

# RESEAU FRANÇAIS DE MECANOSYNTHESE

## Lettre N° 60

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**Mars 2000**

**173 (+4) Groupes de Recherche**  
**(dont 100 (+3) à l'étranger / 35 Pays)**

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

### International Editorial LRFM Committee

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

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**PRIERE DE RENOUVELLER L'INSCRIPTION au RFM au titre de l'Année 2000**  
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- Le RFM fête ses 5 Ans
- Annonce et bulletin d'Inscription JRFM'2000
- NATO General Announcement and Call for Participation
  - Annonce de Congrès
  - Cooperative Research

### 4 Nouvelles Adhésions

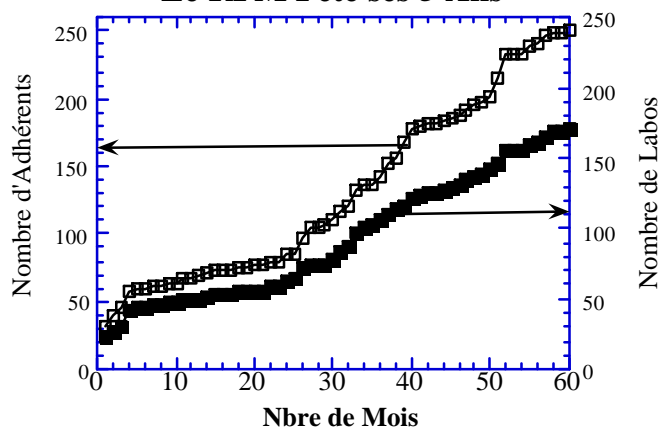
**V. D. Makhaev** - Institute of Problems of Chemicals Physics - Chernogolovka - Russie

**S.T. dos Reis** - Fisics Institute of Sao Paulo - Brésil

**F. Saito** - Institut for Advanced Materials Processing - Tohoku University - Japon

**L. Voron** - GEMPPM - INSA Lyon - France

### Le RFM Fête ses 5 Ans



Evolution du Nombre d'adhérents & de Laboratoires participant au RFM

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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 Intéressants**

- 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>**

**NOUVEAU**  
(11/01/2000)

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From Prof. N. N. Lyakhov  
Website of his Institute : [www.solid.nsc.ru](http://www.solid.nsc.ru)  
Members of the can find there some additional information on the  
address :  
[www.solid.nsc.ru/eng/books/books.html](http://www.solid.nsc.ru/eng/books/books.html)  
which contain the bibliography  
on mechanochemistry and mechanical activation  
begin with 1987 till 1999.  
This document is partly in Russian, but all English references are  
presented in original transcription.  
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**Bulletin d'adhésion 2000 / Subscription Print**

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

**Eric GAFFET**  
UPR CNRS 806  
Groupe "Nanomatériaux : Elaboration et Transitions de Phases Hors Equilibre"  
Université de Technologie de Belfort - Montbeliard

F90010 - Belfort Cedex - France

**Nom/Name :** .....**Prénom / First Name :** .....

**Adresse complète / Full Address :** .....  
.....  
.....

**Téléphone/ Phone:** .....**Télécopie (Fax) :** .....

**e\_Mel. / e-Mail :** .....

désire adhérer au Réseau Français de Mécanosynthèse /want to be a member of the French Mechanical Alloying Network

Chèque ci joint / Check enclosed in the amount of **100FF**

**The check has to be to the order : Reseau Francais de Mecanosynthèse**

(Please do not use Eurocheck, the taxes do correspond to 40% of the amount of the check).

**N.B. :** Pour la rédaction du prochain N° de la Lettre du Réseau Français de Mécanosynthèse, tout(e) article, annonce,  
Eric Gaffet - CNRS UPR A0423  
Groupe "Nanomatériaux : Elaboration et Transitions de Phases Hors Equilibre"

Tél. : 84 - 58 - 31 - 02 / Fax : 84 - 58 - 30 - 27

E-mail : Eric.Gaffet@utbm.fr

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## **JRFM'2000**

5èmes Journées du Réseau Français de Mécanosynthèse  
23 et 24 Mai 2000 - Bordeaux, ICMCB

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### **Thématique retenue**

Caractérisations physico-chimiques des matériaux élaborés par mécanosynthèse

- **Vers la Modélisation de la Mécanosynthèse**  
Prof. Jacques FOCT, Université de LILLE

- **Elaboration et caractérisation de nanotubes élaborés par mécanosynthèse**  
Dr Laurent CHAFFRON, CEA saclay-CEREM-SRMP/DTM, Gif sur Yvette

- **La Mécanosynthèse au Japon**  
Prof. Associé Shin-Ichi ORIMO, Higashi Hiroshima University  
Prof. Associé Koichi TSUCHIYA1, Toyohashi university  
Prof. Etsuo AKIBA1, NIMC

#### **Contacts et pre - inscriptions**

##### **Local Organization :**

Dr. Jean-Louis BOBET  
Institut de Chimie de la Matière Condensée de Bordeaux (ICMCB)  
Avenue du Dr A. Schweitzer  
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Tel : 33-(0)5-56-84-26-53 - Fax : 33-(0)5-56-84-24-80  
E\_Mel : bobet@icmcb.u-bordeaux.fr

##### **Réseau Français de Mécanosynthèse**

Dr. Eric Gaffet (Pdt of the RFM)  
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Jean-Louis Bobet  
et toute l'équipe "Détermination structurale et matériaux magnétiques"  
CNRS - ICMCB  
(UPR 9048 CNRS - Université Bordeaux1)  
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E-mail : Eric.Gaffet@utbm.fr

## NATO Advanced Study Institute

### Functional Gradient Materials and Surface Layers Prepared by Fine Particles Technology

Kiev (Ukraine)

June 18-28, 2000

Sponsored by the North Atlantic Treaty Organization

### General Announcement and Call for Participation

*Source de l'information* ; Dr Marie-Isabelle Baraton (Director for NATO countries)

The Advanced Study Institute on "Functional Gradient Materials and Surface Layers Prepared by Fine Particles Technology" will contribute to the dissemination of the state-of-the-art knowledge about functional gradient materials, coatings and thin films having ultrafine and nanophase structures, and about the underlying physics and chemistry of surface phenomena that have been used to synthesise superior materials with unique properties and to control the interface properties in composites. This ASI will emphasise the connections between the physics and the chemistry of surface layers, as well as the connections between chemical, physical and processing data of nanostructured composite materials, coatings and films.

Round tables are scheduled to discuss physical and chemical characteristics of nanostructured coatings, films and compact materials, including structure and electronic properties, modeling, high-temperature processes, corrosion resistance ...

An exhibition including books and reports display, prototype demonstration, career opportunities will be organized as a parallel session.

Posters sessions will be planned for participants to present their research work.

A book will be published in the NATO ASI Series shortly after the meeting.

#### ASI Directors

Dr Marie-Isabelle Baraton (Director for NATO countries)

SPCTS - UMR 6638 CNRS, University of Limoges

123 Avenue Albert Thomas - F-87060 Limoges (France)

Phone: + 33 555 45 7348 - Fax: + 33 555 77 8100 - e-mail: baraton@unilim.fr

Professor Irina V. Uvarova (Co-Director for Partner Countries)

Institute for Problems in Materials Science

3, Krzhizhanovsky str.

Kiev, 252142 (Ukraine)

Phone: + 380 44 444 1533 - Fax: + 380 44 444 2131 - e-mail: uvar@ipms.kiev.ua

#### Organizing Committee

M.-I. Baraton (France), I.V. Uvarova (Ukraine), J.A. Akkara (USA), R.A. Andrievski (Russia), V.V. Skorokhod (Ukraine), L.L. Kolomietch (Ukraine).

#### Official Language

English

#### Key Speakers

Akkara J.A. (NSF, Arlington, USA)

Functional Polymer Encapsulation of High Performance Nanoparticles

Andrievski R.A. (INCP, Russian Academy of Sciences, Chernogolovka, Russia)

New Superhard Materials Based on Nanostructured High-Melting Compounds - Achievements and Perspectives

Baraton M.-I. (University of Limoges, France)

Chemical Phenomena at the Surface of Nanoparticles and Thin Layers

Berndt C.C. (State University of New York, Stony Brook, USA)

Use of Thermal Spray to Manufacture Biomaterials for Dental and Orthopedic Applications

Chow G.M. (National University of Singapore, Republic of Singapore)

Nanostructured Films and Coatings. Chemical Synthesis and Properties

Eastman J.A. (Argonne National Laboratory, USA)

MOCVD Synthesis and Thermal Properties of Nanocrystalline Oxide Thin Film

Gogotsi Y.G. (University of Illinois, Chicago, USA)

Nanostructured Carbon Coatings: From Nanoporous Carbon to Aligned Nanotubes

Matkivsky A.O. (Carat Co, Lviv, Ukraine)

Perspective of Ultrafine and Nanophase Technology on Optical Manufacturing Research

Noskova N.I. (IMP, Russian Academy of Sciences, Yekaterinburg, Russia)

Local atomic Structure and Shear Modulus of Grain Boundaries in Nanocrystalline Solids

Skorokhod V.V., Ragulya A.V. (IPM, NAS Ukraine, Kiev, Ukraine)

Actual Problems in the Theory and Processing of Nanostructured Materials

Trusov L.I. (Ultram Co, Moscow, Russia)

Nanocrystalline Materials Work !

