



**RESEAU FRANÇAIS DE  
MECANOSYNTHESE**

**Lettre N°83**

**Février 2002**

**185 Groupes de Recherche  
(dont 111 à l'étranger / 33 Pays)**

**Bureau du RFM : E. Gaffet (Président)  
G. Le Caër (Secr. Gén.), A.R. Yavari (Trés.)**

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**Bulletin d'adhésion 2002 / Subscription Print**

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

Eric GAFFET

CNRS UMR5060 « Métallurgies et Cultures »  
Nanomaterials Research Group

Site de Sévenans (UTBM) - F90010 - Belfort Cedex - France

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

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J'aimerais adhérer au Réseau Français de Mécanosynthèse / want to become a member of the French Mechanical Alloying Network

Chèque ci joint / Check enclosed in the amount of **20 Euros (20€)**

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

Le site web du RFM est :

<http://www.bls.fr/amatech>

Rubrique Pages Sciences et Techniques pour l'Ingénieur (Rubrique Sciences)  
vous y trouverez les anciennes lettres du RFM (accessible par Adobe Acrobat)

les statuts du RFM ainsi que les annonces concernant les JRFM'2001 et quelques éléments mis à jour régulièrement concernant les derniers résultats dans ce domaine.

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Les JRFM'2002 seront intégrées dans le cadre du Congrès  
**Matériaux 2002** (Tours - France, du 21 au 25 Octobre 2002)

Symposium 1 :

**Poudres et Matériaux Nanostructurés, du fondamental aux applications industrielles**

Website : <http://www.materiaux2002.net> : E\_mail : [materiaux@materiaux2002.net](mailto:materiaux@materiaux2002.net)

**Sommaire**

- ⇒ Thèses / Congrès
- ⇒ Bibliographie du mois d'Octobre
- ⇒ Dossiers d'annonces techniques

**Congress and School Announcements**

**SHS 2001**  
**VI International Symposium on Self-Propagating High-Temperature Synthesis**  
**Haifa, Israel - 17 - 21 Février 2002**

<http://www.ism.ac.ru/events/shs2001/shs2001.html>

E-mail: [gotman@techunix.technion.ac.il](mailto:gotman@techunix.technion.ac.il)

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**Nano 2002**

16 - 21 Juin 2002

Orlando, Florida - USA

Website : <http://www.nano2002.com/>

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Workshops

**Gordon Research Conference on Granular and Granular-Fluid Flow**

Plymouth, NH, USA June 30 - July 5 ,2002

<http://sol.rutgers.edu/~shinbrot/gordon2002/gordon2002.html>

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**ICSTR**

**INTERNATIONAL CONFERENCE ON SOLVO-THERMAL REACTIONS**

July 22-26, 2002 - Hilton East Brunswick / East Brunswick, New Jersey,

Abstracts are being solicited for an international conference on solvo-thermal reactions (ICSTR). The meeting will be held on July 22-26, 2002 at the Hilton East Brunswick, in East Brunswick, New Jersey, which is conveniently located near all major forms of transportation. Solvothermal reactions are chemical reactions performed in aqueous and nonaqueous solvents at elevated temperature and pressure. They are a novel low temperature means (soft chemistry approach) for synthesis and processing of materials, nanotechnology, pharmaceuticals, environmental remediation, energy applications, catalysis and many other applications. The abstract submission deadline is March 15, 2002. Reduced rate early-bird registration must occur prior to May 15, 2002.

Reduced registration rates are available  
for graduate students.

More information on this meeting can be found at <http://www.ICSTR.rutgers.edu/>

or by contacting Professor Richard E. Riman at Rutgers University via  
[riman@email.rci.rutgers.edu/732-445-4946\(v\)/732-445-6262\(f\)](mailto:riman@email.rci.rutgers.edu/732-445-4946(v)/732-445-6262(f))

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**RQ11**

Rapidly Quenched and Metastable Materials

25-30 August 2002

Department of Materials, University of Oxford, UK

Contact: RQ11 Conference Organiser, Beggars Roost, Channels End Road,

Comworth Bedford MK44 2NS, U.K.

Tel: +44 (0) 1234 378862

Fax: +44 (0) 1234 376219

E-mail: [mailto:rq11@materials.ox.ac.uk](mailto:mailto:rq11@materials.ox.ac.uk)

Website: <http://www.materials.ox.ac.uk/rq11>

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**10th European Symposium on Comminution**

Heidelberg from 2-5 September 2002.

Org. European Federation of Chemical Engineering

Full information available at <http://www.comminution2002.de>

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International Symposium on

Metastable, Mechanically Alloyed and Nanocrystalline Materials

(ISMANAM-2002)

Seoul, Korea, 8-12 September, 2002.

Web site : <http://anu.andong.ac.kr/~ismanam/>

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L. A. C. A. M. E - 2. 0. 0. 2

EIGHTH LATIN AMERICAN CONFERENCE

ON APPLICATIONS OF THE MÖSSBAUER EFFECT

PANAMA, 22-27, SEPTEMBER, 2002.

E-mail: [mailto:lacame2000@fisica.ciens.ucv.ve](mailto:mailto:lacame2000@fisica.ciens.ucv.ve)

<http://www.up.ac.pa/Eventos/lacame2002/inicio.htm>

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**Matériaux 2002**

Tours - France

21- 25 Octobre 2002

Website : <http://www.materiaux2002.net>

E\_mail : [materiaux@materiaux2002.net](mailto:materiaux@materiaux2002.net)

please post in Bulletin Board

## ICCE/9 San Diego

NINTH INTERNATIONAL CONFERENCE ON COMPOSITES ENGINEERING

July 1-6, 2002

reception starts Sunday evening, June 30, 2002

WYNDHAM Emerald Plaza Hotel, San Diego (Four Diamond Rating)

\$83.00+tax per night for six nights, \$99.00+tax per night for five nights or less

sponsored by International Community for Composites Engineering

The ICCE/9 conference will be held in San Diego, California, USA. Sessions will start Monday morning July 1 and finish Saturday morning July 6, 2002.

The goals of the ICCE/9 San Diego conference is to (1) BRIDGE THE GAP between materials science, mechanics and processing of composites (2) INTERDISCIPLINARY research bridging the gap between aerospace technology, bio-materials, chemistry, electronics, fluid mechanics, infrastructures, magnetic materials, **nanotechnology**, physics, powder metallurgy, sensors/actuators, among others (3) LEVERAGING of research resources and encouraging joint research among participants. The following symposia (among many others not listed here) will be highlighted:

Ageing, Flammability	Materials Sciences ( <b>all</b> areas)
Bio-Dental Composites	Mathematical Modeling
Chemistry	Metallurgy ( <b>all</b> areas)
Coatings and Wear	Metal Matrix Composites
Ceramics Composites	Metals, Alloy, Minerals
Characterization of Materials	Micromechanics
Computational Mech.	<b>Nano-Materials</b>
Damage, Fracture and Fatigue	Non-Destructive Testing
Earthquake Engineering	Packaging Barrier Polymers
Electronic/Magnetic Mat. Particulate Composites	Plastics Engineering
Experimental Techniques	Powder Metallurgy
Fiber/Matrix Properties	Processing MMC, CMC, PAC
Functionally Graded Mat,	Smart Composites
General Applications	Structural Dynamics
Impact Engineering	Textile Composites
Industrial Composites	Vibration, Buckling
Infrastructures Composites.	Wood or Construction Mat.
Interface, Interphase	Wave Propagation
Joints and Adhesion	

**Major symposium: Nano Materials, Magnetic Materials, Thin Films**

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Margherita Clerico	John Hart-Smith	Yun-Hae Kim	Rong Liu	A. Oudjehane	Shiro Suzuki	Masaru Zako
Leonard Daniel	Paul Herrington	Sug-Won Kim	Karl Maile	H. Paradies	K. Takemura	
Lorence Dieval	Peter Hing	Yuuji Kimura	S.T. Mileiko	Joung-Man Park	Vasco Teixeira	

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The ICCE/9 conference proceedings will consist of two-page extended abstracts. Each page has a two-column format. Please use single-line space to save space. The first line is the paper title (spanning two columns) and it should be of size 14 with all capital "bold" typeset. The second line is blank, the third line is the name of the authors in bold face font size 11, centered, spans two columns. The fourth line is the postal mailing address, preferably written in only one line non-bold, font size 11, also centered. The rest of the text is also in font size 11. Please keep approx. 3/4 inch margin on all edges. Laser print quality is required. Please underline the name of the presenter. Abstracts will be reviewed, and must be full of details and results, thus, please include as many figures as possible in these two pages, and fill up the entire second page. Abstracts should be emailed before **May 1, 2002** (always simultaneously postal mail, fax is only temporary photographs must be original)

Due to limited space of over 500 oral paper presentations, each presenter will present no more than one paper.

**ABSTRACT SUBMITTER must agree to ORALLY PRESENT THE PAPER.** We welcome research/educational table exhibitions.

Interested authors should submit **IMMEDIATELY** the tentative PAPER TITLES and a suggested subject category to: Dr David Hui, University of New Orleans, Dept. of Mechanical Engineering, New Orleans, LA 70148. Tel: (504)-280-6652 FAX/Tel:(504)-280-6192, dhui@uno.edu <http://www.uno.edu/~enr/composite>

**Wyndham San Diego** hotel group rate is \$83+tax per night for six nights, \$99+tax per night for five nights or less. Please mention ICCE and reserve from the "local" Wyndham hotel during office hours (7AM-5PM San Diego time), Tel: (619)-239-4500, please do not contact the Wyndham central reservation system because they do not know our reduced group rate. Wyndham Hotel FAX: (619)-239-3274, you will get fax reply if you send fax. Pre-registration fee is \$350 before May 1, 2002, \$400 before June 1, 2002 \$450, after June 1, 2001 and on-site rate is US\$490

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**SOUTENANCES DE THESE**  
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**Sophie Soiron**

**Influence de la mécano-chimie sur les propriétés structurales et catalytiques d'oxydes de structure type pérovskite et spinelle**

16 novembre 2001, à Amiens, Amphi Figlarz, à 14h

**Jury:**

**Rapporteurs:** M. Jean Mimault (*Université de Poitiers*), M. Edmond Payen (*Ecole de chimie de Lill*)

**Examineurs:** M. Luc Aymard (*Université d'Amiens*), M. Christian Julien (*CNRS- Université de Paris VI*), M. G-Abbas Nazri (*General Motors R&D*), Melle. Aline Rougier (*CNRS- Université d'Amien*), M. Bechara Taouk (*Université de Compiègnese*), M. Jean-Marie Tarascon (*Université d'Amien*)

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**F. Dore**

**"Densification de pseudo alliages W - Cu à partir de phases submicroniques"**

Université de Grenoble - 13 Novembre 2001

**Jury :**

E. Gaffet (Rapporteur), J.-L. Jorda (Rapporteur), C. Allibert (Directrice de Thèse),  
C. Martin (Co - Dir. De Thèse), J.-F. Lartigue, M. Soustelle

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**Raphaël JANOT**

Université de Nancy I – 24 octobre 2001

**Mécanosynthèse en milieu liquide de composés graphite-lithium superdenses, de graphite très anisométrique et de maghémite supportée ou non sur graphite**

