



**RESEAU FRANÇAIS DE  
MECANOSYNTHESE**

**Lettre N°76**

**Juillet 2001**

**185 (+1) Groupes de Recherche  
(dont 111 (+1) à l'étranger / 33 Pays)**

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

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**Nouvelle Adhésion**

A. Popovich - Russie

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Full Congress Announcements  
Congress List (related to Nanomaterials)  
New Proposal for Cooperation, PhD, **new Post Doc proposal**, International Relationships)  
Books (related to Nanomaterials)  
Bibliography  
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Le site web du RFM est :

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

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

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

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**Bulletin d'adhésion 2001 / Subscription Print**

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

Eric GAFFET

CNRS UMR5060 « Métallurgies et Cultures »

Thème « Nanomatériaux : Elaboration et Transitions de Phases Hors Equilibre »

Site de Sévenans (UTBM)

F90010 - Belfort Cedex - France

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

Adresse complète / Full Address : .....

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

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

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

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

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

Lettre RFM N°76 - Juillet 2001  
Corresp. : <mailto:Eric.Gaffet@utbm.fr>

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



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**Announce de congres et / Ou Ecoles  
Congress and School Announcements**

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**COLLOQUE SUR LES INNOVATIONS  
DANS LES MATERIAUX FRITTES**

Poitiers-Futuroscope

3-4-5 juillet 2001

consulter le site, <http://www.sf2m.asso.fr/>(rubriques sommaires puis conférences)

**International Conference on the Applications of the Mossbauer Effect**

Oxford, UK

2-7 September 2001

Abstracts are now invited for for the above meeting, which is the next in the ICAME conference series. You are asked to submit your abstract via the conference website <http://www.iop.org/IOP/Confs/ICAME/> by no later than 1 April 2001.

For further information or enquiries please contact the Conference Office at the Institute of Physics, 76 Portland Place, London W1B 1NT, UK.

E-mail should be directed to: <mailto:rebecca.chapple@iop.org>

International Conference

**"FUNDAMENTAL DASES of MECHANOCHEMICAL TECHNOLOGIES"**

Novosibirsk, Russia, August 16-18, 2001

Contact: Prof. N. Lyakhov

Institute of Solid State Chemistry

E-mail: <mailto:Conf@solid.nsc.ru>

Fax: , +7 3832 32 28 47

The first circular is available on WEB-Site of the Institute:

<http://www.solid.nsc.ru/>

**(IPCM 2001)**

La 7eme conference internationale sur les phenomenes d'interface dans les materiaux composites (IPCM 2001) se tiendra au palais des congres d'Arcachon (40 km de Bordeaux) du 11 au 14 septembre 2001.

<http://www.arcachoncongres.com/ipcm2001/>

**(IWSIS-3)**

October, 7-12, 2001.

3rd International Workshop on Surface and Interface Segregation , Island of Porquerolles, French Riviera, This Workshop is devoted to the study of the segregation phenomenon in defects of crystallized solids (surface, grain boundary, interface of interphase...)

INFOS, : <http://www.crmc2.univ-mrs.fr/confs/iwsis>

**"VI International Symposium on Self-Propagating High-Temperature Synthesis, (SHS-2001)"**

Haifa, Israel . October 14-18, 2001.

More information on the

Web site: <http://www.technion.ac.il/technion/materials>

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>

**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>

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



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**Matériaux 2002**

Tours - France  
21- 25 Octobre 2002

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

<http://www.up.ac.pa/Eventos/lacame2002/inicio.htm>  
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**SOUTENANCES DE THESE**  
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**Nathalie Bouad**

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

**Potentialité de la mécanosynthèse d'alliages à base de tellure 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:

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

From, Professor Soon H. Hong

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

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

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

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

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



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

**Singapour (11/06/2001) – From Professor L. Lu**

**Post-doctoral fellow position**

The position requires candidates with an Ph.D. degree materials science. He/she should have a demonstrated track record on synthesis of metallic amorphous materials. Preference will be given to candidates with relevant experience of mechanical alloying.

The application form can be downloaded from the website:

<http://www.nus.edu.sg/NUSinfo/Appoint/RESAPPT.HTML>

Please submit your application to  
Associate Professor L. Lu  
Dept. of Mechanical Engineering  
National University of Singapore  
10 Kent Ridge Crescent  
Singapore 119260  
E-mail: <mailto:mpeluli@nus.edu.sg>

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**Brazil (4 / 6 / 2001) – Post Doc Position from Professor Gerardo F. Goya**

The Magnetic Materials Group at São Paulo University is seeking a postdoctoral associate with experience in powder synthesis and magnetism to work on nanostructured ceramics. The candidate should demonstrate the ability to work independently, contribute to innovative experimental design, and develop new projects in this area. Background in at least three of the following areas is desirable: Mechanochemical synthesis. Mechanical alloying. Magnetism in nanostructured systems. Transport measurements. Mossbauer Spectroscopy Scanning/Transmission Electron Microscopy

The candidate should send a curriculum vitae, three representative publications (preferably with the candidate as a first author) and the names, addresses, email and phone numbers of two references that can comment on the candidate's capabilities. Position is open for applicants within three years of receipt of Ph.D. The postdoctoral contract will be one+one year, with salary US\$ ~15000 /y.

Applicants should send the information before 15-August-2001.

Interested candidates should send **correspondence** to:

Professor Gerardo F. Goya  
Laboratório de Materiais Magnéticos  
Instituto de Física - Universidade de Sao Paulo  
CP 66318 Sao Paulo  
05315-970 SP Brazil  
e-mail: [goya@macbeth.if.usp.br](mailto:goya@macbeth.if.usp.br)  
Fax: (55) 11 3818 6984  
Desk: (55) 11 3818 6885

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**France (28/05/2001) – from V. Nivoix**

Proposition de sujet de thèse du

Laboratoire d'Analyse Spectroscopique et de Traitement de Surface des Matériaux,  
UPRES EA 1290, Université de Rouen

**Relation structure-propriétés d'oxydes mixtes nanométriques élaborés par différentes voies de synthèse**

Les ferrites mixtes manganèse-zinc utilisés dans les composants électroniques sont des ferrites doux toujours très utilisés de nos jours, dont les performances peuvent encore être améliorées.

