (I) assigned to a hydroxylalkyl-type radical at C-1, an anisotropic doublet of doublets (II) assigned to an acylalkyl-type radical at C-2 and/or C-3 as discrete components, and a singlet spectrum (III) assigned to dangling-bond sites (DBS), while those of amylose consist of two kinds of spectral components, I and III. One of the most intriguing facts is that the component radicals are all glucose-derived mid-chain alkyl-type radicals as in the case of plasma irradiation, although it is known that mechanoradicals are produced by the polymer main-chain scission. It can be reasonably assumed, therefore, that the mechanoradicals primarily formed by 1,4-glucosidic bond cleavage of polysaccharides at room temperature underwent a hydrogen abstraction from the glucose units to give rise to the glucose-derived mid-chain alkyl-type radicals. Furthermore, spectrum III was a major component in the simulated spectra of both cellulose

and amylose, unlike those in the case of plasma irradiation; suggesting that cross-linking reactions simultaneously occur accompanied by a decrease in the molecular weight in the course of vibratory milling.

**[43] SYNTHESIS OF MG2NI ALLOY BY BULK MECHANICAL ALLOYING**

Aizawa T. Kuji T. Nakano H. - Journal of Alloys & Compounds. 291(1-2):248-253, 1999

Mg<sub>2</sub>Ni was synthesized by a solid-state reaction from the constituent elemental powder mixtures via bulk mechanical alloying. Since homogeneous refining and alloying takes place efficiently by repeated forging, the process time can be reduced to one fiftieth of time duration necessary for conventional mechanical milling and attrition. This MA Mg<sub>2</sub>Ni alloy can be obtained as a high dense powder compact with a relative mass density in the order of 85%; its average powder particle size ranges from 1 to 3 μm. The prepared Mg<sub>2</sub>Ni alloy has a sufficiently high reaction rate to hydrogen even at relatively lower temperatures and a potential to form a ternary hydride on the pressure-composition isotherms from 523 to 623 K. The enthalpy and entropy for ternary hydride formation were estimated to be -28.7 KJ/mole-H and -46.8 J/K/mole-H, respectively. The present bulk mechanical alloying was found to be adaptive to fabrication of single phase Mg<sub>2</sub>Ni in high productivity.

**[42] STRUCTURE, PHASE TRANSFORMATION AND MAGNETIC PROPERTIES OF SMYFE100-1.5YC0.5Y ALLOYS PREPARED BY MECHANICAL ALLOYING AND RE-MILLING**

Geng DY. Zhang ZD. Cui BZ. Guo ZJ. Liu W. Zhao XG. Zhao T. Liu JW. - Journal of Alloys & Compounds. 291(1-2):276-281, 1999

The structure, phase transformation and magnetic properties of Sm<sub>y</sub>Fe<sub>100-1.5y</sub>C<sub>0.5y</sub> (y=10, 12, 14, 16, 18, 20) alloys prepared by mechanical alloying (MA) have been studied systematically. The Sm<sub>2</sub>Fe<sub>17</sub>C<sub>x</sub> (Sm<sub>2</sub>Fe<sub>14</sub>C) phase tends to form when y is increased (decreased). For the same composition, the Sm<sub>2</sub>Fe<sub>17</sub>C<sub>x</sub> phase is formed more easily when the annealing temperature T<sub>n</sub> is lower. No Sm<sub>2</sub>Fe<sub>14</sub>C phase is observed in the Sm<sub>20</sub>Fe<sub>70</sub>C<sub>10</sub> alloy. In other alloys, annealed between 650 degrees C and 950 degrees C, the Sm<sub>2</sub>Fe<sub>14</sub>C and Sm<sub>2</sub>Fe<sub>17</sub>C<sub>x</sub> phases coexist under a certain condition. Re-milling and re-annealing proved to be suitable for improving the magnetic properties of the MA alloys. After annealing at 800 degrees C for 35 min, re-milling for 1 h and then re-annealing at 600 degrees C for 35 min, the value (BH)<sub>max</sub> = 10.6 MGOe has been achieved for the MA Sm<sub>14</sub>Fe<sub>79</sub>C<sub>7</sub> alloy. After annealing at 800 degrees C for 35 min, the MA Sm<sub>20</sub>Fe<sub>70</sub>C<sub>10</sub> alloy was re-milled for 1 h and then re-annealed at 550 degrees C for 2 h, resulting in the value of H<sub>i</sub>(e) = 7.96 kOe.

**[41] HYDROGEN STORAGE PROPERTIES OF THE MECHANICALLY MILLED MGH<sub>2</sub>-V NANOCOMPOSITE**

Liang G. Huot J. Boily S. Van Neste A. Schulz R. - Journal of Alloys & Compounds. 291(1-2):295-299, 1999

In this paper we present the hydrogen storage properties of a new composite MgH<sub>2</sub>+V prepared by ball milling. Intensive milling of magnesium hydride with vanadium produces a nanocomposite of beta-MgH<sub>2</sub>+gamma-MgH<sub>2</sub>+VH<sub>0.81</sub>. The MgH<sub>2</sub>+5 at.% V composite produced by mechanical milling can desorb hydrogen at 473 K under vacuum and re-absorb hydrogen rapidly even at room temperature. The activation energy of hydrogen desorption was measured to be 62 kJ mol<sup>-1</sup> H<sub>2</sub>. The fast kinetics of the ball milled MgH<sub>2</sub>-V composite primarily comes from the catalytic effect of vanadium and the small powder size. The formation enthalpy and entropy of the composite are identical to that of pure magnesium.

**[40] A STUDY OF THE MICROSTRUCTURE OF NANOCRYSTALLINE AL-TI ALLOYS SYNTHESIZED BY BALL MILLING IN A HYDROGEN ATMOSPHERE AND HOT EXTRUSION**

Moon KI. Lee KS. - Journal of Alloys & Compounds. 291(1-2):312-321, 1999

Nanocrystalline Al-Ti alloy powders were produced by reactive ball milling (RBM) in a hydrogen atmosphere and its microstructure consisted of nano-sized Al and nano-sized TiH<sub>2</sub>. Thermal analysis of as-milled powders showed that the decomposition of TiH<sub>2</sub> and the subsequent formation of Al<sub>3</sub>Ti occurred at 370-480 degrees C. The powder was consolidated by hot extrusion at 500 degrees C. The grain size of as-extruded specimens was about 50-100 nm. The hardness of Al-5 at.%Ti specimens synthesized by RBM and subsequent hot extrusion was 25-75% higher than that of Al-8 wt.%Ti alloys produced by mechanical alloying (MA) in Ar atmosphere and hot extrusion. Room temperature and high temperature (300, 400, 500 degrees C) tensile strength of RBM Al-5 at.%Ti alloys were superior to those of MA Al-8 wt.%Ti alloys. The strength in these alloys appeared to be related to a large extent to the very fine grain size. The ductility of RBM alloys decreased with grain refinement. It is possible that the deterioration in ductility of nanocomposite Al-Ti alloys has to be attributed to the increase of the interface area between Al and Al<sub>3</sub>Ti and its high energy. SEM fractograph showed that fracture progressed intergranularly.

