RESEAU FRANÇAIS DE MECANOSYNTHESE

Lettre N°47

Février 1999

134 Groupes de Recherche

(dont 71 à l'étranger)

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

3 Nouvelles Adhésions

T. Benameur - Dpt Génie Mécanique - ENIM - Tunisie
B. Cantor - Dpt of Materials - Univ. Oxford - UK
P. Schumacher - Dpt of Materials - UK

Prière de Penser à votre Cotisation au titre de l'année 1999 !!

Pour ceux qui n'auraient pas réglé leur cotisation1998 (voire 1997...), Prière de le faire rapidement.
La liste de diffusion sera remise à jour pour 1999

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Le site web du RFM est le suivant
http:://www.bls.fr/amatech
Rubrique Pages Sciences et Techniques pour l'Ingénieur (Rubrique Sciences)
⇒ vous y trouverez les anciennes lettres du RFM (accessible par Adobe Acrobat)
les statuts du RFM ainsi que les annonces concernant les JRFM'99 et quelques éléments mis a jour régulièrement concernant les derniers résultats dans ce domaine.

Une Rubrique "Forum"
est ouvert sur le site web du RFM afin de permettre
de discuter sur tout sujet
"Mécanosynthèse et/ou Nanomatériaux"

L'inscription en ligne aux JRFM'99
est possible sur ce site Web !!!!

Corresp. : Eric.Gaffet@utbm.fr

JRFM'99

4 èmes Journées du Réseau Français de Mécanosynthèse
Dijon (ESIREM) - les 2-3 Juin 1999
2ème circulaire

Thématique 1999 :
Mécanosynthèse : Réactivité et Mécanique

Conférences Invitées

• **L'analyse microstructurale des solides à cristallisation fine par diffraction des rayons X**
D.LOUER (Directeur de Recherches CNRS- LCSIM Cristallochimie - Univ. Rennes)

• **Potentialités du Rayonnement Synchrotron en Mécanosynthèse**
JF.BERAR (Ingénieur de Recherches CNRS- Lab. de Cristallographie de Grenoble / ESRF)

• **Mécanochimie : Bilan et Perspectives**
J.C.MUTIN (Directeur de Recherches LRRS CNRS - Univ Dijon).

Propositions de Communications Orales (0) et Par Affiche (A) (Liste Provisoire)

1) Co - broyage de poudres pharmaceutiques
M. Baron - Ecole des Mines d'Albi (O + A)

2) Mécanochimie de basse énergie : le cas du nitrure de bore
M. Gasnier, H. Szwarc - ICMO - Lab. Chimie Inorganique Orsay (O + A)

3) Etude de la Microstructure d'un Nitrure d'Aluminium broyé
C. Goujon et al. - Ecole des Mines de St Etienne (O)

4) Influence de la mécanosynthèse sur la structure cristallographique TRNi (TR = Gd, Y, Er)
M. Nakhl, B. Chevalier, J.-L. Bobet, B. Darriet - ICMCB - Bordeaux (A)

5) Mécanosynthèse appliquée au système binaire Mg - Co. Réactivité avec l'Hydrogène
J.-L. Bobet, B. Darriet, E. Akiba - ICMCB - Bordeaux (O)

6) Propriétés et choix de capteurs intermétalliques d'hydrogène destinés à un procédé d'oligomérisation
A. Sauvage, M. Mercy, JC Gachon, P. Pareja (A)

7) "Structural disorder in YBa_2Cu_3Ox synthesized by mechanical activation
A. Shlyachtina - Moscou - Russie (A)

8) Etude du frittage de poudres nanométriques W - Cu et Cr - Cu
F. Doré et al. - LTPCM / ENSEEG Grenoble (O)

9) Etude in situ par rayonnement synchrotron de la compressibilité de poudres nanostructurées obtenues par broyage mécanique à puissance controlée (Cas du Fe, Ni, Cu)
E. Gaffet, J.-P. Itié, C. Meunier, S. Vives (Belfort, LURE Orsay et Montbéliard) (A)

10) Etude de la compressibilité de la poudre de Fer Nanométrique élaborée par broyage Mécanique
M. Zougggar et al. - SP2MI Poitiers (O)

11) Mécanosynthèse d'alliages Fe - Mo
T. Ziller, G. Le Caër, P. Delcroix - LSG2M / CNRS Nancy (A)

12) Cinétiques de Transformations de phases induites par broyage à haute énergie de TiO_2 anatase en fonction de la nature du matériau de broyage et du rapport masse de poudres sur masse de billes
T. Girot, S. Begin - Colin, G. Le Caer, A. Mocellin - LSG2M / CNRS - Nancy (A)

13) Rôle de l'activation mécanique sur la synthèse du composé Cu_3Si
H. Souha, F. Bernard, E. Gaffet, JC Niepce - Dijon, Belfort (O)

14) Elaboration du composé FeAl nanométrique par MASHS - Suivi In situ par rayonnement synchrotron
F. Charlot,F. Bernard, E. Gaffet, D. Vrel, JC Niepce - Dijon, Belfort Villetaneuse (A)

15) Elaboration par MASHS de siliciures $FeSi_2$ et $MoSi_2$ nanométriques
Ch Gras, E. Gaffet, F. Bernard, D. Vrel, JC Niepce (A)

16) Suivi in situ de l'élaboration du composé $NbAl_3$ par MASHS et ERAM
V. Gauthier, F. Bernard, E. Gaffet, JP Larpin (A)

17) Effet de la taille des billes lors de broyage successif par attrition : obtention de poudres nanométriques pour applications électrocéramiques
F. Perrot - Sipple, D. Aymes, J.C. Niepce - Dijon (O)

JRFM'99
Appel à Communications

(à renvoyer avant le **31 Janvier 1999** en ce qui concerne les communications orales**)**

Nom: .....Prénom: .....
Adresse:.....
.....
Téléphone : ..... Fax : .....E-mail : .....

Souhaite présenter une communication lors des JRFM99 sous forme de :
communication orale Communication par affiche
Titre :

Fiche d'inscription à retourner à
Frédéric BERNARD (Journées RFM99), Laboratoire de Recherches sur la Réactivité des Solides (UMR5613 CNRS - Université de Bourgogne), 9 avenue Alain Savary, BP 400, 21011 Dijon Cedex. (Tel : 03.80.39.61.25 - fax : 03.80.39.61.67, E-mail : fbernard@u-bourgogne.fr)

Comité d'organisation :

Frédéric Bernard et toute l'équipe " Matériaux à Grains Fins "
Laboratoire de Recherches sur la Réactivité des Solides
(UMR 5613 CNRS - Université de Bourgogne),
9 avenue Alain Savary, BP 400, 21011 Dijon Cedex.
Tel : 03.80.39.61.25 - Fax : 03.80.39.61.67,
E-mail : fbernard@u-bourgogne.fr)
Eric Gaffet - Président du Réseau Français de Mécanosynthèse
Groupe "Nanomatériaux" - CNRS UPR 806
Université de Technologie de Belfort - Montbéliard (UTBM)
90100 Belfort Cedex.
Tél : 03 84 58 31 02 - Fax : 03 84 58 30 27
E-mail : Eric.Gaffet@utbm.fr

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Comme vous le savez, l'An 2000 s'avère d'ores et déjà très chargé en matière de programmation de conférences scientifiques
Ceci nous oblige à prendre dès maintenant des dispositions quant à l'organisation des JRFM de l'an 2000.

les 5èmes Journées du RFM, les JRFM'2000 se tiendront à **Bordeaux**

Organisées par **J.-L. Bobet et al (ICMCB - CNRS / Univ. Bordeaux)**
La Thématique retenue est la suivante :
Caractérisations Physico Chimiques des produits issus de Mécanosynthèse
deux séries de dates sont retenues
16 & 17 Mai 2000 ou 23 & 24 Mai 2000
Les personnes ayant des suggestions ou des remarques sur le recoupement de ces deux séries de dates avec d'autres congrès ou évênements scientifiques déjà programmés sont invitées à nous les communiquer rapidement afin d'éviter des recouvrements génants.