Tsakalacos T. (Rutgers University, Piscataway, USA)

Stress Distributions in Nanostructured Films and Coatings  
Uvarova I.V. (IPM, NAS Ukraine, Kiev, Ukraine)  
State-of-the-art of Ultrafine and Nanosized Materials  
Valakh M.Y. (Institute of Semiconductors, NAS Ukraine, Kiev, Ukraine)  
The Optical Investigation of Silicon-Based Nanostructured Materials  
Vassen R. (KFA, Jülich, Germany)  
Conventional and New Materials for Thermal Barrier Coatings

### **Participation**

This NATO ASI is aimed at the senior Ph.D. students, postdoctoral level and junior scientists, and suitably-qualified senior scientists. All participants (lecturers and students) are expected to remain the whole duration of the ASI (11 days) to ensure full interactions. The participants and their accompanying guests are responsible for the costs of transportation, lodging and meals.

**ASI Registration Fees :** US\$ 300 will be charged to the participants.

**ASI Location :** This ASI will be held in the building of Ukrainian Academy of Government Management, 36/1 Melnikov St., Kiev 03119, Ukraine.

**Lodging and Meals :** The ASI organizers will provide the participants with lodging and meals. The tentative price is US \$ 40-50 per day (including breakfast and two meals).

**Financial Support :** Partial financial support will be provided on a selective basis.

**Liability and Insurance :** Neither NATO nor the organizing committee will be responsible for any liabilities incident to the ASI. Participants are encouraged to obtain their own travel and medical insurance.

**Application :** Participation at this ASI, open to all suitably qualified applicants irrespective of their nationality, will be limited and by invitation only. If you wish to be considered for an invitation to attend, please complete and return the attached application form (see below). After the review of applications, invitations will be issued to selected applicants. Applications from NATO and non-CP countries should be faxed or e-mailed to the director. Applications from the CP countries should be faxed or e-mailed to the co-director.

**THE APPLICATION DEADLINE IS MARCH 7th, 2000.**

Invitations to selected applicants will be issued by the end of March and confirmation of participation must be returned to the directors within 10 days. The participants who will need a visa to enter in Ukraine will receive an official invitation letter.

The application form (see below) is limited to two pages only. The abstract of the presentation should not exceed 100 words. The selected participants will be notified if their contribution will be an oral or a poster presentation.

The definite scientific program will be sent to the selected participants in due course.

More information can be found on: <http://www.materials.kiev.ua/events>

## **APPLICATION FORM**

NATO Advanced Study Institute  
Functional Gradient Materials and Surface Layers Prepared by Fine Particles Technology  
Kiev (Ukraine) - June 18-28, 2000

### **Applicant Information**

Last Name:

First Name:

Title:

Institution:

Address of the Institution:

Phone:

Fax:

e-mail:

Education and Relevant Background:

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### **Presentation Information**

Title:

Author(s):

Institution(s) and Address(es):

Abstract(s) of the presentation(s):  
(One abstract per presentation)  
(No more than 100 words per abstract)

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

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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

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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 et/ou Eric.Gaffet@utbm.fr

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**JRFM'2000**  
23 - 24 Mai 2000 - Bordeaux France  
Wbsite : <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

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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 : euomech@lpmm.univ-metz.fr  
WebSite : <http://www.lpmm.univ-metz.fr/euomech>

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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  
website : <http://www.materials.ox.ac.uk/OCAMAC/ISMANAM/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  
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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**Hugues GUÉRAULT**

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

*28 Janvier 2000*

**TDOCTEUR de IUNIVERSITÉ DU MAINE**

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

Thèse préparée au sein du Laboratoire de Physique de l'Etat Condensé (LPEC), Université du Maine Le Mans

**Jury :**

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

**RESUME**

Ce travail est consacré à l'étude des propriétés structurales et magnétiques de poudres nanostructurées de fluorures ioniques MF<sub>3</sub> (M= Fe, Ga) obtenues par broyage haute énergie. Malgré le caractère fortement réactif des poudres (caractère hygroscopique des fluorures et augmentation de la surface spécifique), nous avons réussi à maîtriser les conditions de synthèse de ces poudres, leur reproductibilité ainsi que leur conditionnement pour

La caractérisation structurale s'appuie sur des techniques de diffraction de rayons X et de sondes locales (Spectrométrie Mössbauer du <sup>57</sup>Fe et Résonance Magnétique Nucléaire <sup>19</sup>F, <sup>69</sup>Ga et <sup>71</sup>Ga) dont la complémentarité des échelles spatiales permet une modélisation de la microstructure des poudres broyées. L'ensemble des résultats avec des grains cristallins de l'ordre de 15nm et des joints de grains désordonnés dont l'épaisseur est fonction de l'intensité de broyage. Ces deux phases, dont l'entité de base reste octaèdre MF<sub>6</sub>, sont ainsi décrites par des empilements pseudo-cubique (grains) et aléatoire (joints de grains) d'unités octaédriques reliées par les sommets.

Les comportements magnétiques statique et dynamique des poudres nanostructurées FeF<sub>3</sub> ont été suivis en fonction de la température par spectrométrie Mössbauer et mesures magnétiques FC et ZFC. A basse température, les grains cristallins présentent un ordre antiferromagnétique alors que les joints de grains ont un ordre de type -paramagnétique des joints de grains accompagné d'un découplage plus ou moins effectif des grains (monodomains), fonction de l'épaisseur des joints de grains comparée à la portée des interactions de superéchange. On observe alors un comportement superparamagnétique de l'aimantation des grains à haute température avec des températures de blocage dépendantes de l'épaisseur des joints de grains.

Broyage Haute Energie, Poudre Nanostructurée de Fluorure, Modélisation Structurale et Magnétique, Empilement d'Octaèdres, Joints de Grains, Spectrométrie Mössbauer, Diffraction de rayons X, Résonance Magnétique Nucléaire, Mesures Magnétiques

**ABSTRACT**

The structural and magnetic properties of nanostructured fluoride powders were investigated by means of several complementary techniques. In spite of both the high reactivity of powders and the high hygroscopic behaviour of fluorides, the synthesis of some nanostructured ionic fluoride powders MF<sub>3</sub> (M= Fe and Ga) was successfully achieved under certain conditions and the conditioning as well to be investigated without any further transformation.

Both X-ray diffraction and local probe techniques (<sup>57</sup>Fe Mössbauer spectrometry, <sup>19</sup>F, <sup>69</sup>Ga and <sup>71</sup>Ga NMR) were applied to these powders because of their different spatial scale efficiency. The mutual analysis of data allows first to describe the microstructure of these powders in terms of crystalline grains and grain boundaries. It also allows to estimate their respective atomic proportions as a function of milling time and milling power. The average diameter is 15 nm while the thickness of the grain boundaries is dependent on the milling power. We propose an average structural modelling which consists of pseudo-cubic and random packing of corner-sharing octahedral units assigned to crystalline grains and grain boundaries, respectively.

Static and dynamic magnetic behaviours were studied as a function of temperature by <sup>57</sup>Fe Mössbauer spectrometry and static magnetic including FC and ZFC measurements. The crystalline grains and the grain boundaries behave as blocked antiferromagnet and speromagnet at low temperature, respectively. The increase of temperature favours the progressive speromagnetic-paramagnetic transition within the grain boundaries over a wide range of temperature. The magnetisation of crystalline grains is dominated by superparamagnetic fluctuations grains at high temperature and the blocking magnetic temperature is found strongly dependent on the thickness of grain boundaries, i.e. milling conditions: the nanostructured ferric fluoride powders behave as an assembly of weakly interacting single domain antiferromagnetic grains.

**Key-words:** Mechanical Milling, Nanostructured Fluoride Powders, Structural and Magnetic Modelling, Octahedral Packing, Grain Boundaries, Mössbauer Spectrometry, X-ray Diffraction, Solid State NMR and Magnetic Measurements.

6 Déc. 1999 - Université de Technologie de Belfort - Montbéliard  
Nanomatériaux : Elaboration et Transitions de Phases Hors Equilibre, UPR 806 CNRS, UTBM - Sévenans.  
Matériaux à Grains Fins, LRSS UMR 5613 CNRS-Université de Bourgogne.

**Jury (Provisoire) :**

G. Bertrand, G.LeCaer (Rapp.) , F.Thévenot (Rapp.) , F.Bernard (Co - Dir.), E.Gaffet (Co - Dir.),

-----  
**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**

**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

**Maîtrise 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

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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, G. Bertrand, F. Bernard (Co - Dir.), E. Gaffet (Co - Dir.)

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**O. Held**

**"Etude des réactions de combustions solide-solide ou solide liquide auto-entretenu pour différents**

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

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

**D. Cracco**

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

CNRS - Thiais - France - 25 Juin 1999

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

**A. Gentil - Sagot**

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

**Elaboration par Métallurgie des Poudres**

Ecole des Mines - Paris - 17 Juin 1999

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

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**Cooperative Research on Related Areas**

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**COREE du SUD (19/01/2000)**

From Professor Soon H. Hong

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

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

E-mail : HYPERLINK mailto:shhong@sorak.kaist.ac.kr / shhong@sorak.kaist.ac.kr

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

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

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

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

**Ph D Position Proposal**

A partir de septembre 2000 (Bourse du Ministère)

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

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

1 - Etude de mélanges composites Mg (ou Mg<sub>2</sub>Ni) + intermétalliques élaborés par mécanosynthèse (structure, composition chimique, capacités d'absorption d'hydrogène, morphologie, surface, granulométrie, ) ;

2 - Réalisation d'électrodes négatives à partir de ces mélanges et études électrochimiques.