**[39] XANES SPECTRA OF MECHANICALLY ALLOYED YFE**

Tokuyoshi T. Nakai I. - Japanese Journal of Applied Physics Part 1-Regular Papers Short Notes & Review Papers. 38(Suppl 1):476-479, 1999.

To investigate change in electronic states by phase transformation during the mechanical alloying, we measured X-ray absorption near edge structure (XANES) spectra for mechanically alloyed YFe (m-YFe). The m-YFe powders were synthesized from a mixture of pure element powders. The structure of m-YFe powders was characterized by X-ray diffraction. After 120 h of milling the pattern becomes a broad reflection, which means that the specimen is in an amorphous state. We have found that a peak in Fe edge XANES spectra increases with the milling time. The energy of the pre-edge also shifts to the lower energy.

**[38] XANES STUDIES OF MECHANICAL ALLOYING Y2CO**

Nakai I. Tokuyoshi T. - Japanese Journal of Applied Physics Part 1-Regular Papers Short Notes & Review Papers. 38(Suppl 1):480-483, 1999.

We report x-ray absorption near edge structure (XANES) and x-ray diffraction (XD) measurements for mechanically alloyed Y<sub>2</sub>Co. Mechanical alloying for 90 h produces a homogeneous amorphous Y<sub>2</sub>Co from mixture of crystalline Co and Y powders. During the milling till 90 h, the intensity of a pre-peak in the Co K edge XANES spectra shows a remarkable grow and its energy position shifts to the lower energy side. On the other hand, they are hardly changed after 90 h till 165 h. We discuss these behavior from the viewpoint of the Co-Co bonding.

**[37] XANES STUDY ON PHASE TRANSFORMATION OF AMORPHOUS Y2FE**

Nakai I. Tokuyoshi T. - Japanese Journal of Applied Physics Part 1-Regular Papers Short Notes & Review Papers.

38(Suppl 1):484-487, 1999

Phase transformation in mechanical alloying Y2Fe has been investigated by means of x-ray absorption near edge structure (XANES) and x-ray diffraction (XRD) measurements. Milling changes mixture of crystalline powders of Fe and Y to an inhomogeneous amorphous material Y2Fe, which is subsequently transformed into some crystalline phase with homogeneous amorphous matrix. The pre-edge of Fe K absorption grows during the crystalline to amorphous transformation and decays for changing from the amorphous phase to the amorphous-crystalline mixed state. The energy of the pre-edge also shifts in the sequential phase transformation.

**[36] XANES SPECTRA IN THE SOLID STATE REACTION OF Y2Ni**

Nakai I. Tokuyoshi T. - Japanese Journal of Applied Physics Part 1-Regular Papers Short Notes & Review Papers. 38(Suppl 1):488-491, 1999

X-ray absorption near edge structure (XANES) and x-ray diffraction (XRD) investigations for a mechanically alloyed Y2Ni system are presented. From XRD measurements we find a phase transformation from mixture of crystalline Ni and Y into a single phase of an amorphous state by mechanical alloying for 80 h. The XANES spectrum at the Ni K edge have a pre-peak, which increases remarkably with increasing the milling time and becomes as sharp as that of a melt spun amorphous Y67Ni33 alloy. The energy position of the pre-peak also shifts to the lower energy side with the milling time.

**[35] BULK NANOSTRUCTURED METASTABLE ALLOYS PREPARED BY SEVERE PLASTIC DEFORMATION**

VV Stolyarov, RZ Valiev - ADVANCES IN NANOCRYSTALLIZATION (Series: MATERIALS SCIENCE FORUM), 1999, Vol 307, pp 185-190

The present paper describes results of structural investigations and properties of several metastable nanostructured materials prepared by severe plastic deformation. Among the investigated materials are two Al-based alloys, multi-phase hard magnetic Pr20Fe73.5B5Cu1.5 alloy. Development of ageing effects during heating of these alloys, which leads to appearance of unusual mechanical and magnetic properties, was studied.

**[34] GRAIN-BOUNDARY STRUCTURE IN NANOCRYSTALLINE BALL-MILLED FeRh**

A Hernando, E Navarro, AR Yavari, D Fiorani, M Rosenberg - ADVANCES IN NANOCRYSTALLIZATION (Series: MATERIALS SCIENCE FORUM), 1999, Vol 307, pp 191-196

Starting from four different FeRh compositions, a nanocrystalline ferromagnetic phase has been obtained, for each case, through mechanical alloying by ball-milling. At low temperatures, some characteristic behaviors typical of the spin-glass or superparamagnetic state have been observed in all the compositions: a maximum of AC and ZFC: susceptibility at a temperature T-max weakly dependent on the frequency and on the applied field, which increases with the grain size, and a broad distribution of hyper-fine fields in the Mossbauer spectra at T < T-max. Actually, all the results seem to indicate that the observed behavior is intrinsic to the nanocrystalline nature of ferromagnetic FeRh. The shifted hysteresis loop observed after field cooling as well as the remarkable irreversibility in high fields indicate that T-max corresponds to the freezing temperature of the strongly deformed grain boundaries, which behave as spin-glass-like phase.

**[33] MICROSTRUCTURE, CONDUCTIVITY AND HARDNESS OF Cu AND Ag-BASED COMPACTS WITH IMMISCIBLE ELEMENTS**

K Tousimi, AR Yavari, JH Ahn, A Sulpice - ADVANCES IN NANOCRYSTALLIZATION (Series: MATERIALS SCIENCE FORUM), 1999, Vol 307, pp 223-229

Cu and Ag matrix Cu-W and AgSiW composites were prepared by ball milling followed by thermomechanical compaction. Si and W being immiscible in Cu and Ag, the compacts were found to have good conductivities. TEM, SEM and x-ray diffraction analyses indicated that the Si particles are nanometric. However, while the W coherent domain (crystal) size is nanometric both in the powders and the compacts, the W particles are micrometric. The hardness values are still lower than the best available electrical contact materials because of high volume-fractions of remaining porosity. We expect to obtain higher densities and good hardness values through optimised thermomechanical treatment of the compacts.

