

# RESEAU FRANCAIS DE MECANOSYNTHESE

## Lettre N°41

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Août 1998

**124** Groupes de Recherches  
(dont 61 (+2) à l'étranger)

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

### 3 Nouvelles Adhésions

**R.E. Riman** - Dpt Ceramic and Materials Engineering - Rutgers University - Piscataway - USA  
**C. Roques - Carmes** - LMS/ENSMM - Besançon - France  
**L. Wei** - Institute of Metal Research - Shenyang Chine

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**ANNONCE DE CONGRES ET / OU ECOLES**  
**CONGRESS AND SCHOOL ANNOUNCEMENTS**  
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All the details may be obtained by E-Mail to E. Gaffet

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**ECM - 18**

**18th European Crystallographic Meeting**  
Praha - République Tchèque - 15 - 20 Août 1998  
**Symposium B3 ; Nanocrystalline Materials**  
Chairman : H.E. Schäffer, K.H. Ehses  
Conf. Secretary : Kuzel@karlov.mff.cuni.cz

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**Intelligent Processing of Nanostructured Ceramics**

Materials Science Summer Institute - New Brunswick - 20 - 29 Août 1998  
Contact : L.C. Klein Rutgers University - E-Mail ; Licklein@RCI.Rutgers.Edu

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**"JMC6" & "CMD17"**

**6èmes Journées de la Matière Condensée et**  
**17th General Conference of the Condensed Matter Division of the European Physical Society**  
**Grenoble** - France - 25 - 29 Août 1998  
Org : Société Française de Physique et European Physical Society  
Website : <http://www.polycnrs-gre.fr/eps.html>

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**Fatigue Damage of Structural Materials II**

Engineering Foundation Conference  
Cape Cod - Massachusetts - USA 31 Août - 4 Septembre 1998  
Org. A.K. Vasudevan, J.C. Cammett, T. Nicholas, K. Jata  
E-Mail : engfnd@aol.com

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**ESTAC 7 et EUROSOLID - 5**

**Baltonfüred** - 30 Août - 4 Septembre 1998  
Contact : Prof. G. Liptay - Hungarian Chemical Society - Fő u. 68, Budapest - H - 1027 Hongrie

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**5th International Conference on Nanometer scale Science and Technology (NANO 5)**

Birmingham - UK - 31 Aout - 4 Septembre 1998  
Site : <http://www.iop.org/IOP/Confs/IVC>

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**Ninth International Symposium on Small Particles and Inorganic Clusters (SSPIC 9)**

Lausanne - Suisse - 1 - 5 Septembre 1998

Website : <http://ipent.epfl.ch/isspic9>

**Magnetism of Nanostructured Phases - MNP Conference  
EMMA Satellite Meeting**

San Sebastian (Espagne) - 4 / 6 Septembre 1998

E-Mail : [wupdocal@sp.ehu.es](mailto:wupdocal@sp.ehu.es)

**9th European Symposium on Comminution and Classification**

Albi (France) - 8 - 10 Septembre 1998

sous l'égide de l'European Federation of Chemical Engineering

Contacts : J. Dodds - Chairman of the Organizing Committee

WebSite - <http://www.enstimac.fr/manif/comminution98>

**First Joint ESF - NSF Symposium on Aerosols for Nanostructured Materials and Device**

Edinburgh - Ecosse - 12 Septembre 1998

Contact : [h.fissan@uni-duisburg.de](mailto:h.fissan@uni-duisburg.de) ou [dyhpui@tc.umn.edu](mailto:dyhpui@tc.umn.edu)

**First International Conference on Inorganic Materials**

Synthesis, Characterisation, Properties and Applications of Inorganic Materials

Versailles - 16 / 19 Septembre 1998 - France

Website : <http://www.elsevier.nl/locate/materials98>

**ISAPM98**

**3<sup>rd</sup> International Symposium on Advanced Powder Materials (ISAPM98)**

23 - 26 September 1998 - KAIST - Tazejong - Corée du Sud

Correspondence : Prof. Suk-Joong L. Kang or Ms. Sung Sook Park - Center for Interface Science and Engineering of Materials (CISEM) - Korea Advanced Institute of Science and Technology (KAIST) - Yusong-gu, Kusong-dong, Taejon, 305-701 Korea - Tel:82-(0)42-869-4113, 8919 / Fax: 82-(0)42-869-8920

E-mail : [sjkang@sorak.kaist.ac.kr](mailto:sjkang@sorak.kaist.ac.kr) / [e\\_cisem@cais.kaist.ac.kr](mailto:e_cisem@cais.kaist.ac.kr)

**Solid State Chemistry : Novel Syntheses and New Materials**

Bordeaux - France - 24/26 Septembre 1998

Website : <http://chemistry.rsc.org/rsc/conf.htm>

**Workshop on**

**Nanoscale Structure and Kinetics at Solid Interface**

Halle - Allemagne - 28 - 30 Septembre 1998

Website : <http://www.ep3.uni-halle.de/workshop/nanosk.html>

**The Reh binder Memorial International Conference  
on Colloid Chemistry and Physical Chemical Mechanics**

Moscou (Russie) - 4 - 8 Octobre 1998

Contact : Prof. N.B. Uriev - Institute of Physical Chemistry - Leninsky Prospect 31 - 11795 Moscow - Russie

E-Mail : [Rehbinder98rehb.chem.msu.su](mailto:Rehbinder98rehb.chem.msu.su) ou <http://www.chem.msu.su>

**Powder Metallurgy 98**

Granada - Espagne - 18 - 22 Octobre 1998

Site Web : <http://www.epma.com/congress/>

**Gorham / Intertech Conference on "Commercializing Nanotechnology 98 : Bridging New Materials to Market"**

19 - 21 Octobre 1998 Nashville - TN USA

Contact : C.E. Spear - Intertech Corporation : Fax 207 - 781 - 2150

**JA 98**

Paris - 27 - 29 Octobre 1998

Symposium 1 : Phénomènes déterministes et aléatoires en science des matériaux

Symposium 2 : Matériaux poreux et mousses : élaboration, structures et propriétés

Symposium 3 : Les hydrures métalliques

Symposium 4 : Propriétés Thermophysiques et thermomécaniques des matériaux de la mesure à la simulation de procédés industriels

Symposium 5 : Analyse d'images et reconnaissance de formes en matériaux

Symposium 6 : Lois de comportement et calcul de structures

Org. SF2M - Contact : [SFMM@wanadoo.fr](mailto:SFMM@wanadoo.fr)

**Fifth International Symposium on Quantum Confinement : Nanostructures**

194th Meeting of the Electrochemical Society

1 - 6 Novembre 1998 - Boston - MA - USA

<http://www.electrochem.org>

**Nouveau**

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**Fine, Ultrafine and Nano Powders '98**  
Crowne Plaza Hotel - LaGuardia Airport - 8 - 10 Novembre 1998  
Contact : E-Mail : Tombcc@aol.com

**Nouveau**

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**Sixth Foresight Conference  
on Molecular Nanotechnology**  
Westin Hotel - Santa Clara - 12 15 Novembre 1998  
California Satellite Conference to Nano'98  
Contact : E-Mail : globus@nas.nasa.gov

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**Symposium on Advanced Technologies for Particle Production**

AIChE Annual Meeting  
15 - 20 November - Miami Beach - FL - USA  
Technical Sessions and ChairPersons  
1/ Particle Synthesis in Dispersions and Supercritical Fluids-R. Davis/MT Harris/D. Tomasko  
2/ Sol - Gel Synthesis of Particles - A McCormick/PN Kumta/T. Okubo  
3/ Chemical Kinetics during Particle Formation - J. Floess, K. Higashitani, S. E. Pratsinis  
4/ In-Situ Diagnostics during Particle Formation-Ph. W. Morrison,R.M. Carangelo, D.T. Spicer  
5/ Agglomerate Particle Dynamics - G. Fotou, SK Friedlander, Takahashi  
6/ Computational Fluid Dynamics during Particle Formation and Growth - L. Collins, K. Kontomaris  
7/ Aerosol Reactors - A.W. Weimer, M. Kamal Akhtar  
8/ Particle Charging - T. Matsoukas  
9/ Film synthesis by Particle Technologies - G. Grader, S. Bhandarkar  
**10/ Nanoparticles - M. Senna, T.J Mountziaris, H. Glicksmn**  
11/ Particulate deposits : Transport mechanisms, microstructure and properties : D. Rosner  
12/ Posters on Advanced Technologies for Particles Production : G. Beaucage, H. Riemenschneider  
Web Site : www.aiche.org

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**ISMANAM98**

International Symposium on Metastable, Mechanically Alloyed and Nanocrystalline Materials  
Wollongong (Sydney) - Australie - 7 - 12 Décembre 1998

**International Advisory Committee :**

V.V. Boldyrev, R.W. Cahn, S. Enzo,H. Fecht, E. Gaffet, A. Garcia - Escorial, A.L. Greer, E.Y. Gutmanas,  
K. Lu, M. Mammoun, M.T. Mora, H. Mori, M.A. Morris, L. Schultz, M. Senna, A. Slawska - Waniewska,  
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R. Bormann, G. Cocco, A. Hernando, C.C Koch, M. Magini, R. Schulz, A.R. Yavari

**Contact :** A. Calka E-Mail : Andrzej\_Calka@uow.edu.au et

**WebSite :** http://www.uow.edu.au/conferences/ismanam98

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**Satellite Symposium on Mechanochemistry / ISMANAM98**

(Mechanochemical Synthesis and Mechanochemistry)

Wollongong - Australie 7 /12 Decembre 1998

**International Advisory Committee :**

E. Ivanov (Chairman), A. Calka, V. Bodyrev, P. Butyagin,  
E. Gaffet, E. Gutman, M. Senna, C. Suryanaryana, R. Schwarz

**WebSite :** http://www.uow.edu.au/conferences/ismanam98

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**Nanostructured Hybrid Materials**

Symposium TMS Annual Meeting - San Diego CA - USA - 28 Février 4 Mars 1999

Contact : gmchow@anvil.nrl.navy.mil

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**4th International Workshop on Metastable Phases (IV IWOMP)**

7 - 9 Avril 1999 - Bologne - Italie

Contact : Bonetti@df.unibo.it

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**12th International Conference on Wear of Materials**

Atlanta - Georgie / USA - 25 - 29 Avril 1999

contact : Amy Richardson E-Mail A.Richardson@elsevier.co.uk  
or web site : http://www.elsevier.nl/locate/wom99

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**Nanostructured Materials Symposium at the 5th IUMRS International Conference  
on Advanced Materials**

(IUMRS - ICAM'99)

Beijing - Chine - 13 - 18 Juin 1999

Contact : Kelu@imr.ac.cn

WebSite - http://www.chimeb.edu.cn

**10th International Conference on Rapidly Quenched and Metastable Materials (RQ10)**

Bangalore - Inde - 23 - 27 Août 1999

Website : <http://www.metalrg.iisc.ernet.in/rqten/>

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**Annonces de Soutenance de Thèses**  
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**Transformations antiferromag - ferromag - paramagnétiques - verre de spin dans les alliages de Fe Rh nanocristallisés par Broyage**

**E. Navarro** - Université de Complutense - Madrid - Espagne - 18 Mai 1998

Co directeurs : A. Hernando - A.R. Yavari  
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**Modifications morphologiques et microstructurales du matériau actif des cathodes de batteries à l'ion lithium induites par broyage et traitement thermique**

**Ph. Perrot** - Université de Poitiers - 6 Mai 1998

Co - Directeurs : E.L. Mathe, M. Grosbras

**Jury** : J. Mimault, H. Van Damme, A. Dauger, M. Broussely, P. Goudeau, E.L. Mathe, M. Grosbras  
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**Effects of the mechanical milling on carbons : negative electrode materials of Li - ion batteries"**

**F. Salver Disma** - Université de Picardie Jules Verne - 4 Février 98

**Jury** : Aymard L., Beguin F., Coulon M., Furdin G., Lassegues JC, Percheron Guegan A., Rouzard JN, Tarascon JM.  
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**"Elaboration et Caractérisations de Cermets Alumine - Métal à partir de poudres obtenues par Mécanosynthèse"**

**J.-L. Guichard** - INPL - Nancy - 23 Janvier 1998

**Jury** : A. Simon, C. Carry, F. Thévenot, G. Le Caër, A. Mocellin  
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**"Spinelles nanométriques à valence mixte et à fort taux de lacunes cationiques : Transfert électronique dans un ferrite de molybdène Fe<sub>2.47</sub>Mo<sub>0.53</sub>O<sub>4</sub>, de la synthèse aux propriétés magnétiques dans le système fer - vanadium Fe<sub>3-x</sub>V<sub>x</sub>O<sub>4</sub> (0<sup>2</sup>x<2).**

**V. Nivoix** - Université de Bourgogne - 17 Décembre 1997

**Jury** : M. Lenglet, H. Pascard, G. Bertrand, E. Gaffet, M. Guyot, M. Lallemand, A. Rousset, B. Gillot  
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**"The Preparation of Nitrides and Carbides by Mechanical Treatment - Phases and Structures"**

G.M. Wang - School of Physics, University College, The University of New South Wales - Australian Defence Force Academy - Canberra, ACT 2600 - Australia - 10/12/97

Supervisor - S.J. Campbell - Co - Supervisors: W.A. Kaczmarek and A. Calka  
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**"Suivi par Diffraction X en Temps Réel de la Formation par Combustion des intermétalliques des systèmes Al - Ni, Al - Ti, Al - Ni - Ti"**

**J. F. Javel** - Université de Nancy I - 3 Octobre 1997

**Jury** : J.F. Berar, F. Bernard, M. Bessiere, M. Dirand, J.C. Gachon, P. Galez, J.C. Jorda  
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**"Contribution à l'Etude de la Transformation - Tribologique Superficielle en Fretting"**

**E. Sauger** - Ecole Centrale de Lyon - Génie des Matériaux - 26 Septembre 1997

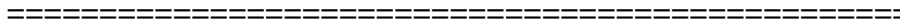
**Jury** : L. Mora - Ponsonnet, P. Blanchard, K. Dang Van, C. Esnouf, E. Gaffet, E. Rosset, A.B. Vannes, L. Vincent

## Sites internet à découvrir

### Site sur la cristallographie / Soft + Littérature

<http://www.lmcp.jussieu/sincris-top/logiciel>

N.B. : si vous connaissez d'autres sites en relation avec les thèmes développés par le RFM, faites nous les connaître



### Post Doc Position Proposals

#### Belgique

The Department Metallurgy and Materials Engineering (MTM) of the K.U.Leuven (Belgium) has a research position available. Candidates are asked to contact the responsible staff member.

