

# RESEAU FRANCAIS DE MECANOSYNTHESE

## Lettre N°40

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**Juillet 1998**

**121 Groupes de Recherches**  
(dont 59 (+9) à l'étranger)

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

### 10 Nouvelles Adhésions

#### **A. Gedanken - Israel**

**L.B. Kiss** - Dpt Materials Science - Uppsala - Suède

**K. KRISTIAKOVA** - Institute of Physics - Bratislava - Slovaquie

**S. Lenhard** - Univ. Metz - France

**J. Li** - Lanzhou University - Dpt Materials Science - Chine

**K. Lu** - State Key Lab for RSA - Chine

**M. Lozovan** - Nat. Inst. R&D for Technical Physics - Roumanie

**P. Reynders** - Merck - Allemagne

**P. Shashi** - De Montfort Univ. - Emerging Technologies Research Centre - Leicester - Royaume Uni

**A.Y. Zubarev** - U.S.U - Russie

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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

#### **4th Russian Conference on the Physics and Chemistry of Ultra Dispersed Systems"**

Obninsk - Russie - 29 Juin 3 Juillet

Contact : P.N. Martynov E-Mail : Sta@ippe.rssi.ru

#### **Journées Francophones des Jeunes Physico-Chimistes 7-9 juillet 1998 - Montpellier**

La 4e édition de ces journées multidisciplinaires propose aux étudiants et jeunes chercheurs un lieu de rencontre et d'échange autour de la Physico-Chimie. Trois grands thèmes seront abordés: - Biologie, Santé, Environnement;  
- Matière, Matériaux;

- Réactivité, Surfaces, Interfaces.

Chaque thème sera introduit par une personnalité scientifique, développé par une série de communications orales et complété par une séance de communications par affiche.

Responsables: Josette Olivier-Fourcade, Jean-Claude Jumas, Pierre-Emmanuel Lippens

Renseignements: Pierre-Emmanuel Lippens

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Tél.: 04 67 14 45 48 / Fax: 04 67 14 42 90 / E-mail : jfjpc@crit.univ-montp2.fr

Internet : <http://ubik.crbm.cnrs-mop.fr/jfjpc98/jfjpc-98.html>

#### **High Temperature Nanostructured Materials**

Gordon Conference - New Hampshire - USA - 19 - 24 Juillet 1998

Contact : Nanthan.S.Jacobson@lerc.nasa.gov

**EuroMAT'98**

Lisbonne - Portugal - 22 - 24 Juillet 1998

E-Mail : lfspm@lemac.ist.utl.pt

**Nouveau  
New**

**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

**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

**"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>

**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

**ESTAC 7 et EUROSOLID - 5**

Baltonfüred - 30 Août - 4 Septembre 1998

Contact : Prof. G. Liptay - Hungarian Chemical Society - Fö u. 68, Budapest - H - 1027 Hongrie

**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>

**Ninth International Symposium on Small Particles and Inorganic Clusters (ISSPIC 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

**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

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

Edinburgh - Ecosse - 12 Septembre 1998

Contact : h. Fissan@uni-duisburg.de ou 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**

**3rd 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, Taejeon, 305-701 Korea - Tel:82-(0)42-869-4113, 8919 / Fax: 82-(0)42-869-8920

E-mail : sjkang@sorak.kaist.ac.kr / 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/confs.htm>

**Nouveau  
New**

**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 :** Reh binder98rehb.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

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

194th Meeting of the Electrochemical Society

1 - 6 Novembre 1998 - Boston - MA - USA

<http://www.electrochem.org>

**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, TJ 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.iche.org](http://www.iche.org)

**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,

R. Schwarz, R.W. Siegel, M. Umemoto

**ISMANAM Steering Committee:**

J.H. Ahn, M.D. Baro, A. Calka, S. Gialanella, A. Inoue, G. Le Caer, D.G. Morris, P.H. Shingu, H. Bakker,

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](mailto:Andrzej_Calka@uow.edu.au) et

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

**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 (ICAM'99)**

Beijing - Chine - 31 Mai - 5 Juin 1999  
Contact : Kelu@imr.ac.cn

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**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. Dager, M. Broussely, P. Goudeau, E.L. Mathe, M. Grosbras

L'objectif de ce travail est d'apporter des améliorations au mélange cathodique (LiNiO<sub>2</sub> + Graphite + Suie) des cathodes des batteries à l'ion lithium. En premier lieu, nous avons cherché à augmenter la conductivité électrique macroscopique du mélange cathodique et du LiNiO<sub>2</sub> (matériau actif) par un broyage mécanique de ce dernier : le suivi des modifications morphologiques a été réalisé par granulométrie LASER, mesures de surface spécifiques BET, etc... Ensuite l'observation de la configuration microstructurale du LiNiO<sub>2</sub>, dans l'état morphologique conduisant à la meilleure percolation, nous a conduit à envisager un traitement thermique adéquat de la poudre broyée. Il est en effet primordial d'assurer non seulement une bonne conductivité électronique mais également une bonne diffusion des cations Li<sup>+</sup> dans la cathode lors du cyclage électrochimique. Les méthodes complémentaires de caractérisation sont la diffraction des rayons X en géométrie symétrique et la microscopie électronique en transmission.