**[32] FORMATION OF TiB2 FROM RUTILE BY ROOM TEMPERATURE BALL MILLING**

Welham NJ. - Minerals Engineering. 12(10):1213-1224, 1999

A mixture of rutile, boron oxide and magnesium has been ball milled together with the intention of inducing a reaction to form titanium diboride. The resultant powders were examined by differential thermal analysis, isothermal annealing and X-ray diffraction to determine the effect of milling on the formation of TiB2. Complete reaction was found to occur in the mill between 10 and 15 h forming TiB2 and MgO with no residual Mg. The unwanted phase, MgO, was readily removed by leaching in acid leaving a fine powder composed of aggregates of TiB2. The feasibility of using this relatively simple, low technology route to value add for minerals is also discussed.

**[31] OXIDATION BEHAVIOR OF (Al2OC)(1-x)(AlN)(x) AND ALN IN TG-DTA**

Tsuchida T. Azuma Y. - Thermochimica Acta. 334(1-2):67-71, 1999

The oxidation behavior of a commercial AlN and (Al2OC)(1-x)(AlN)(x) (x is approximately equal to 0.80), which was obtained by the self-combustion reaction induced by the mechanical activation of the powder mixtures of aluminum metal with natural graphite, was compared in TG-DTA runs. A weight increase due to the oxidation of a commercial AlN began at temperatures above 600 degrees C, followed by a very slow increase with increasing temperature up to 1000 degrees C, whereas that of (Al2OC)(1-x)(AlN)(x) began at temperatures above 700 degrees C, followed by a rapid increase in temperature range from 850 degrees C to 1000 degrees C. The fractional conversion estimated by assuming the weight increase up to 1000 degrees C in TG corresponding to the oxidation reaction of AlN or (Al2OC)(1-x)(AlN)(x) to Al2O3 was about 8% and 23%, respectively. The difference in oxidation reactivity of AlN and (Al2OC)(1-x)(AlN)(x) is discussed.

**[30] MECHANOCHEMICAL FABRICATION OF SINGLE PHASE PMN OF PEROVSKITE STRUCTURE**

Wang J. Xue JM. Wan DM. Ng WB. - Solid State Ionics. 124(3-4):271-279, 1999

Single phase lead magnesium niobate, Pb(Mg1/3Nb2/3)O-3 (PMN), of high sintered density has been successfully prepared via a novel mechanochemical fabrication route. Nanocrystallites of perovskite PMN are triggered to form in a highly activated oxide composition consisting of PbO, MgO and Nb2O5. Unlike in the solid state reaction of

constituent oxides at high temperatures, intermediate pyrochlore phases were not observed in the activated oxide matrix prior to the formation of perovskite PMN phase. A single phase PMN powder of perovskite structure, which consists of more or less spherical particles of 20-30 nm in size together with a minimized degree of particle agglomeration, was obtained when the constituent oxides were mechanically activated for 20 h. It was sintered to a density of similar to 99% theoretical density at 1050 degrees C for 1 h. Sintered PMN exhibits a peak dielectric constant of 18 083 at the Curie temperature at - 11 degrees C when measured at a frequency of 100 Hz.

**[29] MECHANICAL-MILLING-INDUCED AMORPHIZATION OF SE: A CRYSTALLITE DESTABILIZATION MODEL**

Zhao YH. Jin ZH. Lu K. - Philosophical Magazine Letters. 79(9):747-754, 1999

Complete solid-state amorphization has been realized in elemental Se by means of mechanical milling of crystalline Se powder. Quantitative X-ray diffraction and thermal analyses were employed to characterize the amorphization process and indicated that the amorphization onset corresponds to a critical crystallite size and a drop in microstrain. During the major amorphization process, the remaining crystallite size remains unchanged with a constant lattice expansion. A new kinetics model of crystallite destabilization is proposed for the solid-state amorphization which satisfactorily explains the experimental observations.

**[28] SURFACE MODIFICATION OF BARIUM TITANATE POWDER PARTICLES**

Voltzke D. Gablenz S. Abicht HP. Schneider R. Pippel E. Woltersdorf J. - Materials Chemistry & Physics. 61(2):110-116, 1999

The surfaces of n-doped barium titanate (BaTiO<sub>3</sub>) powder particles were modified by milling in a Ca(NO<sub>3</sub>)<sub>2</sub> solution both at room temperature and under hydrothermal conditions before they were analyzed by electron energy loss spectroscopy and transmission electron microscopy down to the nanometer range. The room temperature treated powders showed BaTiO<sub>3</sub> particles covered with a CaO/BaO layer. Heating these particles caused a shell of (Ca,Ba)TiO<sub>3</sub> solid solution. Under hydrothermal conditions rectangular CaTiO<sub>3</sub> crystallites precipitated on the surface of the BaTiO<sub>3</sub> grains, which during heating transformed into a series of (Ba<sub>1-x</sub>Ca<sub>x</sub>)TiO<sub>3</sub> particles. These modifications of the shell structure change the sintering behavior and thus the microstructure as well as the electrical properties of the final ceramics. Especially the room temperature resistance and the voltage withstanding of the ceramic increased compared to that of conventionally prepared materials of identical composition.

**[27] BEHAVIOR OF OXYGEN AND CARBON IN BALL-MILLED Si<sub>3</sub>N<sub>4</sub> POWDERS DURING HEAT TREATMENT AND SINTERING**

Wada S. Kondo Y. Sudo E. Maki Y. - Nippon Seramikkusu Kyokai Gakujutsu Ronbunshi-Journal of the Ceramic Society of Japan. 107(7):611-614, 1999

Three kinds of Si<sub>3</sub>N<sub>4</sub> powders, DW, TX and E10, with and without sintering additives were ball-milled using ethanol as medium. Oxygen and carbon contents in the mixed powders increased due to the mechanochemical reaction between the powders and ethanol. The increases of oxygen and carbon were almost the same for the three powders. After heat treatment at 800-1200 degrees C in vacuum or nitrogen gas, most of the carbon was desorbed. On the other hand, the increment of oxygen remained in the ceramic body even after sintering at 1750 degrees C.