**Jury :**

J. Conard (Rapp.), M. Danot (Rapp.), P. Ehrburger, D. Guérard (Dir. Thèse), R. Marassi, A. Rougier

\*\*\*\*\*

**Thierry Girot**

**"Cinétique et modélisation des transformations de phases induites par broyage à haute énergie dans TiO2 anatase "**

Thèse INPL, 15 Octobre 2001

**Jury :**

J. Focet, D. Michel (R), J.C. Niepce (R), F. Radjai, G. Le Caer, A. Mocellin, S. Begin (Dir. Thèse)

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**Sébastien Lehnard**

**"Texture, Microstructure et Propriétés d'un Alliage Fe-40 Al à grains fins obtenu par métallurgie des poudres et extrusion : Influence des paramètres du procédé et de traitements thermiques"**

Université de Metz - 5 octobre 2001-08-23

**Jury :**

R. Schwarzer (Rapp.), E. Gaffet (Rapp.), JP Morniroli, V Skrotzi, R. Baccino, A. Hazotte,

F. Wagner (Dir. Thèse), Th. Grosdidier (Co. Dir. Thèse)

\*\*\*\*\*

**Nathalie Bouad**

**"Mise au point d'un procédé d'élaboration de matériaux thermoélectriques pour thermogénérateur.**

**Potentialité de la mécanosynthèse d'alliages à base de tellurure de plomb"**

**Montpellier, Université Montpellier II, 10 mai 2001**

**Jury :**

J. Foct, J.C. Niepce, H. Scherrer, R. Griot, A.M. Bouchardy, J. Delallée, Y. Lacrouts-Cazenave, M. Ribes, J.C. Tédénac, R.M. Marin-Ayral (directeur de thèse)

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**Cooperative Research on Related Areas**

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**France (12/04/2001)**

Le portail Internet "France Contact" a été lancé: ce portail s'adresse aux chercheurs étrangers séjournant ou ayant séjourné en France et permettra le suivi et l'animation du réseau que constituent les milliers de chercheurs étrangers ayant effectué un séjour scientifique au sein des établissements et des organismes de recherche français:

Website : <http://www.francecontact.net>

**Europe (6/03/2001)**

The ESF, on the recommendation of the scientific Standing Committee for Physical and Engineering Sciences (PESC), will support, in fields related to PESC's remit, approximately 10 ESF Exploratory Workshops to be held in 2002.

Each workshop will allow 20-25 leading European scientists to explore novel ideas at the European level with the challenging aim to "spearhead" new and preferably inter-disciplinary areas of research.

In specific terms, PESC's 2001 Call is for workshop proposals on R&D subjects which are NOVEL AND PREFERABLY INTERDISCIPLINARY and which concern emerging fields within any of the following areas: chemistry, physics, mathematics, information sciences, fundamental engineering sciences, materials sciences, and technologies research in these areas.

The PESC Call is available at <http://www.esf.org/physical/WorkshopCalls/Call2001.htm>



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**Job Vacancies, Ph D Position and, Post Doc Position  
Requests – Proposals**

**De L. Aymard CNRS / Amiens (France)  
1/10/2001**

**Pour la Rentrée Universitaire 2001 - 2002**

Le Laboratoire de Réactivité et de Chimie des Solides à Amiens recherche pour la rentrée prochaine :

- un étudiant pour une thèse de 3ème cycle. (Durée 3 ans) à partir de septembre 2001
- un étudiant pour un Stage Post Doc (12 mois prolongement possible) à partir de septembre 2001.

Domaine de Recherche: Stockage d'énergie, Hydrures Métalliques.

Envoyer vos CV avant la fin juillet à L. Aymard LRCS

Email : <mailto:luc.aymard@u-picardie.fr>

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## Bibliographie Récente

### Livres ou "Special Issues"

(21/06/2001)

**From Christian Wohlbier (Scientific. Net Webmaster)**

This is a service of <http://www.scientific.net>

\*\*\* **Materials Science Forum** \*\*\*

Materials Science Forum specializes in the rapid publication of international conference proceedings and stand-alone volumes on topics of current interest. It covers all areas of Materials Science, Solid State Physics and Solid State Chemistry. The periodical is indexed in Science Citation Index and covered by all major abstract media.

Volume 246 until 246 [Surface Coatings for Advanced Materials] and

Volume 207 until 209 [Intergranular and Interphase Boundaries in Materials]

<http://www.scientific.net/msf>

\*\*\* **Solid State Phenomena** \*\*\*

Solid State Phenomena specializes in the rapid publication of international conference proceedings and stand-alone volumes on topics of current interest in the field of solid state physics and its applications to materials science related topics. The periodical is indexed in Science Citation Index and covered by all major abstract media.

Volume 61 until 62 [Contemporary Studies in Condensed Matter Physics],

Volume 59 until 60 [Interfaces and Plasticity] and

Volume 57 until 58 [Gettering and Defect Engineering in Semiconductor Technology]

<http://www.scientific.net/sfp>

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(07/06/2001)

« **Strategic and Technological Watch on Nanomaterials** »

by **E. Gaffet** (1998 – 2000) – 4 CD reports (6.000 analysed references)

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

Website : <http://www.innovation128.fr/>

(28/05/2001)

**Advanced Ceramic Materials**

\*\*\* **Key Engineering Materials, Volume 122 until 124** \*\*\*

In spite of the very great progress made in ceramic science, and the elegance and excitement of the research which has been performed, the real driving force for developments in ceramics remains their potential applications. The opportunity for dramatic scientific advances was certainly one reason for the "ceramic fever" of a decade ago, but there is also no doubt that the

prediction of an annual market for fine ceramics, amounting to 6 billion Yen played a role. The challenge is to ensure that ceramics can be successfully introduced into the full breadth of applications where their properties have long made them so appealing. The present volume takes a refreshing and firm step towards the realization of this aim. The publication of a book which sets out to present ceramics from the specific point of view of applications is an event greatly to be welcomed. Systematic organization into various types of application ensures that the reader can fully appreciate the outstanding opportunities offered; and the present limitations. Armed with such a survey, the engineer and scientist will be fully alert to possibilities for progress whenever these arise. 1. Introduction. 2. Electrical and Electronic Functions. 3. Magnetic Functions. 4. Chemical and Physical Functions. 5. Mechanical and Thermal Functions. 6. Biological Functions. 7. Nuclear Applications. 8. Ceramic Coatings. 9. Selected Ceramics with Multi-Applications.

<http://www.scientific.net/kem>

(11/2000) **Information from Fritsch (A. Kohler)**

The subject of the sixth forum part, Fritsch Forum Part VI scheduled for September 14/15th, 2000, will be "high-energy fine grinding". Research and Development demand general-purpose grinding processes which simultaneously exactly define the required energy and the type of stress. This is the only way that reliable results can be achieved when determining activation energies or the mechanical alloying. It must be possible to reproducibly adjust all of the grinding parameters affecting the grinding results.

Participants from research, development and industry will report on demands and novel technological solutions in developing innovative milling technologies. One of the highlights of the event will be FRITSCH's new Vario-planetary mill "pulverisette 4". This planetary ball mill can simulate ball mills of conventional construction, precisely copy the types of stresses that occur there, and thus reproduce or optimise grinding processes. Due to the great flexibility when selecting the grinding parameters, it is possible to produce results that are unattainable with other ball mills. It is the ideal mill for mechanical activation and alloying. The main applications are in the area of material research and naturally wherever a powerful, innovative laboratory planetary mill is needed.

An extensive report has been written about this event which details and makes readily available the relevant parts of the lectures and the extensive results of the discussions. Anyone interested can request a copy of the complete report for this forum part VI event on the topic "high-energy fine grinding". Please contact Andrea Köhler, FRITSCH GMBH, Industriestrasse 8, D-55743 Idar-Oberstein, (Phone: 0049/6784/7046, E-Mail: koehler@fritsch.de)

(7/07/2000) - From Victor Riecanaky Publisher



Lettre RFM N°83 - Février 2002  
Corresp. : <mailto:Eric.Gaffet@utbm.fr>

Cambridge International Science Publishing <http://www.demon.co.uk/cambsci/homepage.htm>

## MACROMOLECULAR MECHANOCHEMISTRY

Volume 1: Polymer Mechanochemistry - by Cleopatra Vasiliu OPREA & Florin DAN

Department of Macromolecules, Gh. Asachi, Technical University, 6600 Iasi, Romania

Macromolecular Mechanochemistry presents from theoretical and experimental point of view the main problems of this field, including the results obtained in more than a century of research. It is organised in two volumes: Polymer Mechanochemistry and Polymers with Chemomechanical Functions, respectively. The present volume deals with: Chained Polystage Character of Mechanochemical Process (1), Mechanochemistry of Polymers Deformation (2); Mechanochemistry of Polymer Fracture (including also the Fracture of Composite Materials) (3), and Mechanochemical Processes for Energy Conversion (4). In this frame, the theoretical and experimental material is organised in correlation to the reaction mechanism, the type of mechanical solicitation, and the nature of environmental medium. This book is addressed to professors, students, and researchers involved in the field of polymer science, to engineers from the industry of synthesis and processing of plastic materials, elastomers and fibres, as well as to specialists from all technical domains that exploit polymer-based materials. They will find in the book examination of the theoretical, experimental and applied problems and wide access to the basic literature in this field. Contents

1. Chained polystage mechanism of mechanochemical processes
2. Mechanochemistry of polymers deformation
3. Mechanochemistry of Polymer Fracture
4. Mechanochemical Processes for Energy Conversion

Volume 1 (ISBN 189832672X) will be published in September 2000, approx. 500 pages, cased, approximate price £ 80.00; (volume 2 will be published at the end of - 2000)

Send your preliminary order to <mailto:orders@cisp.demon.co.uk>

(9/06/2000)

**"Mechanical Alloying : FABRICATION OF ADVANCED MATERIALS AT ROOM TEMPERATURE" by M. Sherif El-Eskandarany**

(ISBN: 977-299-089-7) Published by DAR AL-FIKR AL-ARABI, Cairo-Egypt.

The price of the book is \$50, and a special discount (20%) is offered to all the RFM member.

Preface

Mechanical alloying (MA) process using ball-milling and/or rod-milling techniques, has received much attention as a powerful tool for fabrication of several advanced materials, including equilibrium, nonequilibrium (e.g., amorphous, quasicrystals, nanocrystalline, etc.), and composite materials. In addition, it has been employed for reducing some metallic oxides by milling the oxide powders with metallic reducing agents at room temperature. The MA is unique process in that a solid state reaction takes place between the fresh powder surfaces of the reactant materials at room temperature. Consequently, it can be used to produce alloys and compounds that are difficult or impossible to be obtained by the conventional melting and casting techniques.

This book intended primarily to serve as an introduction to the MA process, including general description of the process, starting material requirements, the equipment, characterizations of the milled powders, and consolidation techniques, which used to compact the powder into fully-dense bulk materials.