Les principaux résultats obtenus sont les suivants :

- Une étude préliminaire de la microstructure des cathodes a montré que les grains de LiNiO<sub>2</sub> étaient micrométriques, qu'ils présentaient des dislocations et qu'en cours de cyclage, ils étaient bordés d'une zone appauvrie en lithium,
- Le broyage du LiNiO<sub>2</sub> avant mélange permet d'améliorer la percolation électrique macroscopique du mélange cathodique associé. En utilisant des conteneurs métalliques, il est possible de transférer des éléments d'addition à la poudre. Pour éviter toute pollution du matériau, susceptible d'influer sur ses propriétés électriques, les broyages de poudres utilisées pour préparer des cathodes ont été réalisés dans des conteneurs en agate. On montre que la diminution de la résistivité de la poudre en fonction du temps de broyage est due majoritairement à l'augmentation de sa surface spécifique.
- Cependant, le broyage modifie la microstructure des grains de LiNiO<sub>2</sub> :
  - il est possible de désordonner la structure cristalline par modification de la répartition des cations Lithium et Nickel sur leurs sites,
  - les domaines cohérents de diffraction deviennent nanométriques ce qui doit faciliter la diffusion intergranulaire des cations Li<sup>+</sup>,
  - parallèlement des microdistorsions importantes du réseau apparaissent au sein de ces domaines. Ces microdéformations constituent vraisemblablement un handicap au déplacement intragranulaire des Li<sup>+</sup>.

- Un traitement thermique sous Argon permet de réarranger les dislocations en sous - joints et de relaxer fortement les microdéformations introduites lors du broyage sans modifier la taille des domaines de diffraction.
- Divers mélanges cathodiques ont été sélectionnés pour préparer des piles - bouton soumises à des cyclages galvanostatiques (charge et décharge). Les performances en puissance et en cyclabilité sont nettement augmentées dans le cas d'une pile où le LiNiO<sub>2</sub> broyé et recuit possède une morphologie optimum et une microstructure idéale (nanodomains, microdistorsions relaxées).

L'ensemble de ces résultats montre les rôles indissociables des configurations macroscopiques et microscopique du LiNiO<sub>2</sub> sur les performances des batteries. Les phénomènes qui se produisent lors des divers traitements subis par la poudre ouvrent la voie à de nouveaux champs d'investigation scientifique et à plusieurs applications industrielles (téléphones mobiles miniaturisés, véhicules électriques plus autonomes, etc...)

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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., Rouzaud 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 électroniques 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

=====  
**"Contribution à l'Etude de la Transformation - Tribologique Superficielle en Fretting"**

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

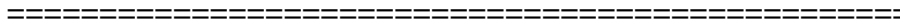
**Jury** : L. Mora - Ponsonnet, P. Blanchard, K. Dang Van, C. Esnouf, E. Gaffet, E. Rosset, A.B. Vannes, L. Vincent

## 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)

#### [34] IMPROVED P/M SILVER-ZINC OXIDE ELECTRICAL CONTACTS

Joshi PB. Krishnan PS. Patel RH. Murti NSS. Gadgeel VL. Ramakrishnan P. - International Journal of Powder Metallurgy. 34(4):63 ff., 1998

Electrical contact grade Ag-ZnO composite powders have been produced by three processing routes: conventional mixing/blending of silver and zinc oxide powder particles; coprecipitation; and mechanical alloying. The powders were characterized in terms of apparent density, tap density, flow rate, particle size, size distribution and also phase analysis by x-ray diffraction. They were then pressed sintered repressed and hot-pressed to near theoretical density. Sintered density, hardness, electrical conductivity and microstructure of the sintered compacts we've assessed. The mechanically alloyed compacts were also subjected to electrical performance evaluation via International Electrotechnical Commission's Standard IEC 947-4-1. Mechanical Alloying has been found to result in an excellent combination of physical microstructural and electrical contact properties.

#### [33] THE STRENGTHENING EFFECT OF AL<sub>3</sub>Ti IN HIGH TEMPERATURE DEFORMATION OF AL-AL<sub>3</sub>Ti COMPOSITES

Wang SH. Kao PW. - Acta Materialia. 46(8):2675-2682, 1998

A series of Al-Al<sub>3</sub>Ti composites with systematic variation of Al<sub>3</sub>Ti content were prepared by mechanical alloying. Microstructural observations have indicated that among these composites, the only distinct variable is the Al<sub>3</sub>Ti content, while the other microstructural variables are essentially the same. The high temperature (623-773 K) deformation behavior of these composites was found to be similar to that of dispersion-hardened aluminum. By considering the presence of a threshold stress, the plastic flow in these composites can be described by lattice-diffusion controlled dislocation creep in the aluminum matrix with a "constant structure". The presence of Al<sub>3</sub>Ti particles can increase the creep strength of these alloys significantly. By considering the load-sharing effect of Al<sub>3</sub>Ti, an analysis based on continuum mechanics approach has been conducted, which can successfully account for the creep rate of these Al-Al<sub>3</sub>Ti composites. The threshold stress for creep in these composites was found to increase with increasing Al<sub>3</sub>Ti content, which could be attributed to the load-sharing effect of Al<sub>3</sub>Ti particles. Similarly, by considering the load-sharing of SiC particles, the proposed analysis can also account for the volume fraction effect of SiC on the threshold stress for creep in Al-SiC composites.

#### [32] OXALATE COPRECIPITATION OF DOPED CERIA POWDER FOR TAPE CASTING

Vanherle J. Horita T. Kawada T. Sakai N. Yokokawa H. Dokiya M. - Ceramics International. 24(3):229-241, 1998

Twenty cation per cent yttria doped ceria Ce<sub>0.8</sub>Y<sub>0.2</sub>O<sub>1.9</sub> was prepared by an optimised oxalate coprecipitation route to give fine sinterable powder. This was formulated into slurries for tape casting of the doped ceria solid electrolyte used in solid oxide fuel cells. Prior dry milling of the powder was found to be most effective to further reduce the average agglomerate size, which allowed to sinter dense membranes at 1400 degrees C for 2 h, among the best results obtained so far for tape cast doped ceria. Ball milling of the powder, composition and viscosity of the slurry as well as sintering conditions of different tapes are discussed.