**[26] EFFECT OF ALCOHOL SOLVENT ON MIXING OF FINE CERAMIC POWDERS AND SOME PROPERTIES OF CERAMIC-BASED COMPOSITES [JAPANESE]**

Nakahira A. Takeda S. Shiomi H. Ohnishi H. - Nippon Seramikkusu Kyokai Gakujutsu Ronbunshi-Journal of the Ceramic Society of Japan. 107(7):662-667, 1999

Effect of alcohol solvent in mixing process on the microstructure and sintering behavior of ceramic-based composites was investigated in detail. The mixture of fine ceramic powders was prepared through the conventional ball-milling method with various alcohol media. The particle distributions of fine ceramic powders were strongly dependent on the kind of alcohol employed during ball-milling, Ceramic-based composites were fabricated by hot-pressing the mixture of fine ceramic powders, Their microstructures and some mechanical properties of the ceramic-based composites were evaluated. It was found that the viscosity of alcohol, surface tension and contact angle greatly affect the sinterability and some of the mechanical properties of ceramic-based composites.

**[25] EFFECT OF COARSE QUARTZ GRAIN SIZES ON MECHANICAL STRENGTH OF ROOFING TILE BODY [JAPANESE]**

Yokota K. Yamasaki T. Nakato T. Kondo Y. - Nippon Seramikkusu Kyokai Gakujutsu Ronbunshi-Journal of the Ceramic Society of Japan. 107(7):678-681, 1999

Clay roofing tiles have been widely used for roof of detached houses. After the Hanshin Awaji great earthquake, there is a sharp decrease in demand for clay roofing the, because it's weight per unit area is the heaviest among all kinds of roofing materials. In the development of a light clay roofing the with high mechanical strength, it is not convenient to use raw materials of high grade due to an increase of the production cost, In this study, the usually employed raw materials, which contained a large fraction of quartz grains with large size, were ground by wet ball milling, and the influence of the maximum quartz grain size on the mechanical strength of the roofing the bodies was studied. Upon grinding the quartz of raw material to a smaller grain size, a roofing the body with high mechanical strength could be obtained, The flaw size for mechanical strength of the roofing tile body seems to mainly depend on the maximum quartz grain size.

**[24] EVALUATION OF POWDER PROPERTIES IN AQUEOUS PROCESSING OF MICROWAVE DIELECTRIC CERAMICS**

Hirata Y. Higashi M. Yamamoto T. - Nippon Seramikkusu Kyokai Gakujutsu Ronbunshi-Journal of the Ceramic Society of Japan. 107(8):702-706, 1999

As-received calcium titanate and neodymium aluminate of median sizes 4.0 and 1.2 μm, respectively, showed fast sedimentation within 10 min because of the formation of hard agglomerates about 1-10 μm. When ball-milling time became longer, the larger particles were selectively ground and the particle size distributions became narrow. The dispersibility of 96 h-milled powders was greatly improved by addition of polyacrylic ammonium (PAA) of 2 mass%. The viscosity of suspension depended on the amount of PAA adsorbed and was lower for CaTiO<sub>3</sub> suspension

than NdAlO<sub>3</sub> suspension. Consolidation of the suspensions and sintering behavior of the CaTiO<sub>3</sub>-NdAlO<sub>3</sub> system were also studied.

**[23] SYNTHESIS, MICROSTRUCTURE AND THERMAL STABILITY OF NOVEL AL-V-FE ALLOY POWDERS BY MECHANICAL ALLOYING**

Lin JX. Pang H. Jia W. Zheng LJ. Tseng MK. - Journal of Materials Science Letters. 18(21):1743-1744, 1999.

Nanocrystalline NiZn ferrite synthesized by high energy ball milling

Jiang JS. Gao L. Yang XL. Guo JK. Shen HL. - Journal of Materials Science Letters. 18(21):1781-1783, 1999

**[22] STRUCTURAL INVESTIGATIONS OF NANOCRYSTALLINE TI-AL AND NI BY PAC**

Lauer S. Guan Z. Wolf H. Wichert T. - Hyperfine Interactions. 121(1-8):307-312, 1999.

Nanocrystalline Ti-Al and Ni are investigated on an atomic scale by perturbed angular correlation spectroscopy in combination with X-ray diffraction. Ti-Al compounds produced by mechanical alloying consist of disordered solid solutions. Annealing of the samples effects a transformation into ordered intermetallic compounds, which is observed via the occurrence of characteristic electric field gradients. The local magnetic properties of nanocrystalline Ni, produced by pulsed electrodeposition, are investigated. Besides the local magnetic field known from polycrystalline Ni, a second component is detected, which is attributed to a magnetic perturbation due to grain boundaries. The width of this magnetic boundary is estimated to 3-4 nm.

**[21] HYDROGEN-ABSORBING ALLOYS [REVIEW]**

Akiba E. - Current Opinion in Solid State & Materials Science. 4(3):267-272, 1999

Improvement of hydrogen capacity in hydrogen-absorbing alloys has been achieved in recent years. Mg-based alloys which were synthesized by ball milling showed lower dehydrogenation temperatures than intermetallic Mg-based alloys. This technique is also effective for preparing a novel Mg-based amorphous alloy, MgNi, and its hydride. Besides conventional intermetallic compounds such as LaNi<sub>5</sub>, solid solution alloy, 'Laves phase related BCC solid solution' with body-centered-cubic structure showed a hydrogen capacity of 2.2 mass% at room temperature. Alanate, which is not an interstitial hydride, was found to react with gaseous hydrogen reversibly with a catalyst, and its hydrogen capacity was more than 3 mass%.

**[20] CONTROLLED CHANGES IN TECHNOLOGICAL PROPERTIES OF PHOSPHATES IN MECHANICAL ACTIVATION WITH ZEOLITES**

Shumskaya LG. Kirillova YA. Yusupov TS. - Journal of Mining Science. 35(1):96-100, 1999

A study has been made of the solid-phase mechanochemical modification of the natural zeolite-clinoptilolite by calcium phosphate in grinding equipment of increased energy intensity. It is shown that the extraction of phosphorus into a citric acid solution is very nearly unchanged as the intensity of mechanical actions is increased. Feasibility has been established for controlled change in the ratio of water-soluble and agrochemically active citric-soluble forms of P<sub>2</sub>O<sub>5</sub>.