The book contains several typical examples of selected advanced materials that have been fabricated by MA. This book is aimed at either senior undergraduate/post graduate students or materials scientists/metallurgists. - M. Sherif El-Eskandarany - April 2000 - Cairo - Egypt

(3/02/2000)

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

1. MECHANICAL ALLOYING - FUNDAMENTALS AND APPLICATIONS <http://www.demon.co.uk/cambsci/book52.htm>

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

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

Energy transfer and energy maps;

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

Modelling mechanical alloying (mechanistic models, deformation, coalescence and fragmentation, evolution of particle size, milling time, powder heating, powder cooling, atomistic model, thermodynamic and kinetic model) Joining of mechanically alloyed materials; Rapid solidification and mechanical alloying; Applications (nickel-based superalloys, Al-based materials, supersaturated solutions, magnetic materials, mechanically alloyed powders for spray coatings, superplasticity, tribological materials, composites, amorphous solids, nanocrystalline materials, solid-state chemical reactions, etc). ISBN 1898326568, 160 pages 234 156 mm, cased, £45.00, 1999

**DISPERSION STRENGTHENED ALUMINIUM PREPARED BY MECHANICAL ALLOYING, by M Besterçi - <http://www.demon.co.uk/cambsci/book51.htm>**



Lettre RFM N°83 - Février 2002  
Corresp. : <mailto:Eric.Gaffet@utbm.fr>

**1. Characteristics of dispersion-strengthened systems** 2. **Mechanical alloying** (kinetics and mechanism of preparation of the Al-C system by mechanical alloying; compaction of powders and heat treatment of compacts);  
3. Microstructure and quantitative evaluation of parameters of dispersion-strengthened materials (definition and properties of interparticle distance; experimental possibilities of determination of structural objects; models of heterogeneous structures and their evaluation; simulation of model structures; analysis of the spatial distribution of particles in the Al-Al<sub>4</sub>C<sub>3</sub> material)  
4. Static and dynamic mechanical properties (mechanical properties at elevated temperatures; mechanical properties at 20 °C; effect of interface on the mechanical properties; superplastic properties of the system; thermal stability of the system; creep characteristics; creep-fatigue characteristics)  
References - ISBN 189832655X, 90 pages, 234 156 mm, soft laminated cover, £25.00, 1999

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**"Mechanical Alloying : Fundamentals and Applications"**

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

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

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

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

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

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

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

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



## Patent / Brevet

### ONE STEP SYNTHESIS AND CONSOLIDATION OF NANOPHASE MATERIALS

Z.A. Munir, F. Charlot, F. Bernard, E. Gaffet – International patent WO 0112366 (publié le 22.02.2001)

Solid reaction products with a dense nanocrystalline structure are formed from reactant particles with diameters in the nano – scale range by compacting the particles into a green body, then passing an electric current through the body causing Joule heating sufficient to initiate the reaction to form the reaction product while simultaneously applying pressure to the reacting body to densify it to a density approaching the theoretical density of the pure product. Surprisingly, this process results in a reaction product that retains the nanocrystalline structure of the starting materials, despite the fact that a reaction has occurred and the materials have been subjected to highly stringent conditions of electric current, heat and pressure.

L'adresse du site web où trouver le texte complet du brevet...

<http://164.195.100.11/netacgi/nph-Parser?Sect1=PTO2&Sect2=HITOFF&p=1&u=/netahtml/search-bool.html&r=1&f=G&l=50&co1=AND&d=ft00&s1=gaffet.INZZ.&OS=IN/gaffet&RS=IN/gaffet>

ou encore pour la version brevet d'application

<http://12.espacenet.com/dips/bnsviewer?CY=ep&LG=en&DB=EPD&PN=WO0112366&ID=WO+++0112366A1+I+>

## Périodiques

### [58] CHARACTERIZATION OF NANOSCALED HETEROGENEITIES IN MECHANICALLY ALLOYED AND COMPACTED CUFE

Wanderka N. Czubyko U. Naundorf V. Ivchenko VA. Yermakov AY. Uimin MA. Wollenberger H. - Ultramicroscopy. 89(1-3):189-194, 2001

Cu<sub>80</sub>Fe<sub>20</sub> and Cu<sub>50</sub>Fe<sub>50</sub> were mechanically alloyed from the pure elements by ball milling for 36 h. The alloy powder was compacted into tablets at room temperature by applying a pressure of 5 GPa. Characterization of the Cu<sub>80</sub>Fe<sub>20</sub> and Cu<sub>50</sub>Fe<sub>50</sub> alloys was carried out by high-resolution transmission electron microscopy (HREM), atom probe field ion microscopy and three-dimensional atom probe (3DAP). The grain size of the nanocrystalline microstructure of the ball-milled alloys observed with HREM varies between 3 and 50 nm. Atom probe and 3DAP measurements indicate that the as-prepared state is a highly supersaturated alloy, in which the individual nanocrystals have largely varying composition. Fe concentration in Cu was found to range from about 8 to 50 at%. It is concluded that by ball milling and compacting an alloy is produced which on a nanometer scale is heterogeneous with respect to morphology and composition.

### [57] EFFECT OF AL-RICH OFF-STOICHIOMETRY ON SUPERPLASTIC FLOW STRESS IN FINE-GRAINED Ni3Al ALLOYS

Doi Y. Matsuki K. Aida T. Akimoto H. - Scripta Materialia. 45(3):301-307, 2001

### [56] TiB<sub>2</sub>/TiC NANOCOMPOSITE POWDER FABRICATED VIA HIGH ENERGY BALL MILLING

Li JL. Li F. Hu K. Zhou Y. - Journal of the European Ceramic Society. 21(16):2829-2833, 2001

TiB<sub>2</sub>/TiC nanocomposite powder was fabricated via high-energy ball milling processing, and microstructure development of the powder mixtures was monitored by X-ray diffraction, transmission electron microscopy. Ball milling B<sub>4</sub>C and elemental Ti powder mixture at ambient temperature resulted in the formation of TiC prior to the formation of TiB<sub>2</sub>. The bulk of TiC and TiB<sub>2</sub> formation accomplished after 5 h of milling. The final product consisted of nanosized TiC particles and microscale TiB<sub>2</sub> particles

### [55] ELECTRICAL PROPERTIES AND THERMAL EXPANSION OF CORDIERITE AND CORDIERITE-MULLITE MATERIALS

Camerucci MA. Urretavizcaya G. Castro MS. Cavalieri AL. - Journal of the European Ceramic Society. 21(16):2917-2923, 2001

Commercially available cordierite and mullite powders were used to obtain cordierite and composite materials with mullite content up to 65 wt.% by attrition milling, uniaxial pressing and sintering. The employed cordierite powders were the coarse, medium and fine single granulometric fractions and the binary mixtures of them with 30, 50 and 70 wt.% of the smaller size component. Mullite powder employed in composites was a 7 h-attrition milled one. The dielectric constant (epsilon), dielectric loss tangent (tan delta), resistivity (rho) and thermal expansion coefficient (alpha) were measured. The influence of the porosity, mullite and glassy phase contents and grain size in the electrical parameters was analyzed. The thermal expansion coefficient as a function of the composition was studied.

### [54] FABRICATION OF CERAMIC COMPOSITE COATINGS USING ELECTROPHORETIC DEPOSITION, REACTION BONDING AND LOW TEMPERATURE SINTERING

Wang ZH. Shemilt J. Xiao P. - Journal of the European Ceramic Society. 22(2):183-189, 2002

We have developed a novel combination of electrophoretic deposition (EPD), reaction bonding and low temperature sintering techniques for the fabrication of yttria stabilized zirconia (YSZ)/alumina composite coatings on FeCrAlloys. A mixture of ethanol and acetylacetone solvent was found to be an effective medium for YSZ and aluminium particle suspension. With the particle size of YSZ and aluminium being significantly reduced during ball milling. By using the EPD process, uniform green form coatings containing YSZ and aluminium particles were produced on FeCrAlloys. After oxidation of aluminium at 500 degreesC and sintering at 1200 degreesC, a dense and adherent YSZ/Al<sub>2</sub>O<sub>3</sub> coating was produced. The presence of aluminium in the green form coatings not only contribute to the bonding between the coating and the metal substrate, but also compensate for the volume shrinkage of the coatings during sintering by the volume expansion arising from oxidation of aluminium to alumina

### [53] HIGH GRAIN SIZE STABILITY OF NANOCRYSTALLINE AL PREPARED BY MECHANICAL ATTRITION

Zhou F. Lee J. Dallek S. Lavernia EJ. - Journal of Materials Research. 16(12):3451-3458, 2001

Grain growth in nanocrystalline (nc) Al with a grain size of 26 nm produced by cryogenic mechanical milling was studied through x-ray diffraction, transmission electron microscopy, and differential scanning calorimetry. Grain growth kinetics resembled those of ball-milled nc Fe. For homologous temperatures (T/T<sub>m</sub>) of 0.51-0.83, the time exponent n from D-1/n -



$D-0(1/n) = kt$  was 0.04-0.28, tending toward 0.5 as  $T/T-M$  increased. Two grain-growth regimes were distinguished: below  $T/T-M = 0.78$  growth ceased at an approximate grain size of 50 nm while at higher temperatures, grain growth proceeded steadily to the submicrometer range. Grain growth over the range of temperatures studied cannot be explained in terms of a single thermally activated rate process. The observed high grain size stability was attributed primarily to impurity pinning drag associated with the grain growth process

**[52] ORIGINS OF STORED ENTHALPY IN CRYOMILLED NANOCRYSTALLINE ZN**

Zhang XH. Wang HY. Kassem M. Narayan J. Koch CC. - Journal of Materials Research. 16(12):3485-3495, 2001

Nanocrystalline Zn was prepared by cryomilling (mechanical attrition at liquid nitrogen temperature). Differential scanning calorimetry (DSC), x-ray diffraction, and transmission electron microscopy were used to study the structural changes and grain size distribution with milling time and subsequent annealing. Maxima in both stored enthalpy (for the low-temperature DSC peak) and lattice strain on the Zn basal planes were observed at the same milling time. Dislocation density on the basal planes is proposed as a major source for lattice strain and the measured stored enthalpy. The released enthalpy that might be due to grain growth is very small

**[51] OXIDATION AND SPLATS OF STAINLESS STEEL PARTICLES COATED WITH AN ALUMINA SHELL**

H Ageorges, P Fauchais - PROGRESS IN PLASMA PROCESSING OF MATERIALS 2001, 2001, pp 537-544

The effect of an alumina shell (4 pm thick), obtained by mechanofusion, on stainless steel particles ((d) over bar = 55 pm) used in plasma sprayed has been studied by examining the corresponding composite spraying particles collected in flight, their resulting splats and coatings. Without alumina, the collected sprayed stainless steel particles exhibit dark spots within their clear cross section, dark spots which display chromium and oxygen elements prominently which seems to be CrO (XRD). The composite sprayed particles are less oxidized. Part of them keep their alumina shell broken but still covering the whole stainless steel particles. The EDS analysis of the distribution of the various elements in the corresponding splats shows that the alumina is dispersed in pieces and frozen over the surface of the stainless steel splat. The other part have their alumina shell completely fused and entrained by the flow to the tail of the moving droplet. In this case, the alumina is spread exactly on the stainless steel splat. Whatever may be the type of particles, without preheating the substrate the splats are extensively fingered and become circular when the stainless steel substrate surface is preheated over 200 degreesC. Finally, when comparing coatings on a rough (Ra approximate to 6.7 +/- 0.3 Vm) stainless steel substrate preheated to 400 degreesC, both hardness and adhesion/cohesion are found to be improved for the alumina coated particles.

**[50] STUDIES ON PA6-PP-WOLLASTONITE COMPOSITE COMPATIBILISED BY PP-GRAFT-MALEIC ANHYDRIDE PREPARED VIA PAN MILLING**

Wang Q. Liu CS. Chen YH. - Plastics Rubber & Composites. 30(8):363-369, 2001.