Les propriétés magnétiques, largement conditionnées par la répartition cationique dans les sites de la phase spinelle, dépendent également de la microstructure et plus particulièrement de la porosité et de la taille moyenne des grains dans le matériau final.

Le procédé industriel actuel par voie céramique ne permet pas d'obtenir une microstructure dense à grains fins pourtant très favorable. De nouvelles voies de synthèse sont actuellement explorées, notamment dans le domaine de la "chimie douce".

Nous nous proposons d'élaborer ces oxydes sous forme de poudres nanométriques par broyage à haute énergie et par voie hydrothermale puis de comparer leurs caractéristiques physiques et structurales.

La synthèse par broyage à haute énergie se fera à l'aide d'un broyeur planétaire nouvelle génération (P4 de Fritsch) permettant d'optimiser le broyage par une meilleure maîtrise des paramètres techniques.



Pour mener à bien la caractérisation complète de ces matériaux nous disposerons de différentes techniques telles que la diffraction des rayons X, la spectrométrie IRTF et la spectrométrie Mössbauer pour la caractérisation structurale, la microscopie électronique à balayage haute résolution ou à transmission et la DRX pour la taille et la morphologie, un SQUID pour les propriétés magnétiques. D'autres méthodes d'analyse pourront être mises en œuvre selon les besoins de l'étude. Le candidat ou la candidate devra avoir des connaissances en chimie des solutions aqueuses et sur la caractérisation des matériaux (diffraction des rayons X, spectrométrie IR, spectrométrie Mössbauer, mesure de magnétisme ...)

**Financement :** Nous ferons une demande de financement auprès du ministère de la recherche ou de la région Haute-Normandie.

**Contacts :** Virginie NIVOIX ou Malick JEAN - Université de Rouen - LASTSM-IUT  
76821 Mont Saint Aignan Cedex  
tel 02 35 14 63 59 fax 02 35 14 63 58  
email : malick.jean@univ-rouen.fr

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**SPAIN (03/04/2001)- From J.J. Suñol (joan josep.sunyol@udg.es)**

### **Postdoctoral researchers required Universitat de Girona**

Soft magnetic materials obtained by mechanical alloying and rapid solidification: thermal and structural characterization. Analysis of nanocrystallization process.

The research position will involve aspects of: materials processing by mechanical alloying, thermal and structural characterization by DSC, TG., XRD, SEM, TEM, TMS; kinetic modeling.

The position will begin with effect from september 2001 to september 2002.

Interested candidates should send correspondence to: Dr. J.J. Suñol.

Department of Physics., EPS (P II). Girona University. E-17071. Girona, Spain. Fax: 34-972418098.

E-mail: <mailto:joan josep.sunyol@udg.es>

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### **(From Paul J. Warren – 6/03/2001)**

A Research Training Network on nanostructured Aluminium alloys is urgently looking for researchers. There are opportunities for Post-doctoral and Pre-doctoral researchers available immediately in Oxford(UK), Grenoble(F), Turin(I), Stockholm(S), Madrid(E), Waterford(IRL), Ioannina(G), Warsaw(PL), and Bratislava(SK). Please pass this information to anyone who may be interested.

For further details please visit the website <http://www.materials.ox.ac.uk/nano-al>.

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### **Post Doctoral Fellow Positions - University of Toronto (5/03/2001)**

The University of Toronto is seeking three postdoctoral fellows for a major new initiative in laser nanofabrication, material diagnostics, and nano-optics fabrication. State-of-the-art laser-processing facilities and optical material fabrication and diagnostic infrastructure are funded from various government and industrial sources. The research is centered in the Photonics Group of the Department of Electrical and Computer Engineering, and directed by Professor Peter R. Herman.

**1 - F2-Laser Nanofabrication Facility** The post doctoral fellow will drive the development of precise optical tools and nanofabrication processes in one of the world's forefront facilities for F2-laser nanofabrication research. The record short-wavelength light of 157 nm drives strong interactions in challenging materials at sub-micron feature sizes that are attractive for widespread application in fabricating photonic components, optical circuits, lab-on-a-chip systems, and wireless electronic circuits. Responsibilities include co-supervision of graduate students, coordination of research activities with scientists from academia and industry, and co-management of the novel facility. The \$1 million program also includes establishing state-of-the-art optical communication diagnostics. The research goals are new micromachining and photosensitivity processes for fabricating and/or tuning optical circuits, 3-D photonic devices, photonic bandgap structures, and binary optics. These studies are to be integrated with related program in ultrafast laser processing.

**2 - Photonic-Bandgap Integrated Optical Circuits** An outstanding candidate is sought for a collaborative project to integrate photonic bandgap structures into functional optical circuits. Three-dimensional diffractive optical structures offer in theory, powerful capabilities in managing light in optical circuits. The goal is to practically harness this capability within the structure of our existing photonics fabrication program (the F2-laser nanofabrication facility and ultrafast-laser processing laboratory) and through collaboration with a leading photonic-bandgap group at the University of Toronto. The project is suited to a technically strong and creative individual motivated to revolutionize the future manufacturing of photonic circuits.

**3 - Laser-Induced Breakdown Spectroscopy of Aluminum** A laser-spectroscopy specialist is required to drive an industrially sponsored research program in laser-induced breakdown spectroscopy of recycled aluminum. The goal is to develop novel laser and diagnostic technology for collecting accurate assays of aluminum metal for a future large-scale pilot project in automobile recycling by Alcan International. One project is the study of a new laser interaction - invented at the University of Toronto - that entails high-repetition 'bursts' of ultrafast laser pulses. This approach promises to cleanly remove surface oxides and precisely probe the underlying bulk aluminum within a single burst. Research centers on fundamental laser interactions and defining laser processing windows in cooperation with our industrial partner.

The research positions entail extensive academic collaboration within the Engineering Faculty, the Department of Physics, the Department of Chemistry, and Photonics Research Ontario ([www.pro.on.ca](http://www.pro.on.ca)) and with other academic research centers: Laser Laboratory, Goettingen, Germany; National Research Council, Canada; and Optical Fibre Technology Centre, Australia. Research also includes close interaction with world-leading photonics and manufacturing companies in Canada



(i.e. JDS Uniphase, Mitel, Raytheon Elcan Optical Technology, Alcan International) and internationally (i.e. Photonics Integrated Research, Lambda Physik, MicroLas). Our principle goals are forefront science and engineering research for public dissemination in high-quality journals and the generation of intellectual property. Successful candidates will lead one of the following three research areas.