Area of research :

Metals and Alloys, Polymer Matrix Composites, Intelligent Processing of Materials, Surface Engineering and Tribology, Metal Forming and Mechanical Behaviour of Materials, Quality Control and Non-Destructive Testing of Materials, Ceramics, Thermodynamics, Corrosion, Nuclear Engineering

Description of research task

Tailor made powders by mechanical alloying of Fe and Cu based materials. Application field: specific composite materials, to be prepared by conventional PM consolidation techniques. Research activities: parametric study of MA, alloy design, microscopic

Staff member to be contacted

Prof. Dr. Ir. L. Froyen

Katholieke Universiteit Leuven - Dept. MTM

de Croylaan 2 - B-3001 Leuven (Belgium)

Tel. +32/16/22.09.31

#### Japon

Our group: Nanocomposite Group, Department of Composite Materials, National Institute of Materials and Chemical Research, Tsukuba, Ibaraki, Japan is now looking for post-doc researchers

The candidates would be integrated in the Nanocomposite Group of the Department of Composite Materials. The research interests of the group are mainly focused on nanocomposite preparation and its optical/chemical functionalities. Research projects currently under way aim to develop nanostructured and optically/chemically active thin films by sputtering, laser ablation and so on. For additional information about the Institute and group :

<http://www.nimc.go.jp/>

<http://www.aist.go.jp/NIMC/fcg/index.html>

Experience in the fields of materials science (ceramic or metal) is required.

There are two types of post-doc positions.

1. Long-term: from 6 months to 2 years

2. Short-term: from 1 to 3 months

If you or someone in your laboratory is interested in this fellowship, please contact as soon as possible to:

Dr. Naoto Koshizaki

Department of Composite Materials

National Institute of Materials and Chemical Research(NIMC) 1-1 Higashi, Tsukuba, Ibaraki 305-8565 JAPAN

Tel: +81-298-54-6335

Fax: +81-298-54-6252

E-mail: [koshizaki@nimc.go.jp](mailto:koshizaki@nimc.go.jp)

<http://www.aist.go.jp/NIMC/fcg/index.html>

## Bibliographie Récente

**N.B. : En cas de difficultés à vous procurer une copie des articles suivants, n'hésitez pas à contacter E. Gaffet (CNRS / IPSé - Belfort)**

### Livres ou "Special Issues"

**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 assurée grâce au concours de M<sup>me</sup> TAUZIN - FIN BiPSé)

#### [75] THERMOELECTRIC PROPERTIES OF P-TYPE BI-SB-TE BASED MATERIAL PREPARED BY PIES METHOD WITH CONVENTIONAL BALL MILLING PROCESS

S Kagawa, H Satake, M Sakamoto, A Yamamoto, T Ohta - PROCEEDINGS ICT'97 - XVI INTERNATIONAL CONFERENCE ON THERMOELECTRICS, 1997, pp 85-88

For the development of energy saving processes for producing thermoelectric materials, the PIES (Pulverized and Intermixed Elements Sintering) method is known as a suitable one. In this study, a high figure of merit p-type Bi-Sb-Te based thermoelectric material was obtained by the oxygen free PIES method. The electrical resistivity of p-type  $(\text{Bi}_2\text{Te}_3)_0.175(\text{Sb}_2\text{Te}_3)_0.825$  material was reduced to  $0.8 \times 10^{-5}$  ohm.m and Seebeck coefficient was obtained  $2.4 \times 10^{-4}$  V/K by fabrication in the oxygen free atmosphere process. The maximum ZT value was almost 1.0. Moreover, it was demonstrated that the improvement in figure of merit value was obtained even if the intermixed powder of elements was prepared by a conventional ball milling process in stead of the high energy ball milling process. The PIES methods using a low energy ball milling process is suitable for mass-production because of its cost efficiency.

#### [74] EFFECTS OF A REDUCTION TREATMENT AND TE DOPING ON THERMOELECTRIC PROPERTIES OF $(\text{Bi}_{1-x}\text{Sb}_x)_2\text{Te}_3$ FABRICATED BY MECHANICAL ALLOYING

HC Kim, JS Choi, HJ Kim, DB Hyun, TS Oh - PROCEEDINGS ICT'97 - XVI INTERNATIONAL CONFERENCE ON THERMOELECTRICS, 1997, pp 127-130

$(\text{Bi}_{1-x}\text{Sb}_x)_2\text{Te}_3$  (0.75 less than or equal to x less than or equal to 0.85) thermoelectric materials were fabricated by mechanical alloying and hot pressing at 550 degrees C for 30 minutes with and without a reduction treatment of the mechanically alloyed powders. When the mechanically alloyed  $(\text{Bi}_{1-x}\text{Sb}_x)_2\text{Te}_3$  powders were hot-pressed without a reduction treatment, a maximum figure-of-merit of  $2.92 \times 10^{-3}/\text{K}$  at 300K was obtained for x = 0.8 composition. Although the electrical resistivity of the mechanically alloyed  $(\text{Bi}_{1-x}\text{Sb}_x)_2\text{Te}_3$  decreased with a reduction treatment of the powders, the figure-of-merit was lowered due to the substantial decrease of the Seebeck coefficient. With addition of 1 wt% excess Te, the figure-of-merit of the reduction-treated  $(\text{Bi}_{0.2}\text{Sb}_{0.8})_2\text{Te}_3$  could be improved to be  $3.33 \times 10^{-3}/\text{K}$ .

#### [73] INFLUENCE OF THE CONSOLIDATION TECHNIQUE ON THE THERMOELECTRIC PROPERTIES OF MECHANICALLY ALLOYED BI-SB

R MartinLopez, A Dauscher, X Devaux, B Lenoir, H Scherrer, M Zandona - PROCEEDINGS ICT'97 - XVI INTERNATIONAL CONFERENCE ON THERMOELECTRICS, 1997, pp 184-187

The aim of this work is multiple : first the synthesis by mechanical alloying of homogeneous  $\text{Bi}_{85}\text{Sb}_{15}$  polycrystalline powder alloys, that have been achieved with an adequate ball to powder weight ratio (10:1), second the consolidation of the powders either by sintering or by hot extrusion in order to try to introduce a texture in the material and finally to measure the thermoelectric properties of the consolidated samples in the 77-300 K temperature range.

#### [72] EFFECT OF POWDER TREATMENT BY MECHANOFUSION PROCESS ON THERMOELECTRIC PROPERTIES OF $\text{FeSi}_2$ SYSTEM

T Kita, K Nogi, H Nagai, M Kohno - PROCEEDINGS ICT'97 - XVI INTERNATIONAL CONFERENCE ON THERMOELECTRICS, 1997, pp 311-314

The thermoelectric properties of hot-pressed  $\text{Fe}_{0.91}\text{Mn}_{0.09}\text{Si}_2$  (mean diameter:  $D_m = 5-10 \mu\text{m}$ ) were measured with changing the powder treatment. In order to improve the thermal conductivity and the electrical conductivity, carbon (0, 1 and 2wt%,  $D_m = 0.02-0.3 \mu\text{m}$ ) was added and the mechanofusion process was used. The mechanofusion process can produce composite powders with high shear stress and compressive force, allowing a more homogenous compact to be fabricated. As a result, the thermal conductivity was remarkably improved in this system. EPMA analyses showed that extremely fine particles of carbon were dispersed along grain boundaries without aggregation. However, the electrical conductivity decreased in this case. This is probably because the electrical resistivity of carbon black is very high. However, when graphite are added, the MF process would be very effective to improve the figure of merit? because the process can increase the grain boundary with additives and improves the electrical conductivity.

#### [71] EFFECT OF DISPERSED SI-PHASE ON THERMOELECTRIC PROPERTIES OF $\text{FeSi}_2$ PREPARED BY MECHANICAL ALLOYING AND SINTERING

BG Min, DH Lee - PROCEEDINGS ICT'97 - XVI INTERNATIONAL CONFERENCE ON THERMOELECTRICS, 1997, pp 315-320

While melt-cast  $\text{FeSi}_2$  requires careful heat-treatment of homogenization and phase transformation to achieve thermoelectric beta-phase, the mechanical alloying(M/A) and sintering method is expected to simplify the process. By using the M/A method with elementary Fe and Si coarse powders, very fine powders consisting of FeSi- and Si-phases were obtained. Especially when excess Si was deliberately introduced in the starting materials, the desired

microstructure containing finely distributed Si-phase in the matrix of beta-FeSi<sub>2</sub> was produced by subsequent sintering. The volume fraction of dispersed Si-phase could be controlled within a range of 0.26 similar to 0.38 by varying the excess Si content. The shape of the dispersed Si phase was something like the coral colony of fine finger-shaped protrusions (diameter less than or equal to 0.5 μm and length less than or equal to 3 μm). The size and volume fraction of the Si-phase in the beta-FeSi<sub>2</sub> matrix was analysed being effective in controlling the thermal conductivity of the sintered mass due to the phonon scattering.

**[70] CHARACTERIZATION OF NANOSTRUCTURED ALPHA-Fe<sub>2</sub>O<sub>3</sub>-SnO<sub>2</sub> SOLID SOLUTIONS PREPARED BY HIGH ENERGY BALL MILLING**

JZ Jiang, R Lin, S Morup - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 449-454

Solid solutions of SnO<sub>2</sub> in alpha-Fe<sub>2</sub>O<sub>3</sub> with SnO<sub>2</sub> contents up to 20 +/- 4 mol% have been prepared by mechanical alloying of alpha-Fe<sub>2</sub>O<sub>3</sub> and SnO<sub>2</sub> powder blends. X-ray diffraction and Mossbauer spectroscopy investigations show that the mechanical milling results in alloying on an atomic scale and that true solid solution formation occurs. We suggest that the high defect concentration and the chemical enthalpy of Fe<sup>3+</sup>-O<sup>2-</sup>-Sn<sup>4+</sup> interfaces between nanostructured alpha-Fe<sub>2</sub>O<sub>3</sub> and SnO<sub>2</sub> regions may serve as a driving force for the formation of a solid solution in the immiscible ceramic system.

**[69] STRUCTURAL AND MAGNETIC PHASES IN BALL MILLED STRONTIUM FERRITE**

WA Kaczmarek, SJ Campbell, E Wu, B Idzikowski, KH Muller - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 455-460

Prolonged milling of SrFe<sub>12</sub>O<sub>19</sub> in air and vacuum results in mixtures of nanostructured iron oxides. The hexagonal ferrite structure is altered to form mixtures of nanoparticles of alpha-Fe<sub>2</sub>O<sub>3</sub> and vacancy defected Fe<sub>3-x</sub>O<sub>4</sub>. Mechanical activation in air for 800 h results in the formation of similar to 10 nm alpha-Fe<sub>2</sub>O<sub>3</sub> particles with both similar to 10 nm alpha-Fe<sub>2</sub>O<sub>3</sub> (similar to 56%) and similar to 3 nm Fe<sub>3-x</sub>O<sub>4</sub> particles (similar to 44%) being obtained in the vacuum milled state. The vacancy defected magnetite particles are likely to form as a result of mechanochemical reduction of alpha-Fe<sub>2</sub>O<sub>3</sub> in the oxygen free environment. The range of nanoparticle sizes obtained on milling is reflected by the relaxation effects observed at room temperature and 4.2 K in the Mossbauer spectra of the milled products. A spin glass-like irreversibility in magnetisation is observed below similar to 150 K.

**[68] STRUCTURAL PROPERTIES AND THERMAL STABILITY OF GLASSY (Zr<sub>0.65</sub>Al<sub>0.075</sub>Cu<sub>0.175</sub>Ni<sub>1</sub>)(100-X)Fe-X (X <= 20) ALLOYS PREPARED BY MECHANICAL ALLOYING**

R Lin, M Seidel, JZ Jiang, J Eckert - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 461-466

Amorphous (Zr<sub>0.65</sub>Al<sub>0.075</sub>Cu<sub>0.175</sub>Ni<sub>1</sub>)(100-x)Fe-x (x less than or equal to 20) alloys have been synthesized by mechanical alloying of elemental powders. They exhibit significant supercooled liquid regions except the sample with an iron content of 20 at.%. No iron-rich cluster was detected in amorphous alloys. Alloying processes are found to be similar in three samples (x=10, 15, and 20). It is suggested that a chemical segregation starting at much lower temperature than the onset crystallization temperature could cause the observation of no supercooled liquid region in the amorphous (Zr<sub>0.65</sub>Al<sub>0.075</sub>Cu<sub>0.175</sub>Ni<sub>1</sub>)(80)Fe-20 alloy.

**[67] PHASE STABILITIES AND MAGNETIC PROPERTIES OF FE-CU SOLID SOLUTION PREPARED BY MECHANICAL ALLOYING**

K Tokumitsu - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 467-472

Fe<sub>100-x</sub>Cu<sub>x</sub> solid solutions with X=5, 10, 15, 20 have been obtained by mechanical alloying of elemental Fe and Cu powders. The structure and the atomic configuration of these solutions have been studied by X-ray diffraction and Fe-57 Mossbauer spectroscopy. X-ray diffractions showed that bcc Fe(Cu) solutions were formed after 15, 20 and 30 hours milling respectively. Mossbauer spectra were composed of 3 or 4 magnetically splitted sextets. Each hyperfine fields were (A) 327 similar to 329 kG, (B) 316 similar to 318 kG, (C) 302 similar to 304 kG, (D) 284 kG. These components correspond to (A) Fe atoms surrounded with 8 Fe, (B) Fe with 1 Cu + 7 Fe, (C) Fe with 2 Cu + 6 Fe, (D) Fe with 3 Cu + 5 Fe in bcc lattice. Mossbauer spectra for Fe-Cu solutions could be considered by the distribution change of these components with Cu concentrations.

**[66] STRUCTURE OF THERMAL TREATED MECHANICALLY ALLOYED Fe<sub>50</sub>Cu<sub>50</sub> STUDIED BY ANOMALOUS DIFFRACTION AND EXAFS SPECTROSCOPY**

P Gorria, P Crespo, JM Barandiaran, A Hernando, JS Garitaonandia, JL Hodeau, E Dooryhee - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 479-484

In this article, we report High Resolution Powder Diffraction (HRPD), EXAFS and Mossbauer results obtained from mechanically alloyed samples of nominal composition Fe<sub>50</sub>Cu<sub>50</sub> after heating at 623 and 723K for one hour respectively. The initial structure of the as prepared samples is an FCC solid solution, but after subsequent annealing a decomposition takes place and a new BCC Fe-rich phase appears. The former FCC solid solution is ferromagnetic with a Curie temperature (T<sub>C</sub>) around 500K and a room temperature saturation magnetization (M<sub>s</sub>) of ca. 80 Am<sup>2</sup>/kg(-1). However, a sample annealed at 950K shows the same value for the M<sub>s</sub> but T<sub>C</sub> lies above 1000K. For intermediate annealing temperatures the value of M<sub>s</sub> is below this value. The aim of this work is to analyze the structure and composition of the two coexisting phases, FCC and BCC. The results obtained from structural measurements give additional information to the overall understanding of the striking magnetic behaviour of this compound along the decomposition.