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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

Nanostructured Hybrid Materials
Symposium TMS Annual Meeting - San Diego CA - USA - 28 Février 4 Mars 1999
Contact : gmchow@anvil.nrl.navy.mil`

XXV JEEP
Journées d'Etudes des Equilibres entre Phases 1999
11 - 12 Mars 1999 - Annecy France
E_Mail : Conference.Jeep@univ-savoie.fr

VII International Seminary
Defects, strucure and properties of Nanocrytalline Materials obtained by Nanocrystallization of Amorphous Solids and of Metals with Extreme Distortion of the Lattice
Mars 1999 - Ekaterinburg - Russie
E_Mail : Noskovaimp.uran.ru

Nanocomposite Materials : Design and Applications
28 Mars - 2 Avril 1999 -Alyeska Resort - Alaska
E_Mail : Engfnd@aol.com

4th International Workshop on Metastable Phases (IV IWOMP)
7 - 9 Avril 1999 - Bologne - Italie
Contact : Bonetti@df.unibo.it

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

E_MRS - Spring Meeting
1 - 4 Juin 1999 - Strasbourg - France
Web Site http://www-emrs.C-strasbourg.fr
Symposium A : Phot - Excited Process and Applications
Symposium B : Protective Coating adn Thn Films 99
Symposium C : Progress in Computational Materials Science
Symposiym D : Plasma and Ion Surface Engineering
Symposium E : Advanced Silicon Substrates
Symposium F : Process induced defects in Semiconductors
Symposium G : Material Physics Issue and Applications on Magnetic Oxides
Symposium H : Strain in Materials : Analysis, Relaxation and Properties
Symposium I : Microcrystalline and Nanocrystalline Semiconductors
Symposium J : Materials for Coherent Optics
Symposium K : Materials, Process and Technology for Optical Interconnect
Symposium L : Ab - Initio Approaches to Microelectronics Materials...
Symposium M Basic Models to enhance Reliability in Si based devices and ..

Symposium N : Molecular Optoelectronics : Materials, Physics and Devices
Symposium O : Chalcogenide Semiconductors for Photovoltaics
Symposium P : Optical Characterization of Semiconductor layers and Surfaces

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JRFM'99

4èmes Journées du RFM
2 & 3 Juin 1999 - Dijon - France
Web Site :http:://www.bls.fr/amatech - Web SubSite : Sciences

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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

PM2 Tec 98

1999 International Conference
on Powder Metallurgy and Particulate Materials
Vancouver - 20 / 24 Juin 1999
E_Mail : Info@mpif.org - Website: www.mpif.org

4th Int. Conf. on Materials Chemistry

13 - 16 Juillet 1999 -Trinity College _ Univ. Dublin - Irlande
Web Site : http://www.rsc.org/conferences
Themes :
Inorganic Nano and Micro Particles
Fucntional Polymers
Magnetic Materials
Organic Nanostructures
Molecular Crystals and Crystal Engineering
Computational Chemistry and Materials for Electronic

Advanced Materials - Nanostructured Systems

15 - 17 Juilllet 1999 - Hong Kong
1st workshop of the new IUPAC series :
"New Directions in Chemistry
Theory, Nanoparticles, Quantum Dots,
Bio - Inspired Structures, Applications to Nanotechnolog
Organizing Committee A. El - Sayed - Georgia Tech - Atlanta - USA
J. Portner - President of IUPAC - Tel Aviv - Israel
N. Teng Yu - HKUST - Hong Kong
S. Williams - Hewlett - Packard Co., California USA
Web Site : http://www.iupac.org/symposia/conferences/wam1

NATO Advanced Research Workshop
Investigations and Applications of Severe Plastic Deformation
2 - 6 Aout 1999 - Moscu - Russie
E_Mail : TLowe@lanl.gov and Valiev@ippm.rb.ru

RQ10

10th International Conference on Rapidly Quenched and Metastable Materials
Bangalore - Inde - 23 - 27 Août 1999
Wesite : http:://www.metalrg.iisc.ernet.in/rqten/

ISMANAM 99

International Symposium on Metastable Mechanically Alloyed and Nanocrystalline Materials
and Euro Conference on Gas Phase Synthesis of Nanocrystalline Materials.
Org. : L. Schultz, J. Eckert, H. Hahn
Dresden - 30 Aout - 3 Septembre 1999
E_Mail : ISMANAM99@ifw-Dresden.de
WebSite: http//www.ifw-dresden.de/imw/ismanam/

SMM14

14th International Conf. on Soft Magnetic Materials
8 - 10 Septembre 1999 Balatonfüred - Hongrie
web site : http://www.kfki.hu - Subsite : smm14

Int. Symp. Cluster and Nanostructure Interfaces (ISCANI)
25 - 29 Octobre 1999 - Richmond USA
website : http://www.vcu.edu/ISCANI/

NANO 2000

5th International Conference on Nanostructured Materials
Sendai - 20 - 25 Aout 2000

E_Mail : nano2000@imr.tohoku.ac.jp

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Annonces de Soutenance de Thèses

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Caractérisation et réactivité de la surface de poudres nanométriques d'oxydes métalliques: Analyse par spectrométrie IR-TF et application à l'étude des mécanismes de détection de gaz par capteurs résistifs.

Jérôme Tribout - Université de Limoges, Limoges, France, 14 décembre 1998.
Directrice: Marie-Isabelle Baraton

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Distinct Element Modelling of a Planetary Ball Mill

M.P. Dallimore - Dpt of Mechanical and Materials Engineering - University Western Australia - Australie

The distinct element method of modelling has been used to develop a model of a laboratory scale planetary ball mill. The model has been used to evaluate variations in processing parameters such as the impact frequency and energy associated with different milling configurations. These results have been compared to the kinetics of the Ni/CuO displacement reaction propagated under these conditions.

The model is two dimensional in nature and incroporates a modified Kelvin viscoelastci spring / damper system to describe the impact proces using experimentally derived impact parameters. the simulation enables quantitative analysis of the collision energies and their spatial distribution. Validation of the model was achieved though the use of high speed videao analysis of actual ball trajectories.

The results of the modelling study have demonstrated that kinematic consid"rations alone are insufficient to predict the variation in ball motion and energy dissipation that occur for different mill configruations. The amount of slip between the balls and the vial wal plays a significant role in determining both the tumbling trajectories and the level and mode of energy dissipation.

When comparing the model predictions to the experimental milling results, it was found that reaction rate was particularly sensitive to the spatial distribution of the impacts. Only when the tumbling trajectories remained relatively unchanged, could the reaction rate be tied to the total level of energy dissipated. Both the energy dissipated through impacts and attrition play an important role in promoting the reaction. The results suggest that the spatial distributinn of the impact energy must be considered with respect to the distribution of powder within the vial to fully explain the variations in reaction rates.

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Synthèse et Propriétés de Ferrites Nanométriques : Influence de l'énergie de surface sur les propriétés structurales et magnétiques de ferrites de titane synthétisés par chimie douce et mécanosynthèse

N. Guigue - Millot - 26 Novembre 1998 - LRRS UMR 5613 CNRS - Univ. Bourgogne - Dijon - France
Jury : J. Etourneau, A. Rousset, G. Bertrand, D. Stuerga, G. Le Caër, M. Guyot, O. Isnard, P. Perriat

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

E. Navarro - Universie 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., 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 Fe2.47Mo0.53O4, de la synthèse aux propriétés magnétiques dans le système fer - vanadium $Fe_{3-x}V_xO_4$ ($0 \leq x < 2$).

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

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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

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Post Doc Position Proposals

USA (17/12/98)

Rutgers University is seeking a postdoctoral associate with demonstrated expertise in mechanochemistry to work on research focused on biomaterials. The candidate must be able to work on research focused on biomaterials. The candidate must be able to work as part of multidisciplinary team involving industry and academia focused on making biomedical implant devices. The candidate should demonstrate the ability to work independtly, publish in archival journals and present their work in a public forum. The candidate should send a curriculum vitae, three representative publications (preferably with the candidate as a first author) and the names, address, email and phone numbers of three refences that can comment on the candidate 's capabilities. The position is available immediately at a salary of $32,000 with health benefits included. The position will be posted until a suitable candidate is identified. Interested candidates should send correspondance to

Professor R.E. Riman
Rutgers University
Departement of Ceramic and Materials Engineering
607 Taylor Road
Piscataway, NJ 08854 - 8065
Riman@alumina.rutgers.edu

Brésil

Post doc to work in electron microscopy characterization of nano - structured materials.
Contact : Walter J. Botta. F. - Federal Univeristy o Sao Carlos - Sao Paulo State - Brésil
Adresse : DEMa - UFSCar - CP 676, 13565 - 905 Sao Carlos SP Brésil.
tél : 016 - 2608251 - Fax : 016 2615404.