Required qualifications for all three positions include a Ph.D. in experimental Physics, Engineering Science, or Electrical Engineering, and experience with several of the following areas: F2 or excimer lasers, ultrafast lasers, optical and opto-mechanical design, photonic devices for optical communications, optical waveguide fabrication and modelling, optical communication diagnostics, photonic bandgaps, laser-matter interaction physics, spectroscopy, and material diagnostics (SEM, FTIR, AFM, SEM, EDX, ESR). An independent and highly motivated person with good technical and communication skills is required. Each position entails a supervisory role with graduate students and other researchers. The successful candidate will also be responsible for coordination and administration of research involving visiting scientists and industrial partners in the local booming Photonics industry.

The postdoctoral positions are available immediately and remain open until filled. Provide a CV, relevant publications, three references, and recent university transcripts by mail, electronically, or by fax:

Professor Peter R. Herman

10 King's College Rd. Tel: 416-978-7722 - Dept. of Electrical and Computer Engineering Fax: 416-971-3020

University of Toronto, Toronto, ON [hermanp@ecf.utoronto.ca](mailto:hermanp@ecf.utoronto.ca) - M5S-3G4, CANADA

*The University of Toronto is Canada's top university, located in the center of Canada's largest and most dynamic city.*

*Toronto is home to a large and diverse immigrant population and has low-crime rates. See more at:*

<http://www.utoronto.ca/toronto.htm>

Further Employment Information: [http://www.ecf.utoronto.ca/~hermanp/job\\_available.htm](http://www.ecf.utoronto.ca/~hermanp/job_available.htm)

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**From B. Mhohamed – UK – (10/01/2001)**

**Marie Curie Training Fellowships**

Applications are invited for 3-12 month research fellowships supported by the Marie Curie Training Sites scheme. The successful candidates will be involved with the processing of alloys, intermetallics, nanostructures, or composites for high-temperature, biomedical and/or energy-storage applications. Processing techniques and facilities include ball milling, mechanical alloying, reaction synthesis, tape casting, slurry powder metallurgy, and vacuum cold/hot pressing. Materials characterisation will be carried out by TG/DTA, DSC, MS, optical microscopy, X-Ray, and SEM/TEM techniques. Complementary modelling activities for materials-design, processing, microstructural evolution, and/or property predictions may also be involved as part of the fellowship training programme. Modelling methodologies range from *ab initio* atomistic simulations to finite-element methods. The candidates must satisfy the basic criteria of the training scheme as outlined under <http://www.cordis.lu/improving>. As the fellowship forms part of a higher degree project, the candidates should be a registered full-time PhD research student in a well-recognised institution, working on materials synthesis, characterisation, and/or computer modelling of materials, of an EU nationality (non-UK) and under 35 years of age. Deadline for application: 30 February 2001.

For further details, please contact: Professor Z. Xiao Guo, phone: 0044-20-7882-5569; e-mail: [x.guo@qmw.ac.uk](mailto:x.guo@qmw.ac.uk); or visiting: <http://www.metallicmaterials.com/>.

QMW / University of London is an equal opportunity employer.

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**From A.R. Yavari - France (8/01/2001)**

**EU Postdoc/Ph.D. positions in fields of Nanostructured Materials and Bulk**

**Metallic Glasses** are available immediately in France and several other EU

States. Please check the following web page

<http://www.inpg.fr/BMG-RTN/>

and contact the Coordinator A.R. Yavari at <mailto:euronano@ltpcm.inpg.fr>



## Bibliographie Récente

### Livres ou "Special Issues"

(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](mailto:koehler@fritsch.de))

#### (7/07/2000) - From Victor Rieicansky Publisher

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

Contents

Introduction - Background - History of Mechanical Alloying - Milling - Factors Affecting the Mechanical Alloying, 8 - Types of Mills, 8 - High Energy Ball mill, 9 - Attritor Ball Mill, 9 - Planetary Ball Mill, 11 - Vibratory Ball Mill, 12 - Low Energy Ball Mill, 15 - Tumbler Ball Mill, 15 - Tumbler Rod Mill, 16 - Effect of Ball-to-Powder Weight Ratio, 19 - Effect of Milling Atmosphere, 22 - Mechanism of Mechanical Alloying, 23 - Ball-Powder-Ball Collision, 24 - Necessity of Mechanical Alloying, 25 - References, 27

**PART I, GRAIN REFINING, SIZE CONTROLLING AND HOMOGENIZATION**

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**PART II, ROOM TEMPERATURE REACTIVE MILLING**

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(05/05/2000)

**Extractive Metallurgy of Activated Minerals**

included in series Process Metallurgy, 10

by P. Balaz - Institute of Geotechnics, Slovak Academy of Sciences

ISBN : 0 - 444 - 50206 - 8 / Price USD 144, Euro 124.79)

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Description

Mechanical activation of solids is a part mechanochemistry, the science with a sound theoretical foundation exhibiting a wide range of potential application. Mechanical activation itself is an innovative procedure where an improvement in technological processes can be attained via a combination of new surface area and defects formation in minerals.

Mechanical activation is of exceptional importance in extractive metallurgy and mineral processing and this area forms the topic of this book and is a result of more than twenty years of research and graduate teaching in the field.

In pyrometallurgy, the mechanical activation of minerals makes it possible to reduce their decomposition temperatures or causes such a degree of disordering that the thermal activation may be omitted entirely. The potential mitigation of environmental pollutants is becoming increasingly important in this context.



The lowering of reaction temperatures, the increase of the rate and amount of solubility, preparation of water soluble compounds, the necessity for simpler and less expensive reactors and shorter reaction times are some of the advantages of mechanical activation in hydrometallurgy. The environmental aspects of these processes are particularly attractive. Several industrial processes are examined and the flowsheets are presented as successful of activation. In these processes, the introduction of a mechanical activation step into the technological cycle significantly modifies the subsequent steps. The book is designed for researchers, teachers, operators and students in the areas of extractive metallurgy, mineral processing, mineralogy, solid state chemistry and materials science. It will encourage newcomers to the mechanochemistry to do useful research and discover novel applications in this field.