**[65] THE PHASE FORMATION AT MECHANICALLY ALLOYED TI-AL COMPOUNDS**

S Lauer, Z Guan, H Wolf, T Wichert - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp

485-490 The local electric field gradients were used for the investigation of ordered atomic structures in Ti-Al compounds using the radioactive probe In-111. The observed electric field gradients were identified by the comparison with the electric field gradients of the intermetallic compounds  $\gamma$ -TiAl and  $\tau$ -TiAl<sub>3</sub>, prepared by conventional melting techniques, or by comparison with X-ray diffraction data. Mechanical alloying of elemental Ti and Al powders was used for the production of Ti<sub>0.50</sub>Al<sub>0.50</sub> and Ti<sub>0.25</sub>Al<sub>0.75</sub> powders. Annealing these samples at 1060 K led to the formation of  $\gamma$ -TiAl and  $\tau$ -TiAl<sub>3</sub>, respectively. Additionally, the phase Ti<sub>2</sub>AlN was detected in both cases indicating the incorporation of N impurity atoms. A metastable face centred tetragonal structure was observed in a Ti<sub>(0.50)</sub>Al<sub>(0.50)</sub> Sample milled for 4 h and tempered at 880 K. In the case of the Ti<sub>0.25</sub>Al<sub>0.75</sub>, a metastable compound with a lattice structure of the D0(23) type was formed in the temperature range between 690 K and 1020 K.

**[64] NEW PREPARATION METHOD OF CU/ZNO CATALYSTS FOR METHANOL SYNTHESIS FROM CARBON DIOXIDE HYDROGENATION BY MECHANICAL ALLOYING**

H Fukui, M Kobayashi, T Yamaguchi, H Kusama, K Sayama, K Okabe, H Arakawa

ADVANCES IN CHEMICAL CONVERSIONS FOR MITIGATING CARBON DIOXIDE (Series: STUDIES IN SURFACE SCIENCE AND CATALYSIS), 1998, Vol 114, pp 529-532

Hydrogenation of carbon dioxide to methanol was investigated over Cu/ZnO catalysts prepared by mechanical alloying (MA) method, which is suitable for excellent mixing of different materials to make alloys or composites. The catalytic activity increases with mechanical milling time, and methanol yield over the catalyst milled for 120 hour is about 1.5 times higher than that of conventional coprecipitated Cu/ZnO catalyst. The reason for increasing catalytic activity by MA method can be attributed to the preparation of well mixed structure of Cu and ZnO nanocrystals.

**[63] NANOSTRUCTURED MATERIALS - FROM FUNDAMENTALS TO APPLICATIONS**

AL Greer - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 3-9

Nanostructured materials are known to possess properties, or combinations of properties, which can be novel or even outstanding. Continued interest in these materials, however, will depend not only on good properties, but also in finding applications in which those properties can be exploited. A few topics are selected from this broad field, focusing on the length scale of the microstructure in the materials - how it affects properties, how it might have optimum values, and how it is affected by processing. Some key developments for applications are briefly reviewed.

**[62] WHAT HAVE WE LEARNED ABOUT NANOSCALE MATERIALS? THE PAST AND THE FUTURE**

DG Morris - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 11-13

**[61] CURRENT UNDERSTANDING OF MECHANICALLY ALLOYED POLYMERS**

WJD Shaw - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 19-29

The history covering the development of mechanically alloyed polymeric materials as evolved at the University of Calgary are covered in this paper. The work is based upon investigations of single polymer materials, polymer/polymer materials, polymer/ceramic materials and polymer/metal materials. The processing of these various materials was conducted using standard mechanical alloying techniques without the use of any control agent or compatibilizer. The equipment, methods and analysis techniques and their interaction with some specific mechanical properties are described. The process results in extremely small domains along with internally stored energy and very reactive surfaces being created. The end material results in both surface bonding and chain movement as controlled by time and temperature. An understanding of mechanisms and property relationships are elucidated. The possibilities based upon other experiments are also presented.

**[60] MECHANICAL ALLOYING OF FULLERENE WITH VARIOUS METAL ELEMENTS**

M Umemoto, K Masuyama, K Tsuchiya - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 31-36

The effect of mechanical milling on C-60(C-70) (a mixture of 87 vol.% C-60 and 13 vol.% C-70) and mechanical alloying on metal-0.3 mol% C-60(C-70) (M-C-60(C-70)), where M=Al,Cu,Fe,Ni,Sn or Zn was studied. Liquid chromatography and infrared absorption measurements done on the mechanically milled C-60(C-70) showed that when milled the C-60(C-70) was stable for 3600 ks. However, milling results in a reduced fraction of C-60 and for the same time, showed the formation of probably a dimer of C-60 and C-60 polymer. After milling C-60(C-70) with a metal for 720ks, a small fraction of the initial C-60(C-70) retained its identity in Al- and Zn-C-60(C-70) systems, whereas the molecular structure of C-60(C-70) is lost when the metal was Cu, Fe, Ni or Sn. The sintered compacts made from the MA powder of a M-C-60(C-70) and a M-graphite showed similar hardness in the cases of M=Cu, Fe and Ni. However, the sintered compacts made from an Al-C-60(C-70) MA powder showed about a 40% increase in hardness compared with that made from an Al-graphite MA powder. While in the case of Zn, the hardness of the sintered compact made from the Zn-C-60(C-70) MA powder was less than half of that made from a Zn-graphite MA powder. The stability of C-60 in a metal after MA was discussed according to which element group the metal belonged to on the periodic table.

**[59] FORMATION OF CERAMIC/METALLIC GLASS COMPOSITE BY MECHANICAL ALLOYING**

C Moelle, IR Lu, A Sagel, RK Wunderlich, JH Perepezko, HJ Fecht - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 47-52

Recently discovered Zr-based bulk glass alloys can be produced by rapid quenching from the melt or as an alternative route by mechanical alloying. Here we present the formation of amorphous matrix-ceramic composite powders by mechanical alloying of an elemental powder mixture with the composition Zr<sub>65</sub>Al<sub>7.5</sub>Ni<sub>10</sub>Cu<sub>17.5</sub> together with SiC particles. The effect of the added ceramic particles on glass formation and thermal stability was investigated with

structural and thermal analysis. During the milling process an amorphous matrix with a homogeneous dispersion of SiC particles develops. This composite material reveals a glass transition followed by crystallization. The onset temperatures for these transformations are found to be increased by about 10 K in comparison to the pure Zr-based alloy. This observation may be explained by small compositional changes in the amorphous matrix during the milling process. No indication for SiC dispersoids acting as heterogeneous nucleation sites was found. The kinetics of primary crystallization was found to be delayed. As such, mechanical alloying represents a useful method for preparation of amorphous-ceramic powders combining the improved mechanical properties of the amorphous state and particle dispersions.

**[58] RECENT PROGRESS IN THE CLEAN MECHANICAL ALLOYING OF ADVANCED MATERIALS**

PS Goodwin, TMT Hinder, A Wisbey, CM WardClose - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 53-62

There is an increasing interest in the use of mechanical alloying (MA) as a means of synthesising advanced materials. Many of these are difficult to process by conventional means and the fine, homogeneous microstructures produced by MA offer great potential in terms of enhanced processability and significantly improved mechanical properties. However, many of these materials, such as those based on titanium or niobium, are very sensitive to contamination and can only be successfully synthesised by MA if careful attention is given to contamination control during processing. Considerable progress has been achieved in this area, making MA a viable route for the synthesis of a number of exciting advanced materials, such as titanium aluminide alloys, particulate reinforced titanium and iron based MMCs, lightweight titanium alloys (eg. Ti-Mg) and other novel materials. This paper reviews the recent results obtained in applying clean MA processing to these types of materials

**[57] THE ROLE OF IMPURITIES IN THE CRYSTALLIZATION OF BALL-MILLED AMORPHOUS ALLOYS**

J Balogh, L Bujdosó, D Kaptas, T Kemeny, Vincze - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 63-67

Melt spun amorphous Fe<sub>83</sub>B<sub>17</sub> ribbons were pulverized in a planetary ball mill. Two parallel sets of experiment were made, where the material of the vial made the only difference in the experimental conditions. In the first kind of experiments tungsten carbide balls and vials were used, while in the second series the tungsten carbide balls were put in chrome steel vials. This way the effect of impurities on the mechanically induced crystallization could be studied and distinguished from other possible effects. Changes of the amorphous phase and the appearance of crystalline phases were followed by Mossbauer spectroscopy and DSC measurements. The results indicate that contamination from the milling tools causes a shift of the chemical composition of the milled amorphous material. In the case of steel milling tools the appearance of bcc nuclei initiate crystallization during milling. Instead of this bcc structure, a modified amorphous phase with a broader hyperfine field distribution - attributed to the significant amount of dissolved tungsten-is observed when the amorphous alloy is milled with tungsten carbide balls and vials. The differences detected under experimental conditions where only the material of the vial was different confirm that the specific impurities introduced in the milling process play a decisive role in the crystallization of amorphous alloys under ball-milling conditions.

**[56] THE FORMATION OF B2 STRUCTURE BY MECHANICAL ALLOYING OF AL<sub>50</sub>FE<sub>50</sub>-XNiX POWDER MIXTURES**

VK Portnoy, AV Leonov, VI Fadeeva, H Matyja - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 69-74

The aim of the present work was to study the influence of Ni on the formation of ordering BCC B2 phase by mechanical alloying. The X-ray, DSC and Mossbauer methods were utilized. The behavior of the lattice parameter, LRO parameters, crystallite size and microstrains during MA of the elemental metallic powders has been studied. It was established that Ni addition to Al<sub>50</sub>Fe<sub>50</sub>-xNiX extends aptitude for the ordering. Different mechanisms of ordering for the different composition Al<sub>50</sub>Fe<sub>50</sub>-xNiX alloys have been shown. Thermal stability of MA alloys has been investigated as well.

**[55] CAN RARE EARTH-TRANSITION METAL COMPOUNDS BE CARBURIZED BY MECHANICAL ALLOYING?**

G Rixecker, KHJ Buschow, H Bakker - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 75-80

Rare earth-iron interstitial carbides and nitrides are candidate materials for advanced permanent magnets. However, since the thermal stability of the parent compounds is generally limited, it has not been possible to date to synthesize single-phase materials using the usual gas-phase nitriding and carburizing processes. Very recently, it has been shown by Mao, Strom-Olsen, Altounian and Yang (J. Appl. Phys. 79 (1996), p.4619.) that two-phase Sm<sub>2</sub>Fe<sub>17</sub>CX/alpha-Fe can be produced by ball milling Sm<sub>2</sub>Fe<sub>17</sub> with graphite and a subsequent heat treatment. Here, we perform ball milling experiments on TbFe<sub>9.5</sub>Mo<sub>2.5</sub>, TbFe<sub>9</sub>Mo<sub>3</sub>, Gd<sub>2</sub>CO<sub>17</sub>, Gd<sub>2</sub>Fe<sub>17</sub> and Sm<sub>2</sub>Fe<sub>17</sub> in order to study whether these compounds can be directly carburized by mechanical alloying with one of the following carbon sources: graphite, glass-like carbon, toluene, solid paraffin or iron carbide. At present, we conclude that a heat treatment is always necessary to start the diffusion of carbon into the host lattice, resulting in the partial decomposition of the compounds.

**[54] INVESTIGATION OF AMORPHIZATION IN AN INTERMETALLIC POWDER MIXTURE OF BULK GLASS FORMING COMPOSITION**

A Sagel, RK Wunderlich, HJ Fecht - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 81-86

Glass formation by ball milling of a mixture of intermetallic phases with composition Zr<sub>60</sub>Al<sub>10</sub>Ni<sub>9</sub>Cu<sub>18</sub>Co<sub>3</sub> was

investigated by X-ray diffraction and thermal analysis and compared with glassy phases produced by liquid undercooling and ball milling of an elemental powder mixture of the same composition. The so obtained amorphous phase reveals a distinct glass transition and a large stability range of the highly undercooled melt of 95 K. It is believed that this is by far the widest temperature range of a highly undercooled melt found for ball milled amorphous materials.

**[53] STRUCTURE AND MAGNETIC PROPERTIES OF NANOCRYSTALLINE SPINEL FERRITES OBTAINED BY HIGH ENERGY BALL MILLING**

D Arcos, N Rangavittal, M Vazquez, M ValletRegi - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 87-92

The mechanochemical synthesis of zinc, manganese and zinc-manganese nanocrystalline ferrites obtained by mechanical milling from precursors: a) oxides and carbonates, b) ceramic products and c) hydroxides and oxides is presented. Final products have been characterised by X-ray diffraction (XRD), differential scanning calorimetry (DSC); Mossbauer spectroscopy and vibrating sample magnetometry (VSM). Results are compared with the ferrites synthesised by the ceramic method. Depending on the Mn/Zn ratio, mechanical treatment leads to ferrites having nanocrystalline structure with improved or decreased magnetic properties compared to ceramic ones. Subsequent thermal treatment of the disordered mechanochemical synthesized ferrites leads to its crystallisation at temperatures which are lower than those at which the crystalline ferrites are formed by the ceramic method.

**[52] MECHANICAL ALLOYING OF THE PEROVSKITE-TYPE STRUCTURED POWDER OF  $\text{La}_{2/3}\text{-xLi}_x\text{TiO}_3$  SHOWING LITHIUM ION CONDUCTION**

S Takai, M Moriyama, T Esaka - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 93-97

A mechanical alloying technique was applied to prepare the oxide precursor powder of  $\text{La}_{2/3}\text{-xLi}_x\text{TiO}_3$  (lithium ion conductors) in the ternary oxide system. As a typical example of a lithium containing sample in  $\text{La}_{2/3}\text{-xLi}_x\text{TiO}_3$ , we chose the  $x = 0.116$  sample, which corresponds to the composition of the sample showing the highest lithium ion conductivity. X-ray diffraction patterns were taken every 24 hours ball milling until 120 hours. Initially, the pattern showed the typical peaks of the mixture of starting materials of  $\text{La}_2\text{O}_3$ ,  $\text{Li}_2\text{CO}_3$  and  $\text{TiO}_2$ . They generally changed to be broadened according to the milling, and a few characteristic peaks appeared. As these almost corresponded to the data of  $x = 0.116$  sintered sample, the perovskite-type oxide phase that we desired was found to be formed by this method; even using  $\text{Li}_2\text{CO}_3$  as one of the starting material. In the other samples of different compositions, the lithium-containing samples could also be prepared by this method. However, the formation of  $\text{La}_{2/3}\text{TiO}_3$ , known as a defect perovskite-type compound, could not be so clear, which denotes that the lithium component plays an important role in the oxide preparation by mechanical alloying.