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 interseted 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 - http://www.aist.go.jp/NIMC/fcg/index.html

Bibliographie Récente

Livres ou "Special Issues"

DISPERSION-STRENGTHENED ALUMINIUM PREPARED BY MECHANICAL ALLOYING

Michal Besterci, Institute of Materials Research, Slovak Academy of Sciences, Kosice
In the book, the author describes the theoretical and technological fundamentals of mechanical alloying the Al-C system. Special attention is given to material characteristics, the kinetics and mechanism of mechanical alloying, methods of mixture compaction and heat treatment of compacted parts. Models of dispersoid spatial arrangement, dispersoid evaluation and optimisation and experimental possibilities are discussed. The interpretation of the static and dynamic mechanical properties, especially strength and ductility properties at 20 °C, mechanical properties at elevated temperatures are discussed, with emphasis on the effect of interface, superplasticity, creep and creep-fatigue characteristics. Content
Introduction
1. Characteristics of dispersion-strengthened systems 2. Mechanical alloying (kinetics and mechanism of preparation of the Al-C system by mechanical alloying; compaction of powders and heat treatment of compacts;
3. Microstructure and quantitative evaluation of parameters of dispersion-strengthened materials (definition and properties of interparticle distance; experimental possibilities of determination of structural objects; models of heterogeneous structures and their evaluation; simulation of model structures; analysis of the spatial distribution of particles in the Al-Al4C3 material) 4. Static and dynamic mechanical properties (mechanical properties at elevated temperatures;
mechanical properties at 20°C; effect of interface on the mechanical properties; superplastic
properties of the system; thermal stability of the system; creep characteristics; creep-fatigue
characteristics)
References
Index
ISBN 189832655X, 80 pages, 234×156 mm, soft laminated cover, £22.00, January 1999
Cambridge International Science Publishing 7 Meadow Walk, Great Abington, Cambridge CB1 6AZ, England Fax +44 1223 894539; Tel +44 1223 893295 Email: cisp@cisp.demon.co.uk
http://www.demon.co.uk/cambsci/homepage.htm

"Mechanical Alloying"

Auteurs : Li Lü & Man On Lai(National University of Singapore)
Kluwer Academic Publishers
Contents : Preface - Introduction to Mechanical Alloying - Experimental Set - Up - The Mechanical Alloying Process - Formation of New Materials - Characterization of Powders - Densification - Mechanical Properties - Mechanisms of Mechanical Alloying - Modeling of Mechanical Alloying - Index

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

Editors: Kenneth E. Gonsalves, Marie-Isabelle Baraton, Rajiv Singh, Heinrich Hofmann, Jerry X. Chen, and Joseph A. Akkara.
Materials Research Society, Symposium Proceedings Volume 501, 1998
MRS, Warrendale, Pennsylvania, USA (website: http://www.mrs.org/)

"Nanomatériaux"

Auteurs : E. Gaffet, S. Begin - Colin, O. Tillement
Editeur : Innovation 128 - 24 Rue du Quatre Septembre - 75002 Paris - France
Les dernières années ont vu apparaître dans le monde des matériaux avancés le préfixe "nano" (nanostructuré, nanocristallins, nanophase ou nanométrique) ; les conférences et les forums sur Internet se multiplient où s'échangent des informations sur les avancées scientifiques et technologiques dans ce domaine des matériaux nanostructurés qui se distinguent des matériaux polycristallins conventionnels par la dimension des cristallites les composant ou par la dimension des hétérostructures présentes : ces dimensions sont de quelques dizaines d'angströms, voire de quelques nanomètres. A ces dimensions, les propriétés des matériaux changent radicalement.
Au début des années 90, les japonais ont été les premiers a lancé d'ambitieux programmes de R & D puisque le MITI a consacré aux nanomatériaux près de 200 millions de dollars pour la période 1990 - 2000 et que la Science & Technology Foundation a investi presque la même somme pour co - financer des projets de laboratoires publics et privés. Les Etats Unis puis les pays européens ont investi plus tardivement mais déjà ont obtenu des résultats prometteurs (••••••) Certaines applications existent déjà au niveau international, quelque 400 sociétés se partagent aujourd'hui un marché voisin de 1 milliard de dollars mais qui devrait tripler, voire quintupler à l'horizon 2001.(••••••)
(•••) Pour aider les industriels concernés à imaginer les applications qu'ils pourraient s'approprier et identifier les acteurs internationaux, la présente étude dresse un état de l'art complet des nanomatériaux en décrivant leurs procédés d'élaboration actuels ou envisagés et en détaillant leurs différentes propriétés physico - chimiques et les géométries que l'on peut obtenir.
Enfin l'étude permet de cerner les applications actuelles et potentielles•••

CHEMISTRY FOR SUSTAINABLE DEVELOPMENT
Vol. 6, No. 2-3, MARCH-JUNE 1998

Proceedings of 2d International Conference on Mechanochemistry
(INCOME-2), which was held in Novosibirsk in 1997.

Contact : Prof. • N.Z. Lyakhov, Inst. Sol. State Chem.- Russian Acad Sci. - Kutaleladze, 18 - Novosibirsk - 630128 Russia - The Proceedings will be available by the price 80 USD.

Mechanochemistry of Materials
Cambridge International Science Publishing

Emmanuel Gutman - Materials Eng. Dpt - Ben Gurion University - Beer Sheva - Israel

Considerable advances have been made in mechanochemistry in the last couple of decades. Training of experts in this fied with a background in materials science, chemical and mechanical engineering, etc. requires study of the fundamentals of mechanochemistry. There is a need for a textbook in the general and compressed form which would cover many aspects and would be used as a basis for understanding the fundamental principles to control mechanochemical phenomena. This textbook is based on lectures given by Prof. Gutman in a graduate course in the mechanochemistry of materials at the Ben - Gurion University of the Negev. The book contains examples of experimental results to illustrate the mechanochemical phenomena and technologies.

BIBLIOGRAPHY ON MECHANICAL ALLOYING AND MILLING

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

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

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

PERIODIQUES
(Rubrique assurée grâce au concours de M^{me} TAUZIN - FIN BiPSé)

[57] ELECTRON MICROSCOPY OF AL3TI-BASE ALLOYS PREPARED BY MECHANICAL ALLOYING
A Cabrera, M Umemoto, K Tsuchiya, JG CabanasMoreno, HA Calderon - ELECTRON MICROSCOPY 1998, VOL 2, 1998, pp 109-110 - 14TH INTERNATIONAL CONGRESS ON ELECTRON MICROSCOPY; CANCUN, MEXICO. AUGUST 31-SEPTEMBER 4, 1998

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[55] ELECTRON MICROSCOPY STUDY OF A NEW MAGNETICALLY ORDERED FCC STRUCTURE IN NANOCRYSTALLINE BALL-MILLED FE
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[54] FACILITATED SYNTHESIS OF ULTRA-HARD C-BN FROM BALL-MILLED H-BN
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[53] CHARACTERIZATION BY TRANSMISSION ELECTRON MICROSCOPY OF NANOCRYSTALLINE TI-AL-X PRODUCED BY MECHANICAL ALLOYING
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[51] PITTING ACTIVATION OF A PASSIVE STATE AND MECHANOCHEMISTRY OF ZIRCONIUM IN HYDROCHLORIC ACID SOLUTIONS
AA Barkov - PROCEEDINGS OF THE SYMPOSIUM ON PASSIVITY AND ITS BREAKDOWN (Series: ELECTROCHEMICAL SOCIETY SERIES), 1998, Vol 97, Iss 26, pp 781-789 - SYMPOSIUM ON PASSIVITY AND ITS BREAKDOWN AT THE 1ST JOINT INTERNATIONAL MEETING OF THE ELECTROCHEMICAL-SOCIETY/INTERNATIONAL-SOCIETY-OF-ELECTROCHEMISTRY; PARIS, FRANCE. SEPTEMBER 1-5, 1997

[50] CHANGES IN THE STRUCTURAL AND MAGNETIC PROPERTIES OF GDIR2 WITH THE MILLING PROCESS
Modder IW. Bakker H. - Physical Review B-Condensed Matter. 58(21):14479-14483, 1998
Both ac magnetic-field and de low and high magnetic-held measurements, have been used, to study the magnetic property changes in relation to the structural changes, induced by ball milling of GdIr2, which is, in the ordered state, a ferromagnetic cubic Laves phase compound. A ferromagnetic transition is shown to be present for all samples, the value of which depends linearly on the Gd-Gd distance, because of the negligible influence of the 5d electrons of Ir on the exchange interaction between the Gd atoms. After intermediate and longer periods of milling, antiferromagnetic interactions are induced, as a consequence of Gd atoms being transferred to the Ir sublattice. This is shown to result in spin-glass-like behavior: a reentrant spin glass appears to be created.