A composite of PA6 PP-wollastonite compatibilised by PP-g-maleic anhydride has been prepared using pan type milling equipment. and its structure and properties investigated by IR, DSC, melt index measurements, SEM, and mechanical testing. The experimental results show that during pan milling, PP, PA6, and wollastonite are effectively pulverised, reaching better mixing owing to the very strong shear forces and pressure exerted by the pan type milling equipment. In particular, some PA6 polymer chains are grafted onto the wollastonite surface and the pan milling affects the crystallinity of PA6 and PP to some degree. The compatibiliser prepared via solid phase grafting of maleic anhydride onto PP via pan milling shows a reasonably good compatibilising effect on the composite, improving the morphology and therefore the mechanical properties of the composite. If combined with suitable coupling agent. the PA6-PP-wollastonite compatibilised by PP-g-maleic anhydride prepared via pan milling (wollastonite content 30 wt-%) possesses much better mechanical properties. its tensile strength increases from 54.6 to 58.6 MPa. and its notched Izod impact strength increases from 29.4 to 48.7 J m(-1), compared with the uncompatibilised system. Pan milling is a novel way to achieve desired structure and hence improved properties of polymer based materials via the polymer processing procedure

**[49] DEVELOPMENT OF MG-BASED HYDROGEN STORAGE ALLOY**

Wang LB. Wang YJ. Yuan HT. - Journal of Materials Science & Technology. 17(6):590-596, 2001

Mg-based hydrogen storage alloys are considered as a promising candidate for hydrogen system because of its lightweight, high storage capacity, low price and rich mineral resources. In detail, we reviewed the preparation and properties of Mg-Ni-based hydrogen storage alloys. All kinds of attempts have been done to improve the hydriding and dehydriding behaviors. It is found that the partial substitution of foreign elements can decrease the hydrogen absorption temperature, especially the substitution of a more electronegative element, such as Al and Mn. Mechanical alloying (MA) and mechanical grinding (MG) are the most effective methods to improve the hydriding/dehydriding kinetics and electrochemical capacity, and decrease the desorption temperature. but the corrosion resistance is so poor that the 80% of maximum capacity is lost within ten cycles. Microencapsulation is a useful measurement for improving the corrosion resistance and electrocatalytic activity. In order to improve the properties of the alloys for practical application, the alloys should have a large number of defects, which give activated sites, subsequently, MA, MG and electroless plating should be used to improve the hydriding/dehydriding kinetics and protect the surface of alloys, respectively. The new composite Mg-based alloys give a new way for the hydrogen storage material to practical application. Furthermore we put forward several problems which will be discussed in future

**[48] INJECTION MOLDING OF W-NI-FE NANOCOMPOSITE POWDER PREPARED BY MECHANICAL ALLOYING**

Qu XH. Fan JL. Huang BY. - Journal of Materials Science & Technology. 17(6):673-674, 2001

Nanocrystalline 90W-7Ni-3Fe (wt pct) composite powder was prepared by mechanical alloying and mixed with binder to form a feedstock. Its rheological and sintering behaviors were compared with those of the feedstock from the original



powder. It is found that milling can increase the maximum powder loading of feedstock and enhance the sintering densification process.

**[47] SYNTHESIS OF SOLID SOLUTIONS  $\text{YNi}_x\text{Mn}_{1-x}\text{O}_3$  VIA POLYMERIC PRECURSORS [SPANISH]**

Gutierrez D. Tartaj J. Duran P. Moure C. - Boletín de la Sociedad Española de Cerámica y Vidrio. 40(5):339-343, 2001

The solid solutions  $\text{YNi}_x\text{Mn}_{1-x}\text{O}_3$  are semiconducting materials which show perovskite-like structure, orthorhombic symmetry, and spatial group Pbnm. Their electrical and magnetic properties are typical of materials with potential applications in sensor systems or as cathodes in solid oxide fuel cells (SOFC's). Usually, the conventional method of mixing oxides used to obtain these materials requires high-temperature synthesis and vigorous milling. By means of a polymerization method (the Pechini method, slightly modified) we have obtained an amorphous, porous, and soft material from which the Ni-modified yttrium manganite, as a single phase, can be synthesized. The powder was characterized by a nanometric particle size, a low temperature of synthesis within the interval 750-800 degreesC, and it did not require much milling. The formation reaction has been studied for the compound  $\text{YNi}_{0.33}\text{Mn}_{0.67}\text{O}_3$ , using X-Ray diffraction (XRD) and the infrared absorption technique (FTIR). Also, a comparison of the electrical behaviour and the sinterization process is made between the sample prepared by the chemical and the mixing oxides method

**[46] NANOCRYSTALLINE HfN PRODUCED BY MECHANICAL MILLING: KINETIC ASPECTS**

Bab MA. Mendoza-Zelis L. Damonte LC. - Acta Materialia. 49(20):4205-4213, 2001

Nanocrystalline HfN powders were obtained by mechanically assisted gas-solid reaction. An analysis of the reaction kinetics under different milling frequencies and gas pressure is presented. The results shown the formation of an interstitial solid solution  $\alpha\text{-Hf}$ , prior to its transformation to cubic HfN. Rietveld refinements of the X-ray diffraction data were done in order to analyze the progress of the structural change, The short range order was characterized by perturbed angular correlations, which indicated a random distribution of N around Hf. The reaction kinetics were obtained from the measured value of absorbed nitrogen per Hf (n). Two main features were evident; the reaction advance dependence on the dose of mechanical energy and a slowing down in the reaction kinetics for n similar to 0.5 associated with the saturation of surface sites, These findings are discussed taking into account the structural change and the role of surface adsorption and volume diffusion.

**[45] SOLID-STATE CHEMICAL SYNTHESIS OF NANOPARTICULATE ZIRCONIA**

Dodd AC. McCormick PG. - Acta Materialia. 49(20):4215-4220, 2001

Mechanically activated reaction of anhydrous chloride precursors with  $\text{Li}_2\text{O}$  has been used to synthesise ultrafine powders of  $\text{ZrO}_2$ , Mg-PSZ, and Y-TZP. In each case, milling of the reactant mixtures resulted in a combination of amorphisation and overall microstructural refinement. Chemical reaction, with the consequent formation of nanoparticulate zirconia and LiCl, only occur-red during subsequent low temperature heat treatment. The mechanism of reaction was found to depend on the heating rate and also on the presence of LiCl diluent in the initial reactant mixture. In addition to altering the reaction mechanism, the addition of diluent decreased the average crystallite size and increased the proportion of single crystal particles in the final zirconia product. Accompanying the decrease in average particle size with dilution was an increase in the tetragonal volume fraction

**[44] ENHANCED GAS-SOLID REACTION AFTER EXTENDED MILLING**

Welham NJ. - Journal of Materials Science Letters. 20(20):1849-1851, 2001.

**[43] FORMATION MECHANISM AND ANNEALING BEHAVIOR OF NANOCRYSTALLINE FERRITE IN PURE FE FABRICATED BY BALL MILLING**

Yin J. Umemoto M. Liu ZG. Tsuchiya K. - ISIJ International. 41(11):1389-1396, 2001.

Nanocrystalline ferrite formation by ball milling in pure Fe (0.004 mass% C) has been studied through morphology observation and microhardness measurements. It was found that nano-ferrite first formed at the outer surface area in the powders at the early stage of ball milling, which was attributed to the deformation localization. The boundaries between the nano-ferrite and work-hardened regions are quite clear under SEM and the hardness of nano-ferrite region is more than 3 GPa higher than that of the work-hardened region. Further milling led to the particle refinement and formation of nano-ferrite of the whole particles. It has been suggested that the nanocrystalline ferrite formed through a transition from dislocation cell wall created by work hardening during ball milling to grain boundary, which is regarded to contribute to the hardness gap between the work hardened structure and the nanocrystalline ferrite. A relatively high stability against temperature was observed in the nano-ferrite formed by ball milling. The irregular grain boundaries of nano-ferrite annealed at high temperature was attributed to that the grain growth of nanocrystalline ferrite takes place by coalescence of neighboring grains

**[42] PT-CERMET CATHODE FOR REDUCED TEMPERATURE SOFCs**

Sasaki K. Tamura J. Dokiya M. - Solid State Ionics. 144(3-4):223-232, 2001

Polarization characteristics of Pt-(Sc<sub>0.10</sub>Ce<sub>0.01</sub>)Zr<sub>0.89</sub>O<sub>2</sub> (SSZ) cermet cathodes were studied in order to develop new cathodes for reduced temperature solid oxide fuel cells (SOFCs). Several Pt-SSZ cermet electrodes were prepared by mixing Pt and SSZ powders using a high-energy ball mill. A Pt-SSZ cermet cathode on yttria-stabilized zirconia (YSZ) electrolyte achieved high cathodic activity; the cathode interfacial conductivity reached as high as 6.7 S/cm<sup>2</sup> at 973 K and 0.8 S/cm<sup>2</sup> at 873 K. These results suggest that this Pt-SSZ cermet cathode has enough capability at 973 K, but is still not satisfactory at 873 K. This high activity can be obtained by ball milling SSZ/Pt mixture of proper ratios in vacuum and by controlling electrode thickness, sintering temperature, and Ca impurity. These electrodes showed two optima of interfacial conductivity vs. SSZ/Pt ratio and remarkable dependence on cathode thickness. It was considered that the first optimum is based on two-



dimensional reaction sites on YSZ electrolyte surface and the second one originates from three-dimensional expansion of reaction sites into the cermet cathode

**[41] NOBLE METAL ALLOY-ZR(SC)O-2 CERMET CATHODE FOR REDUCED-TEMPERATURE SOFCS**

Sasaki K. Tamura J. Dokiya M. - Solid State Ionics. 144(3-4):233-240, 2001

Polarization characteristics of noble metal alloy-(Sc<sub>0.10</sub>Ce<sub>0.01</sub>)Zr<sub>0.89</sub>O<sub>2</sub> (SSZ) cermet cathodes were studied in order to develop a new cathode for reduced-temperature solid oxide fuel cells (SOFCs). Several noble metal alloy-SSZ cermet cathodes were prepared by mixing Pt, Pd, Rh and/or Ag and their alloy powders with SSZ powder using a high-energy ball mill in vacuum and pasting the cermet onto yttria stabilized zirconia (YSZ) electrolyte. A Pt-Ag/SSZ cermet cathode achieved as high as 12 S/cm<sup>2</sup> of interfacial conductivity, sigma (E), at 973 K and 1.5 S/cm<sup>2</sup> at 873 K in air. The results suggest that this Pt-Ag/SSZ cermet cathode has enough activity not only at 973 K but also at 873 K. This high activity can be obtained by selecting a suitable alloy composition, ball milling a proper ratio of SSZ/noble metal mixture in vacuum and controlling the cathode thickness and the sintering temperature. By replacing the metallic component of cermet from Pt to Pt-Ag alloy (50 wt.% Pt), the quantity of Pt in cermet can be reduced to 19 from 40 Mg/cm<sup>2</sup> in addition to the improvement of activity from 6.7 S/cm<sup>2</sup> at 973 K to 12 S/cm<sup>2</sup> of sigma (E) at 973 K. The activation energies, E-a, of Pt-Ag and Pd-Ag/SSZ cermet were smaller than that of Pt/SSZ cermet. In the case of Pt-Ag/SSZ cermet, the E-a decreased with increasing Ag ratio in the Pt-Ag alloy. The E-a also depends on the SSZ/Pt-Ag ratio. This cathode showed two optima of sigma (E) versus the SSZ/Pt-Ag ratio and a remarkable dependence on cathode thickness. It was considered that the first optimum is based on two-dimensional reaction sites on YSZ electrolyte and the second optimum originates from three-dimensional expansion of reaction sites into the cermet cathode layer.