**[19] AN ANALYSIS OF THE INFLUENCE OF GRINDING AIDS ON THE BREAKAGE PROCESS OF CALCITE IN MEDIA MILLS**

Nair PBR. Paramasivam R. - Advanced Powder Technology. 10(3):223-243, 1999

The effectiveness of calcium stearate, boric acid, and silica as additives for the fine grinding of calcite in media mills, i.e. ball, rod, and vibration mills, were explored, by following the size distribution of the ground product collected at various time intervals from 15 s of grinding up to 30 min, using a single size feed. While calcium stearate could achieve a modified grinding rate factor of 1.5 in the ball mill and had indicated an optimum concentration for maximum performance, both boric acid and silica remained inert materials without indicating any influence. It was also found that the presence of calcium stearate during the initial stages of grinding introduced a retardation effect on the grinding process, with the product being coarser in comparison with the blank sample. To overcome this retardation effect for the breakage process induced by the additive, it was found preferable to delay the introduction of the additive by around 5 min. Analysis of the breakage process was carried out by estimating the breakage parameters using the G-H solution for the size-discretized batch grinding equation. It was found that while the additive reduced the breakage rates of coarser size fractions during the retardation period, it helped to significantly increase the breakage rates of finer particles during the finer stages of grinding. The higher breakage rates at finer stages of grinding and the reduced agglomeration observed during this period indicate the acceptability of the alterations in the flow behavior of the mill charge as the possible mechanism for the additive in media milling.

**[18] CHARACTERIZATION OF V<sub>2</sub>O<sub>5</sub>-TiO<sub>2</sub> CATALYSTS PREPARED BY MILLING BY ESR AND SOLID STATE H-1 AND V-51 NMR**

Lapina OB. Shubin AA. Nosov AV. Bosch E. Spengler J. Knozinger H. -Journal of Physical Chemistry B. 103(36):7599-7606, 1999

The interaction between V<sub>2</sub>O<sub>5</sub> and TiO<sub>2</sub> under milling in a ball mill tan (alternative procedure of catalyst preparation) was characterized by H-1, V-51 MAS, and V-51 static (wide line) NMR spectroscopy supplemented by simulations of V-51 NMR spectra. Additionally, ESR and magnetic susceptibility measurements were carried out for the characterization of the paramagnetic V<sub>4</sub><sup>+</sup> and V<sub>3</sub><sup>+</sup> sites. It has been shown that after milling, two different types of octahedrally coordinated vanadium (V<sub>5</sub><sup>+</sup>) species (V<sub>5</sub><sup>+</sup>)(I) and V<sub>5</sub><sup>+</sup>(II) strongly bonded to TiO<sub>2</sub> are formed. At the same time, the appearance of V<sub>3</sub><sup>+</sup> ions and an increase of their concentration is observed during milling-calcination processes, along with the formation of at least three different types of paramagnetic V<sub>4</sub><sup>+</sup> species corresponding to (i) V<sub>4</sub><sup>+</sup> centers in O-deficient V<sub>2</sub>O<sub>5</sub>(-x); (ii) VO<sub>2</sub><sup>+</sup> vanadyl species (V<sub>4</sub><sup>+</sup>)(I) with the vanadium centers in octahedral symmetry with axial distortion; (iii) V<sub>4</sub><sup>+</sup>(II) species with vanadium centers also in octahedral symmetry, but with different bond lengths and strengths as compared to V<sub>4</sub><sup>+</sup>(I). Relative amounts of different V<sub>4</sub><sup>+</sup> and V<sub>5</sub><sup>+</sup> species depend on the milling time, the presence of H<sub>2</sub>O in the system, and the subsequent calcination procedure (temperature and calcination time). Thus, V<sub>5</sub><sup>+</sup>(I) species formed predominantly during milling, whereas V<sub>5</sub><sup>+</sup>(II) species formed after thermal treatment. For the structural characterization of these species, complete sets of the quadrupole and chemical shielding tensor parameters, including relative tensor orientations, have been estimated. This allows us to conclude that the octahedral environment of vanadium in V<sub>5</sub><sup>+</sup>(II) species is less distorted than in

V5+(I) and in both cases the distortion is less axial than in V2O5. Combined NMR, ESR, and magnetic susceptibility measurements indicate that all vanadium species (V3+, V4+, and V5+) are isolated from each other on the TiO2 support.

**[17] FESBO4 SEMICONDUCTOR CERAMICS: A NEW MATERIAL FOR SENSING LIQUID-PETROLEUM GAS**

Zhang TS. Hing P. - Journal of Materials Science-Materials in Electronics. 10(7):509-518, 1999

FeSbO4-based semiconducting ceramics used as a promising candidate for sensing liquid-petroleum gas (LPG) are presented here for the first time. Precursor powders of FeSbO4 were prepared by two different methods (i.e., ball-milling and chemical coprecipitation). The solid-state reaction in the Fe2O3-Sb2O3 system was investigated by means of thermal gravimetric-differential thermal analysis (TG-DTA) and X-ray diffraction (XRD). Based on our experimental results and previous work, the diffusion of antimony oxide onto alpha-Fe2O3 is assumed to be a controlling step of the solid-state reaction. Scanning electron microscopy (SEM) and the BET method were used to characterize the samples calcined at 550 to 1000 degrees C. It was found that a sudden change in specific surface area, crystallite size and particle size takes place between 550 degrees C calcining and 600 degrees C calcining, which has an obvious influence on gas-sensing properties. FeSbO4-based sensors operating at 370 degrees C show a high sensitivity and selectivity to liquid-petroleum gas (LPG) over H2 CO or i-C4H10. Addition of Pd shows a fair increase in sensitivity to LPG, but a remarkable improvement in response and recovery times which is advantageous for its practical application.

**[16] PHASE SELECTION IN A MECHANICALLY ALLOYED CU-IN-GA-SE POWDER MIXTURE**

Suryanarayana C. Ivanov E. Noufi R. Contreras MA. Moore JJ. - Journal of Materials Research. 14(2):377-383, 1999

Formation of a homogeneous nanocrystalline CuIn0.7Ga0.3Se2 alloy was achieved by mechanical alloying of blended elemental Cu, In, Ga, and Se powders in a planetary ball mill. X-ray diffraction and transmission electron microscopy and diffraction techniques were employed to follow the structural evolution during milling. It was observed that, depending upon the milling conditions, either a metastable cubic or a stable tetragonal phase was produced. The grain size of the mechanically alloyed powder was about 10 nm. The mechanically alloyed powder was consolidated to full density by hot isostatic pressing the powder at 750 degrees C and 100 MPa for 2 h. irrespective of the nature of the phase in the starting powder, the hot isostatically pressed compact contained the well-crystallized tetragonal CuIn(0.7)Gao(0.3)Se(2) phase with a grain size of about 50 nm.