(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 234x156 mm, cased, £45.00, 1999

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

1. Characteristics of dispersion-strengthened systems 2. Mechanical alloying (kinetics and mechanism of preparation of the Al-C system by mechanical alloying; compaction of powders and heat treatment of compacts);

3. Microstructure and quantitative evaluation of parameters of dispersion-strengthened materials (definition and properties of interparticle distance; experimental possibilities of determination of structural objects; models of heterogeneous structures and their evaluation; simulation of model structures; analysis of the spatial distribution of particles in the Al-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, 234x156 mm, soft laminated cover, £25.00, 1999

"Mechanical Alloying : Fundamentals and Applications"

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

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

"Non Equilibrium Processing of Materials"

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

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

web site : <http://www.elsevier.nl/locate/isbn/0080426972>

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

A. Inoue

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

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

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

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

DISPERSION-STRENGTHENED ALUMINIUM PREPARED BY MECHANICAL ALLOYING



Lettre RFM N°76 - Juillet 2001  
Corresp. : <mailto:Eric.Gaffet@utbm.fr>

Michal Besterci, Institute of Materials Research, Slovak Academy of Sciences, Kosice

In the book, the author describes the theoretical and technological fundamentals of mechanical alloying the Al-C system. Special attention is given to material characteristics, the kinetics and mechanism of mechanical alloying, methods of mixture compaction and heat treatment of compacted parts. Models of dispersoid spatial arrangement, dispersoid evaluation and optimisation and experimental possibilities are discussed. The interpretation of the static and dynamic mechanical properties, especially strength and ductility properties at 20 °C, mechanical properties at elevated temperatures are discussed, with emphasis on the effect of interface, superplasticity, creep and creep-fatigue characteristics. Content

Introduction

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

Index : ISBN 189832655X, 80 pages, 234x156 mm, soft laminated cover, £22.00, January 1999

Cambridge International Science Publishing 7 Meadow Walk, Great Abington, Cambridge CB1 6AZ, England Fax +44 1223 894539; Tel +44 1223 893295 Email: cisp@cisp.demon.co.uk  
http://www.demon.co.uk/cambsci/homepage.htm

### "Mechanical Alloying"

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

Kluwer Academic Publishers

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

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

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

Materials Research Society, Symposium Proceedings Volume 501, 1998

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

### "Nanomatériaux"

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

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

Les dernières années ont vu apparaître dans le monde des matériaux avancés le préfixe "nano" (nanostructuré, nanocristallins, nanophase ou nanométrique) ; les conférences et les forums sur Internet se multiplient où s'échangent des informations sur les avancées scientifiques et technologiques dans ce domaine des matériaux nanostructurés qui se distinguent des matériaux polycristallins conventionnels par la dimension des cristallites les composant ou par la dimension des hétérostructures présentes : ces dimensions sont de quelques dizaines d'ångströms, 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 lancé d'ambitieux programmes de R & D puisque le MITI a consacré aux nanomatériaux près de 200 millions de dollars pour la période 1990 - 2000 et que la Science & Technology Foundation a investi presque la même somme pour co - financer des projets de laboratoires publics et privés. Les Etats Unis puis les pays européens ont investi plus tardivement mais déjà ont obtenu des résultats prometteurs (.....) Certaines applications existent déjà au niveau international, quelque 400 sociétés se partagent aujourd'hui un marché voisin de 1 milliard de dollars mais qui devrait tripler, voire quintupler à l'horizon 2001.(.....)

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

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

### Mechanochemistry of Materials

#### Cambridge International Science Publishing

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

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

### BIBLIOGRAPHY ON MECHANICAL ALLOYING AND MILLING

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



Lettre RFM N°76 - Juillet 2001  
Corresp. : <mailto:Eric.Gaffet@utbm.fr>

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

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

### [76] MATERIALS FOR RECHARGEABLE BATTERIES AND CLEAN HYDROGEN ENERGY SOURCES [REVIEW]

Wronski ZS. - International Materials Reviews. 46(1):1-49, 2001.

Materials for energy sources, such as rechargeable batteries and fuel cells, are functional materials. Their development presents special challenges since they are designed for more than one function and must satisfy multiple and mostly interlocking requirements. A unique combination of properties is achievable in complex structures such as functional composites, multi-element and multiphase intermetallics, cermets, layered insertion compounds, and new nanostructured and disordered phases. This review deals with a variety of such materials being developed for use in clean energy storage, which is mostly, but not entirely, hydrogen energy storage. The scope of the work encompasses hydrogen gas storage alloys and intermetallics used for electrochemical hydrogen storage, insertion compounds for Li batteries, and ceramics and metal catalysts for fuel cells. It also includes materials used in lead-acid, nickel metal hydride, and lithium rechargeable batteries as well as in solid oxide, proton exchange membrane, and direct methanol fuel cells. Special topics highlight new nanostructured materials obtained by rapid quenching, mechanical alloying, and other processes. The review is complemented by a brief discussion of present trends in assessment of materials requirements for batteries and fuel cells for electric vehicles and portable telecommunication. Also addressed are aspects of recycling and life-cycle analysis

### [75] MATERIAL REMOVAL MECHANISM IN CHEMICAL MECHANICAL POLISHING: THEORY AND MODELING

Luo JF. Dornfeld DA. - IEEE Transactions on Semiconductor Manufacturing. 14(2):112-133, 2001

The abrasion mechanism in solid-solid contact mode of the chemical mechanical polishing (CMP) process is investigated in detail. Based on assumptions of plastic contact over wafer-abrasive and pad-abrasive interfaces, the normal distribution of abrasive size and an assumed periodic roughness of pad surface, a novel model is developed for material removal in CMP. The basic model is  $MMR = \rho(w)NVol(removed)$ , where  $\rho(w)$  is the density of wafer,  $N$  the number of active abrasives, and  $Vol(removed)$  the volume of material removed by a single abrasive. The model proposed integrates process parameters including pressure and velocity and other important input parameters including the wafer hardness, pad hardness, pad roughness, abrasive size, and abrasive geometry into the same formulation to predict the material removal rate (MRR). An interface between the chemical effect and mechanical effect has been constructed through a fitting parameter  $H-w$ , "dynamical" hardness value of the wafer surface, in the model. It reflects the influences of chemicals on the mechanical material removal. The fluid effect in the current model is attributed to the number of active abrasives. It is found that the nonlinear down pressure dependence of material removal rate is related to a probability density function of the abrasive size and the elastic deformation of the pad. Compared with experimental results, the model accurately predicts MRR. With further verification of the model, a better understanding of the fundamental mechanism involved in material removal in the CMP process, particularly different roles played by the consumables and their interactions, can be obtained.