[49] A MODEL FOR THE REACTION ZONE IN POWDERS MECHANICALLY ACTIVATED IN A PLANETARY MILL
Zyryanov VV. - Inorganic Materials. 34(12):1290-1298, 1998
A semiquantitative model is proposed for describing the reaction zone in inorganic powders mechanically activated in a planetary mill. The model considers macro-, meso-, and microprocesses and their time evolution, Some parameters of the reaction zone are evaluated.

[48] MICROSTRUCTURE OF SUPERSATURATED FCC AL-FE ALLOYS: A COMPARISON OF RAPIDLY QUENCHED AND MECHANICALLY ALLOYED AL98FE2
Dunlap RA. Dahn JR. Eelman DA. MacKay GR. - Hyperfine Interactions. 116(1-4):117-126, 1998
Alloys of the composition Al98Fe2 have been prepared by rapid quenching from the melt and mechanical alloying methods and have been studied by X-ray diffraction techniques and room temperature Fe-57 Mossbauer effect methods. Results may be summarized as follows: The rapidly quenched sample is a single phase supersaturated fee Al-Fe alloy. Mossbauer effect spectra indicate the presence of a substantially greater degree of Fe clustering than is expected for a random distribution of atoms on the lattice sites. Mechanically alloyed samples have been studied as a function of milling time and show the initial formation of a supersaturated fee phase with microstructural properties which are quite similar to those of the rapidly quenched sample. Further milling results in the reduction of the average grain size and the formation of an amorphous phase. Mossbauer studies and previously reported phase diagrams suggest that a substantial fraction of the Fe resides in this phase.

[47] STABILITY OF A NANOCRYSTALLINE STRUCTURE IN TIAL-BASED ALLOYS
Senkov ON. Ovecoglu ML. Srisukhumbowornchai N. Froes FH. - Nanostructured Materials. 10(6):935-945, 1998
Two fully dense nanocrystalline TiAl-based compacts, i.e. Ti-47Al-3Cr and Ti-48Al-2Nb-2Cr, were synthesized by hot isostatic pressing (HIP'ing)from mechanically alloyed powders. Microstructure evolution and phase

transformations during mechanical alloying, HIP'ing, and heating were studied with the use of TEM, SEM, XRD and DTA. Formation of amorphous phases was detected in both powders after 15-hour mechanical alloying. During HIP'ing, crystallization of the amorphous phases occurred and a very fine equiaxed grain structure was formed The grain size decreased when the temperature of HIP'ing was decreased, and it was 42 nm after HIP'ing at 725 degrees C. Grain growth occurred during annealing in both alloys. An analysis showed that the grain growth in the TiAl-based nanocrystalline alloys can be satisfactorily described by a single thermally activated process following equation D-n-D-0(n) = K(0)t.exp(-Q/RT), with the parameters n = 4, Q = 335 kJ/mol and K-0 = (2-4).10(22) nm4/h. The most probable reason for the high value of n is the presence of impurities which retard the grain boundary mobility by pinning and dragging effects.

[46] SUPERPARAMAGNETIC TRANSITION AND LOCAL DISORDER IN CUFE2O4 NANOPARTICLES

Goya GF. Rechenberg HR. - Nanostructured Materials. 10(6):1001-1011, 1998

We present X-ray diffraction (XRD), Mossbauer spectroscopy (MS) and d.c. magnetization measurements performed on ball-milled CuF2O4 samples. The average particle size < d > was found to decrease to the nanometer range after t=15 min of milling. Room temperature Mossbauer data showed that the fraction of particles above the blocking temperature Tg increases with milling time, and almost complete superparamagnetic samples are obtained for < d > = 7(2) nm. Magnetization measurements below Tg suggest spin canting in milled samples. The values of saturation moment Cts reveal that site populations are slightly affected by milling. Mossbauer resonant intensities are accounted for on the basis of local disorder of Fe3+ environments, and the development of sample inhomogeneities of CuxFe3-xO4 composition.

[45] THERMOELECTRIC PROPERTIES OF BETA-FESI2 MECHANICALLY ALLOYED WITH SI AND C

Nagai H. Nagai K. Katsura T. Katsuyama S. Majima K. Ito M. - Materials Transactions Jim. 39(11):1140-1145, 1998

The thermoelectric properties of the hot-pressed n-type Fe0.98Co0.02Si2 and p-type Fe0.92Mn0.08Si2 mechanically alloyed with Si and C powders have been investigated. It has been found that mechanical alloying (MA) even for a short period is very effective for forming the beta-FeSi2 phase from the mixture of the alpha-Fe2Si5 and epsilon-FeSi phases during hot-pressing at 1173 K for 1 h. Both of the hot-pressed n-type and p-type samples are composed of mostly the beta-phase with a dispersion of a small amount of epsilon-phase particles. The amount of the F-phase decreases with increasing amounts of (Si+C) addition. A lot of fine alpha-SiC particles around 20 nn form in the samples mechanically alloyed for 20 h and hot-pressed at 1173 K for 1 h. The addition of (Si+C) markedly increases the thermoelectric power of both n-type and p-type FeSi2 and the thermoelectric power values are at a maximum at 3-4 mass%(Si + C) for both cases. The electrical resistivity increases with increasing amount of (Si + C) addition for both n-type and p-type FeSi2. The addition of (Si + C) markedly decreases the thermal conductivity of both n-type and p-type FeSi2 due to the dispersion of fine alpha-SiC particles, which results in the marked increase in the figure of merit. The figure of merit values are maxima at 3-4 mass%(Si+C) for both n-type and p-type FeSi2.

[44] INTERNAL KINEMATICS OF TUMBLER AND PLANETARY BALL MILLS: A MATHEMATICAL MODEL FOR THE PARAMETER SETTING

Schilz J. - Materials Transactions Jim. 39(11):1152-1157, 1998

This paper discusses the internal mechanics of tumbler and planetary ball mills. On the basis of classical mechanics, trajectories of mass particles under the different acceleration conditions in these mills are calculated. This allows the derivation of impact velocities and impact powers of the grinding masses - important quantities which characterize milling conditions. It is shown that, under the assumption of single particle movements, the trajectories and thus the impact energies of mass particles depend only on the ratio of planetary and system wheel radii and on the ratio of their angular velocities. As functions of those proportions, movements of particles are calculated and recorded in order to enable the reader to find the (theoretical) optimum mill setting for a desired purpose. For example, the mechanical alloying of brittle materials requires near-normal collisions with a minimum of sheer component. The applicability of the developed model is discussed.

[43] PREPARATION OF FUNCTIONALLY GRADED MG2SI-FESI2 THERMOELECTRIC MATERIAL BY MECHANICAL ALLOYING PULSED CURRENT SINTERING PROCESS [JAPANESE]

Sugiyama A. Kobayashi K. Ozaki K. Nishio T. Matsumoto A.- Journal of the Japan Institute of Metals. 62(11):1082-1087, 1998

The formation process of functionally graded Mg2Si-FeSi2 thermoelectric material made by the mechanical alloying (MA) and the spark plasma sintering (SPS) was investigated. The MA was performed in a planetary ball mill using three elemental powders. The powders were prepared by the mixing of Mg2Si-xmass%FeSi2 (x=0, 20, 40, 60,80, 100). The MA powders were consolidated by the SPS method. During SPS, the heating program was held at 643 K for 600 s, at 973 K for 300 s and at 923 K for 600 s, in order to attain densification and phase transformation to beta-FeSi2. The structural evolution during milling and after sintering was detected by X-ray diffraction (XRD). The heat treatment was monitored by differential scanning calorimetry (DSC). For Mg-Si the formation of the Mg2Si phase is observed after milling for 1080 ks. But the Mg2Si phase was easily formed during milling when a third element such as Fe was included. For Mg2Si-xmass%FeSi2 phases of Mg2Si, FeSi, Fe and Si were observed after milling for only 180 ks. The Mb powder was functionally graded with high density by SPS. However, the alpha-FeSi2 and FeSi phase were observed in the sintered sample.