**[51] IRON-CHROMIUM OXIDE SOLID SOLUTIONS PREPARED BY MECHANICAL ALLOYING**

D Michel, L Mazerolles, E Chichery - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 99-104

Mechanical alloying was applied in order to prepare  $(\text{Fe,Cr})_2\text{O}_3$  solid solutions at room temperature by high energy milling. The lattice constants of hematite-type solid solutions were compared with that of samples prepared by conventional solid state reaction. A particle size about 20 nm was determined from the profile of XRD lines and TEM observations. Microanalyses by EDX were performed in order to check the chemical homogeneity of the samples.

**[50] MECHANICALLY ACTIVATED LOW TEMPERATURE SYNTHESIS OF SR DOPED LANTHANUM MANGANITE**

F Padella, E Incocciati, CA Nannetti, C Colella, S Casadio, M Magini - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 105-110

Alkaline earth doped lanthanum manganites ( $\text{La}_{1-x}\text{M}_x\text{MnO}_{3+\delta}$ , with  $\text{M}=\text{Ca, Sr, Ba} \dots$ ) are attracting materials because of their peculiar properties displayed in giant magnetoresistance and electrocatalytic activity. Classical synthesis routes of the material make use of solid state reaction, among suitable precursors, at temperatures generally above 900 - 950 degrees C. However such temperatures promote morphological and microstructural characteristics, such as low surface area and large particle size, lowering the catalytic activity. Mechanochemical synthesis technique has been therefore attempted on a mixture of  $\text{La}_2\text{O}_3$ ,  $\text{MnO}_2$  and  $\text{SrCO}_3$  in order to verify the possibility of obtaining  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$  with high specific area and small grain size.  $\text{LaMnO}_3$  has been also synthesized in the same way for comparison. The behavior of the mixtures has been monitored by x-ray diffraction. Thermogravimetry (TG) and differential thermal analysis (DTA) have been used to follow the thermal behavior compared to that of simply blended mixture. TG and DTA thermograms indicate that the synthesis of manganite, already present in the milled samples, is completed at temperatures much lower (650 - 700 degrees C) than those of conventional routes. Preliminary measurements of specific surface area confirm that mechanically activated synthesis of lanthanum manganite is able to produce powders having higher surface area and smaller particle size than those obtained by conventional solid state reactions.

**[49] THE SOLID-STATE SYNTHESIS OF W-25WT% RE USING A MECHANICAL ALLOYING APPROACH**

EY Ivanov, BD Bryskin - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 111-116

Stress-assisted precipitation of sigma-phase from a metastable solid solution are observed in deformed W-25-26wt% Re alloys aligned with the working directions. They are accounted for local chemical inhomogeneities based on concentration gradient of Re and resulted in stress appearance following by microcracks initiation and growth. The formation of nanocrystalline tungsten - rhenium solid solution by mechanical alloying in a planetary ball mill using tungsten - rhenium solid solution by mechanical alloying a planetary ball mill using tungsten containers with tungsten

milling bodies was investigated. The process was monitored by x-ray diffraction (XRD), differential thermal analysis (DTA), particle size analysis, and electron microscopy. It was shown that MA of W-Re mixture resulted in the formation of a finely dispersed composite material and, on later stages of MA a solid solution with a nanocrystalline structure.

**[48] CYCLIC CRYSTALLINE-AMORPHOUS TRANSFORMATIONS BY MECHANICAL ALLOYING**

K Aoki, MS ElEskandarany, K Sumiyama, K Suzuki - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 119-125

Structural changes of elemental powder blends with the atomic composition of Co<sub>75</sub>Ti<sub>25</sub> and Co<sub>50</sub>Ti<sub>50</sub> subjected to high-energy ball milling were investigated by means of X-ray diffraction (XRD), differential thermal analysis (DTA) and the magnetization measurement. The results have shown that amorphous a-Co<sub>75</sub>Ti<sub>25</sub> and a-Co<sub>50</sub>Ti<sub>50</sub> alloys were formed after short milling time. These amorphous alloys transform into metastable bcc-Co<sub>3</sub>Ti and bcc-CoTi alloys with a bcc structure, respectively, upon further milling. These Co<sub>3</sub>Ti and CoTi are thermally stable and do not transform to any other phase(s) upon heating up to 1300 K, but they return to a-Co<sub>75</sub>Ti<sub>25</sub> and a-Co<sub>50</sub>Ti<sub>50</sub> alloys upon further milling, respectively. This amorphization occurs similar to mechanical grinding (MG) by the accumulation of lattice imperfections, which raises the free energy from the stable crystalline phase to the amorphous one. Further milling leads to the formation of crystalline and/or amorphous phases depending on the MA time. The present work demonstrates cyclic crystalline-amorphous phase transformations in the Co-Ti system by MA. Such cyclic phase transformations are not caused by the contamination of the powder and/or the increasing in temperature of the vial during the milling.

**[47] THE CRYSTALLINE-TO-AMORPHOUS TRANSFORMATIONS IN THE TI-FE AND TI(H-2)-FE SYSTEMS DURING BALL MILLING**

AA Novakova, OV Agladze, BP Tarasov, GV Sidorova, RA Andrievsky - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 127-132

Elemental equiatomic Ti-Fe as well as similar Ti(H-2)-Fe powder mixtures were mechanically alloyed in ball mill. The structural changes as a function of milling time were investigated by X-ray diffractometry and low temperature Mossbauer spectroscopy. During milling the supersaturated solid solutions beta-Ti(Fe) and alpha-Fe(Ti) in wide range of composition, two intermetallic phases FeTi and Fe<sub>2</sub>Ti, formation and transformation up to quasi-amorphous state were observed. Furthermore in Fe-Ti(H<sub>2</sub>) system the MA process proceeds much slower and the formation of FeTi and FeTiH<sub>x</sub> phases and alpha-Fe(Ti) solid solutions were observed.

**[46] LOW TEMPERATURE MAGNETIC PROPERTIES OF FCC FERH OBTAINED BY BALL MILLING**

E Navarro, D Fiorani, R Yavari, M Rosenberg, M Multigner, A Hernando, R Caciuffo, D Rinaldi, S Gialanella - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 133-138

Starting from four different FeRh compositions, a nanocrystalline fcc phase, paramagnetic at room temperature, has been obtained, for each case, through mechanical alloying by ball-milling. The starting selected compositions were: ferromagnetic and bcc-ordered Fe<sub>65</sub>Rh<sub>35</sub>, antiferromagnetic and also bcc-ordered Fe<sub>50</sub>Rh<sub>50</sub>, paramagnetic and fcc Fe<sub>28</sub>Rh<sub>72</sub> and Fe<sub>26</sub>Rh<sub>74</sub>. The average grain size of the resulting fcc phase has been found to vary in the 6-12 nm range, depending on the Rh content of the precursor. At low temperatures, some characteristic behaviors typical of the spin-glass or superparamagnetic state have been observed in all the compositions: a maximum in the AC susceptibility, at a temperature (T<sub>m</sub>) which increases with the grain size, a maximum in the measurements of the thermal variation of the ZFC and FC magnetization and a wide distribution of hyperfine fields in the Mossbauer spectra at T < T<sub>m</sub>. The possibility that this behavior could be determined by the presence of iron impurities, deriving from the stainless steel vial and balls used for the milling procedure, has been definitely removed since similar effects have been also observed in samples milled in a vial and with balls of agate. However, the analysis of the de field-cooled magnetization measurements, revealing the absence of any critical behavior of the non-linear susceptibility first coefficient in the vicinity of T<sub>m</sub>, rules out the possibility of a true spin-glass state. Actually, all the results seem to indicate that the observed behavior is intrinsic to the nanocrystalline nature of fcc FeRh.

**[45] THE STRUCTURE TRANSFORMATIONS IN MECHANICALLY ALLOYED FE-MN AND FE-CO COMPOSITION**

VV Tcherdyntsev, SD Kaloshkin, IA Tomilin, EV Shelekhov, YV Baldokhin - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 145-150

Fe-Mn and Fe-Co alloys were prepared by the method of mechanical alloying (MA) of elemental powders in high-energy planetary ball-mill in wide concentration ranges of components. The phase compositions of MA samples of both systems were similar to those existing according to the equilibrium phase diagrams but the concentration ranges of existence of single-phase solid solutions were markedly extended. High concentrations of the stacking faults in the alloys with the f.c.c. structure were observed. Kinetics of the structure transformations at the mechanical alloying was investigated for the Fe<sub>50</sub>Mn<sub>50</sub> alloy. The dependence of the hyperfine magnetic field on the alloy concentration was investigated for the Fe-Co system.

**[44] SOLID STATE REACTIONS IN THE FE-SN SYSTEM UNDER MECHANICAL ALLOYING AND GRINDING**

EP Yelsukov, GA Dorofeev, VA Barinov, TF Grigoreva, VV Boldyrev - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 151-156

Structure-phase transformations in the 68Fe-32Sn system by mechanical alloying from pure elements and mechanical grinding of a multi-phase alloy for different conditions of mechanical treatment in planetary ball mills were studied by X-ray diffraction, Fe-57 Mossbauer spectroscopy and magnetic measurements. The initial stages of structure-phase

transformations by mechanical alloying and grinding were found to be different. However, the final product in all cases was nanocrystalline supersaturated solid solution of Sn in alpha-Fe with the bcc lattice parameter  $a = 0.2995$  nm, the magnetic moment per Fe atom  $\mu(\text{Fe}) = 1.9 \mu(\text{B})$  and the Curie temperature  $T_c = 700$  K.

**[43] SOLID STATE REACTIONS IN MECHANICALLY ALLOYED AL-LI AND AL-LI-MG ALLOYS WITH VARIOUS METAL OXIDES**

M Sugamata, J Kaneko, H Higuchi - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 157-162

For the purpose of attaining in situ formation of fine dispersoids formed by selective oxidation of solute Li and Mg in Al, mechanical alloying was carried out for Al-Li and Al-Li-Mg alloys with or without addition of various metal oxides. Metal oxides added were CuO, Fe<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, Nb<sub>2</sub>O<sub>5</sub>, MgO and Li<sub>2</sub>O. Mechanical alloying was carried out by using an attritor ball mill with addition of ethanol as the process controlling agent. The mechanically alloyed powders were degassed and consolidated to P/M materials by hot pressing and subsequent hot extrusion. Constituent phases were determined by X-ray diffraction for the mechanically alloyed powders and the as-extruded and heat-treated P/M materials. Hardness and microstructures were examined for the P/M materials. It has been confirmed that Li is selectively oxidized and LiAlO<sub>2</sub> is generally formed in the Al-Li alloys. In the Al-Li-Mg alloys, Mg is oxidized in preference to Li. After hot extrusion hardness increases are not observed with progressing preferential oxidation and reduction of the added metal oxide. Coarsening of LiAlO<sub>2</sub> particles is observed in the P/M materials after heating at 873K. As-extruded P/M materials showed relatively high hardness and hence dispersion hardened materials of high mechanical strength can be expected by applying preferential oxidation of the solute in aluminum.

**[42] ON THE NANOCRYSTALLINE TO AMORPHOUS PHASE TRANSITION IN AL BASED ALLOYS DURING BALL MILLING**

T Benameur, A Inoue - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 163-168

We have found that mechanical grinding of Al enriched alloys (Al > 80 at. %) containing late transition metals as solute components such as Ni and Co in the composition ranges of 6 to 9 at.% Ni and 3 to 5 at.% Co (with an atomic ratio Ni/Co equal to 2) leads to the formation of nanostructured equilibrium phases: fee-Al solid solution and Al-9(Co,Ni)<sub>2</sub> compound for Al-Ni-Co systems. Significant differences towards the amorphization reaction were observed in these alloys as result of addition of a few atomic percent of Zr or Ti. Upon disordering (Al<sub>0.88</sub>Ni<sub>0.08</sub>Co<sub>0.04</sub>)(100-x)(Zr/Ti)<sub>x</sub> for x = 1 to 5 at.% prealloyed powders, partial to complete amorphization occurs by the following sequence: fee-Al + Al-9(Co,Ni)<sub>2</sub> + Al<sub>3</sub>Zr/or Al<sub>3</sub>Ti --> fcc-Al + amorphous I --> amorphous II. While mechanical grinding is shown to amorphize Al<sub>3</sub>Zr which enters in equilibrium with the fee-Al solid solution, amorphization of Al<sub>3</sub>Ti compound is not evident. However, increasing Zr or Ti content, the thermal stability of the amorphous phase increases without appreciable variation of the melting temperature of the quaternary alloys. Moreover, the amorphization reaction rate was found tow times larger for Ti than for Zr substitution. Presumably due to an increase of the reduction rate of the fee-Al particles allowing a faster dissolution. The melting temperature, the crystallization temperature and enthalpy of the ternary and quaternary prealloyed powders were determined.

**[41] THERMAL STABILITY STUDY OF SOME FE-NI-P-SI ALLOY POWDERS**

JJ Sunol, MT Mora, N Clavaguera - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 175-180

in the present work, three metal-metalloid alloy compositions (Fe<sub>40</sub>Ni<sub>40</sub>P<sub>20-x</sub>Si<sub>x</sub> with x = 6, 10 and 14) were obtained by mechanical alloying. Investigations of the amorphization progress, the microstructure, the thermal stability and properties of the materials so prepared were performed using X-ray diffraction, scanning electron microscopy with energy dispersion X-ray microanalysis and differential scanning calorimetry techniques. After 32 h milling the product powders consists in mixtures of disordered amorphous-like and crystalline phases. The higher the Si content is, the higher the thermal stability of the amorphous phase is. To obtain a quasi-complete amorphization, it is necessary to continue the milling process until 100 h.