#### [30] THE STRENGTHENING EFFECT OF AL<sub>3</sub>Ti IN HIGH TEMPERATURE DEFORMATION OF AL-AL<sub>3</sub>Ti COMPOSITES

Wang SH. Kao PW. - Acta Materialia. 46(8):2675-2682, 1998

A series of Al-Al<sub>3</sub>Ti composites with systematic variation of Al<sub>3</sub>Ti content were prepared by mechanical alloying. Microstructural observations have indicated that among these composites, the only distinct variable is the Al<sub>3</sub>Ti content, while the other microstructural variables are essentially the same. The high temperature (623-773 K) deformation behavior of these composites was found to be similar to that of dispersion-hardened aluminum. By considering the presence of a threshold stress, the plastic flow in these composites can be described by lattice-diffusion controlled dislocation creep in the aluminum matrix with a "constant structure". The presence of Al<sub>3</sub>Ti particles can increase the creep strength of these alloys significantly. By considering the load-sharing effect of Al<sub>3</sub>Ti, an analysis based on continuum mechanics approach has been conducted, which can successfully account for the creep rate of these Al-Al<sub>3</sub>Ti composites. The threshold stress for creep in these composites was found to increase with increasing Al<sub>3</sub>Ti content, which could be attributed to the load-sharing effect of Al<sub>3</sub>Ti particles. Similarly, by considering the load-sharing of SiC particles, the proposed analysis can also account for the volume fraction effect of SiC on the threshold stress for creep in Al-SiC composites.

#### [29] THE INFLUENCE OF MECHANICAL ALLOYING ON THE SYNTHESIS OF Ni-3(Si, Ti) INTERMETALLICS

Vandyck S. Delaey L. Froyen L. Buekenhout L. - Acta Materialia. 46(8):2831-2840, 1998

Reactive powder metallurgy offers an interesting alternative technology for the production of nickel silicide based intermetallic materials. The heat release during reaction and the microstructure of the product can be controlled by mechanical alloying of the reactants. In order to determine the optimal processing conditions for mechanical alloying, the relationships among the processing conditions, the microstructure of the mechanically alloyed powder and the reaction mechanism have been identified. This was done with the aid of a mathematical description of the mechanical

alloying process in a planetary ball mill, which allowed the prediction of the hardness and deformation of the material as a function of the milling conditions. Experimental verification under a broad range of conditions shows a good agreement between the measured and predicted hardness values. The relation between microstructure and milling conditions was quantitatively expressed as an inverse exponential relation between the calculated strain and the crystallite size of the material. The mechanism of synthesis of the Ni-3(Si, Ti) phase in the mechanically alloyed powder was studied. It is shown that the transition of a high-temperature self propagating reaction in the unmilled powder to a low temperature solid-state reaction depends on the crystallite size of the milled powder. By using the relations among milling conditions, microstructural characteristics and reaction behavior of the mechanically alloyed powder, milling maps with "equivalent milling conditions" were calculated. These milling maps represent the sufficient conditions to obtain the desired level of strain in the material. By using these maps, the experimental work for optimization of the mechanical alloying process can be greatly reduced.

**[28] POWER MEASUREMENTS DURING MECHANICAL MILLING - II - THE CASE OF SINGLE PATH CUMULATIVE SOLID STATE REACTION**

Magini M. Colella C. Iasonna A. Padella F. - *Acta Materialia*. 46(8):2841-2850, 1998

The electrical power consumptions during the milling process of a planetary mill have been measured. The results of the experiments, carried out with a suitable chosen system, are perfectly in line with previous milling experiments. The dependence of the power consumption during milling from the third power of the rotation speed and the effect of the filling of the milling device on the energy transfer and power consumption have been clearly established and confirmed. The relevance of in situ data acquisition during milling, that can disclose a deeper understanding of the mechanical alloying process, has been discussed within the frame of present and previous results.

**[27] TWO-PHASE COEXISTENCE IN FE-CU ALLOYS SYNTHESIZED BY BALL MILLING**

Hong LB. Fultz B. - *Acta Materialia*. 46(8):2937-2946, 1998

Mechanical alloying with a Spex 8000 mixer/mill operated at two intensities was used to synthesize a series of Fe<sub>100-x</sub>Cu<sub>x</sub> alloys with Cu concentrations from x = 0 to x = 49. X-ray diffractometry was used to measure the volume fractions of the b.c.c. and f.c.c. phases in the alloys. Mossbauer spectrometry was used to determine Cu concentrations in the b.c.c. phase, and identify inhomogeneities in Cu concentration in the b.c.c. phase. With higher milling intensity, there was a narrowing of the range of compositions for f.c.c. plus b.c.c. coexistence, and a shift towards Fe-rich compositions. The composition range of two-phase coexistence is understood in terms of heterogeneities in both defect density and concentration.

**[26] AMORPHIZATION TENDENCY OF Ti<sub>34</sub>Zr<sub>11</sub>Cu<sub>47</sub>Ni<sub>8</sub> AND Ti<sub>37</sub>Zr<sub>17</sub>Cu<sub>42</sub>Ni<sub>4</sub> DURING MECHANICAL ALLOYING**

Liu XD. Nagumo M. Umemoto M. - *Materials Transactions Jim*. 39(3):343-350, 1998

Pure elemental powders of Ti, Zr, Cu and Ni corresponding to the compositions of Ti<sub>34</sub>Zr<sub>11</sub>Cu<sub>47</sub>Ni<sub>8</sub> and Ti<sub>37</sub>Zr<sub>17</sub>Cu<sub>42</sub>Ni<sub>4</sub> (at%) were subjected to mechanical alloying on a low energy milling machine in the present work. The premixed powders of the above two compositions underwent the following transformations during mechanical alloying up to 100 h: μm-sized (Ti, Zr, Cu, Ni) → nm-sized (Ti, Zr, Cu, Ni) → amorphous + a small amount of nm-sized crystallites. The 100-h milled Ti<sub>34</sub>Zr<sub>11</sub>Cu<sub>47</sub>Ni<sub>8</sub> sample was found to exhibit an excellent thermal stability against crystallization with the crystallization temperature of about 200 K higher than that of the as-quenched amorphous counterpart. Contrary to the previous rapid quenching result, amorphous phase is easier to occur in Ti<sub>37</sub>Zr<sub>17</sub>Cu<sub>42</sub>Ni<sub>4</sub> than in Ti<sub>34</sub>Zr<sub>11</sub>Cu<sub>47</sub>Ni<sub>8</sub> by MA, indicating that the glass forming ability by rapid quenching is different from that by MA.