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

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

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

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

**Contact :**

**Jean-Louis BOBET**

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

33608 Pessac Cedex FRANCE

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

\*\*\*\*\*

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

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

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

\*\*\*\*\*

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

From Professor Soon H. Hong

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

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

E-mail : HYPERLINK mailto:shhong@sorak.kaist.ac.kr /shhong@sorak.kaist.ac.kr

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

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

**ISRAEL (13 / 10 / 1999)**

**\*\*\* 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

**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 débiterait 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 électrogréffés 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

## Bibliographie Récente

### Livres ou "Special Issues"

#### NOUVEAU (3/02/2000)

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

#### 1. MECHANICAL ALLOYING - FUNDAMENTALS AND APPLICATIONS

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

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

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

Energy transfer and energy maps;

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

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

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

#### DISPERSION STRENGTHENED ALUMINIUM PREPARED BY MECHANICAL ALLOYING, by M Besterici

<http://www.demon.co.uk/cambsci/book51.htm> 1. Characteristics of dispersion-strengthened systems 2. Mechanical alloying (kinetics and mechanism of preparation of the Al-C system by mechanical alloying; compaction of powders and heat treatment of compacts);

3. Microstructure and quantitative evaluation of parameters of dispersion-strengthened materials (definition and properties of interparticle distance; experimental possibilities of determination of structural objects; models of heterogeneous structures and their evaluation; simulation of model structures; analysis of the spatial distribution of particles in the Al-Al4C3 material)

4. Static and dynamic mechanical properties (mechanical properties at elevated temperatures; mechanical properties at 20 °C; effect of interface on the mechanical properties; superplastic properties of the system; thermal stability of the system; creep characteristics; creep-fatigue characteristics)

References - ISBN 189832655X, 90 pages, 234 **156 mm, soft laminated cover**, £25.00, 1999

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

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

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

#### "Non Equilibrium Processing of Materials"

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

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

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

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

A. Inoue

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

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

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

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



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

Michal Besterčí, 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)

Index : 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@demon.co.uk](mailto:cisp@demon.co.uk)

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

### **"Mechanical Alloying"**

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

Kluwer Academic Publishers

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

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

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

Materials Research Society, Symposium Proceedings Volume 501, 1998

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

### **"Nanomatériaux"**

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

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

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

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

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

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

## **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.

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**BIBLIOGRAPHY ON MECHANICAL ALLOYING AND MILLING**

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

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

Please send your order to: Book Department - Cambridge International Science Publishing 7 Meadow Walk, Great Abington, Cambridge CB1 6AZ, England Fax: +44 1223 894 539; tel +44 1223 893295, email: orders@cisp.demon.co.uk / Cambridge International Science Publishing  
<http://www.demon.co.uk/cambsci/homepage.htm>

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

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

## PERIODIQUES

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

**[47] A PHASE MIXTURE MODEL OF A PARTICLE REINFORCED COMPOSITE WITH FINE MICROSTRUCTURE**  
Kim HS. Bush MB. Estrin Y. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 276(1-2):175-185, 2000

A phase mixture model is considered in which a mechanically alloyed, particle reinforced metal matrix composite with ultrafine microstructure is treated as a mixture of a matrix phase, a reinforcing particle phase and a boundary phase. The finite element method is employed in conjunction with a unit cell of the composite to investigate the compressive and tensile behaviour of the system. The reinforcing ceramic particles are taken to be elastic. A unified constitutive model based on dislocation density evolution is used to describe the plastic flow behaviour of the matrix; grain size effects are included. A yield criterion for porous materials, including the evolution of density, is applied to the boundary phase. The boundary phase is assumed to have the mechanical properties of a quasi-amorphous material. The effects of the volume fraction of the reinforcing particles, overall effective density, grain size of the matrix, and the density of the boundary phase on the overall mechanical properties are discussed. The calculated stress-strain curves based on the unit cell model are used to simulate an indentation test and are compared with experimental measurements on Al-alumina composites.

**[46] NICKEL-MOLYBDENUM CATALYSTS FABRICATED BY MECHANICAL ALLOYING AND SPARK PLASMA SINTERING**

De la Torre SD. Oleszak D. Kakitsuji A. Miyamoto K. Miyamoto H. Martinez-S R. Almeraya-C F. Martinez-V A. Rios-J D. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 276(1-2):226-235, 2000

The low energy ball milling process has been used to fabricate Ni-43at.%Mo based electro-active powder. Nanometre-sized Mo particles have been embedded into an agglomerated amorphous matrix. The spark plasma sintering (SPS) process has been used to densify the powder in < 6 min at heating rates of about 250 degrees C min<sup>-1</sup> from 400 to 1300 degrees C, applying loads of 120 MPa. High performance porous nickel electrodes having new phases with a particle size range between 1.0 and 8.7 µm have been obtained after rapid SPS operations. Potentiodynamic polarisation techniques have been used to characterise the hydrogen evolution reaction (HER) in KOH 30% solution at 70 degrees C. The cathodic-Tafel slopes beta c have been found from 25 to 150 mV dec<sup>-1</sup> and appear to decrease as the open porosity of electrodes enlarges. It has been confirmed that the larger porosity (surface roughness) of the electrodes the larger exchange current density I<sub>0</sub> and so their electrocatalytic activity to polarise the HER. In addition to a large surface area and a particle size distribution, the presence of metastable Ni-Mo phases coexisting with MoO<sub>2</sub> and the existence of localised galvanic couples are remarkable features conferring the electrodes with unusual reactivity. In this study the microstructure and densification attained are reported on, as well as the preliminary polarisation analysis of these electrodes.

**[45] MECHANICAL PROPERTIES OF A ZrC-DISPERSED MO ALLOY PROCESSED BY MECHANICAL ALLOYING AND SPARK PLASMA SINTERING**

Takida T. Mabuchi M. Nakamura M. Igarashi T. Doi Y. Nagae T. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 276(1-2):269-272, 2000

A ZrC particle-dispersed Mo processed by mechanical alloying showed much higher strength than a recrystallized pure Mo at room temperature and showed a large elongation of 180% at 1970 K. These excellent properties of the particle-dispersed Mo are attributed to a very small grain size of 3 µm.

**[44] IN SITU FORMATION OF TITANIUM SILICIDES-REINFORCED TiAl-BASED COMPOSITES**

Rao KP. Du YJ. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 277(1-2):46-56, 2000

A new method for preparing Ti<sub>5</sub>Si<sub>3</sub>/TiAl in situ composite has been tried. TiAlSi-based metastable precursors were first synthesized by mechanical alloying, and blended with Ti-50Al powder matrix. The composite mix was cold-pressed to form pellets, followed by heat treatment to initiate reactive sintering that led to the formation of Ti<sub>5</sub>Si<sub>3</sub>/TiAl composites. The resultant in situ formed Ti<sub>5</sub>Si<sub>3</sub>/TiAl composites exhibited multiplex structures containing alpha(2) and gamma phases, extra Ti and small silicide precipitates, and showed some morphological traces of liquid phase sintering. The microstructural evolutions during the formation of metastable precursors and the subsequent phase reactions/transitions involved in the formation of composites have been investigated. A phase hierarchy has been observed during in situ formation of the reinforcement, i.e. TiSi<sub>2</sub> --> Ti<sub>5</sub>Si<sub>4</sub> --> Ti<sub>5</sub>Si<sub>3</sub>. Such a phase selection route, though intrinsically forwarded by thermodynamics, is strongly biased by kinetic factors such as diffusion. Nearly fully monolithic Ti<sub>5</sub>Si<sub>3</sub> reinforcements can be obtained by phase selection for metastable precursors with stoichiometry of Ti-14Al-21Si but not Ti-11Al-21Si, though both were subjected to excess Ti as kinetic bias.

**[43] X-RAY DIFFRACTION, MAGNETIZATION AND MOSSBAUER STUDIES OF NANOCRYSTALLINE Fe-Ni ALLOYS PREPARED BY LOW- AND HIGH-ENERGY BALL MILLING**

Jartych E. Zurawicz JK. Oleszak D. Pekala M. - Journal of Magnetism & Magnetic Materials. 208(3):221-230, 2000

Fe-Ni alloys were prepared both by low- and high-energy ball milling processes. Structure and magnetic properties were studied by using X-ray diffraction, differential scanning calorimetry, Mossbauer spectroscopy and magnetization measurements. Mechanical treatment influenced the magnetic properties of Fe-Ni alloys as compared with the equilibrium alloys. Reduction of grain size resulted in the increase of magnetization. Invar anomaly for 35 at% Ni was not detected.

**[42] THE EFFECTS OF LONG TERM ANNEALING AT 1000 DEGREES C FOR 24 H ON THE MICROSTRUCTURE AND MAGNETIC PROPERTIES OF PR-FE-B/ND-FE-B MAGNETS BASED ON ND<sub>16</sub>FE<sub>76</sub>B<sub>8</sub> AND PR<sub>16</sub>FE<sub>76</sub>B<sub>8</sub>**

Corfield MR. Williams AJ. Harris IR. - Journal of Alloys & Compounds. 296(1-2):138-147, 2000

Magnets produced via a hydrogen decrepitation/roller-milling route have been subjected to a post sintering heat treatment of 1000 degrees C for 24 h. Alloys of nominal composition Pr<sub>16</sub>Fe<sub>76</sub>B<sub>8</sub> and Nd<sub>16</sub>Fe<sub>76</sub>B<sub>8</sub> have been studied in terms of both microstructure and magnetic properties to determine the influence of this 24-h annealing treatment. The effect of annealing the Pr<sub>16</sub>Fe<sub>76</sub>B<sub>8</sub> magnets at 1000 degrees C for up to 24 h resulted in a general increase in the overall magnetic properties, especially in the intrinsic coercivity. In contrast to these observations, the same heat treatment was found to be detrimental to all the magnetic properties of the Nd<sub>16</sub>Fe<sub>76</sub>B<sub>8</sub> magnets.