### [74] THE INFLUENCE OF ADDITIVES DURING WET ULTRA-FINE GRINDING IN AGITATOR BEAD MILLS - PART 2: RESULTS AND CONCLUSIONS

Reinsch E. Bernhardt C. Husemann K. - Cfi, Ceramic Forum International/Berichte der Dkg (Deutsche Keramische Gesellschaft). 78(4):E36-E40, 2001

Additives that increase the repulsive interactions between particles can improve the flowability of a suspension. Thus, at constant energy utilization and a higher solids concentration, the mill throughput can be increased. On the other hand, with a constant solids concentration and equivalent end fineness, considerable energy savings can be achieved or, respectively, a significantly higher product fineness at constant specific energy can be realised. The extent of the effects that are seen through the addition of additives in wet fine grinding are determined by: the solids concentration, the particle size and thus the distance between the particles the stress intensity the particle interactions in the initial condition the possible impurities in the product (through wear from the grinding bodies or wall of the mill) that go into solution, thus altering the particle interactions during grinding the amount of energy loss due to viscosity with respect to the total energy used. Experimental investigations were made on two different model substances (limestone and corundum) with the addition of a number of inorganic and organic additives. The relationship found between the particles interactions, theological properties and comminution parameters are of interest, primarily in the grinding techniques in nano-technology, since only through the addition of appropriate additives a very larger percentage of particle sizes in the range  $<1 \mu m$  can be achieved

### [73] MAGNETIC PROPERTIES OF BALL-MILLED MN NANOPARTICLES

Abdul-Razzaq W. Wu M. - Superlattices & Microstructures. 29(4):273-279, 2001

Nanoparticles of Mn of sizes  $<500 \text{ \AA}$  were prepared by the ball-milling technique. The temperature dependence of the magnetic susceptibility  $X$  showed systematic variation with particle size. Peaks observed in  $chi$  were attributed to the magnetic ordering of the oxides  $Mn_3O_4$  and  $MnO$ . Peaks found in partial derivative( $chi/T$ )/partial derivative $T$  were associated with the Neel temperature of  $\alpha$ -Mn. We estimated that our samples contain about 0.4% of  $Mn_3O_4$ . This low concentration of  $Mn_3O_4$  was not detected by X-ray diffraction experiments but contributed significantly to the magnetization measurements

### [72] MICROSTRUCTURAL SIZE EFFECTS IN HIGH-STRENGTH HIGH-CONDUCTIVITY CU-CR-NB ALLOYS

Anderson KR. Groza JR. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 32(5):1211-1224, 2001

Microstructural refinement to further improve the strength and stability of high-strength high-conductivity Cu-Cr-Nb alloys was attained by mechanical milling (MM). Mechanically milled Cu-4Cr-2Nb and Cu-8Cr-2Nb exhibited an increase in hot-pressed Vickers hardness of 122 and 96 pct, respectively. Mechanical milling produced a corresponding decrease in electrical conductivity of similar to 33 pct for both alloys. The increase in hardness was more due to Cu grain-size refinement than to second-phase particle-size refinement. The drop in conductivity was due to second-phase particle-size refinement, which both increased particle/matrix interfacial area and solute solubility. Mechanically processed Cu-4Cr-2Nb displayed an enhanced thermal stability. Hot-pressed 4-hour milled Cu-4Cr-2Nb experienced a 30 pct increase in conductivity with only a 22 pct drop in hardness when annealed at 1273 K for 50 hours. Such changes were largely due to an increase in dispersed-



particle size (i.e., a decrease in solute and interfacial electron scattering) and Cu grain size (reduced Hall-Petch effect), respectively. The optimum hardness and conductivity combination was found in 4-hour milled and hot-pressed Cu-4Cr-2Nb material.

**[71] DISPERSION STRENGTHENED ALLOY DUE TO THE PRECIPITATION OF CARBIDE DURING MECHANICAL ALLOYING**

Wang CG. Qi BS. Bai YJ. Wu J. Yang JF. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 308(1-2):292-294, 2001

After 60 h of ball milling, Fe-based and Cu-based supersaturated solid solutions form in the two alloy powders of Fe-1.7 C-15Ti, and Cu-3.3 C-13.3Ti, from which TiC dispersion strengthened alloys can be obtained after further milling to 100 h, or by vacuum heat treatment at 800 degreesC for 1 h

**[70] NANO-SIZED AMORPHOUS ALUMINA PARTICLES OBTAINED BY BALL MILLING ZNO AND AL POWDER MIXTURE**

Wu JM. - Materials Letters. 48(6):324-330, 2001

Solid state reduction reaction of ZnO and Al powder mixtures induced by mechanical alloying has been investigated. It is revealed that the reduction product consists of crystalline zinc and amorphous alumina particles with nanometer sizes of 10-50 nm. The zincite is found to be reduced by Al through a diffusion-controlled mechanism, rather than a rapid self-sustaining combustion reaction process. This is attributed to the fact that the calculated theoretical adiabatic temperature for the reaction is far below the critical one to trigger the mechanical alloying induced combustion when evaporation of zinc is taken into consideration. The amorphous structure of the produced alumina, which consumes excess enthalpy released during the reaction, further lowered the theoretical adiabatic temperature. The gradually proceeding redox reaction sustains a relatively low temperature environment, which favors the formation of the amorphous alumina.