**[25] THE CHEMOMECHANICAL EFFECT AND THE MECHANOCHEMICAL EFFECT ON HIGH-PERFORMANCE CONCRETE SUBJECTED TO STRESS CORROSION**

Schneider U. Chen SW. - *Cement & Concrete Research*. 28(4):509-522, 1998

This paper describes the behavior of high-performance concrete under both chemical attacks and mechanical stresses. The specimens of the concrete C80 and C95 were subjected to flexural loads with a level of 30% of their initial strengths and immersed into a 5% ammonium sulfate solution, a 10% ammonium nitrate solution, and water saturated with Ca(OH)<sub>2</sub>, respectively. The development of strength of the concrete was determined at certain time intervals. The simultaneous action of corrosive media and mechanical stresses on the concrete leads to a stress corrosion effect. The difference of strength of the specimens, which were immersed in the water saturated with Ca(OH)<sub>2</sub> and the salt solutions and subjected to the same loading conditions, is defined as a chemomechanical effect (CME). The difference of strength of the unloaded and loaded specimens, both of which were immersed in the same salt solutions, is defined as a mechanochemical effect (MCE). The CME and the MCE of the concrete are discussed in the present paper.

**[24] FULL DENSIFICATION OF LOOSELY PACKED W-CU COMPOSITE POWDERS**

Moon IH. Kim EP. Petzow G. - *Powder Metallurgy*. 41(1):51-57, 1998.

The metal injection moulding (MIM) technique appears to have potential for mass production of small and delicately shaped W-Cu parts, primarily for microelectronics, although problems in densification, owing to the inherently poor sinterability of the W-Cu system as well as the low volume fraction of MIM debound parts, need to be overcome. Several attempts have been made to attain full densification of the loosely packed W-Cu composite powders in MIM brown parts. The use of the nanocomposite W-Cu powders, prepared either by co-reduction of fine W and Cu oxide powder or by mechanical alloying of W and Cu powders, was shown to enhance the sinterability, resulting in the full densification of W-Cu MIM parts. Similar effects were observed in the case of tailored W-Cu powder mixtures with high mixing homogeneity and packing density. The high sinterability of the pertinent W-Cu systems is attributed partly to the nanosintering effect, owing to the intrinsic homogeneous and fine mixed state of the W-Cu phases, and partly to activated sintering which is promoted by impurities introduced during the extensive milling process.

**[23] MECHANOCHEMICAL TREATMENT OF ALPHA-Fe<sub>2</sub>O<sub>3</sub> POWDER IN AIR ATMOSPHERE**

Zdujic M. Jovalekic C. Karanovic L. Mitric M. Poleti D. Skala D. - *Materials Science & Engineering A-Structural*

Materials Properties Microstructure & Processing. 245(1):109-117, 1998

Powder of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> was mechanochemically treated in a planetary ball mill in an air atmosphere. Structural changes were followed by X-ray diffraction analysis, magnetization measurements and differential scanning calorimetry after various milling times. It was found that complete transformation of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> to Fe<sub>3</sub>O<sub>4</sub> is possible during milling in an air atmosphere under appropriate milling conditions. Presumably, the decrease in the oxygen partial pressure during milling has a critical influence on promoting the dissociation of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>. Before nucleation of the Fe<sub>3</sub>O<sub>4</sub> phase, the crystallites of the  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> phase are reduced to a minimal size accompanied by the introduction of atomic-level strain. Local modeling of a collision event, coupled with a classical thermodynamic assessment of the Fe<sub>2</sub>O<sub>3</sub>-Fe<sub>3</sub>O<sub>4</sub> system, were used to rationalize the experimental results. It is proposed that the mechanochemical reactions proceed at the moment of impact by a process of energization and freezing of highly localized sites of a short lifetime. Excitation on a time scale of similar to 10<sup>(-5)</sup> s corresponds to a temperature rise of the order of (1-2) x 10<sup>(3)</sup> K. Decay of the excited state occurs rapidly at a mean cooling rate higher than 10<sup>(6)</sup> K s<sup>(-1)</sup>.

#### [22] MICROSTRUCTURAL EVOLUTION IN MELT-QUENCHED AMORPHOUS SE DURING MECHANICAL ATTRITION

Guo FQ. Lu K. - Physical Review B-Condensed Matter. 57(17):10414-10420, 1998

Microstructural evolution of an as-quenched amorphous Se sample during ball milling was investigated by use of x-ray diffraction, a differential scanning calorimeter, infrared and Raman spectroscopy, as well as x-ray photoemission spectroscopy. It was found that the as-quenched amorphous Se is crystallized completely into a nanocrystalline trigonal Se phase after milling for 30 min. Further milling of the as-milled nanocrystalline trigonal Se resulted in a transformation into another amorphous Se phase, which exhibits a lower crystallization temperature and a smaller crystallization enthalpy compared to those for the as-quenched amorphous Se according to thermal analysis. Infrared and Raman spectroscopy measurements revealed that the as-milled amorphous Se is mainly composed of Se-n polymeric chains that are fundamentally different from the as-quenched amorphous Se in which the Se-8 ring structure is predominant. However, despite the phase evolution and changes in molecular structure in Se, x-ray photoemission measurements indicated that no detectable changes in electronic properties (e.g. the density of valence states, the binding energies of core levels, and the characteristic energy loss functions) were observed among different states of Se samples.