[42] A NEW MODEL AND GAS SENSITIVITY OF NONEQUILIBRIUM XSNO(2)(1-X)ALPHA-FE2O3 NANOPOWDERS PREPARED BY MECHANICAL ALLOYING

Zhu W. Tan OK. Jiang JZ. - Journal of Materials Science-Materials in Electronics. 9(4):275-278, 1998

Nano-sized xSnO(2)-(1-x)alpha-Fe2O3 materials have been prepared using the high energy ball milling technique and their structural and gas sensing properties have been characterized. Based on experimental results, we propose a new structure model, square(1/3) --> Sn-1/3(4+) + 2(1-y) O-s(2-) + 4yO(s)(-), for these non-equilibrium, nano-sized xSnO(2)-(1-x)alpha-Fe2O3 materials. This model can explain not only the lattice expansion of the milled samples,

but also takes into account the charge balance by adding oxygen dangling bonds at the particle surfaces, which can be visualized in the nano-sized powders. The thick film gas sensors made by such mechanically alloyed materials have high ethanol gas sensitivity values of 289 in air and 1016 in nitrogen at 1000 p.p.m. and very good gas selectivity to ethanol over CO and H-2 gases. It is believed that the high ethanol gas sensitivity of these materials is related to the enormous defects such as O- and O2- dangling bonds at the particle surfaces of these nano-sized materials.

[41] HIGH-TEMPERATURE STABILITY OF NANOCRYSTALLINE STRUCTURE IN A TIAL ALLOY PREPARED BY MECHANICAL ALLOYING AND HOT ISOSTATIC PRESSING

Senkov ON. Srisukhumbowornchai N. Ovecoglu ML. Froes FH. - Journal of Materials Research. 13(12):3399-3410, 1998

A fully dense nanocrystalline compact of the Ti-47Al-3Cr (at. %) alloy was produced by mechanical alloying and hot isostatic pressing at 725 degrees C, Microstructure characteristics and grain growth behavior of this compact were studied after annealing for up to 800 h in the temperature range of 725 to 1200 degrees C, using analytical transmission electron microscopy techniques. The temperature and time dependencies of the grain sizes and the grain size distributions were determined, The grain growth occurred, with a time- and temperature-invariant single-peak grain size distribution (when normalized by the mean grain size), which was consistent with normal grain growth. The experimentally measured grain growth exponent decreased from 10 to 4.6 when the temperature was increased. The grain growth kinetics was described by a single thermally activated rate process limited by a permanent pinning force on the grain boundaries. The microhardness decreased on annealing and followed the Hall-Fetch relationship with the parameters H-upsilon o = 5.8 GPa and K-H = 1.6 MPa.m(0.5).

[40] MECHANOCHEMICAL REACTIONS IN THE SYSTEM FETIO3-SI

Chen Y. Williams JS. - Journal of Materials Research. 13(12):3499-3503, 1998

Mechanochemical reactions in the system FeTiO3-Si have been investigated as functions of the powder composition and milling conditions, using x-ray diffraction and thermal analyses. Reduction reactions of FeTiO3 by Si were observed during room-temperature milling with the formation of alpha-Fe, amorphous SiOx, nanocrystalline TiO2, or intermetallic compounds, depending on the Si content. The mechanochemical reaction process consists of a mechanical activation stage and a reaction stage. Higher milling intensity leads to a shorter activation step and a higher reaction rate.

[39] ORIGIN OF SUPERPLASTIC ELONGATION IN ALUMINUM ALLOYS PRODUCED BY MECHANICAL MILLING

Hasegawa T. Yasuno T. Nagai T. Takahashi T. - Acta Materialia. 46(17):6001-6007, 1998

The effects of alloying (Mg, Cu, Ge and/or Si) and prior rolling and annealing on tensile stress-strain behaviors were examined for mechanically milled, powder metallurgy aluminum alloys at 748 K in the strain rate range of 1 x 10(-4)-7.5 x 10(2)/s. Lowering the temperature of prior rolling and increasing the anneal temperature and time result in an increase in the strain rate sensitivity, m. Especially alloying effectively increases the m value as well as the strain to fracture, epsilon(f), at the intermediate strain rate of approximately 10(0)/s; m and epsilon(f) reach 0.4 and 440% for a case of alloying both 1.1 at.% Mg and 1.2 at.% Cu, though they are smaller than 0.1 and 100% at lower (<1 x 10(-2)/s) and higher (>5 x 10(1)/s) strain rates. TEM and SEM revealed that the microstructure of superplastic alloys, compared to pure Al, consists of thermally recovered, fine equiaxed grains (smaller than 1 mu m in diameter) with smooth grain boundaries of large misorientation. It is then postulated that superplastic elongation occurs when smooth boundaries slide under a plastically stable condition due to a large m value.

[38] FORMATION MECHANISM OF NIAL/TIB2 NANOCOMPOSITE BY MECHANICAL ALLOYING ELEMENTAL POWDERS

Zhou LZ. Guo JT. - Journal of Materials Science & Technology. 14(6):491-496, 1998

A NiAl/TiB2 nanocomposite is synthesized by mechanical alloying elemental powders. Upon milling for a certain time, an abrupt exothermic reaction occurs and a large amount of NiAl and TiB2 compounds form simultaneously. It is suggested that two separate chemical reactions, i.e. Ni+Al --> NiAl and Ti+2B --> TiB2, are involved during the exothermic reaction. Addition of Ti and B to Ni-Al system impedes the structural evolution of Ni and Al powders and delays the abrupt reaction. The final products are equilibrium phases without any metastable phases formed. This type of reaction is suggested to be suitable for alloy systems with two large heat release reactions.

[37] A MOSSBAUER STUDY ON THE MECHANICALLY ALLOYED CU-SN ALLOYS

Yang YZ. Zhu YL. Li QS. Ma XM. Dong YD. Chuang YZ. - Journal of Materials Science & Technology. 14(6):551-554, 1998

Nanocrystalline epsilon and eta electron compounds and supersaturated solid solution of the Cu-Sn system have been prepared by mechanical alloying of elemental Cu and Sn powders. The atomic alloying and microstructure of the resultant alloys have been investigated by XRD, DSC and Sn-119 Mossbauer spectroscopy. A little amount of SnO2 was detected by Mossbauer spectroscopy, although no trace of diffraction peaks occurred in the XRD pattern. Thus the spectra for all the milled samples should be fitted using two quadrupole-splitting doublets: one corresponding to SnO2, the other corresponding to the resultant alloys. The composition dependence of the hyperfine parameters has been extensively discussed and explained well with respect to oxidation, surface effect resulting from grain refinement, coordination environment asymmetry and distortion caused or/and induced by mechanical alloying.

[36] MECHANOCHEMICAL POLISHING OF SI WAFER USING BACO3 COATED LAPPING TAPE

Kitajima K. Nakai M. Tottori T. Yasunaga N. Suzuki K. - International Journal of the Japan Society for Precision Engineering. 32(3):194-195, 1998

[35] INVESTIGATION OF MECHANO-CHEMICAL EFFECTS AND KINETICS OF AMORPHISATION OF SANITARY WARE FORMULATION DURING DRY MILLING

Yekta BE. Alizadeh P. Fazeli SA. Solati-Hashjin M. - British Ceramic Transactions. 97(5):227-231, 1998

The effects of dry ball milling a sanitary ware formulation were studies by XRD, particle size measurement, and

simultaneous thermal analysis for different milling times. The results show that, with increase in milling time, the peak intensity related to the dehydroxylation of kaolinite and the maximum temperature of reaction decreased. The kinetics of dehydroxylation of kaolinite as a function of milling time were studied and the activation energy calculated. The dehydroxylation reaction was found to be first order with a rate constant of 0.85 x 10(-6) s(-1).