**[69] MECHANOCHEMICAL-HYDROTHERMAL PREPARATION OF CRYSTALLINE HYDROXYAPATITE POWDERS AT ROOM TEMPERATURE**

Shuk P. Suchanek WL. Hao T. Gulliver E. Riman RE. Senna M. TenHuisen KS. Janas VF. - Journal of Materials Research. 16(5):1231-1234, 2001

Crystalline hydroxyapatite (HAP) powders were prepared at room temperature from heterogeneous reaction between Ca(OH)<sub>2</sub> powders and (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> solutions via the mechanochemical-hydro thermal route. X-ray diffraction, infrared spectroscopy, thermogravimetric characterization, and chemical analysis were performed, and it was determined that the room temperature products were phase-pure, thermally stable HAP with a nearly stoichiometric composition. Dynamic light scattering revealed that the dispersed particle size distribution of the room temperature HAP powders was in the range of 0.15-3.0 μm with a specific surface area of approximate to 90 m<sup>2</sup>/g. Both specific surface area measurements and scanning electron microscopy confirmed that the HAP powders consisted of agglomerates containing hundreds of approximate to 20 nm HAP crystals

**[68] MECHANOCHEMISTRY OF THE TITANIUM-SILICON SYSTEM: COMPOSITIONAL EFFECTS**

Doppiu S. Monagheddu M. Cocco G. Maglia F. Anselmi-Tamburini U. Munir ZA. - Journal of Materials Research. 16(5):1266-1279, 2001

The mechanochemical behavior of the Ti-Si system was investigated across the whole composition range at a constant milling intensity. At low Si content the mechanical alloying process leads progressively to an amorphous structure. In the central range of the equilibrium diagram, crystalline intermetallic compounds form with a combustion-like behavior. A nanostructured composite of TiSi<sub>2</sub> gradually evolves above the Ti<sub>25</sub>Si<sub>75</sub> stoichiometry. Transformation behaviors relate to the thermodynamic and thermochemical properties of the tested mixtures as well as with their mechanical features and structural refinement, which change drastically within the explored composition range

**[67] SYNTHESIS OF DENSE NANOMETRIC MoSi<sub>2</sub> THROUGH MECHANICAL AND FIELD ACTIVATION**

Orru R. Woolman J. Cao G. Munir ZA. - Journal of Materials Research. 16(5):1439-1448, 2001

The effect of mechanical and field activation on the synthesis of dense nanometric MoSi<sub>2</sub> was investigated. Powders of Mo and Si, milled separately or comilled in a planetary ball mill, were reacted in a spark plasma synthesis (SPS) apparatus under different electric current conditions. Milled powders reacted faster and required less current than unmilled powders. Mixtures of powders which were milled separately (to nanometric size) reacted in the SPS to produce micrometric alpha -MoSi<sub>2</sub>. Similar results were obtained for samples comilled to produce nanometric reactants which did not contain detectable amounts of the product phase. When products form during milling, they contain both the alpha and beta modifications of MoSi<sub>2</sub>. The product after the SPS reaction was nanometric MoSi<sub>2</sub> with a crystallite size of 140 nm.

**[66] EFFECT OF MECHANICAL MILLING ON SOLID STATE FORMATION OF BaTiO<sub>3</sub> FROM BaCO<sub>3</sub>-TiO<sub>2</sub> (RUTILE) MIXTURES**

V. Berbenni, A. Marini and G. Bruni - Thermochimica Acta vol. 374(2) (2001) pp.151-158

Barium metatitanate (BaTiO<sub>3</sub>) is widely used because of its high dielectric constant, ferroelectric properties and positive temperature coefficient of electrical resistivity. The present work reports the results obtained in the set up of a preparation method of BaTiO<sub>3</sub> by milling and annealing mixtures of BaCO<sub>3</sub> and TiO<sub>2</sub> (rutile). High energy milling of a BaCO<sub>3</sub> - TiO<sub>2</sub> (rutile) equimolecular mixture resulted in a noticeable temperature drop of the temperature where the reaction can occur. The formation of single phase barium metatitanate (BaTiO<sub>3</sub>) was confirmed by Thermogravimetric Analysis (TGA) [both alone and coupled with Fourier Transform Infrared Spectroscopy (FT-IR) evolved gas analysis], by Differential Scanning Calorimetry (DSC) and X-ray powder diffractometry (XRPD). The formation of phases like Ba<sub>2</sub>TiO<sub>4</sub> (orthotitanate), which are known to be detrimental for BaTiO<sub>3</sub> end performances, has only been revealed as an intermediate minor phase that disappears by annealing at temperatures as low as 750°C. At such temperatures the crystal size of the product is 400 Å. BaTiO<sub>3</sub> formation occurs either by a slow heating (2 K/min) of the milled mixture or by a rapid heating followed by an isothermal annealing of 12h.

**[65] HIGH-TEMPERATURE OXIDATION OF TWO-PHASE NANOCRYSTALLINE Ag-Cr ALLOYS IN 1 ATM O<sub>2</sub>**

Niu Y. Song JX. Gesmundo F. Farne G. - Oxidation of Metals. 55(3-4):291-305, 2001

Two nanocrystalline two-phase Ag-Cr alloys prepared by mechanical alloying and containing approximately 30 and 50 wt.% Cr were oxidized in 1 atm O<sub>2</sub> at 700 and 800 degreesC. Under all conditions, a continuous layer of chromia formed at the



surface of the alloys, in spite of the very low solubility of Cr in Ag. A layer of AgCrO<sub>2</sub> also formed externally to the chromia layer. In the case of the Ag-30 Cr alloy, some Ag particles were also present on the scale, directly in contact with the gas phase. Moreover, Cr particles dissolved in the subsurface region of the alloy, while internal oxidation of Cr, was absent. Ag-Cr alloys prepared by powder metallurgy with coarse grain sizes were able to form an irregular thin chromia layer only at a Cr content of 69 wt.%, while an alloy containing 35 wt.% Cr corroded much more rapidly than the nanocrystalline Ag-30Cr alloy. This difference in the scaling behavior is attributed to the large reduction in the alloy grain size, which favors the dissolution of the Cr-rich particles in a Cr-depleted silver matrix and thus provides a faster supply of chromium from the alloy to the scale.