#### [21] NMR STUDY OF ORDERING KINETICS IN Ni<sub>3</sub>Al ALLOYS

Scherrer P. Dimitropoulos C. Borsa F. Rubini S. - Physical Review B 57(17):10462-10469, 1998

Al-27 nuclear magnetic resonance (NMR) and relaxation has been applied to the study of the effects of ball milling on the long-range order (LRO) of fcc L1(2) Ni<sub>3</sub>Al as well as to the subsequent microscopic reordering process through annealing. Structural changes due to the mechanical treatment are correlated to the appearance of a strong magnetization in the disordered phase. In the absence of significant Fe contamination, this magnetic property is attributed to the existence of magnetic moments of the order of 0.23  $\mu$ (B) ( $\mu$ (B) = Bohr magneton) localized at the Ni sites. Al-27 NMR spectra in both the ordered and the disordered phase are presented. The random distribution of atoms combined with the magnetic properties in the disordered phase cause a substantial broadening of the Al-27 NM line. The linewidth is proportional to the fractional change of disorder and is therefore used to monitor the ordering transformation as a function of annealing time and temperature. The changes of spin-lattice relaxation rates (T<sub>1</sub><sup>(-1)</sup>) and Knight shifts during transformation are also examined. The overall ordering behavior as observed by NMR is described in terms of a stretched exponential for the time dependence of the untransformed fraction, implying a time-dependent transformation rate. The activation energy of the Ni vacancy migration mechanism responsible for the transformation was determined to be E = 1.8 eV +/- 0.2 eV. The average distance covered by the atoms during the ordering is estimated by means of a simple random-walk model.

#### [20] STRENGTH OF SINTERED ALUMINA AT LOW TEMPERATURE DUE TO BALL MILL ABRASION POWDER SEEDING

Yoshizawa Y. Toriyama M. Kanzaki S. Saito F. - Nippon Seramikkusu Kyokai Gakujutsu Ronbunshi-Journal of the Ceramic Society of Japan. 106(4):444-446, 1998

Sinterability of low-temperature calcined  $\alpha$ -alumina powder and the bending strength of sintered bodies were investigated. The  $\alpha$ -alumina powder was made from commercial aluminum hydroxide with seeding of abrasion powder worn in the wet ball milling. The low-temperature calcined  $\alpha$ -alumina powder was very fine and achieved the density of more than 3.8 Mg/m<sup>(3)</sup> by hot pressing at 1200 degrees C. The microstructure of the sample sintered at low temperature was composed of very fine equiaxial grains in spite of low purity. The bending strength of this sample was more than 800 MPa.

#### [19] CATALYSIS OF HYDROTALCITE-LIKE COMPOUNDS IN LIQUID PHASE OXIDATION - (II) - OXIDATION OF P-CRESOL TO P-HYDROXYBENZALDEHYDE

Liu YM. Liu ST. Zhu KZ. Ye XK. Wu Y. - Applied Catalysis A-General. 169(1):127-135, 1998

Hydrotalcite-like compounds (HTLcs): CoMAlCO<sub>3</sub>-HTLcs (M=Cu<sup>2+</sup>, Ni<sup>2+</sup>, Mn<sup>2+</sup>, Cr<sup>3+</sup>, Fe<sup>3+</sup>), were synthesized by coprecipitation and characterized with XRD and IR. The catalysis of these HTLcs and their calcined products were studied in the p-cresol oxidation, and the effects of the temperature of HTLcs calcination, the ratio of Co/Cu, different promoters, reaction temperatures and reaction times on reaction activities were investigated. It has been found that calcined HTLcs have higher activity than uncalcined samples and mechanical mixed oxides in this reaction. The best yield was obtained from the CoCuAlCO<sub>3</sub>-HTLc (Co/Cu/Al=3:1:1) calcined at 450 degrees C. A tentative reaction mechanism was also proposed.

#### [18] SUPERACIDS BY METAL OXIDES, IX - CATALYSIS OF WO<sub>3</sub>/ZRO<sub>2</sub> MECHANICALLY MIXED WITH PT/ZRO<sub>2</sub> FOR REACTION OF BUTANE TO ISOBUTANE

Hino M. Arata K. - Applied Catalysis A-General. 169(1):151-155, 1998

An active catalyst for the skeletal isomerization of butane to isobutane was obtained by mechanically mixing

WO<sub>3</sub>/ZrO<sub>2</sub> and Pt/ZrO<sub>2</sub>. WO<sub>3</sub>/ZrO<sub>2</sub> was prepared by impregnation of Zr(OH)<sub>4</sub> with aqueous ammonium metatungstate, followed by calcination in air at 700 degrees C (15 wt% W); Pt/ZrO<sub>2</sub> was prepared by impregnation of the hydroxide with aqueous H<sub>2</sub>PtCl<sub>6</sub> · 6H<sub>2</sub>O, followed by calcination at 700 degrees C (0.5 wt% Pt). The effect of mixing was specific to Pt/ZrO<sub>2</sub>; no effectiveness was found for Pt/TiO<sub>2</sub>, Pt/SiO<sub>2</sub>, Pt/Al<sub>2</sub>O<sub>3</sub>, Pt/Fe<sub>2</sub>O<sub>3</sub>, and Pt/SnO<sub>2</sub>. Pt-WO<sub>3</sub>/ZrO<sub>2</sub> prepared by co-impregnation of zirconia with the W and Pt materials showed quite low activity; the first impregnation with Pt followed by W gave an activity value much higher than that obtained by the reverse order. XPS of Pt/ZrO<sub>2</sub> showed the Pt species to be cationic, while those of Pt/TiO<sub>2</sub>, WSiO<sub>2</sub>, and Pt-WO<sub>3</sub>/ZrO<sub>2</sub> were metallic.