**[41] A WET BALL MILLING TREATMENT OF Zr-BASED AB(2) ALLOYS AS NEGATIVE ELECTRODE MATERIALS**

Liu BH. Li ZP. Okutsu A. Suda S. - Journal of Alloys & Compounds. 296(1-2):148-151, 2000

A wet ball milling fluorination treatment was successfully developed to improve rate discharge capabilities of Zr-based AB<sub>2</sub> alloys. In this work, treatment conditions such as constituents in the treating solution, ball milling speed and time were optimized. It was found that the Ni-rich surface layer formed through the Ni<sup>2+</sup> reduction by alloy component elements played an important role in the initial activation property. The size of alloy powder particles can be easily adjusted by changing the ball milling speed and time. Although a small size of alloy powder particle is beneficial for quick kinetics, excessive pulverization resulted in a decreased capacity at low discharge rates.

**[40] MAGNETIC PROPERTIES OF COBALT FERRITE/SiO<sub>2</sub> NANOCOMPOSITE**

Mekala SR. Ding J. - Journal of Alloys & Compounds. 296(1-2):152-156, 2000

Nanocrystalline cobalt ferrite dispersed in silica was prepared by mechanical alloying and subsequent heat-treatment. For Co-ferrite with 15 wt% silica, a high coercivity of up to 3.3 kOe and a magnetization of 68 emu/g were obtained after an optimized heat treatment (in air at 900 degrees C). Experiments were conducted to study the optimum amount of silica required to obtain optimum magnetic properties. It was found that a small amount of 4.5 wt% silica (SiO<sub>2</sub>) allows the composite to possess good magnetic properties (coercivity of .3 kOe and saturation magnetization of 72 emu/g).

**[39] MICROSTRUCTURE AND MECHANICAL PROPERTIES OF NANOCRYSTALLINE AL-TI ALLOYS CONSOLIDATED BY PLASMA ACTIVATED SINTERING**

Ryu JR. Moon KI. Lee KS. - Journal of Alloys & Compounds. 296(1-2):157-165, 2000

Nanocrystalline Al-Ti alloys were produced by reactive ball milling and subsequently plasma activated sintered (PAS). The consolidation behavior and microstructure of the nanocrystalline alloys were studied as a function of sintering pressure and temperature with constant holding time of 60 s. This showed that the pressure and temperature had large effects on the final density and grain size. The PAS consolidated Al-Ti alloys exhibited full density (99% of theoretical density) while retaining a grain size of the order of 50-100 nm at a temperature of 500 degrees C with a pressure of 75 MPa. This alloy showed a much more uniform grain distribution and finer grain size compared with Al-Ti alloys manufactured by conventional consolidation methods. At room temperature, the compressive yield strength (about 692 MPa) of PAS alloy was much higher than that of hot pressed Al-Ti alloys. The higher yield strength is considered to be due to the effect of grain refinement strengthening. At all temperatures above 300 degrees C, the PAS alloys exhibited plastic strain beyond 60%.

**[38] FABRICATION OF NANOCRYSTALLINE WC AND NANOCOMPOSITE WC-MGO REFRACTORY MATERIALS AT ROOM TEMPERATURE**

El-Eskandarany MS. - Journal of Alloys & Compounds. 296(1-2):175-182, 2000

Reactant material powders of pure WO<sub>3</sub>, Mg and graphite have been milled at room temperature using a high-energy ball mill. After a few kiloseconds of milling (11 ks), numerous fresh surfaces of the reactant materials are created as a result of the repeated impact and shear forces generated by the balls. After 86 ks of milling, a mechanical solid state reduction is successfully achieved between the fresh Mg and WO<sub>3</sub> particles to form a product of nanocrystalline mixture of MgO and W. A typical mechanical solid state reaction takes place between the W particles and graphite powders to obtain fine grains of nanocrystalline WC. Towards the end-stage of ball-milling (173 ks), the nanocrystalline MgO grains (10 nm) are embedded into the fine matrix of WC to form fine nanocomposite powders (1 μm in diameter) of WC-18% MgO material with spherical-like morphology. This composite powder was then consolidated under vacuum at 1963 K, with a pressure ranging from 19.6 to 38.2 MPa for 0.3 ks, using a plasma activated sintering method. In addition, pure nanocrystalline WC powders (7 nm in diameter) obtained by removing the h<sub>4</sub>g<sub>0</sub> from the milled powders, using a simple leaching technique have been also consolidated by the same consolidation technique. The consolidation step does not lead to a dramatic grain growth and the compacted samples that are fully dense still maintain their unique nanocrystalline characteristics. The elastic properties and the hardness of both consolidated samples have been investigated. A model for fabrication of refractory nanocrystalline WC and nanocomposite WC-18% MgO materials at room temperature is proposed.

**[37] METASTABLE ALLOY BULK BODIES IN THE Fe-W SYSTEM PREPARED BY MECHANICAL ALLOYING AND SHOCK COMPRESSION**

Huang XS. Mashimo T. - Journal of Alloys & Compounds. 296(1-2):183-190, 2000

Bulk bodies of metastable alloys including supersaturated solid solutions and amorphous phases in the iron (Fe)-tungsten (W) system were prepared by mechanical alloying (MA) and shock compression. The X-ray diffraction patterns of the W solid solutions were obtained for the MA-treated powders in the Fe<sub>x</sub>W<sub>100-x</sub> system with Fe content of x less than or equal to 30 mol%, and that of Fe solid solution was obtained with an Fe content of x=90 mol%. For the mixed powders with Fe content of 40 less than or equal to x less than or equal to 70 mol%, the peaks of Fe completely disappeared, and the amorphous halo-like patterns were observed around the (110) peak of W solid solution. For the mixed powder with an Fe content of 80 mol%, an X-ray diffraction pattern of a two-phase mixture of Fe and W solid solutions was obtained. For the MA-treated powders, the lattice parameters of W solid solutions were smaller than that of pure W, and those of Fe solid solutions were larger than that of pure Fe. No large crack could be observed in shock-consolidated bulk bodies, and the cross sections of the bulk bodies showed a metallic gloss. The X-ray diffraction patterns of shock-consolidated bulk bodies formed in a specific low pressure range did not change significantly from those of the MA-treated powders, which indicated that the metastable phases were successfully

consolidated by shock compression without decomposition or recrystallization. Above a driving shock pressure of 40.1 GPa in capsule for the 30:70 mol% Fe-W system and that of 30.5 GPa for the 50:50 mol% Fe-W system, the X-ray diffraction patterns of the recovered bulk bodies showed the appearance of the peaks of Fe<sub>7</sub>W<sub>6</sub> intermetallic compound and the peaks of Fe. The recovered specimens of the metastable solid solution phases in the 80:20 mol% Fe-W system did not recrystallize or decompose up to a driving shock pressure of 39.5 GPa. It was confirmed by the Electron Probe Micro Analysis (EPMA) that Fe and W dispersed well at the submicron level in the shock-consolidated bulk bodies. The Vickers hardnesses of the bulk bodies were much higher than those of pure Fe and pure W polycrystals.

**[36] SOLID STATE SYNTHESIS OF TUNGSTEN CARBIDE IN AN INERT COPPER MATRIX**

Baikalova YV. Lomovsky OI. - Journal of Alloys & Compounds. 297(1-2):87-91, 2000

A copper-tungsten-carbon system was chosen to study the possibility of synthesis of tungsten carbide phases from elements in an inert matrix. Copper played the role of the inert matrix and the composition W<sub>50</sub>C<sub>50</sub> was investigated. A planetary type mill with ball acceleration up to 600 m s<sup>-2</sup> was used as an activator for mechanical treatment. Thermal annealing at 870-940 degrees C was carried out in an argon atmosphere. The influence of the initial Cu-W-C mixture content and preliminary mechanical treatment time on the final product phase content and grain size was studied. High tungsten content is found to retard WC synthesis and W<sub>2</sub>C is obtained as the dominating product. Low tungsten content leads to the enhancement of WC in the products. A decrease in product grain size down to the submicron level with an increase of preliminary mechanical treatment time is demonstrated for Cu<sub>94</sub>W<sub>3</sub>C<sub>3</sub> composition, as an example.

**[35] STABILIZATION OF PEROVSKITE PHASE AND DIELECTRIC PROPERTIES OF 0.95PZN-0.05BT DERIVED FROM MECHANICAL ACTIVATION**

Ling TY. Xue JM. Wang J. - Journal of Alloys & Compounds. 297(1-2):92-98, 2000

0.95Pb(Zn<sub>1/3</sub>Nb<sub>2/3</sub>)O<sub>3</sub>-0.05BaTiO<sub>3</sub> (0.95PZN-0.05BT) of perovskite structure has been successfully synthesized by mechanically activating a powder mixture of PbO, Nb<sub>2</sub>O<sub>5</sub>, ZnO, BaO, and TiO<sub>2</sub>. Nanosized 0.95PZN-0.05BT perovskite particles were triggered to form in the powder mixture mechanically activated for 20 h. The resulting perovskite phase undergoes a decomposition reaction to form pyrochlore phase with increasing heat treatment temperature over the range of 450 to 700 degrees C. Further increasing the heat treatment temperatures above 700 degrees C results in a recovery of the perovskite phase as a consequence of the stabilization of PZN by BaTiO<sub>3</sub>. Sintering at 1100 degrees C for 1 h led to similar to 94% perovskite phase and a sintered density of similar to 96% theoretical in 0.95PZN-0.05BT. Sintered 0.95PZN-0.05BT exhibits a peak dielectric constant of 14000 and a dielectric loss of 0.02 at the Curie temperature of 98 degrees C when measured at a frequency of 100 Hz.