**[40] DEOXIDATION OF IRON OXIDE BY BALL-MILLING**

K Tokumitsu, T Nasu, K Suzuki, AL Greer

MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 181-186

The possibility of deoxidation of iron oxide by ball-milling was studied by XRD, SEM and Mossbauer spectroscopy. Iron oxide (alpha-Fe<sub>2</sub>O<sub>3</sub>:hematite) powder and titanium (Ti) powder were charged into a container for ball-milling with stainless steel balls. The container was filled with argon gas. Milling was interrupted at 50, 60,80,100, 200, 300, 400, 600, 800 and 1000 hours to take samples for analysis. An ordinary rotatory ball-milling machine was used for the experiment. The Bragg peaks of alpha-Fe(110),(200) and (211) appeared in the XRD pattern at 80 hours of milling. At the same time, the intensity of the Bragg peaks of Fe<sub>2</sub>O<sub>3</sub> and Ti decreased rapidly and almost disappeared at 600 hours of milling. The changes of morphology of the samples were observed by SEM. The shape of iron oxide powder was spongy at the first stage of milling. After 100 hours of milling the shape was more lump-like. The results of Mossbauer spectroscopy show that the alpha-Fe is formed by deoxidation of Fe<sub>2</sub>O<sub>3</sub> after just 50 hours of ball-milling. The amount of the alpha-Fe increased and Fe<sub>2</sub>O<sub>3</sub> decreased with milling time. A small amount of TiFe<sub>2</sub>O<sub>4</sub>-like ferrite and Ti<sub>1-x</sub>Fe<sub>x</sub>O-like wustite remained at 1000 hours of milling.

**[39] STRUCTURAL TRANSITION OF ZIRCONIA DURING MECHANICAL ATTRITION**

M Qi, HJ Fecht - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 187-191

Microstructural refining of materials by ball milling is an important method for improving their properties, especially for enhancing toughness of brittle ceramic materials. The present work investigated the structural transition of zirconia ceramics during ball milling by X-ray diffraction and DSC. The results show that besides refining of microstructure, the structural change of zirconia happens too. Three couple of strong diffraction peaks formerly

belonged to monoclinic lattice system combine together respectively, and form three new strong diffraction peaks. Two peaks of (022)<sub>m</sub> and ((2) over bar 20)<sub>m</sub> combine into one peak first, then that of (020)<sub>m</sub> and (200)<sub>m</sub>, finally that of ((1) over bar 11)<sub>m</sub> and (111)<sub>m</sub> combine into one. After 40 h milling, the diffraction peaks are well consistent with that of orthorhombic zirconia. That is, milling makes structure of a material vary to one with higher symmetry which corresponds a phase existing more stably at high temperature. Based on the experiment results, the form and change of stored energy in materials during milling are discussed. And a term so called as body intergrain energy was proposed.

**[38] EVOLUTION OF NANOSTRUCTURE AND CHEMICAL REACTIVITY OF CARBON DURING BALL-MILLING**  
H Hermann, W Gruner, N Mattern, HD Bauer, F Fugaciu, T Schubert - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 193-198

Nanostructured carbon is produced from graphitic powder by ball-milling in a planetary mill. The size of the nanoparticles is about 3 to 5nm. There is no indication for crystal-like graphitic atomic order in the nanoparticles. The dramatic structural changes caused by the milling process are accompanied by a substantial increase of the chemical reactivity of the nanostructured carbon. The enhancement of the chemical reactivity is essentially a consequence of the distortion of the atomic short-range order.

**[37] HOT-CONSOLIDATION OF MECHANICALLY MILLED AMORPHOUS ZR-CU POWDERS**

JH Ahn, M Zhu - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 201-206

Sinterability of mechanically milled amorphous powders has been examined in the Zr-Cu system. The focus of the study is to see the effect of pressure and additive elements on sintering, microstructural evolution and grain growth during annealing. The results of sintering experiments showed that ball-milled powders exhibited a poor sinterability in spite of high surface and grain boundary energy produced by mechanical milling. The sinterability could be improved by the external applied pressure. Soft metals were used as binder phases or additive elements to enhance the ability of consolidation or to produce composite structures. The results showed that high pressure and the use of additives can assist in consolidating the mechanically milled metastable powders.

**[36] RAPID SINTERING OF NANOSTRUCTURAL POWDER TO FORM Si<sub>3</sub>N<sub>4</sub>**

D Wexler, A Calka, SJ Colburn - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 219-224

Reactive ball milling, using the magneto-mechanochemical method, was performed on silicon in an ammonia atmosphere. X-ray analysis and transmission electron microscopy revealed a nanostructural product, confirmed by energy dispersive spectroscopy to comprise predominantly Si and N. Various combinations of powder/binder (Y<sub>2</sub>O<sub>3</sub> binder, MgO binder) were pressed and rapidly sintered under Argon in an induction heated graphite crucible. Sintered pellets, which were examined using XRD, SEM-EDS, and DTA, were found to comprise Si<sub>3</sub>N<sub>4</sub> with minor amounts of FeSi and binder phases. The densities of the products were high in comparison to those of conventionally prepared Si<sub>3</sub>N<sub>4</sub>, and the hardnesses, obtained using ultra-micro indentation, were found to be in the highest possible range for Si<sub>3</sub>N<sub>4</sub>. Preliminary studies of the production of Si<sub>3</sub>N<sub>4</sub> from nanostructural powder using no binder also gave promising results, with sintered pellets of density and hardness equivalent to that currently achieved using conventional production techniques with binders. The exceptional sintering properties were attributed to the particular nanostructural form of the milled powder.

**[35] MECHANICAL ACTIVATION AND ITS APPLICATION IN TECHNOLOGY -**

VV Boldyrev - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 227-234

The problems related to the application of mechanical activation and apparatus for mechanical activation to technology are considered. Some examples of the application of mechanical activation in metallurgy and chemical industry are presented.

**[34] MECHANOCHEMICAL SYNTHESIS OF NANOCRYSTALLINE COMPLEX OXIDES**

TF Grigorieva, AP Barinova, GN Kryukova, VD Belykh, EY Ivanov, VV Boldyrev - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 235-240

The nanocrystalline complex oxides were produced by mechanochemical activation for high exothermal reactions of barium peroxide with metals (Ti, Zr, Mo, Al, Sn, Si, Fe). Mechanochemically synthesized complex oxides were studied by x-ray diffraction and by electronic microscopy methods.

**[33] MECHANOCHEMICAL SYNTHESIS OF INDIUM-TIN OXIDE POWDER**

E Ivanov, TF Grigorieva - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 241-246

Submicron indium oxide powder was obtained by the oxidation of metal and urea-hydrogen peroxide adduct (UHPA) mixture reactively milled with hydrogen peroxide in high energy mill according to the reaction: 2In + 3 (NH<sub>2</sub>)<sub>2</sub>CO . H<sub>2</sub>O<sub>2</sub> --> In<sub>2</sub>O<sub>3</sub> + 3H<sub>2</sub>O + (NH<sub>2</sub>)<sub>2</sub>CO Reactive milling (RM) of In with UHPA and hydrogen peroxide resulted in the formation of RM precursor - a complex mixture of submicron metal particles, traces amount of In<sub>2</sub>O<sub>3</sub> dispersed in an organic matrix. High resolution transmission and scanning electron microscope were used to study the microstructure of the precursor. Study of a dynamic change of IR spectra of In-UHPA in H<sub>2</sub>O<sub>2</sub> allowed us to suggest formation of In-urea complex. Reactive milling of UHPA with In and H<sub>2</sub>O<sub>2</sub> resulted in gradual disappearance of O-O stretching band and formation of a new bond in the 500-600 cm<sup>-1</sup> range, presumably due to In-O, while carbamide bands did not change. Thermal treatment of this RM precursor at 473K for 3-4 hours resulted in complete disappearance or evaporation of organic compounds. Further heating up to 573-673K in air/oxygen led to the oxidation with the formation of indium oxide, having typical IR spectra identical to one synthesized by wet chemistry and annealed at 673K.. Our study showed that reactive milling with combination of thermal treatment can be used to

syntheses submicron powders of a indium, tin oxides and indium-tin oxides (ITO). Sintering process of ITO powder obtained by RM was also studied.

**[32] REDUCTION AND CHANGE OF MAGNETIC PROPERTIES OF CO(OH)(2) ON MILLING WITH AL**

T Kamei, T Isobe, M Senna, T Shinohara, F Wagatsuma, K Sumiyama, K Suzuki - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 247-252

The reduction process and change of the magnetic properties have been investigated for a mixture comprising Co(OH)(2) and metallic Al as a function of mechanical milling. Dehydration and apparent thermit reaction by milling are detected by thermal analyses (TG-DTA). With increasing the milling time, the magnetization, M, becomes temperature-independent above 50K, whereas M at 5K rapidly decreases. At above 100K, M shows a maximum after milling for 12h due to reduction to form metallic Co. Zero-field Co-59-spin echo NMR signals are detected for the milled mixture. Marked negative Knight shift of Al-27 NMR by milling is induced by the polarization of 3d spins of neighboring Ca atoms. At the later stage of milling, partial re-oxidation of Co and compounding with aluminium takes place. The neighboring structure of cobalt atoms is widely distributed as confirmed by the separate Co-59 NMR signals.

**[31] MECHANOCHEMICAL REACTIONS OF ILMENITE**

Y Chen, JS Williams, SJ Campbell - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 253-258

Natural ilmenite samples (containing both FeTiO<sub>3</sub> and Fe<sub>2</sub>Ti<sub>3</sub>O<sub>9</sub> phases) have been ball milled in two atmospheres (vacuum and air). Different mechanochemical reactions were observed and the reaction processes were investigated by analysing the as-milled samples using X-ray diffraction and Mossbauer spectroscopy. A reduction reaction occurred during milling in vacuum because of the iron contamination, while an oxidation reaction was induced in the atmosphere of air with formation of thermally metastable phases, which corresponds to a low-temperature oxidation reaction in a thermal annealing process. The different products of milling in two atmospheres result in different chemical reactivities for the milled ilmenite samples.

**[30] TRANSFORMATIONS IN OXIDES - DRY AND WET MECHANOCHEMICAL PROCESSES**

WA Kaczmarek, SJ Campbell - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 259-264

In the past few years mechanochemistry has proved to be an exciting technique for modification of existing materials and the synthesis of new materials. For example, a broad range of ceramics and ceramic-metal composites, amorphous and nanocrystalline alloys as well as high temperature phases of nitrides, carbides and silicides have been produced by high energy ball milling, with one of the main developments being the mechanochemistry of oxide materials. An overview of the morphological, structural and stoichiometric changes which take place on dry and wet milling a variety of oxides is presented. Among the systems studied are ZnFe<sub>2</sub>O<sub>4</sub>, BaFe<sub>12</sub>O<sub>19</sub>, alpha-Fe<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub>.

**[29] NITROGENATION DURING BALL MILLING OF STAINLESS STEEL**

A Calka, D Wexler, J Zhou, D Dunne - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 265-270

Reactive ball milling, using the magneto-mechano-chemical method, was performed on type 304 stainless steel powder in order to investigate structural development and nitrogenation. Experiments were performed in argon, nitrogen and ammonia gases. The allotropic transformation from austenite to ferrite or martensite, which was believed to be stress induced, occurred after milling in all three gases with the reverse transformation occurring after longer milling times. Milling in both nitrogen and ammonia resulted in nitrogenation of the stainless steel powder, in the case of ammonia to higher levels (greater than 7 wt. %) than those achieved using conventional high-pressure-high-temperature diffusion processing. Rapid uptake of N during milling in ammonia is associated with formation of Fe<sub>3</sub>N. Annealing of nitrated powders at 1000 degrees C resulted in precipitation of the phases in the following order with increasing nitrogen concentration; beta-Cr<sub>2</sub>N, beta-Cr<sub>2</sub>N+CrN and CrN.

**[28] TEMPERATURE EFFECTS OF SI MILLING IN NH3**

ZL Li, JS Williams, A Calka - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 271-276

alpha-Si<sub>3</sub>N<sub>4</sub> has been formed by ball milling of pure Si in NH<sub>3</sub> and subsequently annealed in Ar. Milling was carried out at room temperature and 100 degrees C and the N content varies with milling temperature. For the case of milling at 100 degrees C, the complete formation of an amorphous SixNy phase was obtained after 48h milling, compared with 168h at room temperature. Both as-milled and annealed samples were investigated by X-ray diffraction, composition analysis, DTA and TGA. The results show that the crystallization temperature of a-Si<sub>3</sub>N<sub>4</sub> decreases or increases in a complex way with increased milling time depending on milling temperature. The results are discussed in terms of competing adsorption/desorption and reaction processes during milling.

**[27] AMORPHOUS PDO FORMATION DURING REACTIVE MILLING OF ALPD ALLOYS UNDER OXYGEN GAS**

K Tousimi, AR Yavari - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 277-282

PdAl was milled under controlled oxygen atmosphere with the aim of obtaining Pd/Al<sub>2</sub>O<sub>3</sub> nanocomposite. However the measured oxygen absorption during milling far exceeded the mole fraction needed to form Al<sub>2</sub>O<sub>3</sub>. A similar result was obtained for PtAl milled under O<sub>2</sub> gas. This initially led to the speculation about possible formation of AlO(2) as reported by Lu and al.[12]. However TEM results, X-ray and thermogravimetric analysis showed clearly that the excess oxygen absorption is due to formation of amorphous PdO. This result was further confirmed by the preparation of amorphous PdO during milling of pure Pd under O<sub>2</sub> gas.

**[26] HYDROGENATION OF CO AND C DURING MECHANICAL TREATMENT OF ZR AND NI CONTAINING**

## SYSTEMS

AN Streletskii, OS Morozova, IV Berestetskaya, AB Borunova - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 283-288

Processes of CH<sub>4</sub> and C<sub>2</sub>H<sub>6</sub> formation during mechanical treatment of NiZrH<sub>x</sub>, ZrH<sub>x</sub>, NiZr, Zr, Ni, and Zr+Ni in the presence of CO + H<sub>2</sub> or CO atmosphere as well as the mechanochemical reaction of hydrides with carbon (graphite) have been investigated using X-ray, chemisorption, thermodesorption, mass-spectroscopy and chromatography. It was found that 1) the rate of methane formation decreases when Ni is included into the systems, 2) 100% hydrogen of hydrides may be converted into methane in mixtures of the hydrides with carbon, 3) in the presence of CO the major part of hydrides hydrogen is evolved in a molecular form while only <15% of H<sub>2</sub> is expended for the methane formation. The results are discussed in terms of mechanochemical reactions of Zr with gases.