**[15] ROOM TEMPERATURE REDUCTION OF SCHEELITE (CAWO4)**

Welham NJ. - Journal of Materials Research. 14(2):619-627, 1999

A mixture of scheelite and magnesium has been mechanically milled together for 100 h, either with graphite or in a nitrogen atmosphere, with the intention of forming tungsten carbide or nitride. The resultant powders were examined by thermal analysis, isothermal annealing, and x-ray diffraction to determine the effect of milling on the reduction of scheelite. With graphite, nanocrystallite W2C was the exclusive tungsten product; WC was not detected even after annealing at 1000 degrees C. No nitride formed in the system milled with nitrogen; however, 10 nm crystallites of elemental tungsten were formed. The unwanted phases, MgO and CaO, were readily removed by leaching in acid, leaving a fine powder composed of impact welded aggregates of either carbide or 99% pure tungsten metal.

**[14] PHASE TRANSFORMATION IN BALL-MILLED IRON-RICH SM-FE(-C) POWDERS**

Mao O. Altounian Z. Strom-Olsen JO. Yang J. - Journal of Materials Research. 14(3):750-762, 1999

Two intermetallic phases, R2Fe17 carbide and R2Fe14C, which are promising candidates for permanent magnets, are formed in the iron-rich R-Fe-C ternary alloy system (R = rare earths). Using x-ray diffraction and thermomagnetometry the phase formation, transformation, and thermodynamic relations between the two structures, prepared by high energy ball milling, are studied quantitatively for R = Sm. The results lead to a free energy diagram for the pseudobinary system of Sm2Fe17 and C. A maximum equilibrium carbon content, x(c), has been established for the carbide Sm2Fe17Cx and its temperature dependence determined. Beyond the equilibrium concentration, Sm2Fe17C, transforms into a mixture of Sm2Fe17Cxc, Sm2Fe14C, and alpha - Fe. Although not thermodynamically stable, Sm2Fe17C, can still be formed through nonequilibrium processes by being kinetically favored over the stable phase(s). This feature is important for the production of Sm-Fe-C-based permanent magnets.

**[13] THE STRUCTURAL TRANSFORMATION OF ANATASE TiO2 BY HIGH-ENERGY VIBRATIONAL BALL MILLING**

Sen S. Ram ML. Roy S. Sarkar BK. - Journal of Materials Research. 14(3):841-848, 1999

The structural transformation of anatase TiO2 by high-energy vibrational ball milling was studied in detail by different analytical methods of x-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). This structural transformation involves both phase transition and nanoparticle formation, and no amorphization was observed. The crystallite size was found to decrease with milling time down to nanometer size similar to 13 nm and approaching saturation, accompanied by phase transformation to metastable phases, i.e., TiO2(II), which is a high-pressure phase and TiO2(B), which was identified in ball-milled powder reported for the first time in this paper. These phases eventually started transforming to rutile by further milling.

**[12] SYNTHESIS AND SINTERING OF RARE-EARTH-DOPED CERIA POWDER BY THE OXALATE COPRECIPITATION METHOD**

Higashi K. Sonoda K. Ono H. Sameshima S. Hirata Y. - Journal of Materials Research. 14(3):957-967, 1999

Doped ceria, which has a higher oxygen ion conductivity than yttria-stabilized zirconia, is one of the possible electrolytes for solid oxide fuel cell at low temperatures. This study concerns powder preparation and densification of rare-earth-doped ceria. Rare-earth-doped ceria powders with a composition of Ce0.8R0.2O1.9 (R = Yb, Y, Gd, Sm, Nd, and La) were prepared by heating the oxalate coprecipitate when a mixed rare earth/ cerium nitrate solution was added to an oxalic solution. The oxalate and derived-oxide powders were characterized by x-ray diffraction (XRD), thermogravimetry differential thermal analysis (TG-DTA), particle size analyzer with laser diffraction, inductively coupled plasma (ICP), transmission electron microscopy (TEM), and scanning electron microscopy (SEM). This method provided the oxalate solid solutions containing Ce and R, which were calcined to form the oxide solid

solutions at 600 degrees C in air. The lattice parameter of oxide powders increased linearly with increasing ionic radius of doped rare earth. The size of platelike particles of oxalates and oxides depended on the concentration of oxalic acid and showed a minimum at 0.4 M oxalic acid. Dry milling of oxide powder with alpha-Al<sub>2</sub>O<sub>3</sub> ball was effective in reducing the size and aspect ratios of particles with little contamination of Al<sub>2</sub>O<sub>3</sub>. These rare-earth-doped ceria powders with various sizes were formed by uniaxial pressing (49 MPa) followed by cold isostatic pressing (294 MPa), and sintered at 900-1600 degrees C in air for 4 h. The micrometer-sized-doped CeO<sub>2</sub> powders were densified above 95% of the theoretical density at 1200 degrees C. The grain size of rare-earth-doped ceria after sintering at 1600 degrees C was larger in the samples with the larger rare-earth element.

**[11] STRUCTURE AND THERMAL STABILITY OF NANOSTRUCTURED IRON-DOPED ZIRCONIA PREPARED BY HIGH-ENERGY BALL MILLING**

Jiang JZ. Poulsen FW. Morup S. - Journal of Materials Research. 14(4):1343-1352, 1999

Fury stabilized cubic zirconia doped with iron oxide has been synthesized by high-energy ball milling from powder mixtures of monoclinic zirconia and hematite. It is found that the iron ions dissolved in cubic ZrO<sub>2</sub> are in substitutional positions with a maximum solubility of approximately 18.5 mol% alpha-Fe<sub>2</sub>O<sub>3</sub>. The unit-cell volume of the cubic ZrO<sub>2</sub> phase decreases with increasing iron content. During heating the cubic-to-tetragonal transition occurs at approximately 827 degrees C and the tetragonal-to-monoclinic transition seems to be absent at temperatures below 950 degrees C. During cooling the tetragonal-to-monoclinic transition occurs at 900-1100 degrees C.

**[10] LEAD ZIRCONATE TITANATE VIA REACTION SINTERING OF HYDROXIDE PRECURSORS**

Xue JM. Wang J. - Journal of Materials Research. 14(4):1503-1509, 1999

Lead zirconate titanate (PZT) has been successfully fabricated via a unique one-step sintering processing route, which is simpler than the traditional precursor-calcination-milling-pelleting-sintering route and is able to deliver an enhanced sintered density at a much reduced sintering temperature. The hydroxide precursor was prepared by coprecipitation from a mixed nitrate solution containing Pb<sup>2+</sup>, Zr<sup>4+</sup>, and Ti<sup>4+</sup> ions, and it was then compacted into pellets without being calcined at a low temperature. The precursor pellets were dehydrated at 400, 500, and 600 degrees C for 4 h, respectively, followed by an isostatic pressing at 350 MPa, prior to being sintered at a high temperature. Dehydration temperature has a large impact on the sintering behavior of these hydroxide-derived PZT ceramics. The PZT dehydrated at 400 degrees C was seriously cracked when sintered at temperatures ranging from 950 to 1150 degrees C, due to the incomplete dehydration. A sintered density of 99.2% theoretical density was obtained at 1050 degrees C for 2 h for the powder pellet dehydrated at 500 degrees C for 4 h. It exhibits a dielectric constant of 1024 and a dielectric loss of 2.1% at a frequency of 1 kHz at room temperature. A calcination at a too-high temperature, e.g., 600 degrees C, results in a reduction in the sinterability of the precipitate-derived PZT ceramic.