**[34] MECHANOCHEMICAL SYNTHESIS OF LAMNO<sub>3</sub> FROM LA<sub>2</sub>O<sub>3</sub> AND MN<sub>2</sub>O<sub>3</sub> POWDERS**

Zhang QW. Saito F. - Journal of Alloys & Compounds. 297(1-2):99-103, 2000

Perovskite-type lanthanum manganite (LaMnO<sub>3</sub>) powder was synthesized by room temperature milling of La<sub>2</sub>O<sub>3</sub> and Mn<sub>2</sub>O<sub>3</sub> powders using a planetary ball mill. The synthesis reaction proceeds with increase in milling time and is finished by about 180 min. The reaction process was quantitatively evaluated by measuring the percentage of free (unreacted) La<sub>2</sub>O<sub>3</sub> in the milled samples. The product powder (LaMnO<sub>3</sub>) has large specific surface area of about 10 m<sup>2</sup>/g, which is comparable to the data obtained by the complex chemical synthesis methods. The mechanochemical process can also be applied to synthesis of manganese complex oxides with other rare earth elements such as Pr, Nd and Sm.

**[33] NANOCOMPOSITES IN THE SN-MN-C SYSTEM PRODUCED BY MECHANICAL ALLOYING**

Beaulieu L. Larcher D. Dunlap RA. Dahn JR. - Journal of Alloys & Compounds. 297(1-2):122-128, 2000

Intermetallic phases and mixtures of intermetallic phases in the Sn-Mn-C ternary system were prepared by mechanical alloying. Studies concentrated on Sn<sub>2</sub>Mn, SnMn<sub>1.77</sub> and SnMn<sub>3</sub>C. Nanoscale two-phase mixtures of Sn<sub>2</sub>Mn-SnMn<sub>1.77</sub> and Sn<sub>2</sub>Mn-SnMn<sub>3</sub>C were prepared and studied by electron microscopy, X-ray diffraction and Mossbauer spectroscopy. These results show that each particle is a mixture of nanoscopic grains of the coexisting phases. The grain sizes of the phases are shown to be in the 10 nm range. On the basis of the results in the Sn-Fe-C system the present investigation suggests that the materials prepared here may be suitable as anode materials for Li-ion cells.

**[32] EFFECT OF SUBSTITUTION OF FE AND NI FOR CO IN THE SYNTHESIS OF MG<sub>2</sub>CO COMPOUND USING THE MECHANICAL ALLOYING METHOD**

Bobet JL. Akiba E. Darriet B. - Journal of Alloys & Compounds. 297(1-2):192-198, 2000

The influence of the elaboration route of Mg<sub>2</sub>Co<sub>1-x</sub>M<sub>x</sub> (M=Fe or Ni and x less than or equal to 0.3) hydrogen storage alloy was investigated. When Co was replaced by Fe, the formation of 'Mg<sub>2</sub>Co' (containing a very small amount of Fe) was observed after about 60 h of ball milling, but the yield of formation was lower. Increasing milling time just led to an amorphization of the 'Mg<sub>2</sub>Co' compound. We also observed, that as expected, no new compound was formed between Mg and Fe. Annealing of unmilled products did not lead to any new compounds and annealing of milled products just led to the crystallization of the existing compounds. When cobalt was replaced by nickel, the major difference was the formation, after annealing, of the Mg<sub>2</sub>Ni compound. This difference is attributed to the higher stability of Mg<sub>2</sub>Ni compared to that of 'Mg<sub>2</sub>Co'. The yield of the 'Mg<sub>2</sub>Co' phase was higher than that with Fe and that without any adding element.

**[31] STRUCTURE AND SOFT MAGNETIC PROPERTIES OF FINEMET ALLOYS**

Raja MM. Chattopadhyay K. Majumdar B. Narayanasamy A. - Journal of Alloys & Compounds. 297(1-2):199-205, 2000

The soft magnetic properties of Fe<sub>73.5</sub>Cu<sub>1</sub>Nb<sub>3</sub>Si<sub>13.5</sub>B<sub>9</sub> (Finemet) alloys prepared by mechanical alloying were studied using various experimental techniques including Mossbauer spectroscopy. The study includes the influence of the milling atmosphere. The results are compared with mechanically alloyed Fe-Si alloys as well as with melt-spun Finemet alloys. The coercivity of the mechanically alloyed powder is much larger than that of the melt-spun ribbons, though the saturation magnetisation is the same. This is ascribed to the absence of a grain boundary amorphous

ferromagnetic phase resulting in a weakening of the exchange coupling between the nanograins. This study suggests that the interfacial component in Finemet alloys plays a crucial role in achieving good soft magnetic properties. The critical grain size for single domain particle was found to be 10 nm. For very small grain sizes, the existence of superparamagnetism was also studied using Fe-57 Mossbauer spectroscopy.

**[30] STUDY OF THERMAL STABILITY OF MECHANICALLY ALLOYED Ti-75% AL POWDERS**

Zhang F. Lu L. Lai MO. - Journal of Alloys & Compounds. 297(1-2):211-218, 2000

Phase formation during mechanical alloying of Ti-75Al powder mixtures has been investigated using X-ray diffraction method. An Al (Ti) solid solution by the diffusion of Ti into Al was formed at the early stage of the milling. The upper solubility of Ti in Al was determined as 6.7 wt.% in the present study. Further milling led to the formation of L1(2) ordered Al<sub>3</sub>Ti with a lattice parameter of 3.983 Angstrom. The average grain size of this phase was about 15 nm. Thermal stability of the mechanically alloyed powders was systematically studied using differential thermal analyzer. The results showed that during the heat treatment, formation of different phases strongly depended on the duration of milling. For the sample milled for 30 h, the activation energy of L1(2)-Al<sub>3</sub>Ti→D0(23)-Al<sub>3</sub>Ti transformation was determined as 147 kJ mol<sup>-1</sup> using Kissinger's equation, while that of D0(23)-Al<sub>3</sub>Ti→D0(22)-Al<sub>3</sub>Ti was 243 kJ mol<sup>-1</sup>.

**[29] THE STRUCTURE AND MAGNETIC PROPERTIES OF Nd8Fe84-XCuXTi8 (X=0 SIMILAR TO 3) ALLOYS PREPARED BY MECHANICAL ALLOYING**

Cui BZ. Sun XK. Liu W. Geng DY. Zhang ZD. Zhao XG. - Journal of Alloys & Compounds. 297(1-2):219-225, 2000

The effects of a partial substitution of Cu for Fe on the structure and magnetic properties of mechanically alloyed Nd<sub>8</sub>Fe<sub>84</sub>Ti<sub>8</sub> alloys were studied in detail. Compared with Cu-free Nd<sub>8</sub>Fe<sub>84</sub>Ti<sub>8</sub> alloys prepared by mechanical alloying under the same conditions, the temperature necessary for the formation of the Nd(Fe, Ti)(12) phase decreased from 900 to 750 degrees C with the addition of 3 at.% Cu. The addition of Cu negatively influenced the nitrogeneration of the Nd(Fe, Ti)(12) phase. The pretreatment of hydrogenation on the Cu-added alloys at 300 degrees C facilitated the formation of nitrides. With increasing the annealing temperature, the intrinsic coercivity of the nitrides increased until it attained a maximum at 850 degrees C. In the 1 at.% Cu-added alloys annealed at temperatures between 750 and 800 degrees C, the metastable phase Nd(Fe, Ti)(7) was formed. With increasing the annealing temperature, the Nd(Fe, Ti)(7) phase was gradually transformed into the Nd(Fe, Ti)(12) phase. The intrinsic coercivity of the 1 at.% Cu-added nitrides reached a maximum at 900 degrees C. With increasing Cu content, the magnetic properties of the nitrides degraded.

**[28] HYDRIDING PROPERTIES OF A MECHANICALLY MILLED Mg-50 WT.% ZrFe1.4Cr0.6 COMPOSITE**

Wang P. Wang AM. Zhang HF. Ding BZ. Hu ZQ. - Journal of Alloys & Compounds. 297(1-2):240-245, 2000

The ball-milling method was used to produce Mg-50 wt.% ZrFe<sub>1.4</sub>Cr<sub>0.6</sub> composite. The thermodynamic, kinetic, and cycling properties, as well as the resistance to oxidation were examined. This composite possesses excellent kinetic properties, even at moderate temperature. Scanning electron microscopy, energy-dispersive spectrum transmission electron microscopy and X-ray diffraction were used to characterize the composite before and after hydriding. The fine ZrFe<sub>1.4</sub>Cr<sub>0.6</sub> particles covering Mg particles acting as a "catalytic film", a significant amount of imperfections and some alloy powder in the Mg matrix introduced by ball milling all contributed to the enhanced absorption/desorption rate.