**[64] MECHANICAL PROPERTIES AND MICROSTRUCTURES OF AL<sub>2</sub>O<sub>3</sub>-5 VOL.% YAG COMPOSITES**

Wang HZ. Gao L. Shen ZJ. Nygren M. - Journal of the European Ceramic Society. 21(6):779-783, 2001  
Al<sub>2</sub>O<sub>3</sub>-5 vol.% YAG powder mixtures were prepared by three different methods: (i) Calcination of co-precipitated Al- and Y-hydroxides; (ii) precipitation of Al(OH)(3) in a slurry containing nano-sized YAG particles, followed by calcination; (iii) ball milling of Al<sub>2</sub>O<sub>3</sub> and YAG particles. Almost fully dense Al<sub>2</sub>O<sub>3</sub>-5 vol. % YAG compacts were obtained by hot-pressing these powders at a temperature equal to or exceeding 1550 degreesC. Mechanical tests performed at room temperature showed that the bending strength of the Al<sub>2</sub>O<sub>3</sub>-5 vol.% YAG composite prepared from powder (i) was 604 MPa, and the fracture toughness 5.0 MPa m<sup>1/2</sup>, whereas compacts of the other two powders had lower bending strength and fracture toughness values. Microstructure investigations revealed a homogeneous distribution of YAG in the Al<sub>2</sub>O<sub>3</sub> matrix in the compacts prepared from the powders (i) and (ii), although some nano-sized pores were found within the Al<sub>2</sub>O<sub>3</sub> matrix prepared from powder (i).

**[63] DIRECT SYNTHESIS OF HYDROXYAPATITE-SILK FIBROIN NANO-COMPOSITE SOL VIA A MECHANOCHEMICAL ROUTE**

Nemoto R. Nakamura S. Isobe T. Senna M. - Journal of Sol-Gel Science & Technology. 21(1-2):7-12, 2001.  
In the presence of fine silk cocoon (silk fibroin, SF) powder, a low viscosity sol of nano-hydroxyapatite (HAp, Ca-10(PO<sub>4</sub>)(6)(OH)(2))-SF was synthesized by a wet mechanochemical reaction. Nano crystals of HAp are oriented along their c-axis. The secondary structure of SF was changed by milling. A uniform thin gel film was obtained by a simple dip coating on the glass substrate precoated by chitosan.

**[62] FORMATION OF A TIN/TI<sub>5</sub>SI<sub>3</sub> NANO-GRAIN COMPOSITE BY A NON-EQUILIBRIUM PM PROCESS**

Suehiro Y. Ameyama K. - Journal of Materials Processing Technology. 111(1-3):118-121, 2001  
In the present paper, the authors have proposed a non-equilibrium powder metallurgy (PM) process which enables improved formability and the production of a (TiN + Ti<sub>5</sub>Si<sub>3</sub>) composite. The microstructure and high-temperature deformation behavior of Ti-Si<sub>3</sub>N<sub>4</sub> mechanically alloyed (MA) powder compacts were investigated. Powders of the elements Ti and Si<sub>3</sub>N<sub>4</sub> of composition Ti-20 mass%Si<sub>3</sub>N<sub>4</sub> were blended for MA. The MA process for 720 ks resulted in the formation of amorphous and alpha -Ti phases. These phases changed to TiN, Ti<sub>2</sub>N and Ti<sub>5</sub>Si<sub>3</sub> phases after heat treatment at elevated temperatures. A (TiN + Ti<sub>5</sub>Si<sub>3</sub>) ultra-fine microduplex structure was obtained after heat treatment at 1473 K for 3.6 ks. The amorphous and alpha -Ti phases were stable even after vacuum hot pressed (VHP) at 803 K, while they disappeared and alpha -Ti, Ti<sub>2</sub>N and Ti<sub>5</sub>Si<sub>3</sub> phases appeared in 813 and 823 K VHP specimens. Compression tests revealed that the 803 K VHP specimen with the non-equilibrium phases had the lowest flow stress at 993 K and an initial strain rate of 4.2 x 10<sup>(-4)</sup> s<sup>(-1)</sup> in these three VHP specimens. The slower strain rate test produced a larger amount of harder phases such as TiN, Ti<sub>2</sub>N and Ti<sub>5</sub>Si<sub>3</sub>. Therefore, there exists an appropriate condition for a low temperature and high strain rate forming process. A (TiN + Ti<sub>5</sub>Si<sub>3</sub>) microduplex structure was also obtained in the specimen compressed to 25% after annealing at 1473 K for 3.6 ks.

**[61] SHAPES AND SIZES OF NANOSCALE PB INCLUSIONS IN AL**

Johnson E. Johansen A. Dahmen U. Chen S. Fujii T. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):187-193, 2001

Al-Pb alloys are monotectic and characterized by a large miscibility gap in the liquid phase area and extremely limited mutual solubility in the solid phase. Due to the extent of the miscibility gap the alloys are difficult to make in conventional processing. However, alloys with relatively homogeneous microstructures of fine Pb inclusions in an Al matrix can be made by metastable processing such as rapid solidification, ion implantation, ball milling and physical vapor deposition. The first two techniques have been employed to make alloys of Al with 0.5-3 at.% Pb. The alloys contain fine dispersions of nanoscale Pb : implantation and from about 10-500 nm after rapid solidification. Inclusions inclusions with sizes in the range from 1 to about 20 nm after ion embedded in the Al matrix are single crystalline, and they grow in parallel cube alignment with the matrix. They have cuboctahedral shape with atomically smooth {111} and {100} facets determined from a minimization of the interface energy. Using high resolution TEM, two types of deviations from the classical Wulff construction which alter the shape of the inclusions, have been studied. The smallest inclusions, less than about 20 nm in size, adopt a series of magic sizes that can be related to the occurrence of periodic minima in the residual strain energy. Likewise, in this size range, the energy contribution from the cuboctahedral edges becomes non-negligible leading to an increase in the aspect ratio of the inclusions with decreasing size. Inclusions located in grain boundaries in general adopt a single crystal morphology where one part is faceted and grows in parallel cube alignment with the matrix grain, while the other part has a shape approximating a spherical cap. In special cases such as twin boundaries and {111} twist boundaries, the inclusions are bicrystalline where each part is aligned with the respective grain and the two parts are separated by a boundary similar to that of the matrix. These shapes can be explained using the Cahn-Hoffman xi -vector construction, which generalizes the Wulff construction to determine equilibrium shapes at anisotropic interfaces and their junctions