**[17] EFFECT OF POWDER DISPERSION ON THE (1)A(1)REVERSIBLE-ARROW-T-5(2) SPIN TRANSITION IN FE(II) COMPLEXES WITH 4-AMINO-1,2,4-TRIAZOLE**

Varnek VA. Lavrenova LG. - Journal of Structural Chemistry. 38(5):742-747, 1997

A series of Fe(II) complexes with 4-amino-1,2,4-triazole ground in an agate mortar for 10 min is studied by Mossbauer spectroscopy. Strong effects of powder dispersion both on the (1)A(1) reversible arrow T-5(2) spin transition and on the structure dynamic characteristics of the complexes are found. Thus at 295 K the high-spin form of Fe(II) appears in the samples or its fraction increases; the ionicity of Fe-N bonds and the extent of distortion of the octahedral environment of iron atoms for the low-spin phases of the complexes also increase. It is established that powder dispersion markedly affects the probability of the Mossbauer effect and the vibrational spectrum of the lattice of coordination compounds. For both the low- and high-spin phases of the complexes, it is reported that the vibrational spectrum is "softened." The main reason for these effects is supposed to be defectiveness rather than the size of the particles due to mechanical activation of the powder.

**[16] MECHANOSYNTHESIS MECHANISM OF TiC POWDERS**

Wu NQ. Lin S. Wu JM. Li ZZ. - Materials Science & Technology. 14(4):287-291, 1998

During ball milling of a powder mixture of elemental titanium and graphite, TiC is synthesised by a combustion reaction. The factors controlling the reaction kinetics have been investigated using X-ray photoelectron spectroscopy and differential thermal analysis. In the incubation period before a combustion reaction, the carbon atoms diffuse along the grain boundaries of Ti, resulting in the mixing of the reactants on a nanometre scale. A transitional beaded state (Ti...C) is formed, reducing the ignition temperature for a combustion reaction. In addition, a minimum adiabatic temperature of 1800 K is necessary for the occurrence of combustion during the mechanochemical process.

**[15] MECHANOCHEMICAL TREATMENT OF CaCO<sub>3</sub> FOR REDUCING HYDROPHILICITY**

Popa M. Popa AA. - Polymer-Plastics Technology & Engineering. 37(1):115-126, 1998.

The article discusses the modification of CaCO<sub>3</sub> hydrophilicity by mechanochemical methods, in view of its compatibilization with hydrophobic polymeric matrices. The observation was made that by ultrahigh-speed stirring of the solutions of polybutadiene and acrylic acid--in which CaCO<sub>3</sub> powder has been suspended--as well as by vibratory milling of CaCO<sub>3</sub> in the presence of isoprene, modifications in the hydrophilicity of the anorganic particles occur. The treatment's efficiency depends on two process parameters: duration and monomer/charge ratio.

**[14] CHEMICAL ORDER AND STRUCTURE OF THE MECHANICALLY MILLED Fe<sub>2</sub>Hf Laves PHASE**

Xia SK. Saitovitch H. Silva PRJ. Gomez JA. Assuncao FCR. Baggiosaitovitch E. - Journal of Physics-Condensed Matter. 10(15):3457-3466, 1998

The effects of mechanical milling on the chemical order and structure of the Fe<sub>2</sub>Hf C14 Laves phase have been studied by x-ray diffraction and Mossbauer spectroscopy. The results show that ball milling leads to a segregation of two phases. One, with a lower degree of chemical disorder, maintains the structure of the original intermetallic phase and the other, with a higher degree of chemical disorder, exhibits a disordered structure. At 4.2 K, the two phases exhibit similar hyperfine magnetic fields which are smaller than that of the original intermetallic phase, but much larger than that of the amorphous alloy with the same composition, prepared by sputtering deposition. Differing from the crystalline phase, the structurally disordered phase shows a Curie temperature lower than room temperature.

**[13] MECHANOCHEMICAL EFFECTS ON YTTRIUM-ALUMINUM GARNET**

Minkova N. Tzvetkov G. - Materials Letters. 35(1-2):135-138, 1998

The phase changes of yttrium-aluminum garnet as a result of mechanoactivation in a planetary ball mill have been studied. In addition to the structural changes the long term activation causes mechanolysis. The latter provokes the formation of YAlO<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub> and Y<sub>2</sub>O<sub>3</sub>. The subsequent treatment of activated samples with water and 18% HCl at 363 K leads to the conversion of similar or equal to 26% Of the garnet into a soluble form.

**[12] NITRIDING BEHAVIOR OF NANOCRYSTALLINE NdFe<sub>10.5</sub>VTi<sub>0.5</sub> COMPOUNDS**

Jin ZQ. Tang W. Zhang JR. Lu LY. Tang SL. Du YW. - J.Magnetism & Magnetic Materials. 185(1):71-76, 1998

The effects of nitrogenation on the structure and magnetic properties of nanocrystalline NdFe<sub>10.5</sub>VTi<sub>0.5</sub>N<sub>x</sub> compounds have been studied using X-ray diffraction and magnetic measurement. The introduction of nitrogen atoms results in a lattice expansion, which is larger in the basal plane than along the c-axis of the unit cell. After milling for 5 h. NdFe<sub>10.5</sub>VTi<sub>0.5</sub> compounds present an average grain size of about 20 nm. When nitriding the nanocrystalline compounds at 350 degrees C, a coercive force of 3.7 kOe is obtained although the nitrogenation is still not complete up to 40 degrees C. Upon nitriding at 450 degrees C, unmilled coarse-grained NdFe<sub>10.5</sub>VTi<sub>0.5</sub>N<sub>x</sub> compounds remain as the ThMn<sub>12</sub>-type structure; however, the milled nanocrystalline compounds exhibit a transition to amorphous phase. The amorphization mechanism has been explained from the thermodynamics point of view.