[34] MECHANO-ACTIVATED REACTIONS OF HALOGEN SUBSTITUTION IN ALKYLHALIDES - V - ACTIVE STATES GENERATED BY MECHANICAL TREATMENT OF KCL AND CAUSING REACTIONS IN RI (R = ME, ET) [RUSSIAN]

Mitchenko SA. Dadali YV. Kovalenko VV. - Zhurnal Organicheskoi Khimii. 34(9):1293-1296, 1998

[33] SYNTHESIS AND PROCESSING OF A CU-IN-GA-SE SPUTTERING TARGET

Suryanarayana C. Ivanov E. Noufi R. Contreras MA. Moore JJ. - Thin Solid Films. 332(1-2):340-344, 1998

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

[31] PROTON TRANSFER IN THE SOLID STATE: MECHANOCHEMICAL REACTIONS OF FLUORIDES WITH ACIDIC SUBSTANCES

Fernandez-Bertran J. Reguera E. - Solid State Ionics. 112(3-4):351-354, 1998

Crystalline KF reacts with solid acidic substances by milling in an agate mortar. Proton transfer takes place between the acid and KF with formation of KHF2 and the K salt of the acid. The reactions were monitored by IR and XRD techniques in the solid reaction mixture. The following substances react readily with KF: KHSO4, KH2O4, (NH4)(2)SO4, and organic acids (oxalic, maleic, benzoic, p-chlorobenzoic, p-nitrobenzoic, miristic and nicotinic). NH4F, NaF are less reactive in that order while LiF and CaF2 are inert.

[30] KINETIC PROCESS OF MECHANICAL ALLOYING IN FE50CU50

Huang JY. Jiang JZ. Yasuda H. Mori H. - Physical Review B-Condensed Matter. 58(18):R11817-R11820, 1998

It is shown that mechanical alloying in the immiscible Fe-Cu system is governed by the atomic shear event and shear-induced diffusion process. We found that an alpha-to-gamma phase transformation, as evidenced by the Nishiyama-Wasserman orientation relationship, occurs by simultaneous shearing process when the grain size reduces to about 20 nm and Cu content reaches 20 at. % in bcc-FerichCu grains in the intermediate stage of milling of Fe50Cu50. Further milling promotes the interdiffusion between fcc-FerichCu and fcc-FeCurich, which is favored because of the similarity of the lattice structures, until a complete fee Fe-Cu solid solution is formed. The results provide significant insight into the understanding of recent experiments showing that chemical mixing of immiscible elements can bd induced by mechanical alloying.

[29] INFLUENCE OF MAGNETIZATION ON THE REORDERING OF NANOSTRUCTURED BALL-MILLED FE-40 AT. % AL POWDERS

Hernando A. Amils X. Nogues J. Surinach S. Baro MD. Ibarra MR. - Physical Review B-Condensed Matter. 58(18):R11864-R11867, 1998

Deformation and nanocrystallization created by ball milling in Fe-40 at. % Al powders give rise to a progressive structural disorder and a spontaneous magnetization. Annealing tends to restore the structurally ordered paramagnetic state. The ordering-disordering process brings about a volume change clearly related to the saturation magnetization. This effect is associated with variation of the density of states at the Fermi level. The reordering temperature exhibits an anomalous field dependence, which can be explained considering the combined effect of directional magnetic order and structural order.

[28] PROCESSING OF AMORPHOUS FE-W REINFORCED FE MATRIX COMPOSITES

Stawovy MT. Aning AO. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 256(1-2):138-143, 1998

Metal matrix composites in which crystalline Fe was reinforced by 2-phase particulates composed of an amorphous Fe alloy and nanocrystalline W were produced using a two-step mechanical alloying (Mh) process. The particulates, which had a composition of Fe-40 at.% W, were prepared first by MA in a SPEX(TM) mill. The composite powders were then formed by blending crystalline Fe with a range (2.6-33.7) of volume percentages of the reinforcing particulates using MA, employing both SPEX(TM) and attritor mills. Bulk samples were produced via cold pressing and annealing and analyzed for reinforcement distribution, porosity content, and hardness. The MA blending technique was effective at producing a uniform distribution of reinforcement particles. An increase in sample hardness was seen as volume fraction reinforcement increased. Unfortunately, the effectiveness of the compaction technique decreased with increasing volume fraction reinforcement, for porosity was found to increase monotonically with the volume fraction of the reinforcement.

[27] EFFECTS OF MECHANICAL ALLOYING ON THE REACTION SINTERING OF ZRSIO4 AND AL2O3

Khor KA. Li Y. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 256(1-2):271-279, 1998

Mechanical alloying was employed to study its effects on reaction sintering of zircon-alumina mixture to form mullite. Ball milling duration and materials were the main factors to investigate during mechanical alloying. It was found that zircon and alumina became amorphous during mechanical alloying. Ball milling media affects the recrystallization of amorphous phases upon subsequent heat treatment. The temperature of recrystallization of zircon and crystallization of zirconia is 30-70 degrees C higher with alumina ball-milling material than with zirconia ball-milling material, from 940 to 950 degrees C and from 880 to 910 degrees C. Ball milling time had important effect

on the temperature of mullite formation. The mullite formation temperature decreased with increasing ball-milling time, from 1556 degrees C for 5 h ball-milling to 1420 degrees C for 40 h ball-milling.

[26] EFFECTS OF MILLING AND PARTICLE SIZE DISTRIBUTION ON THE SINTERING BEHAVIOR AND THE EVOLUTION OF THE MICROSTRUCTURE IN SINTERING POWDER COMPACTS

Shiau FS. Fang TT. Leu TH. - Materials Chemistry & Physics. 57(1):33-40, 1998

In this investigation, it was observed that the contamination arising from the milling medium has a great influence on the sintering behavior. The densification rate of the powder milled with ZrO2 balls is largely inhibited, and the activation energy of ZrO2-doped alumina is significantly enhanced. The possible reasons for the enhancement of the activation energy were discussed. The effects of particle size distribution of powder compacts on the microstructural evolution of alumina during sintering were proposed and discussed.

[25] EFFECT OF SOLVENT FOR WET MILLING ON TENSILE PROPERTIES OF GLASS-CERAMICS GREEN SHEET [JAPANESE]

Moriya Y. Yamade Y. - Nippon Seramikkusu Kyokai Gakujutsu Ronbunshi-Journal of the Ceramic Society of Japan. 106(11):1079-1083, 1998

In this study, the cause of the variation of strength and elongation of glass ceramics green sheet as a function of the dispersion medium of wet milling was investigated. Green sheets were made from a mixture of glass powders ground by wet milling, ceramic fillers, plastic binder and solvent, The used glass was consisted of MgO, Al2O3, SiO2, B2O3 and K2O, In case of using water as a dispersion medium, wet milled glass powder became porous, because MgO and B2O3 ingredients were highly dissolved into water solvent, Therefore, it was considered that a mechanical adhesion between glass powder and binder was enhanced, and green sheet with high tensile modulus and with low elongation was produced. On the other hand, in case of using xylene as a dispersion medium, the surface area of glass powder was much smaller than that by using water, because components of the glass did not dissolve. It was considered that the adhesive strength between glass powder and binder was low so that the elongation of green sheet was the almost same as that of binder only.

[24] INFLUENCE OF POLYACRYLIC AMMONIUM ADDITION ON SUBMICRON GRINDING OF ALPHA-ALUMINA POWDER USING WET ROTATION BALL MILLING

Yokota K. Hashizuka Y. Kondo Y. - Nippon Seramikkusu Kyokai Gakujutsu Ronbunshi-Journal of the Ceramic Society of Japan. 106(11):1144-1146, 1998

The wet grinding of or-alumina powder with water is known to cause formation of aluminum tri-hydroxide on the particle surface of the powder by mechanochemical effects. The present authors have found that wet submicron-grinding of alpha-alumina powder using polyacrylic ammonium (PAN) as a, dispersant can be performed without generation of hydroxide on the particle surface. In this study, the relationship between the amount of PAN and the formation of aluminum tri-hydroxide in wet rotation ball milling of alpha-alumina powder using water as a solvent was investigated. Without addition of PAN, hydroxide generated on the particle surface of or-alumina powder after 24-h grinding. On the other hand with the addition of 1.7 mass% PAN, hydroxide did not generate after 168-h grinding. PAN was considered to be absorbed onto the AlOH2+ sites of the particle surface of alpha-alumina powder and, thus, to disturb the generation of hydroxide. Grinding efficiency was not improved by addition of PAN during submicron grinding.