**[40] DEVELOPMENT OF AMORPHOUS AL65CU35-XTIX ALLOYS BY MECHANICAL ALLOYING**

Chattopadhyay PP. Gannabattula RNR. Pabi SK. Manna I. - Scripta Materialia. 45(10):1191-1196, 2001

**[39] THERMAL STABILITY OF ULTRA FINE-GRAINED STEEL CONTAINING DISPERSED OXIDES**

Belyakov A. Sakai Y. Hara T. Kimura Y. Tsuzaki K. - Scripta Materialia. 45(10):1213-1219, 2001

Annealing behaviour of an ultra fine-grained steel was studied at temperatures of T = 600-900 degreesC. At T less than or equal to 800 degreesC, recovery develops more readily at grain boundaries as compared to grain interiors, leading to a fast release of lattice distortions associated with the non-equilibrium state of grain boundaries

**[38] SYNTHESIS AND CHARACTERIZATION OF NANOCRYSTALLINE MG2COH5 OBTAINED BY MECHANICAL ALLOYING**

Chen J. Takeshita HT. Chartouni D. Kuriyama N. Sakai T. - Journal of Materials Science. 36(24):5829-5834, 2001.

The stoichiometric mixture of 2MgH<sub>2</sub> + Co was ball milled under a hydrogen atmosphere to synthesize nanocrystalline metal hydride Mg<sub>2</sub>CoH<sub>5</sub>. Upon milling, the mixture was analyzed by X-ray powder diffraction (XRD) and thermal methods employing the techniques of differential scanning calorimetry (DSC), thermogravimetry (TG) and differential thermal analysis (DTA). Hydrogen absorption and desorption measured by pressure-composition-temperature (P-C-T) curves indicated that the capacity loss was small after 20 consecutive cycling tests. The enthalpies associated with hydride formation and decomposition were measured to be -69.5 and -83.2 kJ mol<sup>-1</sup> H<sub>2</sub>, respectively. At the temperatures of this study (553 to 653 K), hysteresis decreases with increasing temperature

**[37] MAGNETIC STRUCTURE OF FEXCU100-X MAGNETORESISTIVE ALLOYS PRODUCED BY MECHANICAL ALLOYING**

Socolovsky LM. Sanchez FH. Shingu PH. - Hyperfine Interactions. 133(1-4):47-52, 2001.

FexCu<sub>100-x</sub> magnetoresistive alloys were produced by mechanical alloying. X-ray diffraction shows fcc structure. The room-temperature Mossbauer spectra evolves from an asymmetrical doublet below x = 25%, to a broad magnetic hyperfine field distribution above this concentration. Quadrupole splitting of the doublet varies between 0.48 and 0.57 mm/s, and its isomer shift from 0.16 to 0.29 mm/s. Low-temperature Mossbauer spectroscopy displays a B-hf distribution. Magnetization measurements display different features depending on concentration, from mictomagnetism to ferromagnetism. Low-temperature magnetoresistance is measured. Samples with x similar to 20% exhibit larger magnetoresistivity ratios. Bulk and hyperfine magnetic properties are correlated in order to explain magnetoresistivity features of these samples

**[36] CHARACTERIZATION OF IRON NITRIDE POWDERS FORMED BY MECHANICAL ALLOYING AND ATMOSPHERIC HEAT TREATMENT TECHNIQUES**

Lin CK. Chen GS. Chen JS. Chin TS. Lee PY - Chung-Kuo Kung Ch'Eng Hsueh K'An/Journal of the Chinese Institute of Engineers. 24(6):753-760, 2001

Iron nitrides were manufactured by mechanical alloying of Fe<sub>2.33</sub>N and Fe powder mixtures at starting compositions of Fe<sub>12</sub>N<sub>2</sub> and Fe<sub>16</sub>N<sub>2</sub> and annealed in air at 200 and 240 degreesC. It was found that the supersaturated nanocrystalline alpha-Fe solid solution phase was produced after MA, and iron nitrides were formed after heat treatment. The saturation magnetization (sigma) of the heat-treated powders is higher than those of the as-milled powders. The sigma values for the specimens heat treated at 200 degreesC are higher than those heat treated at 240 degreesC, and the highest value among all of the samples is 218 e.m.u./g which is obtained by annealing of as-milled Fe<sub>12</sub>N<sub>2</sub> powders in air at 200 degreesC for 6



hours. The possible existence of the  $\alpha$ -Fe<sub>16</sub>N<sub>2</sub> phase is responsible for the high saturation magnetization in mechanically alloyed Fe<sub>16</sub>N<sub>2</sub> and Fe<sub>12</sub>N<sub>2</sub> powders.

**[35] CRYSTALLIZATION OF LEAD NIOBATE GLASS BY MECHANICAL ACTIVATION**

Xue JM. Wang J. - Journal of the American Ceramic Society. 84(11):2691-2695, 2001

Mechanical activation-triggered crystallization in PbNb<sub>2</sub>O<sub>6</sub>-based glass was dependent on the initial presence of nuclei. The crystallization cannot be initiated by mechanical activation in a highly amorphous glass composition quenched from 1350 degreesC where PbNb<sub>2</sub>O<sub>6</sub> nuclei did not exist. The steady growth of nanocrystallites of PbNb<sub>2</sub>O<sub>6</sub> was observed with an increasing degree of mechanical activation in the glass quenched from 1300 degreesC, where a density of PbNb<sub>2</sub>O<sub>6</sub> nuclei existed before mechanical activation. The inability to nucleate in the highly amorphous oxide glass by mechanical activation is consistent with the much higher structural stability as compared with that of metallic glasses, such as Fe-Si-B. The mechanical activation-grown PbNb<sub>2</sub>O<sub>6</sub> nanocrystals were 10-15 nm in size as observed using HRTEM and their crystallinities were further improved by thermal aging at an elevated temperature in the range of 550 degrees to 650 degreesC

**[34] MICROSTRUCTURE AND ELECTROCHEMICAL CHARACTERISTICS OF ADVANCED COMPOSITE ZR-BASED HYDROGEN STORAGE ALLOY**

Wen MF. Chen LA. Tong M. Chen DM. Cheng HM. Zhai YC. Tian YW. - Journal of Materials Science & Technology. 17(Suppl 1):S31-S34, 2001.

Zr<sub>0.9</sub>Ti<sub>0.1</sub>(Ni,Co,Mn,V)<sub>(2.1)</sub> as-cast alloy was prepared by melting under Ar atmosphere; and then, four composite alloys were prepared by ball-milling after hydrogen absorbing agent was added. The initial discharge capacity was as high as 430 mAh .g(-1) for composite alloy electrode c discharged at 60 mA .g(-1) current density and its maximum capacity reached 505 mAh .g(-1). All composite alloy electrodes have good activation behavior, being fully activated in one or two cycles. At 300 mA .g(-1) current density, the composite alloy electrode c was found to have good cycle stability

**[33] EFFECT OF SiC PARTICULATES ON THE MICROSTRUCTURE OF AL-6Ti-6Nb MATRIX COMPOSITES FABRICATED BY MECHANICAL ALLOYING TECHNIQUE**

Jia DC. Meng QC. Zhou Y. Lei TQ. - Journal of Materials Science & Technology. 17(Suppl 1):S61-S64, 2001.

Millimeter sized SiC (m-SiCp) and nanometer sized SiC (n-SiCp) particulates reinforced Al-6Ti-6Nb matrix composites were prepared by mechanical alloying (MA) and later hot-press sintering. Their microstructure was investigated to know the influence of the incorporated SiC particles. The secondary Al<sub>3</sub>Ti, AlNb<sub>2</sub> intermetallics particle size, the Al matrix grain size and their submicrostructure are strongly affected, and they are correlated with the thermal stability of the composites.

**[32] OXIDATION CONTROL AND NON-EQUILIBRIUM PHASE FORMATION IN CU-CR ALLOYS DURING MECHANICAL ALLOYING**

Cui XL. Wang L. Qi M. - Journal of Materials Science & Technology. 17(Suppl 1):S89-S92, 2001.

Using X-ray diffraction (XRD), scanning electron microscopy (SEM), differential scanning calorimeter (DSC) and optical microscopy, phase transformation of Cu-Cr alloys with various compositions during mechanical alloying process has been investigated. Besides the formation of supersaturated solid solution, the results show that a kind of amorphous oxide formed in the process, and the addition of carbon has obviously effect on the suppression of oxidation and the deoxidization of oxide. The reactive milling has a remarkable effect on the behavior of oxidation.

**[31] MICROSTRUCTURE EVOLUTION OF Ti/BN POWDER BLEND DURING BALL MILLING AND HEAT TREATMENT**

Li JL. Hu K. Zhong Y. - Journal of Materials Science & Technology. 17(Suppl 1):S101-S104, 2001.

Ball milled Ti/BN composite powder was prepared by high energy ball milling for 40 h, using Ti and BN (the molar ratio of Ti/BN is 3:2) as starting materials. The as-milled composite powder consists of TiN, Ti and amorphous phase. TiN formed while the milled powder was annealed at 400 degreesC. The heat treatment at 700 degreesC led to the formation of TiB<sub>2</sub> and TiB. The nanocrystalline Ti and amorphous phase converted to TiN and TiB<sub>2</sub> when the powder was heated to 1300 degreesC

**[30] AN X-RAY DIFFRACTION STUDY OF NANOCRYSTALLINE TITANIUM PREPARED BY HIGH-ENERGY VIBRATIONAL BALL MILLING**

Chatterjee P. Sen Gupta SP. - Applied Surface Science. 182(3-4 Special Issue SI):372-376, 2001

A detailed X-ray line-broadening study in ball-milled Ti sample is presented. It is observed that ball milling leads to the formation of nanocrystalline grains and transform partially to an f.c.c. Ti-phase after 10 h of milling. Using a Rietveld analysis, the volume fraction of f.c.c. phase was found to be similar to 24% with a crystallite size of similar to 20 Angstrom. The origin of high strains is probably the dislocations present in the sample. Anisotropic line broadening of the fault-unaaffected reflections is explained in terms of anisotropic dislocation contrast corresponding to  $\langle a \rangle$  and  $\langle a + c \rangle$  dislocations. Both edge and screw dislocations were predicted with a significant basal slip. High dislocation density similar to 10(11) cm/cm(3) has been found in the milled samples. A large increase in the isotropic thermal parameter was also observed for the h.c.p. Ti-phase after 10 h of milling

**[29] SYNTHESIS OF NANOSIZED TITANIUM POWDER BY HIGH ENERGY MILLING**

Dabhade VV. Mohan TRR. Ramakrishnan P. - Applied Surface Science. 182(3-4 Special Issue SI):390-393,

Titanium powders of about 2  $\mu$  particle size were subjected to high energy attrition milling in an argon atmosphere. Selecting suitable milling parameters, nanosize ( $< 100$  nm) titanium powders were prepared after 15 h of milling. An average particle size of 35 nm was obtained at 30 h of milling after which the particle size stabilized with continuation of milling to 75 h. The powders after milling for various durations were characterized by TEM, ICP and XRD, and these results are reported and discussed.