**[11] THE FORMATION OF Mg FERRITE BY MECHANICAL ALLOYING AND SINTERING**

Moustafa SF. Morsi MB. - Materials Letters. 34(3-6):241-247, 1998

The kinetics of Mg-ferrite formation during ball milling of iron and magnesium oxides powder have been examined. A mixture of Fe<sub>2</sub>O<sub>3</sub> and MgO powders corresponding to nominal composition of one mole each, was subjected to milling, using a stainless steel ball mill. The weight ratio between the steel balls and the powders was approximately 8:1. Powders, after different milling times, were examined by X-ray diffractometry and optical microscopy. For the mill-operating condition investigated, the particle sizes in the mill generally decreased with milling time, indicating a

greater tendency for particle fracture than agglomeration (welding). After a milling time of 23 h, Mg-ferrite was formed. The milled powders were sintered for 2 h at temperatures of 800, 1000, and 1300 degrees C. For comparison, unmilled powders were also sintered at the same temperatures and time. Complete formation of Mg-ferrite was obtained only after milling followed by sintering at 1300 degrees C for 2 h, while unmilled particles showed partial formation of Mg-ferrite when sintered at the same temperature for the same time.

**[10] STRUCTURAL AND ELECTROCHEMICAL PROPERTIES OF TI-RU-FE-O ALLOYS PREPARED BY HIGH ENERGY BALL-MILLING**

Yip SH. Guay D. Jin S. Ghali E. Vanneste A. Schulz R. - J. Materials Research. 13(5):1171-1176, 1998

The structural and electrochemical properties of the Ti-Ru-Fe-O system have been studied over the whole ternary metal compositional range, keeping constant the oxygen content at 30 at. %. The phase diagram was explored systematically by varying the composition of the material along one of the following axes: (i) constant Ru content of 16 at. %; (ii) constant Ti/Ru ratio of 2; (iii) constant Ti/Fe ratio of 1.6. For O/Ti ratios equal or below unity, the most prominent peaks observed in the x-ray diffraction patterns belong to a B2 structure. For O/Ti ratio larger than unity, stable titanium oxide phases are formed, which coexist with a cubic Fe-like or hcp-Ru like phases depending on the Fe/Ru ratio. Powder compositions with stoichiometry close to  $Ti_2RuFeO_2$  are of interest due to good electrocatalytic properties, long-term stability, and low Ru content.

**[9] MECHANICAL ALLOYING STUDIES IN THE GAMMA(Fe<sub>3</sub>Zn<sub>10</sub>) AND GAMMA(1)(Fe<sub>5</sub>Zn<sub>21</sub>) SINGLE AND MIXED PHASE COMPOSITIONS**

Jordan A. Liu ZT. Uwakweh ONC. - Journal of Materials Research. 13(5):1177-1185, 1998

Homogeneous or uniform crystalline materials are obtained following the ball milling of pure elemental powders of Fe and Zn in proportions to yield single phases Gamma(Fe)<sub>3</sub>Zn(10), Gamma(1)(Fe<sub>5</sub>Zn<sub>21</sub>), and Gamma + Gamma(1) mixed phase (Fe<sub>25</sub>Zn<sub>75</sub>). Differential scanning calorimetry (DSC) measurements of the as-milled materials show characteristic stages in the temperature range of 50-600 degrees C prior to establishing stable equilibrium. The activation energies determined from kinetic analyses range from 49 to 189 kJ/mole in these materials. A characteristic stage at 130 degrees C marking the distinct evolution of the Gamma and Gamma(1) phases from the intermediate or mixed phase composition is identified from XRD measurements. The identification of a unique Fe site with a quadrupole splitting (QS) of 1.5 mm/s in corroboration with x-ray diffraction (XRD) shows that this stage marks the onset of an in situ transformation prior to the distinct evolution of the homogeneous phases. The Mossbauer effect measurement of the as-milled materials are resolved in terms of four unique Fe sites with QS of 1.1, 0.241, 0.073, and 0.0772 mm/s.

**[8] FORMATION OF INTERMETALLIC NANOCOMPOSITES IN THE TI-AL-SI SYSTEM BY MECHANICAL ALLOYING AND SUBSEQUENT HEAT TREATMENT**

Liu KW. Zhang JS. Wang JG. Chen GL. - Journal of Materials Research. 13(5):1198-1203, 1998

Formation of nanocrystalline Al<sub>3</sub>Ti, TiAl, Ti<sub>3</sub>Al, and Ti<sub>5</sub>Si<sub>3</sub> composites by mechanically alloying (MA) in the Ti-Al-Si system and subsequent annealing treatment are investigated. Microstructure development was monitored by x-ray diffraction, differential thermal analysis, and transmission electron microscopy. An amorphous phase could be generated through milling for 100 h. The results of annealing at different temperatures on this amorphous phase show that the formation of titanium aluminides (Al<sub>3</sub>Ti, TiAl, and Ti<sub>3</sub>Al, according to the initial relative amount of Ti and Al) and Ti<sub>5</sub>Si<sub>3</sub> (the only silicide produced by the crystallization reaction) take place. Annealing produces nanocrystalline composites of Al<sub>3</sub>Ti, TiAl, Ti<sub>3</sub>Al, and Ti<sub>5</sub>Si<sub>3</sub> with a grain size less than 20 nm. With increasing annealing temperature, the crystalline sizes of the phases increased.

**[7] MECHANICALLY ACTIVATED REDUCTION OF NICKEL OXIDE WITH GRAPHITE**

Yang H. McCormick PG. - Metallurgical & Materials Transactions B-Process Metallurgy & Materials Processing Science. 29(2):449-455, 1998

The reduction of nickel oxide with graphite during ball milling at both ambient and elevated temperatures was investigated using X-ray diffraction (XRD), simultaneous thermogravimetry and differential thermal analysis (TG/DTA), and electron microscopy. It was found that milling at ambient temperature did not result in the reduction of nickel oxide to nickel. However, milling significantly reduced the critical reaction temperature for the reduction, from 1350 K for the unmilled sample to similar to 650 K for samples milled for 12 hours or longer. This reduction in reaction temperature is rationalized in terms of the microstructural refinement observed in the milled samples. The reduction of nickel oxide to nickel was observed to occur at elevated temperatures during milling. The thermodynamics and kinetics of the reduction reaction are discussed.