### [25] MICROSTRUCTURAL CHARACTERIZATION AND SINTERING OF FINE POWDERS OBTAINED BY SHS REACTION DURING MILLING

EMJA Pallone, DE Hanai, R Tomasi, WJ Botta - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 289-294

High-energy ball milling of highly exothermic mixtures can result in SHS type reactions which, depending on milling conditions, can lead to very fine distribution of the product powders. In this work we present the microstructural characterization of the powder mixtures produced by reactive milling of the metallic phases Al, Zr (or the intermetallic Al<sub>2</sub>Zr) with the oxide Nb<sub>2</sub>O<sub>5</sub>, and we discuss the sintering behaviour of the resulting fine Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub>-Nb product powders. Milling was performed at different ball/mass ratio and milling times and the transformations during milling were studied by X-ray diffraction (XRD), transmission electron microscopy (TEM) and scanning electron microscopy (SEM). XRD indicated that the reaction was completed after 2h milling even with ball/mass ratio of 2:1; however the product powder was strongly aggregated into large particles. SEM observations in the cross-section of these aggregates revealed a dense, heterogeneous microstructure, of Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> ceramic matrix with large inclusions of Nb, typical of a self-propagating high-temperature synthesis for the same reaction. Sintering of dry pressed powders was carried out at 1650 degrees C under vacuum of 10(-5)mbar for 2h and resulted in a dense and uniform microstructure in the metallic and the ceramic phases.

### [24] TRANSMISSION ELECTRON MICROSCOPY STUDIES OF MECHANICAL ALLOYING IN THE IMMISCIBLE FE<sub>2</sub>O<sub>3</sub>-SnO<sub>2</sub> SYSTEM

DG Rickerby, JZ Jiang, R Lin, S Morup - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 351-356

Microstructural development and nanoscale compositional variations in mechanically alloyed Fe<sub>2</sub>O<sub>3</sub>-SnO<sub>2</sub> powders have been examined by transmission electron microscopy and energy dispersive X-ray spectrometry. The mean grain size was found to stabilize around 10 nm after 19 h milling time, in close agreement with that estimated from X-ray diffraction line broadening measurements, whereas dissolution of SnO<sub>2</sub> grains was incomplete even after 110 h. Isolated grains with the SnO<sub>2</sub> cassiterite structure, of diameter >10 nm, persisted up to the maximum milling time. These observations are discussed in relation to previous measurements in the same system by X-ray diffraction and Mossbauer spectroscopy, which suggested that alloying on the atomic scale occurred after 110 h milling. The present studies confirm that the amount of Sn dissolved in the Fe<sub>2</sub>O<sub>3</sub> hematite lattice increases with longer milling times, indicating that a supersaturated solid solution is formed, but that mixing may be locally inhomogeneous at the atomic level. Similar conclusions have been reported for studies of mechanical alloying in immiscible metallic systems. The tendency for SnO<sub>2</sub> grains above a certain critical size to remain undissolved, while smaller grains can more easily enter into solid solution with Fe<sub>2</sub>O<sub>3</sub>, is consistent with the expected behaviour due to the increased chemical contribution to the interfacial energy with decreasing grain size. Mossbauer results also showed that some of the SnO<sub>2</sub> had not reacted with Fe<sub>2</sub>O<sub>3</sub> after 110 h milling.

### [23] HRTEM IMAGE PROCESSING STUDY OCCURRING IN MECHANICALLY ALLOYED ZrO<sub>2</sub>-10MOL.%Y<sub>2</sub>O<sub>3</sub> POWDERS

AM Tonejc, A Tonejc, GW Farrants, S Hovmoller - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 357-362

We have investigated, using transmission electron microscopy (TEM), high resolution electron microscopy (HRTEM) and image processing, the early stages of the mechanical alloying process of a mixture of zirconia and yttrium oxide powders. We focused our investigation to two regions: to a grain boundary region and to the region of stacking faults near grain boundary. Fourier filtering revealed on the atomic level one possible sequence of alloying that occurred in the grain boundary and on stacking faults.

### [22] DIFFRACTION OF AMORPHOUS AND NANOCRYSTALLINE ALLOYS PREPARED BY SOLID STATE REACTIONS

S Enzo - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 363-372

In this paper we review some of the existing issues concerning the structural characterization of end-products obtained after extensive mechanical treatment and deformation. After recalling the basic aspects of diffraction broadening at small and wide angles, we suggest that the presence of nanostructures is testified in the diffraction patterns by the strong Cauchy or super-Cauchy character of peak profiles. Good data for this kind of experiments and analyses are currently made available at the large scale facilities for X-rays and neutrons. From the mathematical point of view the determination of crystallite or particle size distribution functions is an ill-posed problem, that is, small fluctuations of the experimental data can affect considerably the final solution. It is then necessary to start with

the full control of the critical steps in data analysis corroborated with the knowledge of the thermodynamics of the systems under investigation.

**[21] MICROSTRUCTURAL CHARACTERISATION OF METASTABLE STRUCTURES IN INTERMETALLIC COMPOUNDS**

L. Lutterotti, S. Gialanella - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 373-378

Mechanical milling, rapid solidification, heavy ion bombardment are extensively used to produce metastable and non-equilibrium structures in materials. Nanograined metallic and intermetallic powders can be attained by ball-milling as a consequence of the progressive grain size reduction promoted by plastic deformations. The microstructural characterisation of such samples may face some difficulties related to the defect structures or to the changes introduced by the sample preparation techniques. In this study a few examples of microstructures, as investigated by X-ray diffraction methods, present in ball-milled intermetallic powders are discussed. Disorder induced by the increase in the concentration of some kinds of defects is discussed with particular reference to Ni<sub>3</sub>Al and FeAl alloys. Transformations to new polymorphs are investigated in the case of a TiAl base alloy.

**[20] MECHANICALLY ACTIVATED SHS REACTION IN THE FE-AL SYSTEM: IN-SITU TIME RESOLVED DIFFRACTION USING SYNCHROTRON RADIATION**

E. Gaffet, F. Charlot, D. Klein, F. Bernard, J.C. Niepce - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 379-384

The Mechanical Activation Self propagating High temperature Synthesis (M.A.S.H.S.) processing is a new way to produce nanocrystalline iron aluminide intermetallic compounds. This process is mainly the combination of two steps; in the one hand, a mechanical activation where the Fe-Al powder mixture was milled during a short time at given energy and frequency of shocks and in the other hand, a Self propagating High temperature Synthesis (S.H.S.) reaction, for which the exothermicity of the Fe + Al reaction is used. This fast propagated MASHS reaction has been in situ investigated using the Time Resolved X-Ray Diffraction (TRXD) using a X-ray synchrotron beam and an infrared thermography camera, allowing the coupling of the materials structure and the temperature field. The effects of the initial mean compositions, of the milling conditions as well as of the compaction parameters on the MASHS reaction are reported.

**[19] THE STRUCTURE OF MECHANICALLY ALLOYED ALXFE(1-X) END-PRODUCTS AFTER ANNEALING**

S. Enzo, G. Mulas, R. Frattini - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 385-390

Al-Fe specimens were mechanically alloyed as a function of composition for extensive times of treatment. From analysis of X-ray diffraction data, supported by the powder pattern fitting with the Rietveld method, it was found that the end-products are mainly BCC extended solid solutions, even in those cases where the major component in the parental mixture was FCC aluminium. The dissipative processes of the solid state reaction are followed with small angle neutron scattering curves of mechanically alloyed Al<sub>66</sub>Fe<sub>34</sub> specimens, which show a slope in the log I vs log Q plot decreasing as a function of the consumption of FCC aluminium. The solid state dissolution of aluminium is also suggested by strong asymmetry of X-ray peak profiles. In particular, the asymmetry of the most intense (110) peak profile of the BCC phase increases linearly as a function of the aluminium/iron atomic ratio. Annealing of the Al-Fe end-products in a differential scanning calorimeter produces varying exothermal features. Analysis of XRD patterns by the Rietveld method helps to define the structural transformations induced in mechanically and thermally treated specimens.

**[18] STRUCTURAL EVOLUTION OF AL<sub>66</sub>FE<sub>34</sub> AND AL<sub>75</sub>FE<sub>25</sub> POWDERS PREPARED BY MECHANICAL ALLOYING**

S. Enzo, R. Frattini, G. Mulas, F. Delogu - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 391-396

Al<sub>66</sub>Fe<sub>34</sub> and Al<sub>75</sub>Fe<sub>25</sub> alloys were produced by extensive mechanical treatment of elemental powder mixtures. X-ray diffraction data as a function of time of treatment show that iron strongly affects the end-products. Notwithstanding FCC aluminium is in the largest concentration, the final habit is a single phase BCC structure. An important dissolution of iron in the lattice of aluminium seems to be ruled out, whilst the dissolution of aluminium in iron appears to proceed according to a two-stage process. From an analysis of the integral peak broadening it appears that the elastic properties of the BCC end-product are isotropic and similar to aluminium.

**[17] STRUCTURAL REFINEMENT OF AG-FE BLENDS DURING HIGH ENERGY BALL MILLING**

M. Angiolini, A. Deriu, F. Malizia, G. Mazzone, A. Montone, F. Ronconi, M. VittoriAntisari, J.S. Pedersen - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 397-402

The effect of high energy ball milling on the structural refinement of the binary system Ag-Fe has been studied by Transmission and Scanning Electron Microscopy, Small Angle Neutron Scattering and Mossbauer spectroscopy. Samples containing 10 wt% of Fe were submitted to high energy ball milling for times up to 50 hours. The process produces a fine dispersion of Fe into the Ag matrix; the size distribution function is quite broad with an average radius of the order of a few nm. The minimum average particle size is obtained after about 10 hours of milling, longer term processing leads to a detectable particle coarsening. TEM analyses give evidence of a well defined orientation relationships between the Fe particles and the Ag matrix. Mossbauer spectroscopy reveals a modification of the hyperfine field experienced by the Fe atoms only for short term milling. On the basis of the experimental results one can assume that the fine dispersion of Fe into the Ag matrix occurs by mutual solubilisation probably induced by plastic shear, followed by fast decomposition by precipitation. After this step, further milling can induce only coarsening of the microstructure.

**[16] CONTRIBUTION OF MOSSBAUER SPECTROMETRY TO THE STUDY OF MECHANICALLY ALLOYED MATERIALS AND OF NANOMATERIALS**

G LeCaer, P Delcroix, J Foct - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 409-418

Significant aspects of mechanically alloyed materials and of nanomaterials that are more particularly evidenced by Mossbauer spectrometry are discussed. Some examples include Mossbauer investigations of the intermixing process by grinding and of the mechanosynthesis of iron nitrides. Unconventional applications of new Mossbauer techniques to the field of nanomaterials are also mentioned.

**[15] MECHANICALLY ALLOYED AND RAPIDLY QUENCHED FE-ZR-B-CU: MOSSBAUER INVESTIGATION**

C Stiller, E Wu, SJ Campbell, A Kerr, WA Kaczmarek, JS Williams, J Eckert, L Schultz - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 425-430

Ferromagnetic Fe<sub>86</sub>Zr<sub>7</sub>B<sub>6</sub>Cu<sub>1</sub> alloys have been prepared by both mechanical alloying of elemental powders and rapid quenching. The mechanically alloyed powders are magnetically harder than their rapidly quenched counterparts. The fractions of the main phases-an alpha-Fe type phase, an amorphous phase and a grain boundary region-present in the as prepared and the heat treated materials have been estimated from the spectral components in their room temperature Mossbauer spectra. The magnetic hardening of the mechanically alloyed material is likely to be due to a number of effects including: an inhomogeneous microstructure; distortions in the grain boundary region, and inhomogeneous long-range stress as well as microstrain at the atomic level.

**[14] MOSSBAUER STUDY OF FENBCUSIB MECHANICAL ALLOYING PROCESS**

JS Garitaonandia, JM Barandiaran, P Gorria, L Righi - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 431-436

In the present work we report the preparation of nanocrystalline alloys, of composition Fe<sub>73.5</sub>Nb<sub>3</sub>Cu<sub>1</sub>Si<sub>13.5</sub>B<sub>9</sub>, formed directly by mechanical alloying of the constituents. Powders of the pure elements, in the appropriate proportions, were mixed in a Reitch planetary ball mill for upto 120 hours. The composition and the amount of the crystalline phases evolve during the milling and have been followed by Mossbauer Spectroscopy. A metastable FeSi solid solution is-formed with up to 18% Si content. This phase contains about 50% of the Fe present in the sample. At the same time other Fe disordered alloys-are produced. These are characterised by an inhomogeneous distribution of hyperfine fields.

**[13] THE LOCAL ATOMIC STRUCTURE OF NANOCRYSTALLINE MECHANICALLY GROUND FE-SI ALLOYS**

VM Fomin, EV Voronina, EP Yelsukov, AN Deev - MECHANICALLY ALLOYED, METASTABLE AND NANOCRYSTALLINE MATERIALS, PART 1 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 269-2, 1, pp 437-442

The local atomic structure of disordered mechanically ground Fe-Si alloys with the Si concentration from 14 to 50 at. % is studied by X-ray diffraction, Mossbauer and EXAFS-spectroscopy. The alloys studied are shown to be monophase disordered nanocrystalline ones. It is obtained from the Mossbauer data that the concentration behaviour of the probabilities of the local atomic surroundings of the Fe atom does not agree with the chaotic distribution in crystalline lattice with the coordination number 8. On the other hand, the values of Cowley short range order parameters calculated from the Mossbauer and EXAFS data exceed essentially the limit values and are not satisfactory either quantitatively or qualitatively. The local atomic structure of disordered mechanically ground Fe-Si alloys with the Si concentration form 14 to 50 at. % is studied by X-ray diffraction, Mossbauer and EXAFS-spectroscopy. The alloys studied are shown to be monophase disordered nanocrystalline ones. It is obtained from the Mossbauer data that the concentration behaviour of the probabilities of the local atomic surroundings of the Fe atom does not agree with the chaotic distribution in crystalline lattice with the coordination number 8. On the other hand, the values of Cowley short range order parameters calculated from the Mossbauer and EXAFS data exceed essentially the limit values and are not satisfactory either quantitatively or qualitatively.

**[12] COMMINATION AND MECHANICAL ACTIVATION OF PORTLAND CEMENT IN DIFFERENT MILL TYPES**

Sekulic Z. Popov S. Milosevic S. - Ceramics-Silikaty. 42(1):25-28, 1998.

The experiments of comminution (grinding-mechanical activation) were performed in laboratorial ball mill, vibro mill with balls and vibro mill with rings, using portland cement from the regular production. A noticeable mechanical activation was registered in vibro mill with rings (as confirmed by XRD analysis). In all three mill types, mechanical properties of ground cement were improved due to an increase of specific area to approximately the same value (400 m<sup>2</sup> kg<sup>-1</sup>). The highest compressive and bending strengths were achieved in Portland cement comminuted in vibro mill with rings (55 MPa and 9 MPa, in comparison with the original PC values 43 MPa and 7.5 MPa respectively), as registered after 28 day solidification period. Thereby, the compressive strength was about 10% higher and bending strength about 3.5% higher then in the cement treated in mill with balls until the same specific area was reached.