**[28] MECHANICALLY ACTIVATED MIXTURES OF CALCIUM CHLORIDE WITH SODIUM BETA-DIKETONATES**

Dedlovskaya EM. Zaitseva IG. Kuz'mina NP. Petrova LA. Borisov AP. Makhaev VD. - Russian Journal of Coordination Chemistry. 27(11):767-770, 2001

The preparative chemical method, X-ray diffraction analysis, and vacuum sublimation method are used to show that the mechanical treatment of mixtures of calcium chloride with sodium salts of acetylacetonone, trifluoroacetylacetonone, dipivaloylmethane, or acetylacetoinimine induces their interaction, forming volatile calcium beta -diketonates and sodium chloride. Heating of the reaction mixtures results in sublimation of the obtained calcium beta -diketonate and nonreacted sodium beta -diketonate; this is impeded by the formation of poorly volatile forms.

**[27] DENSIFICATION AND STRUCTURAL CHANGE OF MECHANICALLY ALLOYED W-CU COMPOSITES**

Kecskes LJ. Trexler MD. Klotz BR. Cho KC. Dowding RJ. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 32(11):2885-2893, 2001

Fine-grained, high-density (97+ pct of theoretical density (TD)), 80W-20Cu wt pct (58W-42Cu at. pct) composites have been prepared using nonconventional alloying techniques. The W and Cu precursor powders were combined by a high-energy ball-milling procedure in air or hexane. The mechanically alloyed W + Cu powder mixtures were then cold pressed into green compacts and sintered at 1523 K. The milling medium and milling time were varied to increase product densities with a concomitant order-of-magnitude decrease in grain size. For densification, air was found to be a more effective medium than hexane. From microhardness measurements, it was concluded that the W-Cu alloys were dispersion and solution hardened, but were sensitive to entrapped residual impurities. X-ray diffraction (XRD), energy-dispersive X-ray spectroscopy (EDS), and scanning electron microscopy (SEM) analyses were used to demonstrate that the as-milled and sintered W-Cu alloy structures were metastable, decomposing into the starting W and Cu components upon heating at or above 723 K

**[26] COMPETITIVE ADSORPTION PHENOMENA IN NONAQUEOUS TAPE CASTING SUSPENSIONS**

Blackman K. Silaty RM. Lewis JA. - Journal of the American Ceramic Society. 84(11):2501-2506, 2001

Competitive adsorption phenomena in alumina ( $\alpha$ -Al<sub>2</sub>O<sub>3</sub>)polyvinyl butyral (PVB)-Menhaden fish oil (MFO) suspensions are studied using diffuse reflectance Fourier transform infrared spectroscopy and thermogravimetric analysis. PVB and MFO commonly serve as binder and dispersant species, respectively, in nonaqueous tape casting systems. Adsorption isotherm measurements reveal that both PVB and MFO have an affinity for  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> surfaces, with corresponding plateau coverages of 3.4 and 2.3 mg/m<sup>2</sup>, respectively. Sequential competitive adsorption of these species occurred during two-stage milling. MFO was fully adsorbed onto  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> powder in the absence of PVB during first-stage milling. PVB was then introduced to the system during second-stage milling and found to displace preadsorbed MFO species from such surfaces. At the culmination of milling, PVB composed 25-35 wt% of the adsorbed organic layer. Complete displacement of MFO was not observed, which limited PVB adsorption on  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>

**[25] CRYSTALLIZATION OF LEAD NIOBATE GLASS BY MECHANICAL ACTIVATION**

Xue JM. Wang J. - Journal of the American Ceramic Society. 84(11):2691-2695, 2001

Mechanical activation-triggered crystallization in PbNb<sub>2</sub>O<sub>6</sub>-based glass was dependent on the initial presence of nuclei. The crystallization cannot be initiated by mechanical activation in a highly amorphous glass composition quenched from 1350 degreesC where PbNb<sub>2</sub>O<sub>6</sub> nuclei did not exist. The steady growth of nanocrystallites of PbNb<sub>2</sub>O<sub>6</sub> was observed with an increasing degree of mechanical activation in the glass quenched from 1300 degreesC, where a density of PbNb<sub>2</sub>O<sub>6</sub> nuclei existed before mechanical activation. The inability to nucleate in the highly amorphous oxide glass by mechanical activation is consistent with the much higher structural stability as compared with that of metallic glasses, such as Fe-Si-B. The mechanical activation-grown PbNb<sub>2</sub>O<sub>6</sub> nanocrystals were 10-15 nm in size as observed using HRTEM and their crystallinities were further improved by thermal aging at an elevated temperature in the range of 550 degrees to 650 degreesC

**[24] MIXED FLUORIDE ION CONDUCTORS PREPARED BY A MECHANICAL MILLING TECHNIQUE: EFFECT OF GRAIN SIZE AND STRAIN ON THE IONIC CONDUCTIVITY**

Kumar M. Sekhon SS. - Journal of Physics D-Applied Physics. 34(19):2995-3002, 2001

Mixed metal fluoride ion conductors which have been prepared by a mechanical milling process have been found to have higher value of conductivity than single fluorides. Specific results for the synthesis of mixed metal fluorides of composition xPbF(2) (.) (1 - x)SnF<sub>2</sub> and NH<sub>4</sub>F-2SnF(2) are presented. The variation of conductivity with composition (x), milling time and temperature has been studied. The grain size and the strain factor were calculated from the linewidth of different x-ray diffraction lines and their correlation with the conductivity value and the milling time has been studied.

**[23] ANTIMONY OXIDE-MODIFIED VANADIA-BASED CATALYSTS - PHYSICAL CHARACTERIZATION AND CATALYTIC PROPERTIES**

Spengler J. Anderle F. Bosch E. Grasselli RK. Pillep B. Behrens P. Lapina OB. Shubin AA. Eberle HJ. Knozinger H. - Journal of Physical Chemistry B. 105(44):10772-10783, 2001

Antimony-modified vanadia-on-titania catalysts were prepared for the selective oxidation of o-xylene to phthalic anhydride by ball milling of powder mixtures followed by calcination. A binary Sb<sub>2</sub>O<sub>3</sub>-V<sub>2</sub>O<sub>5</sub> system was also prepared for comparison purposes. The resulting materials were physically characterized by surface area measurements, X-ray diffraction analysis (XRD), laser Raman spectroscopy, X-ray absorption fine structure (XAFS) spectroscopy, electron spin resonance (ESR), magnetic susceptibility determination, and V-15 solid-state NMR. The catalytic performance of the TiO<sub>2</sub>-supported materials was tested for o-xylene oxidation. After calcination of the Sb<sub>2</sub>O<sub>3</sub>-V<sub>2</sub>O<sub>5</sub> binary mixture at 673 K, Sb<sup>3+</sup> is almost quantitatively oxidized to Sb<sup>5+</sup>, while both V<sup>3+</sup> and V<sup>4+</sup> are detected. V<sup>3+</sup> and some V<sup>4+</sup> are most likely located in a



nonstoichiometric VSbO<sub>4</sub>-like structure, while the majority of V<sup>4+</sup> preferentially concentrates within shear domains in oxygen-deficient V<sub>2</sub>O<sub>5-x</sub> particles. In the titania-supported catalyst system, both Sb<sub>2</sub>O<sub>3</sub> and V<sub>2</sub>O<sub>5</sub> spread on the anatase surface. Sb<sup>3+</sup> is oxidized to Sb<sup>5+</sup>, and V<sup>3+</sup>, V<sup>4+</sup>, and V<sup>5+</sup> are detected. VSbO<sub>4</sub>-like structures are not observed. The presence of antimony leads to the formation of presumably V<sup>3+</sup>-O-V<sup>5+</sup> redox couples. The paramagnetic centers-in contrast to the binary mixture-are largely isolated. Antimony preferentially migrates to the surface and appears to exhibit a dual function catalytically. It is inferred from the experimental data that the addition of antimony leads to site isolation and to a reduction of surface acidity. We suggest that V-O-V-O-V domains or clusters are interrupted by incorporation of Sb to form V-O-Sb-O-V species. As a consequence of this site isolation and a reduction of surface acidity, overoxidation of o-xylene is reduced. These two effects are therefore most probably responsible for the improved selectivity of the ternary catalyst system over the binary one toward phthalic anhydride

**[22] REVERSIBLE HYDROGEN STORAGE VIA TITANIUM-CATALYZED LiAlH<sub>4</sub> AND Li<sub>3</sub>AlH<sub>6</sub>**

Chen J. Kuriyama N. Xu Q. Takeshita HT. Sakai T. - Journal of Physical Chemistry B. 105(45):11214-11220, 2001

A vibrating-mill technique, which can activate the reaction system by bringing the reagents into very close contact at the preparative scale and by providing extra mechanical energy, much more effectively than the well-known ball-milling method, was used to prepare titanium(III) chloride (TiCl<sub>3</sub>).(1/3)AlCl<sub>3</sub>-doped lithium tetrahydridoaluminate (LiAlH<sub>4</sub>) and lithium hexahydridoaluminate (Li<sub>3</sub>AlH<sub>6</sub>) powders with nanocrystallites. The phase structure and dehydrogenating/rehydrogenating properties were characterized by using X-ray diffraction (XRD), scanning electron microscopy (SEM), thermogravimetry (TG), and differential scanning calorimetry (DSC). The mechanism of reversible dehydrogenation and rehydrogenation was examined by means of X-ray photoelectron spectroscopy (XPS). Thermodynamic and kinetic measurements showed a distinct change for the dehydrogenating/rehydrogenating reactions over the temperature range 25-250 degreesC. From the Arrhenius plot of hydrogen desorption kinetics, apparent activation energies were found to be 42.6 and 54.8 kJ/mol H<sub>2</sub> for the hydride decompositions of LiAlH<sub>4</sub> and Li<sub>3</sub>AlH<sub>6</sub>, respectively. The results based on the properties of reversible hydrogen storage and catalysis function indicate that both the homogeneous distribution of Ti-catalyzed nanocrystalline complex hydrides and the Ti-catalyst with a Ti-O double left right arrow Ti<sup>3+</sup> (Ti<sup>0</sup>/Ti<sup>2+</sup>/Ti<sup>3+</sup>) defect site play important roles in optimizing the reversible hydrogen storage.

**[21] DEGREE OF CRYSTALLIZATION AND CONTAMINATION EFFECT ON FERROELECTRIC CERAMIC POWDER PRODUCED BY FINE GRINDING**

Munekata M. Kamata O. Shirasaka T. Higuchi T. - Japanese Journal of Applied Physics Part 1-Regular Papers Short Notes & Review Papers. 40(9B):5638-5641, 2001

Fine grinding of ferroelectric materials is indispensable and elemental in order to produce electronic components of small size and high-performance ceramics. The deterioration of the electrical properties occurs due to contamination during conventional fine grinding. In addition, the crystallinity (degree of crystallization) of ground fine particles is low, and in mass-production there was the problem of the deformation of the dimensions caused by the sintering process. In our experiment, by using the wet cycloid mill, it was possible to control the amorphizability (lattice distortion) and fine particles with high crystallinity and good electrical properties could be obtained

**[20] SYNTHESIS AND ELECTROCHEMICAL PROPERTIES OF NOVEL DIMERIC FULLERENES INCORPORATED IN A 2,3-DIAZABICYCLO[2.2.2]OCT-2-ENE FRAMEWORK**

Murata Y. Suzuki M. Komatsu K. - Chemical Communications. (22):2338-2339, 2001.