**[6] ENERGETICS OF COLLISION BETWEEN GRINDING MEDIA IN BALL MILLS AND MECHANOCHEMICAL EFFECTS**

Venkataraman KS. Narayanan KS. - Powder Technology. 96(3):190-201, 1998

This paper describes the physical and chemical changes such as decomposition, double decomposition, oxidation, and phase transformation reported in the literature as a consequence of solid surfaces coming into contact under mechanical forces. These changes, generally termed mechanochemical and chemomechanical effects, occur in the near-surface regions when surfaces come into contact as in abrasion between solid surfaces and repeated collisions among grinding media in ball mills. For understanding mechanochemical effects in ball mills, the normal impact between two grinding balls is quantified in terms of collision force, contact radius, contact time, and collision energy. These measures are quantified as a function of ball sizes (diameters varying between 1 and 4 cm), ball material (steel and alumina), and collision velocity (0.4-4 m/s), using expressions developed by previous investigators from the principles of continuum mechanics.

**[5] INDICATION OF DEGREE OF THE MECHANOCHEMICAL ACTIVATION BY OXYGEN DENSITY**

Juhasz AZ. - Hungarian Journal of Industrial Chemistry. 26(1):19-22, 1998.

The destruction of the crystalline order via external mechanical effects, e.g. by intensive grinding, is an important phenomenon of mechanochemistry and is denominated by the author as "structural activation". The mechanochemical

structural activation is actually a result of two opposite processes: one is the destruction of the crystalline structure (amorphisation, AM), the other is a densification in the microenvironment within the grains or between the amorphous grains and also a certain degree recombination. The dynamic balance of the two processes results in the final state of the milling product, which can be assessed by the measurement of oxygen density. The mechanochemical balance can be interpreted as the destruction of the structure of crystalline materials and is immediately followed by partial densification of high free energy amorphous atomic aggregates to a more stable and denser, although still amorphous, product. The dominance of recombination processes of the originally amorphous materials on prolonged milling is demonstrated.

**[4] MAGNETIC PROPERTIES OF MECHANICALLY ALLOYED CO-CU**

Modder IW. Schoonderwaldt E. Zhou GF. Bakker H. - Physica B. 245(4):363-375, 1998

The changes of the structural and magnetic properties of Co<sub>20</sub>Cu<sub>80</sub> during mechanical alloying were studied as well as the structure and magnetic behaviour of mechanically alloyed Co<sub>34</sub>Cu<sub>66</sub>. The magnetic behaviour of the final products was compared to that of similar alloys prepared by different techniques. Clear evidence for superparamagnetism was found in the mechanically alloyed compounds.

**[3] FULLY DENSE AL-PB NANOCOMPOSITE BULK SAMPLES CONSOLIDATED FROM MECHANICALLY MILLED POWDERS**

Zhou F. Sheng HW. Lu K. - Journal of Materials Research. 13(2):249-252, 1998

Powders with a nanostructured mixture of pure Al and Pb phase were produced by mechanical milling of elemental blends of Al and Pb with a composition of Al<sub>90</sub>Pb<sub>10</sub> (wt. %). Under a pressure of 1.5 GPa at 280 degrees C, the as-milled powders were successfully consolidated into bulk, full-density samples (>99.5% theoretical density) while the average grain sizes of Al and Pb in the compacted samples remain unchanged with respect to those in the as-milled powders. The achievement of the full density without grain coarsening in the consolidation process could be reasonably attributed to melting of the nanometer-sized Pb particles of which the melting point is considerably depressed

**[2] INVESTIGATION OF AL-PB NANOCOMPOSITES SYNTHESIZED BY NONEQUILIBRIUM PROCESSES**

Sheng HW. Zhou F. Hu ZQ. Lu K. - Journal of Materials Research. 13(2):308-315, 1998

Two nonequilibrium processes (melt-spinning and ball-milling) were successfully employed to synthesize Al<sub>1-x</sub>Pb<sub>x</sub> (x = 5, 10, 20, 30 wt. %) nanocomposites with distinct microstructures. In the melt-spun (MS) Al-Pb alloys, the nanometer-sized Pb particles are uniformly distributed in the micrometer-grained Al matrix and have an orientational relationship with the matrix, while in the ball-milled (BM) samples, both Pb and Al components are refined with prolonged milling time, forming nanocomposites with Pb particles homogeneously dispersed into the Al matrix. The minimum particle size of Pb in the milled samples linearly increases with the Pb content. The microhardness of the BM Al-Pb samples is much larger than that of the MS samples, which mainly results from strengthening effects of the nanometer scale Al grains following the Hall-Petch relationship. The microhardness for both BM and MS Al-Pb samples varies with the Pb content, and maximum hardness for both samples exists when Pb content is about 5 wt. %, indicating that small amounts of Pb, in the form of nanoparticles, may strengthen the Al matrix.

**[1] DIFFERENT PATHWAYS OF PHASE TRANSITION IN A V-SI SYSTEM DRIVEN BY MECHANICAL ALLOYING**

Liu L. Lu L. Lai MO. Magini M. Fei GT. Zhang LD. - Materials Research Bulletin. 33(4):539-545, 1998

Solid state reactions induced by mechanical alloying of an elemental powder mixture of V and Si with a composition of V<sub>75</sub>Si<sub>25</sub> were investigated using X-ray diffraction and scanning and transmission electron microscopy. It was found that the phase transitions in this system are closely related to the milling intensity. High-energy milling mainly causes the formation of intermetallic compounds, whereas low-energy milling leads to amorphization. The thermodynamics and kinetics related to the different pathways of V-Si phase transition are briefly discussed in this paper. The effect of air contamination on phase transition is also addressed.

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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  
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