**[9] SYNTHESIS OF FE-RICH FE-AL NANOCRYSTALLINE SOLID SOLUTIONS USING BALL MILLING**

Jiang HG. Hu HM. Lavernia EJ. - Journal of Materials Research. 14(5):1760-1770, 1999

The synthesis of nanocrystalline Fe, Fe-4 wt% Al, and Fe-10 wt% Al solid solutions by SPEX ball milling has been studied. The microstructural evolution during ball milling, as well as subsequent heat treatment, has been characterized. The results demonstrate that ball milling promotes the formation of alpha Fe-4 wt% Al and alpha Fe-10 wt% Al solid solutions by reducing the activation energy of these alloys and generating thermal energy during this process. For Fe-10 wt% Al powders milled for various time intervals up to approximately 20 min, the FeAl intermetallic compound is formed. For alloys annealed at temperatures ranging from 600 to 1000 degrees C, the addition of 10 wt% Al to Fe significantly enhances the thermal stability of the nanocrystalline Fe-Al alloys. Interestingly, the addition of Al within the range of 4-10 wt% seems to have little effect on the thermal stability of these alloys annealed under the same conditions. Also, the thermal stability improves for alloys milled in air as opposed to those processed using Ar.

**[8] MICROSTRUCTURE AND 1000-1400 K MECHANICAL PROPERTIES OF CRYOMILLED NiAl-0.7Zr**

Whittenberger JD. Garg A. Hebsur MG. - Journal of Materials Research. 14(6):2418-2429, 1999

An attempt has been made to improve the intermediate temperature strength of cryomilled NiAl by utilizing third element solid solution or precipitation strengthening mechanisms. To this end an NiAl-0.7 (at.%) Zr alloy was cryomilled, densified by hot extrusion, and tested between 1000 and 1400 K. Although over 3 wt% nitrogen was introduced via cryomilling, mechanical testing revealed that the cryomilled NiAl-0.7Zr was significantly weaker than the base alloy between 1000 and 1200 K. Chemical and microstructural analyses revealed that, in addition to similar to 16 vol% AlN, all the Zr had been converted into ZrN. A thermodynamic analysis of cryomilling indicated that the formation of ZrN could have been anticipated since it is a more stable nitride than AlN. While Zr was an unsatisfactory addition in NiAl, thermodynamics also suggest several alloying elements that might lead to good intermediate temperature strength after cryomilling.

**[7] POSSIBLE EVIDENCE FOR THE STABILIZATION OF BETA-CARBON NITRIDE BY HIGH-ENERGY BALL MILLING**

Fahmy Y. Shen TD. Tucker DA. Spontak RL. Koch CC. - Journal of Materials Research. 14(6):2488-2499, 1999

The possibility of stabilizing the theoretically predicted beta-C<sub>3</sub>N<sub>4</sub> phase by high-energy ball milling is investigated. Charges of graphitic carbon were milled with and without minor alloying additions under different atmospheric media, namely gas and/or liquid phases of nitrogen, air, or ammonia. Milling was performed at either of two energy levels for periods of up to 48 h. The beta-C<sub>3</sub>N<sub>4</sub> phase was found to exist as small crystallites in a matrix of primarily amorphous carbon at volume fractions estimated between 5 and 10 at.%. High-resolution electron diffraction and x-ray diffraction indicate that the crystalline nature of the C<sub>3</sub>N<sub>4</sub> phase corresponds with a hexagonal lattice with a = 6.46 Angstrom and c/a = 0.374, which are within 2% of the theoretically calculated lattice parameter values. Analysis of electron energy-loss spectroscopy (EELS), x-ray photoelectron spectroscopy (XPS), and Fourier transform infrared (FTIR) spectra verify the presence of chemically bonded carbon and nitrogen with chemical states reflecting combined sp<sup>2</sup> and sp<sup>3</sup> hybridization. Chemical analysis confirms nitrogen enrichment at levels consistent with the C<sub>3</sub>N<sub>4</sub> stoichiometry and the estimated degree of stabilization. The possible mechanism(s) responsible for the stabilization of

the beta-C<sub>3</sub>N<sub>4</sub> phase are briefly discussed.

**[6] MECHANICAL PROPERTIES OF VAPOR-GROWN CARBON FIBER COMPOSITES WITH THERMOPLASTIC MATRICES**

Tibbetts GG. McHugh JJ. - Journal of Materials Research. 14(7):2871-2880, 1999

This article discusses the mechanical properties of vapor-grown carbon fiber (VGCF)/nylon and VGCF/polypropylene composites. Fibers in the as-produced condition yielded composites with marginally improved mechanical properties. Microscopic examination of these composites clearly showed regions of uninfiltated fibers, which could account for the unsatisfactory mechanical properties. The infiltration of the fibers by both polymers was improved by carefully ball milling the raw fiber so as to reduce the diameter of the fiber clumps to less than 300  $\mu$ m. Properties of composites made with ball-milled material were improved in every respect. VGCF reinforcement in nylon slightly improved the tensile strength and doubled the modulus, while VGCF in polypropylene doubled the tensile strength and quadrupled the modulus compared to unreinforced material. Moreover, the composites were sufficiently improved that differences in fiber surface preparation became important. For example, air-etched fibers and fibers covered with low concentrations of aromatics produced polypropylene composites with significantly better mechanical properties than did fibers whose surfaces were heavily coated with aromatics. Both the tensile strength and the modulus of the composites fabricated with clean fibers exceeded theoretical values for composites made with fibers randomly oriented in three dimensions, indicating that the injection-molding process oriented the fibers to some extent.