Novel dimeric fullerenes incorporated in a 2,3- diazabicyclo[ 2.2.2]oct-2-ene framework, with and without direct interfullerene-cage bonds, were synthesized and fully characterized spectroscopically; the electronic communication between the two fullerene cages was clarified by differential pulse voltammetry

**[19] AN X-RAY DIFFRACTION STUDY OF NANOCRYSTALLINE TITANIUM PREPARED BY HIGH-ENERGY VIBRATIONAL BALL MILLING**

Chatterjee P. Sen Gupta SP. - Applied Surface Science. 182(3-4 Special Issue SI):372-376, 2001

A detailed X-ray line-broadening study in ball-milled Ti sample is presented. It is observed that ball milling leads to the formation of nanocrystalline grains and transform partially to an f.c.c. Ti-phase after 10 h of milling. Using a Rietveld analysis, the volume fraction of f.c.c. phase was found to be similar to 24% with a crystallite size of similar to 20 Angstrom. The origin of high strains is probably the dislocations present in the sample. Anisotropic line broadening of the fault-unaffected reflections is explained in terms of anisotropic dislocation contrast corresponding to <a > and <a + c > dislocations. Both edge and screw dislocations were predicted with a significant basal slip. High dislocation density similar to 10(11) cm/cm(3) has been found in the milled samples. A large increase in the isotropic thermal parameter was also observed for the h.c.p. Ti-phase after 10 h of milling

**[18] SYNTHESIS OF NANOSIZED TITANIUM POWDER BY HIGH ENERGY MILLING**

Dabhade VV. Mohan TRR. Ramakrishnan P. - Applied Surface Science. 182(3-4 Special Issue SI):390-393, 2001

Titanium powders of about 2 mu particle size were subjected to high energy attrition milling in an argon atmosphere. Selecting suitable milling parameters, nanosize (< 100 nm) titanium powders were prepared after 15 h of milling. An average particle size of 35 nm was obtained at 30 h of milling after which the particle size stabilized with continuation of milling to 75 h. The powders after milling for various durations were characterized by TEM, ICP and XRD, and these results are reported and discussed.

**[17] A COMPARISON OF DIFFERENT MODELS FOR MECHANICAL ALLOYING**

Harris JR. Wattis JAD. Wood JV. - Acta Materialia. 49(19):3991-4003, 2001



Five potential models describing the particle size distribution of powder during the mechanical alloying process have been analysed. The models are based on Smoluchowski's coagulation-fragmentation equations and use a range of size dependent aggregation and fragmentation rates. A technique has been developed to determine model parameters from the experimental average particle size. Size distributions predicted by these parameters compare well with the corresponding experimental results. The most successful models identify the most accurate size dependence. The numeric values of some aggregation and fragmentation rate parameters are qualitatively related to material properties of the initial powder charge.

**[16] THE MECHANICAL BEHAVIOR OF A CRYOMILLED AL-10TI-2CU ALLOY**

Hayes RW. Rodriguez R. Lavernia EJ. - *Acta Materialia*. 49(19):4055-4068, 2001

The mechanical behavior of a cryomilled Al-10Ti-2Cu (wt.%) alloy has been studied by performing uniaxial tension tests at temperatures ranging from room temperature to 525 degreesC. Elastic-nearly perfectly plastic stress-strain behavior is observed at all temperatures. Tension-compression asymmetry of the room temperature yield stress is also observed. Those characteristics are in agreement with those recently reported in the literature for single-phase NC materials. The flow stress (700 MPa at room temperature) decreases dramatically with increasing temperature. Testing of material following thermal exposures suggests that microstructural coarsening alone cannot account for the decrease in strength with increasing temperature. From a coarsening standpoint, this material appears to be very thermally stable. The ductility is influenced by several factors. Low levels of internal porosity along with the presence of fine oxide and carbide dispersoids contribute to lower ductility. The absence of work hardening exhibited by the Al-10Ti-2Cu also leads to reduced strain to failure. The features observed on fracture surfaces suggest that fracture occurs by the nucleation and growth of voids at particle-matrix interfaces. Evidence of fracture along prior powder particle boundaries is present as well. The microstructure consists primarily of regions containing grains measuring in the range 30-70 nm. Large grained regions consisting of nominally pure Al ranging in size from 300 to 500 nm are also present. No evidence of dislocation activity within either the fine or large grained regions can be found in the as extruded material. Specimens deformed at room temperature and 93 degreesC reveal evidence of dislocation activity within the large grain regions. Dislocation configurations suggest an Orowan bypass mechanism. No dislocations are found within the 30-70 nm size grains following tensile deformation

**[15] COLD ROLLING OF BULK NANOCRYSTALLINE COPPER**

Lu L. Sui ML. Lu K. - *Acta Materialia*. 49(19):4127-4134, 2001

By means of the electrodeposition technique, a bulk nanocrystalline (nc) copper sample was prepared with a high purity and a high density. The nc Cu sample was found to exhibit extreme extensibility under rolling at room temperature [*Science*, 287 (2000) 1463]. In this paper, the microstructure evolution of the nc Cu during the cold-rolling process was examined by means of X-ray diffraction (XRD) analysis, transmission electron microscopy (TEM) and differential scanning calorimetry (DSC). It was found in the initial stage of cold rolling that the microstrain and grain boundary energy increase substantially, while the crystallite size remains unchanged during the whole process. When the degree of deformation exceeds 1000%, the microstrain and the grain boundary energy tend to saturated values, implying that this deformation process is dominated by grain boundary activity rather than lattice dislocation. This phenomenon agrees well with the observed mechanical behavior of the nc Cu sample

**[14] DRY WEAR BEHAVIOUR AND ITS RELATION TO MICROSTRUCTURE OF NOVEL 6092 ALUMINIUM ALLOY-Ni3Al POWDER METALLURGY COMPOSITE**

Wang Y. Rainforth WM. Jones H. Lieblisch M. - *Wear*. 250(Part 2):1421-1432, 2001

Novel aluminium alloy matrix composites reinforced by 15 vol. % Ni<sub>3</sub>Al intermetallic particles were prepared by a powder metallurgy route. The 6092 aluminium alloy was produced by gas atomisation, followed by blending with Ni<sub>3</sub>Al particles. Consolidation by extrusion at 515 degreesC, with an extrusion ratio of 30:1, produced a uniform distribution of Ni<sub>3</sub>Al in the Al alloy matrix. The wear properties of the aluminium alloy-Ni<sub>3</sub>Al composites and the monoliths were examined by dry sliding against an M2 steel counterface at 0.94m/s and a load in the range 42-140N in a block-on-ring configuration. At 42 and 91 N, the composites offered superior wear resistance to the monoliths. Although, some surface deformation occurred at 42N, the Ni<sub>3</sub>Al largely retained its original shape, allowing it to act as a load-supporting reinforcement. At the highest load (140 N), the monolith offered superior wear resistance to the composite. Gross plastic deformation was observed at the worn surface of the composite, resulting in severe fragmentation of the Ni<sub>3</sub>Al. A mechanically mixed layer (MML) was observed for both materials at all loads, the thickness of which increased from similar to 25 µm at 42 N for both materials to similar to 80 µm for the monolith and similar to 40 µm for the composite at 140 N. Transmission electron microscopy (TEM) of MML identified a complex structure comprising an amorphous phase containing Al, O, a nanoscale Fe-, Al-, O-based phase, found mainly in the monolith, and in the composite, Ni<sub>3</sub>Al fragmented down to the nanometre scale. A linear relationship was found between the wear rate (mm<sup>3</sup>/m) and the depth of deformation, with a similar relationship being observed for both composite and monolith. The relationships between wear rate, surface structural changes and starting microstructure are discussed.

**[13] STUDY ON PHASE TRANSFORMATION OF FE-NI POWDERS DURING MECHANICAL ALLOYING**

Zhu LH. Ma XM. Zhao L. - *Journal of Materials Science*. 36(23):5571-5574, 2001

Fe-Ni ultra-fine particles were prepared by mechanical alloying and phase transformation during mechanical alloying was studied. Results show that phase transformation tendency is different during mechanical alloying of Fe-Ni powders with different nickel content. For Fe-30Ni powder, martensite is the only product obtained with the increase of milling time, while for Fe-35Ni powder, a part of martensite has been transformed into austenite when milling time is prolonged to above 40 h.



It indicates that the nickel content plays an important role in the phase transformation tendency during mechanical alloying of Fe-Ni powders.

**[12] MECHANOCHEMICAL PROCESSING OF ENARGITE (CU<sub>3</sub>AS<sub>4</sub>)**

Welham NJ. - Hydrometallurgy. 62(3):165-173, 2001

A commercial enargite (CU<sub>3</sub>As<sub>4</sub>) concentrate was mechanically milled in inert and oxidising atmospheres for up to 50 h. No substantial effect was observed in either an inert atmosphere or in air other than a broadening and weakening of the diffraction peaks. Milling in oxygen was dramatic with significant reaction evident even after 1 h. The products were As<sub>2</sub>O<sub>3</sub> and CU<sub>2</sub>SO<sub>4</sub>·5H<sub>2</sub>O, both of which are readily soluble; a small amount of SO<sub>2</sub>, was also formed. Dissolution in acid left CuS as the major phase; this phase was thermodynamically predicted to be a minor phase during the oxidation process and appeared to be stable for long periods under milling. Milling for 1 h at 100 degreesC resulted in the same products but showed a greater extent of oxidation. The small fraction of tennantite in the sample was comparatively resistant to oxidation

**[11] SOLID-PHASE SYNTHESIS OF COPPER(II) BETA-DIKETONATES UPON MECHANICAL ACTIVATION**

Petrova LA. Borisov AP. Aleshin VV. Makhaev VD. - Russian Journal of Inorganic Chemistry. 46(10):1501-1506, 2001

We showed that a solid-state reaction of copper(III) chloride with sodium beta -diketonates upon mechanical treatment in a vibration ball mill results in copper(II) beta -diketonates. The influence of milling conditions and the nature of the reactants on the course of the process, product yield, and some properties of activated mixtures was studied. The yield of copper(II) beta -diketonates reaches 70-90%. After mechanical preactivation of the reaction mixture, copper(II) beta -diketonate synthesis can proceed as a self-propagating reaction

**[10] THE REACTION OF FULLERENE C-60 WITH PHTHALAZINE: THE MECHANOCHEMICAL SOLID-STATE REACTION YIELDING A NEW C-60 DIMER VERSUS THE LIQUID-PHASE REACTION AFFORDING AN OPEN-CAGE FULLERENE**

Murata Y. Kato N. Komatsu K. - Journal of Organic Chemistry. 66(22):7235-7239, 2001

The reaction of fullerene C-60 with phthalazine was studied both in solution and in the solid state using the high-speed vibration-milling technique. The reaction in solution gave open-cage fullerene derivative 1 in 44% yield by a one-pot reaction. In contrast, the solid-state reaction afforded dimeric derivative 2 as the sole product. Dimeric derivative 2 was found to undergo intramolecular [2 + 2] cycloaddition between the two C-60 cages located in close proximity to give a new C-60 dimer 6 in quantitative yield. The structures of these new derivatives of C-60 were determined by spectroscopic methods, and the electrochemical behavior of 2 and 6 was also studied.