**[11] DEFORMATION BEHAVIOUR OF PARTICLE STRENGTHENED P/M-ALUMINIUM ALLOYS UNDER CREEP LOADS**

Ismail Y. Nicolini G. Elmagd E. - Metall. 51(10):557-563, 1997

Three different powder metallurgy (P/M) aluminium alloys (AlC001, AlC101 and AlC301) were tested under tension and compression creep loads. The materials were manufactured by mechanical alloying and subsequent consolidation by cold compaction and hot extrusion. The tension and compression tests were carried out at 300 degrees C, 400 degrees C and 500 degrees C. In tension creep tests, the applied stress was chosen to result a fracture life ranging between few minutes and 4000 hours. In compression creep tests, the applied stress was chosen to have a time to the loss of stability from 0.1 to 100 hours. These materials were also microstructurally inspected. The role of interfacial microstructure in the fracture of the strengthened P/M aluminium alloys were examined using SEM- and TEM-methods to study the correlation of the microstructure with the creep behaviour of these materials. It was found that

the creep resistance increases with the increase of the volume fraction of  $Al_2O_3$  and  $Al_4C_3$  in both tension and compression. Also, there is a distinct reduction of the ductility with increasing the volume fraction, but, it can withstand the applications at high temperatures up to 500 degrees C, and the tertiary creep stage does not appear in AIC301 in tension creep tests.

**[10] SEMI-CONTINUOUSLY MECHANICAL ALLOYING IN A PRODUCTION SCALE USING CYCLE OPERATION - PART I**

Zoz H. Ernst D. Mizutani T. Okouchi H. - Metall. 51(10):568-572, 1997

The production of large quantities of powders for industrial application e.g. in paints or soldering materials is an aim followed by Fukuda Metal Foil and Powder Co. Ltd. in Japan. For these applications, particles with a special geometry (diameter to thickness value) are needed. As the prior production route for these special particle geometries used at Fukuda is a very time intensive process, laboratory milling experiments in a Simoloyer CM01 (Zoz horizontal rotary ball mill) with a grinding chamber capacity of 1/2 liter were carried out to receive silver microflakes with the aim to reduce processing time and to increase the efficiency of the process. The results showed that this is a convenient way to achieve the wanted goal. Based on the same conceptual design the Simoloyer CM100s with a chamber volume of 100 liters allows the scaling-up of the process for an industrial application. The present work will focus on the development of a new grinding unit for a semi-continuously production of mechanically alloyed and mechanically particle deformed powders using the Cycle Operation for ductile materials with Critical Milling Behaviour (CMB).

**[9] FORMATION OF  $CaTiO_3$  BY GRINDING FROM MIXTURES OF  $CaO$  OR  $Ca(OH)_2$  WITH ANATASE OR RUTILE AT ROOM TEMPERATURE**

Mi GM. Saito F. Suzuki S. Waseda Y. - Powder Technology. 97(2):178-182, 1998

Room temperature grinding by a planetary ball mill has been applied to four kinds of mixtures prepared from  $CaO$  or  $Ca(OH)_2$  and anatase or rutile ( $TiO_2$ ). Crystalline  $CaTiO_3$  can be mechanochemically synthesized by grinding the mixture of  $CaO$  with both forms of  $TiO_2$  but is difficult to obtain from the mixtures of  $Ca(OH)_2$  and  $TiO_2$ . These observations are in contrast to the results of Avvakumov et al. [E.G. Avvakumov, E.T. Devyatkina, N.V. Kosova, Mechanochemical reactions of hydrated oxides, J. Solid State Chem. 113 (1994) 379-383] and are in agreement with the thermodynamic data. The reactivity of  $CaO$  or  $Ca(OH)_2$  with anatase is slightly higher than is the case with rutile. Heating of the ground mixtures at about 900-1200 K is found to be effective for producing crystalline  $CaTiO_3$ .

**[8] PREMATURE CENTRIFUGING, OSCILLATION AND AXIAL MIXING OF AN INDUSTRIAL GRINDING MILL LOAD**

Vannierop MA. Moys MH. - Minerals Engineering. 11(5):437-445, 1998

A study of the load behaviour of an industrial mill was undertaken to improve the understanding of the grinding-mill process. Load behaviour was measured directly by means of probes inserted through or mounted onto mill finer bolts. Toe and shoulder positions of the load were identified. Load oscillation and premature centrifuging occurred, and the effect of this on power is investigated. Salt tests were conducted to give an indication of axial mixing. Liner bolt movement probes attached to the outside of the mill measured some aspects of load behaviour. The data has provided valuable insights into mill load behaviour.

**[7] EFFECTS OF MIXING CONDITIONS ON THE PROPERTIES OF HOT-PRESSED ALUMINUM NITRIDE HEXAGONAL BORON NITRIDE COMPOSITES [Japanese]**

Kanai T. Iseki T. - Nippon Seramikkusu Kyokai Gakujutsu Ronbunshi-Journal of the Ceramic Society of Japan. 106(5):514-518, 1998

Aluminum nitride-hexagonal boron nitride ceramic composites were sintered by means of uniaxial hot pressing with changing the mixing conditions of raw materials. Mixed powders with a planetary-type milling had a larger surface area than those mixed with a rotary-type milling, which was a consequence of the fragmentation of the h-BN particles. The bulk density of the powder compact increased with increasing the specific surface area of the starting powders independent of the mixing conditions. The density of the isostatically cold pressed body reached about 74% of the calculated theoretical density. These results were thought to be caused by a bimodal particle-size distribution, suitable for dense compact during mixing, which also reduced the friction among particles during forming because of the excellent lubricant action of the B-EN. It was noticed that the maximum density in the sintered materials was achieved in correspondence of a specific surface area of the starting powders. The bulk densities of the sintered materials produced from small specific surface area powders increased with increasing the green density. On the other hand, the sintered density decreased because of the presence of fine h-BN particles which impeded the grain growth and the densification of AlN with a large specific surface area. It was considered that the densification of the composites is dominated by the densification of AlN.

**[6] SINTERING BEHAVIOR OF FINE-GROUND ALUMINA POWDERS BY WET ROTATION BALL MILLING AND MICROSTRUCTURES OF FIRED BODIES [Japanese]**

Yokota K. Kondo Y. - Nippon Seramikkusu Kyokai Gakujutsu Ronbunshi-Journal of the Ceramic Society of Japan. 106(5):519-524, 1998

Sintering behavior of some fine-ground alumina powders with various particle sizes and lattice strains prepared by wet rotation ball milling method were investigated. It was found that easy-to-sintering of powders was dependent on the particle size and independent of the lattice strain; hence, it was taken that mechanochemical effects did not contribute to easy-to-sintering of powders. Platelike grains, which grew in the transverse to c-axis, were formed in the sintered body of all ground powders. These grains grew largely as the particle size of ground powders increased. And it could be suggested that there is a possibility of controlling the platelike grain size by the particle size of the powder.

**[5] FORMATION OF EPSILON- $FeXN$ /BN MAGNETIC NANOCOMPOSITE AND ITS THERMODYNAMIC AND KINETIC ANALYSES**

Liu L. Yao B. Wang HY. Li FS. Ding BZ. Su WH. - Chinese Science Bulletin. 43(6):467-470, 1998

A nanocomposite of nanometer-sized magnetic granular epsilon- $FeXN$  embedded in a nonmagnetic amorphous boron nitride matrix was prepared by ball milling mixture of alpha-Fe and hexagonal boron nitride in argon atmosphere.

The grain size of the epsilon-FeXN alloy was about 10-20 nm. The nitrogen concentration in the epsilon-FeXN alloy increases with extending milling time. Both thermodynamic calculation and the present experiment show that iron and nitrogen atoms have higher alloying driving force than iron and boron atoms. Analyses of thermodynamics and kinetics about formation of the epsilon-FeXN alloy suggested that the formation of the epsilon-FeXN alloy is related to amorphization of the hexagonal boron nitride and refinement of the alpha-Fe. It was found from the present experiment that a critical grain size of the alpha-Fe reacting with nitrogen in the amorphous boron nitride is about 8 nm.

**[4] FORMATION AND INTERFACE STRUCTURE OF TiC PARTICLES IN DISPERSION-STRENGTHENED CU ALLOYS**

Dehm G. Thomas J. Mayer J. Weissgarber T. Pusche W. Sauer C. - Philosophical Magazine A-Physics of Condensed Matter Defects & Mechanical Properties. 77(6):1531-1554, 1998

TiC-dispersion-strengthened Cu alloys were prepared by mechanical alloying and subsequent hot extrusion. The evolution of the microstructure with respect to the preparation process is analysed by transmission electron microscopy techniques. The TiC dispersoids are formed in situ by the reaction of Ti and graphite. Ti diffuses from the pre-alloyed CuTi matrix to C inclusions which are embedded in the matrix after high-energy milling. Heat treatment of the powder mixtures at 400 degrees C leads to heterogeneous nucleation of TiC at the C/Cu interface. Thereby a well defined cube-on-cube orientation relationship is established between TiC and the Cu matrix. A study of the morphology of the TiC dispersoids shows that they are faceted on {111}(TiC), {110}(TiC) and {100}(TiC) planes and possess ledges on the atomic scale. The TiC/Cu interfaces are atomically abrupt and free of interface phases. The {100}(TiC)//{100}(Cu) and [110](TiC)//[110](Cu) topotaxy leads to a misfit of 17.6% between the adjacent lattices. This misfit is accommodated by a dislocation network along [100](Cu) directions.

**[3] MAGNETIC VISCOSITY AND COERCIVITY ANALYSIS IN MECHANICALLY ALLOYED AND MELT-SPUN NDDYFEB MAGNETS**

Villasboas V. Gonzalez JM. Cebollada F. Rossignol MF. Taylor DW. Givord D. - Journal of Magnetism & Magnetic Materials. 185(2):180-186, 1998

In this work we study the temperature dependence of coercivity and magnetic viscosity in mechanically alloyed magnets of composition (Nd1-xDyx)(16)Fe76B8 (0.1 < x < 0.5) and Nd15.5Dy2.5Fe65Co10Ga0.75B6.25, and of melt-spun magnets of composition (Nd1-xDyx)(15)Fe76B9 (0.5 < x < 0.8) for temperatures between 25 and 500 K. The results are discussed in terms of the so-called global model of coercivity which provides information about the magnetic properties in the activation volume v(a), which is evaluated from magnetic viscosity measurements.

**[2] MECHANOMAKING OF HIGH SPEED STEEL AISI M2 POWDERS FABRICATION**

Matteazzi P. Wolf F. - Materials Science & Engineering A- 246(1-2):235-243, 1998

High speed steel AISI M2 was obtained by high energy milling from the elemental powders. The kinetics of synthesis were followed by Mossbauer spectroscopy and X-ray diffraction. About 14 h are required to complete alloying (12 at.% solute content) of the matrix and formation of carbides (M6C and Fe3C) all having grain sizes below 20 nm. The mean particle size was 50 nm and the individual particles were almost fully dense. Limited grain growth to < 100 nm at temperatures up to 1300 degrees C was observed. This new concept for the fabrication of alloys and materials is suggested to be termed mechanomaking.

**[1] STRUCTURAL DEFECTS IN MECHANICALLY ACTIVATED PYRITE**

Zhizhaev AM. Trifonova OV. Smyk AA. - Russian Journal of Applied Chemistry. 71(1):32-34, 1998

Mechanical activation of pyrite was studied by Fourier analysis of the X-ray line profile. Coherent scattering regions and root-mean-square deformation of activated samples were calculated.

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IPSé - F90010 - Belfort Cedex - France

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les personnes précédées par "•" sont inscrites sur la liste de diffusion électronique du RFM

### Allemagne

• K.U. Kainer

**Institut für Werkstoffkunde und Werkstofftechnik** Light Metals, Powder Metallurgy and Composites Group - Agricolastr. 6 - D 38678 - Clausthal Zellerfeld - Allemagne

• P. Reynders

**Merck - Pigments Division** ) Bldg M18 - 64271 Darmstadt - Allemagne

• A. Sagel

**Institut für Metallphysik und Technologie** Technische Universität Berlin - Hardenbergstr.36 - PN 2 - 3 - D - 10623 - Berlin - Allemagne

• M. Veith

**Universität des Saarlandes** Institut für Anorganische Chemie - Postfach 15 11 50 - D 66041 - Saarbrücken - Allemagne

### Angleterre

• P. Shashi

**De Montfort University** -Emerging Technologies Research Centre - SER Centre - Howthron Building - Gateway - Leicester LE1 9BH - Royaume Uni

### Argentine

• F.H. Sanchez (97)

**Dpto de Fisica** - Universidad Nacional de La Plata - CC67 - 1900 La Plata - Argentina

• L. Mendoza - Zelis (97)

**Dpto de Fisica** - UNLP - CC 67 - 1900 La Plata - Argentine

### Australie

• A. Calka(1997)

**Dep. Materials Engineering** -University of Wollongong - NSW 2522 - Australie

• S.J. Campbell

**School of Physics**- University College - The University of New South Wales - Australian Defence Force Academy - Canberra ACT 2600 - Australie.

• Y. Chen (1997)

**Dep.Elec. Mat. & Eng.**- RSPHYSSE - The Australian Nat. Univ. - Canberra ACT 0200 -

• J. Harrowfield (1998)

**Chemistry Department- Un. of Western Australia**- Nedlands - WA 6907 - Australie

• F.J. Lincoln

**Special Research Centre for Advanced Mineral and Materials Processing**

Univ. Western Australia - Nedlands, Perth - Western Australia 6907 - Australie

• J. Nikolov

The Australian National University - Canberra ACT 0200 - Australie.

• W. Kaczmarek

**Dpt Appl. Mathematics** - Institute of Advanced Studies - Research School of Physical Sciences & Engineering - The Australian National University - Canberra ACT 0200 - Australie.