**[5] AN ALPHA-Fe<sub>2</sub>O<sub>3</sub> POWDER OF NANOSIZED PARTICLES VIA PRECURSOR DISPERSION**

Liu XY. Ding J. Wang J. - Journal of Materials Research. 14(8):3355-3362, 1999

An alpha-Fe<sub>2</sub>O<sub>3</sub> powder of nanosized particles has been successfully prepared by effectively dispersing the precipitated hydroxide precursor in a sodium chloride matrix. In particular, the hydroxide precursor was converted into crystalline alpha-Fe<sub>2</sub>O<sub>3</sub> particles approximately 10 nm in size when it was mechanically activated in the sodium chloride matrix for 1 h. The subsequent calcination at 600 °C for 1 h resulted in a limited degree of coarsening in particle size while the crystallinity of alpha-Fe<sub>2</sub>O<sub>3</sub> was further established. The effectiveness of obtaining ultrafine alpha-Fe<sub>2</sub>O<sub>3</sub> powders by mechanical activation in the sodium chloride matrix was demonstrated by comparing the powder with those obtained via other routes, such as mechanical activation without sodium chloride as the matrix, calcination at 600 °C, and then mechanical activation in sodium chloride matrix, respectively. None of these processing routes led to a powder comparable in particle characteristics to that derived by the precursor dispersion.(b).

**[4] STRUCTURE DEVELOPMENT AND PROPERTY CHANGES IN HIGH-DENSITY POLYETHYLENE/CALCIUM CARBONATE BLENDS DURING PAN-MILLING**

Huang H. - Journal of Applied Polymer Science. 74(6):1459-1464, 1999

A new self-designed mechanochemical reactor, inlaid pan-mill, was used in studying high density polyethylene (HDPE) and calcium carbonate (CaCO<sub>3</sub>) blends. The effects of CaCO<sub>3</sub> on the crushing and structure of HDPE matrix and the properties of HDPE/CaCO<sub>3</sub> blends were investigated. Scanning electron microscopy, Fourier transformed IR spectroscopy, dynamical mechanical testing analysis, capillary rheometer, and Instron material testing system were used to characterize the structure of HDPE and evaluate the properties of HDPE/CaCO<sub>3</sub> blends. The introduction of calcium carbonate during milling improved milling efficiency, and time needed for each cycle was greatly reduced. Oxygen-containing groups on HDPE chains, which were produced during milling, increased interfacial interactions and improved the dispersion and distribution of calcium carbonate particles in HDPE/CaCO<sub>3</sub> blends. Rheological, thermal, and mechanical properties were also improved. The elongation at break of milled blends with high concentrations of calcium carbonate was significantly higher than that of unmilled blends.

**[3] STUDY OF ALN AND Si<sub>3</sub>N<sub>4</sub> POWDERS SYNTHESIZED BY SHS REACTIONS**

Bermudo J. Osendi MI. - Ceramics International. 25(7):607-612, 1999.

The particular characteristics of aluminum nitride and silicon nitride powders obtained by SHS technology are reported. The synthesized powders showed high particle sizes that are not suitable for sintering. Consequently, the powders were subjected to an energetic milling process using an attritor with different milling media and times. Silicon nitride powders were milled with Si<sub>3</sub>N<sub>4</sub> balls while for aluminum nitride powders different media (Si<sub>3</sub>N<sub>4</sub>, Al<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub>) were used. Particle size and specific surface area were determined in both powders as a function of the milling variables. The increase in the level of impurities associated with the milling procedure was measured. The morphology of each powder was analyzed before and after milling by scanning electron microscopy. The results were evaluated by comparing with characteristics of typical commercial powders of AlN and Si<sub>3</sub>N<sub>4</sub>, to establish the differences with the SHS powders.

**[2] AMMONIA SYNTHESIS OVER MULTI-PROMOTED IRON CATALYSTS OBTAINED BY HIGH-ENERGY BALL-MILLING**

Jacobsen CJH. Jiang JZ. Morup S. Clausen BS. Topsøe H. - Catalysis Letters. 61(3-4):115-120, 1999

The feasibility of producing ammonia synthesis catalysts from high-energy ball-milling of a simple mixture of the constituent oxides has been investigated. The effect of ball-milling the fused oxidic precursor of the industrial KM1 ammonia synthesis catalyst has also been studied. The results show that high-energy ball-milling offers some interesting possibilities for preparing novel catalytic materials. It is observed that ball-milling of the powder oxides mixture leads to formation of solid solutions and the catalytic activity is significantly higher than that of the starting material. Furthermore, ball-milling of fused oxidic KM1 precursor is seen to give rise to more homogeneous promoter distribution and slightly higher activity. The quite small activity increase observed in this case probably reflects the fact that the fusion process has already resulted in a close to optimal promoter distribution. The choice of atmosphere during ball-milling is also seen to offer possibilities for regulating the phase composition.

**[1] PREPARATION AND REACTION MECHANISTIC CHARACTERIZATION OF SOL-GEL INDIUM/ALUMINA CATALYSTS DEVELOPED FOR NOX REDUCTION BY PROPENE IN LEAN CONDITIONS**

Maunula T. Kintaichi Y. Haneda M. Hamada H. - Catalysis Letters. 61(3-4):121-130, 1999

The impregnation and sol-gel preparation methods were investigated to develop high activity catalysts and understand

the significance of the indium-aluminium interaction on alumina-supported indium catalysts in NO<sub>x</sub> reduction with propene. Active In/alumina catalysts with a very high surface area (270 m<sup>2</sup>/g) and thermal stability were prepared in controlled conditions by sol-gel processing. When Al isopropoxide and In nitrate in ethyl glycol were used as precursors in aqua media, indium atoms were incorporated evenly distributed as a thermally stable form in the aluminium oxide lattice structure. In wet impregnation it was beneficial to use a certain excess of aqueous In solution (volumes of solution : pores = 2 : 1) to have the highest NO<sub>x</sub> reduction activity. The catalyst containing dispersed Al on In oxide (58 wt% In, phase-equilibrium preparation method) showed activity at lower temperatures than any other In-Al oxide catalyst or pure In<sub>2</sub>O<sub>3</sub>. The adsorption of different reaction intermediates on alumina and stable In<sub>2</sub>O<sub>3</sub> sites were detected by FTIR studies. In/alumina catalysts have active sites to oxidize NO to NO<sub>2</sub>, partially oxidize HC, form the actual reductant which contains N-H or N-C bonding and react with NO to dinitrogen. The cooperation with indium and aluminium was evident even in the mechanical mixture of sol-gel prepared alumina (301 m<sup>2</sup>/g) and In<sub>2</sub>O<sub>3</sub> powders (27 m<sup>2</sup>/g), where the probability for molecular-scale intimate contact between indium and aluminium sites was very low (particle size 10-250 μm). Short-lived gaseous intermediates and surface migration are the possible reasons for the high catalytic activities on the two physically separated active sites both necessary for the reaction sequence.

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