**[27] SYNTHESIS OF A LOW-DENSITY Ti-Mg-Si ALLOY**

Senkov ON. Cavusoglu M. Froes FH. - Journal of Alloys & Compounds. 297(1-2):246-252, 2000

A low-density titanium alloy was synthesized from blended elemental powders of TiH<sub>2</sub>, Mg, and Si by mechanical alloying and/or heat treatment. The titanium hydride was used in place of titanium. Phase transformations occurring in the system during heating at a constant rate were studied with the use of DTA and XRD. During heating of the blended elemental powders decomposition of titanium hydride occurred in the temperature range 550-750 degrees C and some silicon went into solid solution in titanium while the majority of the silicon reacted exothermically with magnesium at about 500 degrees C producing an intermetallic phase Mg<sub>2</sub>Si. This phase was stable on heating up to 950 degrees C, where a eutectic component of this phase began to melt leading to formation of a liquid solution of magnesium in silicon, followed by a reaction of the Liquid silicon with titanium and formation of a Ti<sub>5</sub>Si<sub>3</sub> phase. A third reaction in the system was detected at about 1100 degrees C due to formation of MgO, so that after annealing at 1150 degrees C three stable phases, Ti(Si), Ti<sub>5</sub>Si<sub>3</sub>, and MgO, were present in the alloy. No decomposition of the Ti<sub>5</sub>Si<sub>3</sub> phase or formation of Mg<sub>2</sub>Si were detected either during subsequent cooling or a second heating of the alloy. Completely different kinetics of the phase reactions occurred in the mechanically alloyed powders. Magnesium and silicon dissolved in the titanium hydride during mechanical alloying. Decomposition of the titanium hydride occurred at 320-600 degrees C, the Mg<sub>2</sub>Si phase was formed after heating to 450 degrees C, and the Ti<sub>5</sub>Si<sub>3</sub> phase was detected after heating to 570 degrees C. The Mg<sub>2</sub>Si decomposed completely at a temperature of 650 degrees C with the formation of MgO and Ti<sub>5</sub>Si<sub>3</sub>. After heating to 1150 degrees C, three stable phases, TiN<sub>0.3</sub>, Ti<sub>5</sub>Si<sub>3</sub>, and MgO, were present in the alloy. A discussion of the results is given.

**[26] HYDROGEN STORAGE IN MECHANICALLY MILLED Mg-LaNi5 AND MgH2-LaNi5 COMPOSITES**

Liang G. Huot J. Boily S. Van Neste A. Schulz R. - Journal of Alloys & Compounds. 297(1-2):261-265, 2000

Magnesium and magnesium hydride were mechanically milled with LaNi<sub>5</sub> to make a Mg-Ni-La ternary alloy for hydrogen storage. Mechanical milling of MgH<sub>2</sub> + LaNi<sub>5</sub> or milling of Mg + LaNi<sub>5</sub> followed by a full hydrogenation leads to a composite of MgH<sub>2</sub> + LaH<sub>3</sub> + Mg<sub>2</sub>Ni. Upon hydrogen absorption/desorption cycling, a mixture of Mg + LaH<sub>3</sub> + Mg<sub>2</sub>Ni phases is obtained in both cases, but with different powder sizes. The powder size is greatly reduced by using MgH<sub>2</sub> instead of Mg in the milling process. The reduction in powder size gives faster absorption kinetics, and slower desorption kinetics. Adding both Ni and La to Mg-based alloys produces a synergetic effect on the hydrogen absorption/desorption. The ternary Mg-Ti-La alloy showed much better absorption and desorption kinetics than the binary alloys Mg-La and Mg-Ni. Lanthanum hydride has strong catalytic effects on absorption of Mg, but weak effects on desorption. Mg<sub>2</sub>Ni has better catalytic effect than lanthanum hydride at temperatures above 373 K.

**[25] HYDRIDING BEHAVIOR OF MG-AL AND LEACHED MG-AL COMPOUNDS PREPARED BY HIGH-ENERGY BALL-MILLING**

Bouaricha S. Dodelet JP. Guay D. Huot J. Boily S. Schulz R. - Journal of Alloys & Compounds. 297(1-2):282-293, 2000

The structure and hydrogen absorption properties of Mg:Al alloys prepared by high-energy ball milling were studied over the whole compositional range. These materials were prepared in their as-milled and Al-leached forms. The latter are obtained from the former materials by leaching out Al in a 1 N NaOH solution. The structure of the various alloys was determined by X-ray diffraction. The structure of the material in the hydrided state was also determined in some cases. In the as-milled state, hcp Mg(Al) with a small proportion of Mg<sub>17</sub>Al<sub>12</sub> and fcc Al(Mg) are formed at Mg:Al (90:10) and (20:80) compositions, respectively. At intermediate (58:42) and (37:63) compositions, the intermetallic Mg<sub>17</sub>Al<sub>12</sub> and Mg<sub>3</sub>Al<sub>2</sub> phases are formed, respectively. Following leaching, the Al content of Mg:Al (90:10) and (20:80) varies from 10.4 and 77.0 to 3.0 and 51.0 at.%, respectively. In both cases, noticeable change in the XRD pattern confirms that bulk dissolution of Al has been achieved. There is a two-fold increase in the specific surface area of Mg:Al (90:10) following leaching of Al. In the case of Mg:Al with intermediate compositions, dissolution of Al, if any, does not lead to discernable modification in the structure of the material. The measured hydrogen capacity of the as milled material decreases with Al content, from H/M=1.74 for pure un-milled Mg, to 1.38 for Mg:Al (90:10), and then to 1.05 for Mg:Al:Al (75:25). In each case, there is a further 10-15% decline of the hydrogen absorption capacity after leaching. In the case of Mg:Al (58:42), which basically only contains a nanocrystalline Mg<sub>17</sub>Al<sub>12</sub> intermetallic phase, hydriding leads to the formation of MgH<sub>2</sub> and Al. This reaction is totally reversible and Mg<sub>17</sub>Al<sub>12</sub> is recovered upon de-hydriding. In each case, there is an increase in the kinetics of hydrogen absorption and desorption following Al leaching.

**[24] MECHANICAL ALLOYING AND SINTERING OF LEAD TELLURIDE**

Bouad N. Marin-Ayral RM. Tedenac JC. - Journal of Alloys & Compounds. 297(1-2):312-318, 2000

Lead telluride, appropriately doped, has promising good thermoelectric properties. It has often been utilised since the 1960s in thermogenerators, Conventional solidification was used in this case. Now, for industrial applications, we must reduce the cost of fabrication, meanwhile thermoelectric properties must be enhanced. In this study, we develop thermoelectric materials for a new process, mechanical alloying, which permits us to obtain smaller grain size powders. Moreover, we have succeeded in sintering these powders.

**[23] DEFORMATION INDUCED TRANSFORMATIONS AND GRAIN BOUNDARY THICKNESS IN NANOCRYSTALLINE B2FEAL**

Negri D. Yavari AR. Deriu A. - Acta Materialia. 47(18):4545-4554, 1999

Precise measurement of fundamental Bragg peak shifts during milling of nanocrystalline ordered B2 Fe<sub>60</sub>Al<sub>40</sub> has allowed for the first time, the deconvolution of the 110 fundamental Bragg peak intensities of the b.c.c. disordered regions and of the ordered B2 regions from the start and before full disappearance of the latter. The evolution of the lattice parameter  $a(0)$  of the b.c.c. solid solution with milling time shows two characteristics. First a jump to higher values from the initial  $a(0)$  of the B2 phase, with a  $\Delta a(0)$  change of the order of 1% corresponding to a volume per atom  $\Delta V$ -expansion of about 3%. Subsequently,  $a(0)$  continues to increase slowly with further milling at constant grain size  $D$  and in the absence of any B2 phase. This continuing change of  $a(0)$  with further milling up to at least 180 min is attributable to a reduction of chemical short-range order (CSRO) or the number of Al-Fe heteroatomic "bonds". The appearance of two well-defined maxima in the hyperfine field (HF) distributions derived from the Mossbauer spectra indicated the presence of two ferromagnetic environments contributing to the broadened Mossbauer resonance sextet signal. The evolution of the second component of this Mossbauer signal scales with the grain size. Using the mean grain size  $D$  derived from X-ray peak profiles and TEM pictures together with the grain boundary thickness  $d(gb)$  of 1.25 nm determined by Fultz et al. (J. appl. Phys., 1996, 7, 127) for b.c.c. Fe-based alloys, the fraction of grain boundary atoms  $n(gb)/n(\text{total})$  was estimated and found to be consistent with the fraction of Fe atoms contributing to the lower HF component of the Mossbauer sextet signal. The grain boundary atom count using both methods confirms that grain boundaries in materials nanocrystallized by heavy deformation are nearly as dense as in the bulk.

**[22] CARBOTHERMIC REDUCTION OF ILMENITE (FeTiO<sub>3</sub>) AND RUTILE (TiO<sub>2</sub>)**

Welham NJ. Williams JS. - Metallurgical & Materials Transactions B-Process Metallurgy & Materials Processing Science. 30(6):1075-1081, 1999

The mechanically activated carbothermic reduction mechanism of ilmenite has been examined by a combination of steady-state and dynamic thermal techniques coupled with X-ray diffraction. The reaction was found to proceed via an initial, rapid reduction to elemental iron and rutile, which was followed by a slow reduction of rutile to a series of oxides of the general formula  $Ti_nO_{2n-1}$  until Ti<sub>3</sub>O<sub>5</sub> was formed, which was found to be relatively stable. Iron was probably incorporated into the  $Ti_nO_{2n-1}$  lattice only for  $n > 3$ , forming mixed oxides of uncertain composition. The formation of TiC was evident at temperatures as low as 1100 degrees C, but the rate of reaction was extremely slow, presumably due to a solid-state diffusion limitation. Increasing the temperature gave increasing conversion of TiO<sub>2</sub> to TiC until it was the only confirmed product. The effect of iron on the later stages of reduction was removed by examining the reduction of pure rutile. It was found that the reduction of Ti<sub>3</sub>O<sub>5</sub> was enhanced by the presence of iron. The separation of iron from the titanium product proved to be high, with >90 pct of iron removed after the initial reduction. The iron removal increased slowly to almost 100 pct when elemental iron and titanium carbide were the products.