[23] EMF MEASUREMENTS ON NANOCRYSTALLINE COPPER-DOPED CERIA

Knauth P. Schwitzgebel G. Tschope A. Villain S. - Journal of Solid State Chemistry. 140(2):295-299, 1998

Mixed oxide samples of nanostructured CuxCe1-xO2-y of various composition were generated by (i) chemical precipitation and ball milling and (ii) inert gas condensation. X-ray diffraction measurements suggested that copper oxide was dissolved in nanostructured cerium oxide up to concentrations of x = 11.15. Solid electrolyte cells of the type A, Cu2O/CuBr/CuxCe1-xO2-y (A = Cu or CuO) showed reversible cell voltages. The ratio of the formal chemical activities of CuO and Cu2O dissolved in nanostructured cerium oxide were calculated from the cell voltages. The results are discussed in terms of an apparent macroscopic solubility, due to interfacial segregation of copper oxide on nanostructured cerium oxide.

[22] SURFACE CHARACTERIZATION OF CU-M (M = TI, ZR, OR HF) ALLOY POWDER CATALYSTS

Molnar A. Bertoti I. Szepvolgyi J. Mulas G. Cocco G. - Journal of Physical Chemistry B. 102(46):9258-9265, 1998

X-ray photoelectron spectroscopy was used for surface characterization of amorphous Cu-M alloy powders (Cu40Ti60, Cu50Zr50, and Cu65Hf35) produced by mechanical alloying of the constituents, When used as catalyst in the dehydrogenation of alcohols Cu-Zr and Cu-Hf were found to undergo structural changes resulting in surface copper enrichment with a substantial fraction of copper being in fully reduced, metallic (Cu-0) state. This result accounts for the high and stable catalytic activity of Cu-Zr and Cu-Hf in the transformations of alcohols. In contrast, when Cu-Ti is used in inert atmosphere, surface enrichment of titanium is observed. Although segregation of copper occurs in the presence of hydrogen, only oxidized copper species are detected on the surface. In either case, the catalytic activity of Cu-Ti is inferior to the other two alloy samples. TiH2 is shown by XRD to be the characteristic species formed under hydrogen. This is stable in the presence of hydrogen and, therefore, Cu acts as an oxygen scavenger and forms oxidized surface species that do not show catalytic activity.

[21] CHARACTERISATION OF ALPHA-FEOOH GRINDING PRODUCTS USING SIMULTANEOUS DTA AND TG/DTG COUPLED WITH MS

Subrt J. Balek V. Criado JM. Perez-Maqueda LA. Vecernikova E. - Journal of Thermal Analysis. 53(2):509-517, 1998

Effects induced by grinding in synthetic goethite samples were studied. The products of alpha-FeOOH grinding were characterised by means of DTA, TG/DTG coupled with EGA (Mass spectrometry detection), powder X-ray diffraction analysis, and surface area determination.

[20] NANOCRYSTALLINE Y-BA-CU-O POWDER

Fang H. Ravi-Chandar K. - Journal of Superconductivity. 11(5):555-561, 1998
Nanocrystalline Y-Ba-Cu-O precursor was prepared by wet high-energy ball milling. It was found that with an increase of milling time, the orthorhombic 123 phase changes to a metastable (Y0.33Ba0.66)CuO3-x disordered cubic phase, and that the particle size distribution moves toward smaller sizes. The median particle size of the YBCO powder milled for 90 h was found to be less than 20 nm. This metastable cubic phase decomposes into a multi-phase mixture in the temperature range of 200-500 degrees C, and the mixture further tranforms to the pure tetragonal 123 phase at 800-900 degrees C. Furthermore, the temperature for nucleation of 123 from this nanometer-sized precursor was found to be lower than that in micron-sized 123.

[19] LITHIUM INSERTION IN BALL-MILLED GRAPHITE
Wang CS. Wu GT. Li WZ. - Journal of Power Sources. 76(1):1-10, 1998
The effects of mechanical milling on the microstructure, morphology and electrochemical performance of graphite powders with respect to lithium insertion are studied. After 150 h of ball-milling, the well-graphitized graphite has been pulverized into small particles with a size of about 50 nm, in which there are a lot of excess vacancies, microcavities and metastable carbon interstitial phases with sizes around 13 Angstrom. Due to the large surface energy, the merging of single particles is favoured and results in the formation of agglomerates with average size about 1 mu m. Voids are formed among the agglomerated particles. The ball-milled graphite shows reversible specific capacity for lithium of 700 mA h g(-1) (L1.88C6) with large hysteresis. The large reversible capacity is due mainly to Li doping at vacancies, microcavities (or at the edges of the metastable carbon interstitial phase) and voids. The bonding change between the interstitial carbon and the carbon in the aromatic plane that is induced by insertion of Li atoms leads to hysteresis. During charge-discharge cycles, the reversible capacity above I V decreases rapidly, which may be due to some vacancies and microcavities being annihilated by moveable and some bound interstitial carbon and to electrolyte penetrating gradually into voids formed by agglomerated particles during the Li insertion and desertion process.

[18] X-RAY AND NEUTRON DIFFRACTION STUDY OF NANOCRYSTALLINE TI-RU-FE-O COMPOUNDS
Blouin M. Guay D. Huot J. Schulz R. Swainson IP. - Chemistry of Materials. 10(11):3492-3497, 1998
The effect of adding oxygen on the structure of nanocrystalline Ti-Ru-Fe compounds obtained by high-energy ball-milling has been studied by X-ray and neutron diffraction using a Rietveld refinement analysis. It is shown that oxygen atoms readily oxidize Ti to form various types of titanium oxides depending on the oxygen content. In each case, a simple cubic structure (cP2-CsCl) is also formed during milling but with a concentration higher than expected on the basis of various reaction schemes. Through a detailed analysis of the neutron and X-ray diffraction peaks, it is shown that the la site of the CsCl-type unit cell is depleted from Ti atoms by preferential substitution with Fe. At high oxygen concentration, the alloy is a multiphase material containing Ti2-xRu1+yFe1+z, Ti oxides, Ru, and Fe.

[17] USING HIGH-TEMPERATURE CHEMICAL SYNTHESIS TO PRODUCE METASTABLE NANOSTRUCTURED COBALT
Leslie-Pelecky DL. Bonder M. Martin T. Kirkpatrick EM. Liu Y. Zhang XQ. Kim SH. Rieke RD. -Chemistry of Materials. 10(11):3732-3736, 1998
Chemical synthesis at elevated temperature (200 degrees C) produces a highly disordered form of cobalt similar to that produced by mechanical milling. Annealing of the disordered phase produces material with different face-centered cubic (fcc) to hexagonal close-packed (hcp) ratios, depending on the particular thermal treatment and the amount of disorder prior to annealing. The fcc-to-hcp ratio changes with the molecular weight (or boiling point) of the solvent, but this correlation does not appear to be linear. The coercivity and remanence ratio of chemically synthesized cobalt depend on the crystallite size, phases present, and amount of disorder.

[16] CHEMICAL PREPARATION AND CHARACTERIZATION OF METAL-METALLOID ULTRAFINE AMORPHOUS ALLOY PARTICLES
Chen Y. - Catalysis Today. 44(1-4):3-16, 1998
Ultrafine amorphous alloy particles (UAAP) constitute an overlapping area of amorphous alloys and nanophase materials. Special properties of the particles derived from the combination of their long-range disordered structure and nanoscale size are of great interest in catalysis. This paper reviews some fundamental aspects in the chemical preparation of UAAP consisting of transition metal (M) and metalloid elements (B, P) and their applications in catalysis. The three fundamental reaction equations in the preparation of MB UAAP, the autocatalytic nature of the chemical reaction to produce NiP UAAP, and a milling-induced solid state reduction method are discussed. Their interesting catalytic properties, especially their unique selectivities in some of the hydrogenation reactions, have shown promising potential applications of UAAP in catalysis. With regard to the interactions between the transition metal and metalloid elements of the UAAP, experimental results and theoretical calculations have led to the suggestion that the charge transfers between M and B or P are in the same direction but to different extents, i.e., from metal to metalloid elements.