**[9] LOCAL STRUCTURE AROUND A NITROGEN ATOM FOR CR-N AND V-N AMORPHOUS ALLOYS PREPARED BY MECHANICAL ALLOYING**

Fukunaga T. - Journal of Non-Crystalline Solids. 293:187-192, 2001

Cr<sub>81</sub>N<sub>19</sub> and V<sub>76</sub>N<sub>24</sub> amorphous alloys were prepared by mechanical alloying under nitrogen gas atmosphere. Detailed structures for the two samples were studied by neutron diffraction. A coherent scattering length for the N atom for neutron scattering is much larger than that of the Cr atom, and moreover that of the V atom is negative and very small compared to those of Cr and N atoms. Radial distribution functions (RDF(r)) clearly indicate a positive peak around 2 Angstrom for the Cr-N system and also a negative peak around 2 Angstrom for the V-N system. In contrast, only very small positive peak in the vicinity of 2 Angstrom was observed in RDF(r)s by X-ray diffraction with Mo-K alpha radiation for the Cr-N and V-N systems. Therefore, these peaks can be identified as Cr-N and V-N correlations, respectively. Areas of the positive Cr-N and negative V-N peaks mean the coordination numbers of metal atoms (Cr or V) around a N atom, which were calculated to be 5.5 and 5.1 atoms for the Cr-N and V-N systems. The results indicate that the N atom absorbed during the mechanical alloying (MA) process is located at the center of polyhedra formed by 4 and 6 metal atoms for stabilizing the amorphous structure.

**[8] SHORT RANGE STRUCTURES OF AMORPHOUS SE-TE PREPARED BY MECHANICAL ALLOYING**

Itoh K. Misawa M. Fukunaga T. - Journal of Non-Crystalline Solids. 293:575-579, 2001

The time-of-flight neutron scattering measurement was employed in order to elucidate the short range structures for a series of amorphous Se<sub>100-x</sub>Te<sub>x</sub> (x = 0 similar to 20) mixtures synthesized by mechanical alloying (MA). The first peak in the radial distribution functions was resolved into two Gaussian peaks, which correspond to the Se-Se and Se-Te correlations, respectively. Amorphous Se prepared by MA has a lot of dangling bonds. In contrast, for the Se<sub>100-x</sub>Te<sub>x</sub> samples, Te atoms are preferentially bonded with the dangling bonds of Se atoms with increasing Te concentration and, consequently, a twofold chain structure is formed.

**[7] AMORPHIZATION OF GE-GESE MIXTURES IN MECHANICAL ALLOYING PROCESS**

Shirakawa Y. Matsuda T. Tani Y. Shimosaka A. Hidaka J. - Journal of Non-Crystalline Solids. 293:764-768, 2001

Structural changes by mechanical stress of a planetary ball mill have been investigated in Ge-GeSe eutectic mixtures. Amorphization of the GeSe Compound was accomplished after 27 h of grinding under the present condition, Pair distribution function g(r) of the amorphous GeSe is calculated. The nearest neighbour Ge-Se distance is 2.37 Angstrom, which is smaller than that of crystalline GeSe. The only crystalline GeSe phase in the eutectic mixtures selectively changes to the amorphous phase. Ge-GeSe alloying is not performed at the atomic level under the present condition, because the peak positions of interplanar spacings of GeSe are almost constant in the variation with grinding time. The decay rate of integrated intensities of (1 1 1) and (0 0 4) peaks of GeSe is evaluated. The amorphous transition energy related to the decay rate for the crystalline-amorphous transition of GeSe in the mixtures decreases with decreasing GeSe content.

**[6] CRYSTALLINE-AMORPHOUS TRANSITIONS OF GE-SE ALLOYS BY MECHANICAL GRINDING**



Tani Y. Shirakawa Y. Shimosaka A. Hidaka J. - Journal of Non-Crystalline Solids. 293:779-784, 2001  
X-ray diffraction and differential scanning calorimetry (DSC) measurements of Se and GeSe<sub>2</sub> during ball milling have been investigated. In this study, amorphous Se and GeSe<sub>2</sub> are synthesized by mechanical grinding (MG) recognized as a method of inducing a crystalline-amorphous phase transition. The amorphization of Se is caused from distortion between helical chains connected by van der Waals forces. The enthalpy AH for crystallization of Se by rapid quench is lower than that by the MG. This result suggests that the difference of AH corresponds to that of the structures in intermediate range. Monoclinic GeSe<sub>2</sub> is amorphized by disordering between the connected tetrahedra. It is considered that the tetrahedral unit of GeSe<sub>2</sub> by the MG is distorted with wide distributions. GeSe<sub>2</sub> is completely amorphized after 4 h of grinding, and Se after 30 h in the present milling. The difference indicates that disordering between the connected tetrahedral units in monoclinic GeSe<sub>2</sub> is induced more easily than that between chains in trigonal Se

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Zhu LH. Ma XM. Zhao L. - Journal of Materials Science. 36(23):5571-5574, 2001

Fe-Ni ultra-fine particles were prepared by mechanical alloying and phase transformation during mechanical alloying was studied. Results show that phase transformation tendency is different during mechanical alloying of Fe-Ni powders with different nickel content. For Fe-30Ni powder, martensite is the only product obtained with the increase of milling time, while for Fe-35Ni powder, a part of martensite has been transformed into austenite when milling time is prolonged to above 40 h. It indicates that the nickel content plays an important role in the phase transformation tendency during mechanical alloying of Fe-Ni powders.

#### **[4] EFFECT OF MECHANICAL ALLOYING ON COMBUSTION SYNTHESIS OF MOSI<sub>2</sub>**

Park HS. Shin KS. Kim YS. - Journal of Materials Research. 16(11):3060-3068, 2001

Characteristics of the combustion synthesis of MoSi<sub>2</sub> using elemental Mo and Si powder mixtures prepared by mechanical alloying were investigated. The mechanical alloying resulted in powders of pseudolamellar structure and a partial conversion of the elemental powders to hexagonal MoSi<sub>2</sub> phase. Combustion reaction of the mixture was ignited around 670 degreesC, which is much lower than that with the powder prepared by low-energy ball milling. A mathematical model was developed to demonstrate the possibility of the ignition of the combustion reaction of the lamellar structure via a solid-state diffusional process. On the basis of model, effects of mechanical alloying time on the ignition temperature and combustion temperature are discussed

#### **[3] AMORPHIZATION OF SILICON DURING MECHANICAL TREATMENT OF ITS POWDERS: 1. PROCESS KINETICS**

Streletskii AN. Leonov AV. Butyagin PY. - Colloid Journal. 63(5):630-634, 2001

The kinetics of silicon amorphization in the process of mechanical treatment of powders in a vibrating micromill was studied by the X-ray diffraction. The treatment was carried out in the argon atmosphere, the apparatus energy intensity was equal to 18 W/g, and the amount of consumed energy (dose) was as high as 510 kJ/g (14 MJ/mol). The analysis of the shape of X-ray diffraction patterns and the dynamics of the changes in silicon atomic structure were described within the framework of three-fraction model. Fraction 1 composed of large crystalline blocks comprising particles of initial powder; the second fraction is represented by nanocrystalline blocks with the dimensions of not less than 8 nm; and the third fraction is an amorphous phase. A decrease in the content and sizes (from 10(2) to 25 nm) of initial microcrystals of fraction I is accompanied by the formation of X-ray amorphous phase 3. Nanocrystalline blocks of fraction 2 are none other than the intermediate products. They are first accumulated synchronously with the amorphous phase and then disintegrated with a decrease in their sizes from 8 to 4 nm. At the initial stage of experiment, at the dose up to 15 kJ/g and the degree of amorphization up to 40%, the energy yield of the formation of amorphous phase amounts to 1 +/- 0.1 mol/MJ. At the end of experiment (the dose varies from 20 to 510 kJ/g), the yield drops by tens of times, and the content of amorphous phase reaches 70-80%.

#### **[2] AMORPHIZATION OF SILICON DURING MECHANICAL TREATMENT OF ITS POWDERS: 2. HEATS OF RECRYSTALLIZATION**

Streletskii AN. Mudretsova SN. Maiorova AF. van Miltenburg JC. Butyagin PY. - Colloid Journal. 63(5):635-638, 2001

Exothermic effects arising during silicon powder heating after their mechanical treatment were measured by synchronous TG-DSC thermal analysis method. Silicon samples possessed semicrystalline structure. As temperature varied from room temperature to 1200 degreesC at a rate of 10 and 50 deg/min, the heat was released in the range 550-800 degreesC. The crystallization of amorphous phase was also observed in the same temperature range using the X-ray diffraction. As the amount of energy consumed during the mechanical treatment of powders increased, the value of the heat effect rose synchronously with the degree of amorphization. In this case, the ratio of the released heat to the content of X-ray amorphous phase was constant and equal to 6 +/- 2 kJ/mol

#### **[1] AMORPHIZATION OF SILICON DURING MECHANICAL TREATMENT OF ITS POWDERS: 3. SORPTION OF GASES**

Butyagin PY. Streletskii AN. Berestetskaya IV. Borunova AB. - Colloid Journal. 63(5):639-644, 2001

Several series of adsorption measurements were made for a more detailed analysis of the processes resulted in the amorphization of silicon during the mechanical treatment of its powder. Among these measurements are the determination of the specific surface area by the BET method and the registration of "instantaneous" sorption of hydrogen and oxygen directly during the mechanical treatment of powders in the atmosphere of these gases. The sorption of hydrogen is low corresponding approximately to the monomolecular coverage of a freshly formed silicon surface. The sorption of oxygen (P > 100 Torr) is significantly higher than that of hydrogen and its yield is close to that of amorphous phase in the inert environment. The presence of oxygen slows down the formation of amorphous



phase. To explain this result, it was suggested that, in the regions of the concentration of mechanical stresses, silicon is active, disordered, and possesses a high sorption capacity. In the oxygen atmosphere, the sorbed molecules tend to oxidize the active silicon, whereas, in an inert atmosphere, (lie amorphous phase that is stable up to 800 degreesC is formed from active silicon



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## Press release

### “PULVERISETTE 5” PLANETARY BALL MILL

Fast, contamination free grinding below 1 micron

Handles sample volumes from 1 ml up to 4 x 225 ml

**The Fritsch planetary ball mill, the standard of 35 years of grinding experience, has been completely redesigned and upgraded.**

**Features now include:**

**rapid, batchwise grinding down to colloidal fineness, dry or in suspension. For mixing and homogenising of emulsions and pastes. Feed size less than 10 mm edge length.**

**the “pulverisette 5” carries the CE mark for proven safety. (for example: the mill cannot be opened during operation, nor started up with the lid in the open position).**

**no disturbing contamination because grinding bowls and balls are available in 9 different materials**

**reproducible grinding and full recovery in sealed grinding bowls  
simultaneous grinding of either 8 small samples or 4 large samples  
membrane keypad with digital display of operational parameters, including rotational**

**speed of support disc (360 rpm)**

**Integral fan located in the grinding chamber for additional cooling permits longer grinding process**

**Grinding and cooling sequences programmable for any milling process**

**special lids with valves available for milling in an inert atmosphere suitable for mechanical alloying**

**if smaller quantities (up to approximately 40 ml) are to be processed to the fineness of < 1 micron according to the same principle of operation the “pulverisette 7” Planetary Micro-Mill is utilised.**

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