**[21] MICROSTRUCTURAL REQUIREMENTS FOR ALUMINA-SiC NANOCOMPOSITES**

Winn AJ. Todd RI. - British Ceramic Transactions. 98(5):219-224, 1999

The relative ease with which alumina-SiC nanocomposites polish, compared with monolithic alumina, has been noted in the literature and taken to be an example of the beneficial 'nanocomposite effect' stemming from an apparent strengthening of the grain boundaries. The percentage of 'polished' surface, as opposed to areas dominated by grain pullout or fracture, following grinding or polishing, therefore provides an easily obtainable figure of merit for

different nanocomposite materials. A variety of alumina-SiC nanocomposites has been fabricated by co-milling commercially available powders and hot pressing in an inert atmosphere. Using variations in starting powder size and heating schedules, it has been possible to produce nanocomposites with similar matrix grain size but with different proportions of particles on the grain boundaries relative to those within the grains. The microstructures were examined using TEM and FEG-SEM, and the effect of microstructural variations on the mechanical properties of the materials was investigated. Fracture surfaces were examined, and a study was made of the relative amounts of polished surface and grain pullout after both grinding and polishing the materials using standard metallographic techniques. The performance of the nanocomposites was compared with that of a pure alumina of the same grain size. The results indicate that the SiC has a direct effect on crack initiation and propagation in alumina, and that it is the particles on the grain boundaries which are responsible for the beneficial effect of the SiC additions. If sufficient SiC is present on the grain boundaries, crack initiation during abrasive wear can be prevented, resulting in a smoother, plastically deformed surface.

**[20] HOW DIFFERENT IS MECHANICAL ACTIVATION FROM THERMAL ACTIVATION? A CASE STUDY WITH PZN AND PZN-BASED RELAXORS**

Wang J. Xue JM. Wan DM. - Solid State Ionics. 127(1-2):169-175, 2000

Using Pb(Zn<sub>1/3</sub>Nb<sub>2/3</sub>)O<sub>3</sub> (PZN) and PZN-based relaxer ferroelectrics, we demonstrate that the solid state reactions triggered by mechanical activation of mixed oxides can proceed via a completely different route from that in the conventional solid state reactions, where the process is controlled by one or more interfacial reactions and diffusions. Nanocrystallites of perovskite Pb(Zn<sub>1/3</sub>Nb<sub>2/3</sub>)O<sub>3</sub> are formed in a highly activated state of mixed oxides where a degree of amorphization has taken place, followed by a steady growth with increasing degree of mechanical activation. The absence of transitional pyrochlores, such as the Pb<sub>3</sub>Nb<sub>4</sub>O<sub>13</sub>-type phases which have always been observed in the conventional solid state reactions, and the considerably low local temperature at the collision points suggest that the perovskite phase occurs via a process involving nucleation and subsequent diffusionless growth.

**[19] OXIDATION OF 1-ALKYLCYCLOALKANOLS WITH PB-IV AND MN-III COMPOUNDS UNDER MECHANICAL ACTIVATION**

Kapustina NI. Sokova LL. Makhaev VD. Petrova LA. Nikishin GI.- Russian Chemical Bulletin. 48(11):2080-2082, 1999

A mechanoactivated solid-state oxidative decyclization of 1-alkylcycloalkanols under the action of the Pb(OAc)<sub>4</sub>-MX or Mn(OAc)<sub>3</sub>-MX systems (MX is a metal halide) was carried out for the first time. The reaction affords exclusively omega-haloalkanes.

**[18] EFFECT OF SiC PARTICULATE SIZES ON THE MECHANICAL PROPERTIES OF AL-6Ti-6Nb MATRIX COMPOSITES PREPARED BY A MECHANICAL ALLOYING TECHNIQUE**

Jia DC. Li XL. Zhou Y. Han HQ. - Journal of Materials Science Letters. 18(24):2029-2032, 1999

**[17] NANOTUBE GROWTH BY SURFACE DIFFUSION**

Chadderton LT. Chen Y. - Physics Letters A. 263(4-6):401-405, 1999

Nanotubes and other nanoclusters of both carbon and boron nitride are controllably grown by simple surface self diffusion, both homogeneously and heterogeneously, following a mechanical ball-milling preparative stage. Brief comparisons are drawn with the nucleation and growth of nanostructures during ionizing irradiations of varying degree on graphite and molybdenite target surfaces.

**[16] SiCp/Mg COMPOSITES MADE BY LOW-ENERGY MECHANICAL PROCESSING**

Huard G. Angers R. Krishnadev MR. Tremblay R. Dube D. - Canadian Metallurgical Quarterly. 38(3):193-200, 1999

Composite powders containing 10, 20 and 30 vol% SiCp, particulates in a Mg matrix were produced using mechanical processing. A small laboratory ball mill was used to achieve a uniform distribution of the reinforcements in the Mg matrix. The particle size distribution and the microstructure of the composite powders were studied as a function of processing time. Based on these observations, four successive stages of processing were identified. It was shown that a homogeneous distribution of SiCp in a Mg matrix may be obtained under low-energy milling conditions. The influence of milling time and concentration of SiCp, on tensile strength, stiffness, elongation and impact properties of rods extruded from the resulting composite powders were investigated. The hardness of SiCp/Mg composites was also evaluated to determine the influence of SiCp, content.

**[15] A MODEL FOR YOUNG'S MODULUS OF A PARTICLE REINFORCED ULTRAFINE GRAINED COMPOSITE WITH POROSITY**

Kim HS. Estrin Y. Bush MB. - Zeitschrift fur Metallkunde. 90(11):863-867, 1999

In order to investigate the effects of grain size and porosity on the modulus of elasticity of a mechanically alloyed, particle reinforced ultrafine grained metal matrix composite, a phase mixture model was considered in which the composite is treated as a mixture of a matrix phase, a reinforcing particle phase and a porous boundary phase. The boundary phase was assumed to have the mechanical properties of a quasi-amorphous material. Hill's rule of mixtures in conjunction with the phase mixture model was employed. The calculated Young's modulus was compared with the experimental results. The effects of the volume fraction of the reinforcing particles, the overall effective density, the grain size of the matrix, and the density of the boundary phase on the overall modulus of elasticity are discussed.

**[14] MECHANICALLY ALLOYED Mg-BASED METALLIC GLASSES AND METALLIC GLASS COMPOSITES CONTAINING NANOCRYSTALLINE PARTICLES**

Eckert J. Schlorke-de Boer N. Weiss B. Schultz L. - Zeitschrift fur Metallkunde. 90(11):908-913, 1999

Mg-Y-Cu bulk metallic glass-forming alloys were synthesized by mechanical alloying. Milling produces an amorphous phase interdispersed with nanocrystalline particles for a variety of different compositions. In addition, composites based on the Mg<sub>55</sub>Y<sub>15</sub>Cu<sub>30</sub> amorphous alloy blended with 5 vol.% second phase oxide particles for additional strengthening were produced. Despite the presence of nanocrystalline particles, all samples exhibit a supercooled liquid region before crystallization. The distinct decrease in viscosity above the glass transition temperature, T<sub>g</sub>, allows to consolidate the milled powders in the viscous state into bulk specimens. The oxide additions increase the

mechanical strength of the composites compared with the Mg55Y15Cu30 glassy alloy, indicating that both the matrix and the particles contribute to the overall hardness of the material.

**[13] INVESTIGATIONS ON ORDERING AND FORMATION OF ANTIPHASE DOMAINS IN IRON ALUMINIDES WITH CHROMIUM**

Frommeyer G. Jimenez JA. Derder C. - Zeitschrift für Metallkunde. 90(11):930-937, 1999

The effect of chromium and nonequilibrium processing routes on the formation of B2 and DO3 ordering in Fe3Al(Cr) base alloys have been investigated. Three selected iron aluminides, Fe70Al30, Fe60Al30Cr10 and Fe55Al22Cr23 (compositions in at.%), were processed by conventional ingot metallurgy, mechanical milling (MA), and rapid solidification using planar flow casting (PFC) technique. The different processing routes and heat treatments influence strongly the long range order (LRO) parameters, the lattice constants, and the ordered B2 and DO3 structures in these aluminides. The ordered states prior to the A2 → B2 and B2 → DO3 phase transformations and after that were imaged by transmission electron microscopy (TEM) and quantitatively evaluated by X-ray diffraction (XRD). All aluminides possess in the as-cast state the disordered A2 and ordered B2 structures. However, the Fe70Al30 and Fe60Al30Cr10 aluminides show also the DO3 superlattice structure. After mechanically milling these alloys exhibit the disordered state in both compositions. The highest value of the LRO parameter was achieved in melt spun ribbons. The as-spun ribbons of the Fe70Al30, and Fe60Al30Cr10 aluminides possess large B2-antiphase domains (APDs) of the order of 5 to 10 microns and small DO3 domains of 2 to 5 nm in size whereas the Fe55Al22Cr23 aluminide reveals only B2 nano-size domains.

**[12] MECHANOCHEMICAL ARYLATION AND ALKYLATION OF FULLERENE C-60 UNDER THE SOLVENT-FREE CONDITIONS**

Tanaka T. Komatsu K. - Synthetic Communications. 29(24):4397-4402, 1999

The mechanochemical reaction of fullerene C-60 with organic bromides and alkali metals was found to give the corresponding aryl or alkyl C-60 derivatives under the solvent-free conditions.

**[11] PULSE MECHANOCHEMISTRY OF ORGANOELEMENT COMPOUNDS [REVIEW]**

Aleksandrov AI. Aleksandrov IA. Prokofev AI. Bubnov NN. - Russian Chemical Bulletin. 48(9):1599-1614, 1999

The results of studies of fast mechanochemical reactions initiated by elastic wave pulses (EWP) of 100 μs duration on polycrystalline mixtures under high pressures (4-25 kbar) are summarized. A possible mechanism of the EWP-initiated processes was proposed.