[15] INTERCALATION VORTICES AND RELATED MICROSTRUCTURAL FEATURES IN THE FRICTION-STIR WELDING OF DISSIMILAR METALS
Murr LE. Li Y. Flores RD. Trillo EA. Mcclure JC. - Materials Research Innovations. 2(3):150-163, 1998
Dissimilar plates (0.6 cm thick) of copper and 6061 aluminum alloy; and 2024 aluminum alloy and 6061 aluminum alloy were friction-stir welded at various rotation speeds (400-1200 rpm) and traverse speeds of 1 to 3 mm/s, and produced variations of vortex and other swirl-like intercalations, especially near the weldzone/work-piece interface. These vortex-like structures are composed of dynamically recrystallized, fine-grained intercalation microstructures which have been observed by optical metallography, scanning electron microscopy and transmission electron microscopy. They are similar to those characterizing mechanical alloying. Such microstructures are especially fascinating because although they resemble vortex instabilities which occur in turbulent fluids, they are formed by extreme deformation in the solid state since centerline temperatures through the weld zone are about 420 degrees C, or similar to 0.7 T-M in the 6061 aluminum system.

[14] USING HIGH-TEMPERATURE CHEMICAL SYNTHESIS TO PRODUCE METASTABLE NANOSTRUCTURED COBALT

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[7] HYDROGENATION DISPROPORTIONATION DESORPTION RECOMBINATION IN SM-CO ALLOYS BY MEANS OF REACTIVE MILLING

Gutfleisch O. Kubis M. Handstein A. Muller KH. Schultz L. - Appl. Phys. Letters. 73(20):3001-3003, 1998

Sm-Co-type alloys were disproportionated by milling in hydrogen at enhanced temperatures. X-ray diffraction confirmed the disproportionation of the SmCo5 and Sm2Co17 phases into Sm hydride and alpha-Co. This "reactive milling" procedure facilitates the disproportionation of these alloys which are characterized by a very high thermodynamic stability, and therefore are not available for a standard hydrogenation disproportionation desorption recombination treatment. Recombination of the reactively milled powders leads to the formation of the original phases, now with dramatically refined grain sizes of around 25 nm and significant coercivities such as mu(0J) H-C = 3.7 T in the case of the SmCo5 alloy. Exchange coupling between the nanoscaled grains resulted in magnetically single phase behavior despite a multiphase microstructure. In particular, for the Sm2Co17 alloy, a remanence enhancement was observed for recombination temperatures less than or equal to 700 degrees C.

[6] X-RAY AND NEUTRON DIFFRACTION STUDIES OF THE AMORPHOUS ZRPD ALLOYS

Alhajry A. Alassiri M. Cowlam N. - Journal of Physics & Chemistry of Solids. 59(9):1499-1505, 1998

X-ray and neutron diffraction measurements have been made on amorphous ZrPd samples prepared by three different techniques, fast quenching (FQ) from the melt, mechanical alloying (MA) and mechanical milling (MM). An important feature of our samples is the small differences between the weighting factors for the partial structure factors (PSFs) and the pair density functions for both X-ray and neutron radiations. This was exploited to investigate the effect of the preparation method on the final structure of these samples. The results showed that the structure factors S(Q) and the reduced radial distribution functions G(r) of these samples prepared by different methods are similar and independent of the way used for producing them.

[5] PHYSICOCHEMICAL MECHANICS OF GRINDING OF SOLIDS

Khodakov S. - Colloid Journal. 60(5):631-643, 1998

Phenomena occurring in the grinding process and the regularities of these phenomena are considered from the standpoints of physicochemical mechanics pioneered by Rebinder. The main regularities of physicochemical mechanics of grinding are formulated. The resultant equations describe this process within the entire range of dispersity. These equations describe quantitatively the physicochemical features of ground material, power consumption of grinding units, the process conditions, and the effect of the environment. The results of one of the branches of physicochemical mechanics (sorption mechanochemistry) are reported; the mechanism and regularities of mechanosorption interaction of liquids and vapors with mechanically activated surface layers of particles, as well as the effect of such interaction on the energy capacity of limiting plastic deformation and fracture of particles are established. Based on theoretical and experimental studies, the mathematical relationships between the energy consumed by the grinding unit and the dispersity of ground materials are proposed, which can be used in practice. These equations (the laws of grinding) are suitable for the calculation of technological process of grinding in open (without separation) and dosed (with separation) technological cycles.

[4] MECHANICAL ALLOYING OF THE TI-NI SYSTEM

Takasaki A.- Physica Status Solidi A-Applied Research. 169(2):183-191, 1998

There kinds of Ti-Ni elemental powders, Ti45Ni55, Ti50Ni50 and Ti55Ni45, were mechanically alloyed by a planetary ball mill for alloying times up to 10 h, and the alloying process and the microstructures after heating at temperature of 1273 K were investigated by powder X-ray diffractometry. The Ti55Ni45 powder formed an amorphous phase after mechanical alloying for 10 h, while the Ti50Ni50 powders formed a disordered b.c.c.-TiNi phase. The Ti45Ni55 powder also formed the disordered b.c.c.-TiNi phase at an intermediate stage of mechanical alloying but turned to an amorphous state with increasing alloying time. After heating at 1273 K, the Ti2Ni phase has been formed in all powders, and the ordered B2-TiNi (CsCl structure) phase was observed in the Ti50Ni50 and Ti45Ni55 powders, but a monoclinic TiNi phase in the Ti55Ni45 powder. In the Ti45Ni55 powder also the TiNi; phase has been formed. The amounts of these intermetallic phases are dependent on the chemical compositions of the starting powders.

[3] NOVEL MECHANOCHEMICAL METHOD FOR PREPARING CORDIERITE AND CORDIERITE-BASED SUPPORT

Avvakumov EG. Devyatkina ET. Kosova NV. Kirichenko OA. Lyakhov NZ. Gusev AA. - Kinetics & Catalysis. 39(5):663-665, 1998

A new method for the synthesis of cordierite at low temperatures based on the mechanical activation of mixtures containing magnesium, aluminum, and silicon hydroxides or natural aluminosilicates (kaolinite, talc, and hydrargillite) followed by treatment at 1260 degrees C was proposed. The monolith honeycomb catalysts with supported iron oxide exhibiting high activity in the oxidation of butane and carbon monooxide were prepared.

[2] SOLID STATE SI-29 MAGIC ANGLE SPINNING NMR - INVESTIGATION OF BOND FORMATION AND CRYSTALLINITY OF SILICON AND GRAPHITE POWDER MIXTURES DURING HIGH ENERGY MILLING

Xie XQ. Yang ZG. Ren RM. Shaw LL. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 255(1-2):39-48, 1998

In this study, solid state Si-29 nuclear magnetic resonance (NMR), transmission electron microscopy (TEM), and X-ray diffraction (XRD) have been used to study the bond formation and structural changes of Si in Si-graphite powder mixtures during high energy milling. Both TEM and NMR analyses have shown that amorphization of Si occurred

under the milling conditions used in this study. Furthermore, the NMR spectra have provided evidence that even under milling conditions that do not result in the formation of crystalline SiC in-situ during milling, most of the Si and C atoms are intimately mixed at atomic levels in the amorphous phase and the nominal composition of the amorphous phase is about 1:1 of the Si:C molar ratio, corresponding to the Si:C molar ratio of the starting powder mixture. Comparison among XRD, TEM, and NMR analyses indicates that the reduction of the integrated area of Si reflections in the XRD patterns is primarily due to amorphization of Si. Based on these results, it is proposed that amorphization of Si is primarily induced by crystallite refinement and mixing of Si and C at atomic levels is assisted by micro-diffusion during milling.

[1] MECHANICAL ACTIVATION OF THE SOLID-STATE REACTION BETWEEN AL AND TIO2

Welham NJ. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 255(1-2):81-89, 1998

Titanium dioxide and aluminium have been mechanically milled together for times up to 100 h. The onset temperature of the main reaction was observed to decrease from 1050 to 660 degrees C after 5 h of milling, increasing the milling time to 100 h decreased the onset to 560 degrees C. Elemental maps showed that the major decrease was due to increased mixing of the phases. Beyond 5 h, the temperature at which the reaction was half completed was found to be a function of log (milling time) and seemed to be correlated with the decrease in crystallite size. There seemed to be three different rate determining steps, the importance of each changing with milling time. Long range solid state diffusion was predominant for milling times < 5 h, short range diffusion for 5-50 h and, possibly, diffusion along lattice defects after 100 h milling. An onset temperature of 560 degrees C was observed for milling times of greater than or equal to 10 h. This was thought to be due to a change in the rate determining step from mass transport to chemical reaction control.

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