### Brésil

• W.J. Botta - Filho

**Dept. Eng. Mater.**-Univ. Federal Sao Carlos - CP 676 - 13565 - 090 - Sao Carlos Sp. - Brésil

• A. de Matos Dias

**Materials Science & Eng. Dpt**- Universidade Luterana do Brasil - Canoas - RS - Brésil

• R. S. de Figueiredo(97)

**Lab. Magn.& Mat. Magn.**- Dep. de Fisica - Cxp 6030 - 60.455 - 760 Fortaleza CE - Bresil

### Canada

• J. Huot\*

**IREQ - Techn. des Matériaux** -1800 Boul. L. Boulet - Varennes, Quebec, Canada J3X ISI

• J.-Y. Huot

**Noranda Technology Centre**- 240 Hymus Blvd - Pointe Claire - Que, H9R 1G5 - Canada

• L. Roué (1998)

**INRS - En. & Mat.**-1650 Bd Lionel Boulet - Case Postale 1020 -Varennes (Québec) J3X 1S

A. Van Neste

Mines & Métallurgie - Un. de Laval - Pav. Pouliot, Ste - Foy Campus - GIK 7P4 - Quebec - Canada

• W.J. D. Shaw

Dept. Mech. Eng. - University Calgary - T2N 1N4 - Calgary Alberta - Canada

### Chine

• J. Li

**Dept Materials Science** - Lanzhou University - Lanzhou 73000 - Chine

• K. Lu

State Key Lab for RSA - Institute of Metal Research - Chinese Academy of Sciences - Shenyang 110015 - P.R. Chine

• L. Wei

Institute of Metal Research - Chinese Academy of Sciences - Shenyang, 110015 - P.R. China

### Corée du Sud

• J.-H. Ahn

**Dept. Materials Engineering - Andong National University**

388 Songchon - Dong, Andong, Gyungbuk 760 749 - Corée du Sud

• S. H. Hong

**Dept. Mat. Sci. & Eng. - Korean Advanc. Inst. of Science and Technology**

373 - 1 Kusong - Dong, Yusung - Gu - Taejon, 305 - 701 - Corée du Sud

### Croatie

• M. Stubicar (1998)

**Department of Physics**- Faculty of Science, P.O. Box 162 - 10001 Zagreb - Croatie

• Andjelka. Tonejc

**Dpt Physics** - Lab. Microstr. Investig. - Bijenicka 32 - PO Box 162 - 10001 Zagreb - Croatie

• Antun Tonejc

**Dpt Physics** - Lab. Microstr. Investig. - Bijenicka 32 - PO Box 162 - 10001 Zagreb - Croatie

### Danemark

• J. Z. Jiang

**Dept Physics** - Tech. Univ. Denmark - Bldg 307 - DK 2800 - Lyngby - Danmark

### Espagne

- P. Crespo(1997)

CENIM - CSIC, Avda G de Amo, 8 - 28040 - Madrid Espagne

## Grèce

- G. Kiriakidis

**Materials Group** - Institute of Electronique Structure and Laser (IESL)  
Foundation for Research and Technology - Hellas (Forth)  
Science & Technology Park of Crete - Vassilika Vouton, Heraclion Crete  
P.O Box 1527 GR - 71110 - Grèce

## Hongrie

- T. Kemeny (1997)
- L.K. Varga

Research Institute for Solid State Physics - 1525 Budapest - P.O. Box 49 - Hongrie  
Research Institute for Solid State Physics - P.O.B. 49 - H- 1525 - Hongrie

## Inde

- B.S. Murthy

Dpt Metallurgical & Materials Engineering - Indian Institute of Technology -  
Kharagpur - 721 302 - Inde

## Israel

- M.P. Dariel (1998)
- N. Frage
- A. Gedanken
- E. Gutmanas

**Dept. Materials Engineering**- Ben Gurion University of the Negev - Beer Sheva - Israel  
**Dept. Materials Engineering**- Ben Gurion University of the Negev - Beer Sheva - Israel  
**Dpt of Chemistry**- Bar - Ilan University - Ramat - Gan; Israel 52900  
**Technion** - Israel

## Italie

- D. Basset (1998)
- S. Enzo
- M. Magini (1997)
- P. Pierrat\*

M.B.N. srl - Via Roma - 4 - I31020 - San Vendemiano (TV) Italie  
**INFN & Dipartimento di Chimica**- Univ. Sassari - Via Vienna 2 - 07100 Sassari - Italie  
**ENEA** - Dipart INNOVAZIONE - C.R. Casaccia - Via Anguillarese, 302 - I00060 Rome - Italie  
**Dipartimento di Scienze e Technologie Chimiche**  
Università degli Studi di Udine - Via del Cottonificio 108 - 33100 Udine - Italie

## Japon

- J.Y. Huang
- M. Senna
- M. Umemoto

Nat. Inst. Research in Inorganic Materials (NIRIM) - Namiki 1 - 1, Tsukuba, Ibaraki -305 Japon  
Keio Univ.-Fac. Sci. Tech. -Dpt. Appl. Ch.- 3-14-1 Hiyoshi Kohoku-ku-223 Yokohama - Japon  
**Fac. Engin.** - Toyohashi Univ. Technology - Tempaku - Cho Toyohashi Aichi 441 - Japon

## Pologne

- T. Janfa
- D. Oleszak
- B. Weglinski

**Technical University of Wroclaw**- Inst. of Electric Machines & Drive - Wybrzeze Wyspianskiego  
27 - Wroclaw 50 - 370 - Pologne  
**Dept Mater. Sci.& Eng.**- Warsaw Univ. Techn. - Nabutta 85 - 02 - 524 - Pologne  
**Technical University of Wroclaw**- Inst. of Electric Machines & Drive - Wybrzeze Wyspianskiego  
27 - Wroclaw 50 - 370 - Pologne

## Portugal

- B. Oliveira e Costa (97)

**Dpto de Fisica** -Faculdada de Ciencias et Tecnologia - Universidade de Coimbra -  
3000 Coimbra - Portugal

## Roumanie

- M. Lozovan

National Institute of R&D for Technical Physics, 47 Mangeron Blvd, 6600 IASI 3 - Roumanie.

## Russie

- N.Z. Lyakhov
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- A. Y. Yermakov
- A.Y. Zubarev

**Inst. Sol. State Chem**- Russian Acad Sci. - Kutaleladze, 18 - Novosibirsk - 630128 Russia  
**Ufa State Aviation Technical University**- Inst. of Physics of Advanced Materials -  
12 K. Marks Str., UFA 450000 - Russie  
**Inst. Chem. Phys.**- RAS, Dept Kinetics & Catalysis - Kosygina Str. 4 - Moscou - Russie  
**Applied Magnetism Lab** - InstituteMetal Physics - 18 S. Kovalevskaya St. - GSP -  
170 - Ekateringurg - 620219 - Russie  
USU - Russie

## Singapour

- Lu Li

Dpt Mechanical and Production Engineering - The National University of Singapore -  
10 Kent Ridge Crescent - Singapore - 119260 - Singapour

## Slovaquie

- K. Kristiakova

Institute of Physics - Dubravska Cesta 9 - SK - 842 28 Bratislava - Slovaquie

## Suède

- L.B. Kiss
- A. Salwen
- S.J. Savage

Nanomaterials and Noise Projects - Dpt Materials Science - Ångström Lab. Uppsala Univ.  
P.O. Box 534 - Uppsala, SE - 75121 - Suède  
**Swedish Inst. Metals Res.**- Drottning Kristinas V. 48 - Stockholm S 114 - 28 - Suède  
**Dept of Materials** - Defence Research Establishment - SE - 172 90 - Stockholm - Suède

## U.S.A.

- P. Bellon
- E. Y. Ivanov
- J.N. Newkirk
- R.E. Riman
  
- L. Takacs

**Dpt Materials Science and Engineering-** 1304 W. Green St. - Urbana IL 61801 - USA  
**Tosoh SMD Inc,** 3600 Gantz Road, Grove City - Ohio 43123 USA  
**Dept Metallurgical Eng.-** Univ. Missouri - Rolla - Rolla MO 65409 USA.  
Dept Ceramic and Materials Engineering - Rutgers University -  
P.O. Box 909 - Piscataway, NJ 08855 - 0909 USA  
Univ. Maryland - Baltimore Country - Dpt Physics - 1000 Hilltop Circle - USA

## Viet Nam

- Pham Khac Hung

ITIMS - 1 Round, Dai Co Viet, Hanoi Viet Nam

## France

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**Lab. Thermodyn. Trait. Poudres-** Fac. Sciences - 2 Bd Lavoisier - F49045 - Angers Cedex  
**Roucaire Instruments Scientifiques -2,** Avenue du Pacifique, Les Ulis - BP 78  
F91943 Courtaboeuf Cedex  
**LCRS** - 33 Rue de Saint Leu - F80039 - Amiens Cedex  
**LRRS** - CNRS UMR 5613 - Equipe "Matériaux à Grains Fins" - Université de Bourgogne  
UFR Sciences et Techniques - 9 Avenue Alain Savary - BP400 - F21011 Dijon Cedex  
**LMCTS ESA 6015-** Fac des Sciences 123 Avenue A. Thomas - F87060 Limoges Cedex  
**ENSCI** - 47 Avenue A. Thomas - F87065 Limoges Cedex  
**LSG2M- CNRS** Ecole des Mines - F54042 - Nancy Cedex  
**LRRS** - CNRS UMR 5613 - Equipe "Matériaux à Grains Fins"  
Faculté des Sciences de Mirande - BP 138 - F21004 - Dijon Cedex  
**Saft Recherche** - Route de Nozay - F91460 Marcoussis  
ICMCB - Bordeaux  
**CREPI - PSA** - Direction des Methodes et Equipements Industriels - Batiment Forge  
Route de Chalampé - BP 1403 - F68071 Mulhouse Cedex  
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**LRRS** - CNRS UMR 5613 - Equipe "Matériaux à Grains Fins" - Université de Bourgogne  
UFR Sciences et Techniques - 9 Avenue Alain Savary - BP400 - F21011 Dijon Cedex  
**Lab. Métal. Phys.-** URA CNRS 131 - Bd 3, Téléport 2 - BP 179 -F86960 - Futuroscope Cedex  
**LRRS** - CNRS UMR 5613 - Equipe "Matériaux à Grains Fins" - Université de Bourgogne  
UFR Sciences et Techniques - 9 Avenue Alain Savar - BP400 - F21011 Dijon Cedex  
**Schneider Electric** - Dir. Rech. Matériaux/A3 - Rue Henri Tarze - F38050 - Grenoble Cedex 9  
CNRS - Lab de Chimie Métallurgique des Terres Rares - 2 - 8 Rue H. Dunant - F94320 Thiais  
LASIR - HEI (CNRS UPR 2631) - 13 Rue de Toul - F59046 Lille Cedex  
**Lab. de Structure des Mat. Métal.-** Bât. 413 - 414 - Un. Paris Sud - F91405 - Orsay Cedex  
**Ecole des Mines d'Albi** - Campus Jarlard - F81013 Albi Cedex 04  
**LMIT** - IUT Belfort - BP 527 - 90016 - Belfort Cedex  
**Laboratoire de ThermoMécanique-** IPSé - F90010 - Belfort Cedex  
**Lab. Métal. Phys.-** URA CNRS 131 - Bd 3, Téléport 2 - BP 179-F86960 - Futuroscope Cedex  
**Laboratoire de ThermoMécanique-** IPSé - F90010 - Belfort Cedex  
**Lab. Magn. & Appl.-**URA 808 -Univ. Rouen-UFR Sci.& Tech-F76821 - Mt St Aignan Cdx3  
**Lab. de Métallurgie Physique**Univ. Lille 1 - Bat C6 - 2ème Et. -F59655 - Villeneuve d'ascq  
**LPMS - CNRS D0407-** Univ. Montpellier II - Sci. et Techn. du Languedoc  
Place E. Bataillon - F34095 - Montpellier Cedex 5  
**Lab. de Cristallographie** -UPR CNRS 5031 - BP 166 - F38042 - Grenoble Cedex  
**Lab. Thermodyn. Mét.-** URA CNRS 158 - Univ. Nancy I - B.P. 239-54506-Vandoeuvre Cdx  
**CNRS UPR 423**"Elab. et Transitions de Phases Hors Equilibre"-IPSé -F90010 - Belfort Cedex  
**Lab. Central de Recherches-** Thomson CSF Domaine de Corbeville - F91404 - Orsay  
**LSG2M- CNRS** Ecole des Mines - F54042 - Nancy Cedex  
Ecole des Mines - St Etienne - France  
Lab. Fluorures - UPRES CNRS A 6010 - Fac des Sciences - Av. O. Messiaen - 72985-Le Mans  
  
**LRRS** - CNRS UMR 5613 - Equipe "Matériaux à Grains Fins"- Université de Bourgogne  
UFR Sciences et Techniques - 9 Avenue Alain Savary - BP400F21011 Dijon Cedex  
**Eq. Physique de l'Etat Condensé** -Univ. du Maine - Fac Sciences - F72017 - Le Mans Cdx  
LETAM - Université de Metz - 57045 Metz Cedex 01  
**Lab. Chimie du Sol. Miréral - CNRS** - B.P. 239 - Vandoeuvre les Nancy Cedex  
**Université de Compiègne - Génie Chimique** -BP 529 - F60205 - Compiègne  
ENSMSE -**Lab. Physicochimie Matériaux** -158 Cours Fauriel - F42023 St Etienne Cdx  
**Lab. de Génie des Matériaux-** ISITEM - CP3023 - F44087 - Nantes cedex 03  
**LPMS - CNRS D0407-** Univ. Montpellier II - Sci. et Techn. du Languedoc  
Place E. Bataillon - F34095 - Montpellier Cedex 5  
**Lab. Tribologie & Dynamique des Systèmes** UMR CNRS 5513  
Dpt de Technologie des Surfaces - Ecole Centrale de Lyon, BP 163 - F69131 Ecully Cedex  
LMIT - Portes du Jura - F25000 Montbliard  
LCSM - URA CNRS 158 - Univ. H. Poincaré - Nancy I - F54506 Vandoeuvre Cedex  
**Eq. Physique de l'Etat Condensé** -Univ. du Maine - Fac Sciences - F72017 - Le Mans Cdx

- P. Lacorre(1998)  
Cdx  
Lab. Fluorures - UPRES CNRS A 6010 - Fac des Sciences - Av. O. Messiaen - 72985-Le Mans
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**N.B. :** Pour la rédaction du prochain N° de la Lettre du Réseau Français de Mécanosynthèse, tout(e) article, annonce, thèse ... peut être envoyé(e) à : Eric Gaffet - CNRS UPR A0423  
Groupe "Nanomatériaux : Elaboration et Transitions de Phases Hors Equilibre"  
IPSé - F90010 Belfort Cedex  
Tél. : 84 - 58 - 31 - 02 / Fax : 84 - 58 - 30 - 27

E-mail : Eric.Gaffet@utbm.fr