**[10] STRUCTURE, STRENGTH AND TOUGHNESS OF NANOCRYSTALLINE FEAL**

Morris-Munoz MA. Dodge A. Morris DG. - Nanostructured Materials. 11(7):873-885, 1999

Refining grain size to the nanocrystalline level has been suggested as a way of improving strength while enhancing ductility and toughness. In the present study, nanocrystalline bulk FeAl has been prepared by mechanical alloying and hot forging. Powders quickly reach a state of partial order during milling, and low temperature annealing is sufficient to chemically homogenise and give full order. Contamination during milling leads to the formation of carbide and oxide particles, which stabilise fine grains during heating. Bulk materials show grain sizes of 20nm to 100nm depending on the consolidation temperature. Hardness and compression strength show little change over this grain size range. Fracture toughness stays high down to moderately small grain sizes, falling only for consolidation at the lowest temperatures. There appears to be a reasonable range of fine grain sizes (40-100nm) where good interparticle bonding and high densities can be achieved leading to good strength and toughness.

**[9] DRIVING MECHANISMS ON MECHANICAL ALLOYING: EXPERIMENTAL AND MOLECULAR DYNAMICS DISCUSSIONS**

Vasconcelos IF. de Figueiredo RS. - Nanostructured Materials. 11(7):935-946, 1999

Mossbauer spectroscopy, X-rays diffraction and differential scanning calorimetry are used to study a fee Fe 45at.% Cu nanocrystalline solid solution obtained by mechanical alloying. These results supported by molecular dynamics simulations are used to discuss some models in mechanical alloying. The kinetic energy or even a function of it appears to be the parameter driving the reactions inside the mill. Impact frequency and powder mass seem to contribute only on the time scale. A capillary pressure is shown to exist in order to alloy the nanocrystalline grains. Despite this, an extra energy, provided by the shocks, is shown to be necessary to start up the process. Final product appears to be a function of strains/stress fields and material response.

**[8] EFFECTS OF REDUCED COORDINATION NUMBER FOR CA ON THE ELECTRON REDISTRIBUTION DURING CA-O-SI BRIDGE BONDING FROM CAO OR CA(OH)(2) AND SIO2**

Fujiwara Y. Isobe T. Senna M. Tanaka J. - Journal of Physical Chemistry. 103(48):9842-9846, 1999

The discrete variational X alpha method has been conducted to elucidate the formation of a heterobridging bond (HBB), Ca-O-Si, when a Si-O pendant bond of SiO2 comes close to the Ca atom on the surface of calcium oxide or hydroxide. A decrease in the coordination number for Ca in CaO or Ca(OH)2 increases the electron population density (PD) between Ca and O, and decreases the PD between Si and O. These simultaneous changes in PD favor the formation and stabilization of a Ca-O-Si HBB. The calculated results agree well with the experimental observation of precursor formation toward calcium silicate during milling of a mixture of Ca(OH)2 and SiO2.

**[7] MICROSTRUCTURAL EVOLUTION OF NI-NACL MIXTURES DURING MECHANOCHEMICAL REACTION AND MECHANICAL MILLING**

Ding J. Tsuzuki T. McCormick PG. - Journal of Materials Science. 34(21):5293-5298, 1999

The evolution of microstructure of Ni and NaCl mixtures formed by mechanochemical reaction and mechanical milling has been studied using X-ray diffraction, electron microscopy and magnetic measurements. Separate nano-sized Ni particles were formed by continuous solid-state reaction of NiCl2 + 2Na during mechanical milling. Further milling resulted in the growth of clustered particles due to inter-particle welding during collision events. On the other hand, milling of micron-sized Ni and NaCl powders resulted in a layered particle morphology and continuous decrease in particle size with increasing milling time.

**[6] IMPROVED DENSIFICATION BY NANO-SIZED SINTERING AIDS FOR SI3N4**

Wang LW. Sigmund WM. Roy S. Aldinger F. - Journal of Materials Research. 14(12):4562-4569, 1999

The densification of Si<sub>3</sub>N<sub>4</sub> with nano-sized sintering aids that were in situ incorporated by a combustion process was studied in comparison with that of sintering aids mixed by ball milling. The combustion process directly produces amorphous and nano-sized Y-Al oxides within the Si<sub>3</sub>N<sub>4</sub> powder. X-ray diffraction results indicate that amorphous Y-Al oxides begin to crystallize into Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> at about 600 degrees C. Additionally the nano-sized sintering aids are more homogeneously distributed and thereby promote the formation of eutectic melts at lower temperatures during liquid-phase sintering. Therefore, the densification process of Si<sub>3</sub>N<sub>4</sub> during liquid-phase sintering is strongly accelerated. The microstructure of as-sintered parts from combusted powder seems more dense and homogeneous.

**[5] DIFFUSED PHASE TRANSITION IN FINE-GRAINED BISMUTH VANADATE CERAMICS**

Shantha K. Varma KBR. -Journal of Materials Research. 14(12):4651-4656, 1999

Nanocrystalline powders of ferroelectric bismuth vanadate, Bi<sub>4</sub>V<sub>2</sub>O<sub>11</sub> (n-BiV), with crystallite size less than 50 nm, were obtained by mechanical milling of a stoichiometric mixture of bismuth oxide and vanadium pentoxide. The n-BiV powders on sintering yielded high-density, fine-grained ceramics with improved dielectric and polar characteristics. Dielectric studies on samples obtained from milled powders indicated that the ferroelectric-to-paraelectric phase transition temperature is strongly frequency dependent. The Curie-Weiss law is found to be valid only at a temperature away from the transition temperature, confirming the diffused nature of the transition, which is attributed to the presence of compositional inhomogeneity, because of partial reduction of vanadium.

**[4] SYNTHESIS OF LN-BA-CU-O CUPRATES FROM MECHANOCHEMICALLY ACTIVATED MIXTURES**

Panova TI. Malysheva SI. Savchenko EP. Glushkova VB. - Inorganic Materials. 35(12):1274-1277, 1999

LnBa<sub>2</sub>Cu<sub>3</sub>O<sub>(7-x)</sub> and LnBa<sub>2</sub>Cu<sub>4</sub>O<sub>(8)</sub> high-T<sub>c</sub> materials were synthesized from fine powders obtained by mechanochemical activation of the starting mixtures. The heat-treatment conditions were shown to have a crucial effect on the structural perfection and chemical composition of the resultant high-T<sub>c</sub> ceramics. According to thermogravimetric measurements, these materials are stable up to 830 degrees C.

**[3] PHYSICO-CHEMICAL AND CATALYTIC PROPERTIES OF IRON-PROMOTED RANEY-NICKEL CATALYSTS OBTAINED BY MECHANICAL ALLOYING**

Zeifert BH. Salmones J. Hernandez JA. Reynoso R. Nava N. Cabanas-Moreno JG. Aguilar-Rios G. - Catalysis Letters. 63(3-4):161-165, 1999

Raney-type catalysts were prepared by means of a two-step procedure: (i) mechanical alloying of the metals and (ii) alkaline aluminum leaching. Mechanical alloying is a novel alternative related to the synthesis of skeletal Ni catalysts. Catalysts characterization was performed by atomic absorption, X-ray diffraction, electron microscopy, and Mossbauer spectroscopies. Textural studies were also carried out. Binary Al-Ni and ternary Al-Ni-Fe alloys were produced by mechanical alloying from pure metallic powders; in particular, the intermetallic beta-(AlNi) phase was formed with a fine microstructure as a non-equilibrium phase; then, aluminum was selectively removed. After aluminum leaching the beta-(AlNi) phase was transformed into the more stable nickel fcc structure. The effect of iron addition to the Ni-Al catalysts depends on iron concentration and reduction temperature; both parameters determine catalysts composition and activity. This work reports physicochemical properties and benzene hydrogenation activity of these materials, compared with conventional catalysts obtained by melting and leaching.

**[2] MECHANO-CATALYTIC OVERALL WATER SPLITTING (II) NAFION-DEPOSITED CU<sub>2</sub>O**

Hara M. Hasei H. Yashima M. Ikeda S. Takata T. Kondo JN. Domen K. - Applied Catalysis A-General. 190(1-2):35-42, 2000

Some modifications of a mechano-catalyst, Cu<sub>2</sub>O, and optimization of the reaction conditions were attempted to improve its activity for mechano-catalytic overall water splitting. The rates of evolution of H<sub>2</sub> and O<sub>2</sub> were improved by deposition of nafion onto Cu<sub>2</sub>O and the choice of an appropriate stirring rod. The irradiation of visible light ( $\lambda > 550$  nm) was found to maintain the activity in the time course, which gradually decreased without irradiation.

**[1] SIMULTANEOUS IR AND TIME RESOLVED X - RAY DIFFRACTION MEASUREMENTS FOR STUDYING SELF SUSTAINED REACTIONS**

F. Bernard, E. Gaffet, M. Gramond, M. Gailhanou, JC Gachon - J. Synchrotron Radiation, 7 (2000) 27 - 33

Self propagating high temperature synthesis provides an attractive practical method for producing advanced materials such as ceramics, composites and intermetallics. This kind of reaction has been investigated in situ using time resolved x - ray diffraction, with an x - ray synchrotron beam (D43 beamline, LURE, Orsay) coupled to simultaneous IR thermography to study structural transformations and thermal evolution. With short acquisition times (30 ms per pattern), it has been possible to observe several steps before obtaining compounds. Two different compound formation have been described : (i) the different steps of reaction, aluminium melting, subsequent temperature increase and fast reaction between Al and Ni at such temperatures that only Ni and AlNi are solid and all other compositions are liquid and well identified ; ii) the formation of FeAl. Here a portion of the iron seems to transform into its allotropic phase and this transition stabilizes the reaction temperature at 1173K. In addition, the aluminium melting during the reaction explains why the nanostructure induced by the mechanical activation is maintained in the end product.

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