**[60] AMORPHIZATION AND MICROSTRUCTURAL EVOLUTION IN MULTICOMPONENT (FeCO<sub>N</sub>)<sub>(70)</sub>Zr<sub>10</sub>B<sub>20</sub> ALLOY SYSTEM BY MECHANICAL ALLOYING**

Liu YJ. Chang ITH. Bowen P. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):389-393, 2001

High energy ball milling of a mixture of elemental and prealloyed powder for 18-27 h has led to an amorphous structure in the multicomponent (Fe<sub>1-x-y</sub>Co<sub>y</sub>Ni<sub>x</sub>)(70)Zr<sub>10</sub>B<sub>20</sub> (x = 0.1-0.4, y = 0, 0.1, 0.3) alloy system. Initially, the starting powder material with mixed crystalline phases transformed into an almost single nanocrystalline supersaturated alpha -Fe phase after milling for 6 h. Subsequently, the milled powders became less crystalline and more amorphous with further increase in



milling time. Co-free  $(\text{Fe}_{1-x}\text{Ni}_x)_{70}\text{Zr}_{10}\text{B}_{20}$  ( $x = 0.1, 0.2, 0.3$ ) alloys and the Co-containing  $(\text{Fe}_{0.6}\text{Co}_{0.1}\text{Ni}_{0.3})_{70}\text{Zr}_{10}\text{B}_{20}$  alloy tend to readily transform to amorphous structure after milling for 18 h. But Co-free  $(\text{Fe}_{1-x}\text{Ni}_x)_{70}\text{Zr}_{10}\text{B}_{20}$  ( $x = 0.3$ ) alloy produced a mixture of nanocrystalline boron-rich phase and amorphous phase after milling for 18 h. However, Co-containing  $(\text{Fe}_{0.6}\text{Co}_{0.3}\text{Ni}_{0.1})_{70}\text{Zr}_{10}\text{B}_{20}$  and  $(\text{Fe}_{0.8}\text{Co}_{0.1}\text{Ni}_{0.1})_{70}\text{Zr}_{10}\text{B}_{20}$  alloys with a low Ni/Co ratio failed to achieve amorphization even after 27 h. For all milled amorphous alloy powders, the glass transition temperatures, the onset crystallization temperatures lie in 544-577 and 579-619 K range, respectively. The supercooled liquid regions varied from 35 to 42 K, and the crystallization enthalpies varied from 3.2 to 6.9 kJ mol<sup>-1</sup> with a change in Ni/Fe ratio.

#### [59] METASTABLE PHASE FORMATION IN FE-SI SYSTEM USING NON-EQUILIBRIUM TECHNIQUES

Gupta A. Dhuri P. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):394-398, 2001

Metastable alloy phase formation in Fe/Si system with nominal composition Fe<sub>30</sub>Si<sub>50</sub> using the techniques of mechanical alloying (MA), solid state interdiffusion (SSI) and swift heavy ion (SHI) irradiation has been compared. The products of SSI depend upon the structure of the iron layers. In the multilayer with amorphous iron layer, the product of SSI is crystalline FeSi phase, while in the multilayer with crystalline iron layer the product is an amorphous phase. It results in the formation of metastable BCC phase with extended solid solubility. Results of both MA and SSI can be understood in terms of a model in which an asymmetry in the diffusivities of the two constituents is a necessary condition for amorphisation to occur during solid state reaction. Amorphisation during SHI irradiation can be understood in terms of a thermal spike model

#### [58] NANO-SCALED MULTI-LAYERED BULK MATERIALS MANUFACTURED BY REPEATED PRESSING AND ROLLING IN THE CU-FE SYSTEM

Shingu PH. Ishihara KN. Otsuki A. Daigo I. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):399-402, 2001

The repeated application of mechanical deformation of metallic materials has been proved to be an effective technique for producing bulk nano-scaled regulated structures. These materials have been shown to have unique properties characteristic for nano-materials. This paper reports the experimental results on the mechanical strength, magneto-resistivity, and thermoelectricity obtained by repeated pressing and rolling of alternately stacked thin metallic foils in the Cu-Fe system. In the samples which have the layer thickness greater than 35 nm the Hall-Petch relation is primarily obeyed. For the samples with the layer thickness less than 35 nm, the strength and hardness deviate from this relation. Large magneto-resistivity change (GMR), have been confirmed and a noted change in thermo-electricity (EMF) dependent on the layer thickness, as observed for the Ag-Fe system, have also been confirmed.

#### [57] MECHANICAL SOLID-STATE FORMATION OF Y(1-X)CE(X)AL<sub>3</sub> AND ITS APPLICATION AS AN X-RAY SCINTILLATOR

Sakurai K. Guo XM. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):403-407, 2001

Mechanical solid-state reaction with a high-energy ball mill can be used not only for metallurgical reactions, but also for preparing complex oxides. The present paper describes its successful application to the synthesis of cerium-doped yttrium aluminum perovskite, Y<sub>1-x</sub>Ce<sub>x</sub>AlO<sub>3</sub>, which exhibits a promising optical response to X-ray irradiation. The technique uses crystallization from a strongly disordered non-equilibrium phase prepared by ball milling. It has been found that the combination of yttria and aluminum hydroxide powders as the starting materials is satisfactory in avoiding contamination from balls and vials. Mixing with ceria powder and then heating to at most 1150 degreesC enables the transition to the crystalline phase and the substitution of cerium to the yttrium site simultaneously. The synthesis of a similar scintillating material Y<sub>3-y</sub>Ce<sub>y</sub>Al<sub>5</sub>O<sub>12</sub> is reported as well

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#### [56] ORDER-DISORDER STUDIES AND MAGNETIC PROPERTIES OF MECHANICALLY ALLOYED NANOCRYSTALLINE Ni<sub>3</sub>Fe ALLY

Chinnasamy CN. Narayanasamy A. Ponpandian N. Chattopadhyay K. Saravanakumar M. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):408-412, 2001

Ni<sub>3</sub>Fe alloy with different grain sizes has been formed by mechanical alloying technique using a high energy ball mill. Fe-57 Mossbauer studies have been carried out at room temperature and hyperfine field distributions have been obtained for various milling durations. The influence of surface atoms on magnetic hyperfine fields and the effect of grain size on Fe magnetic moment have been investigated. The atomic ordering is found to be faster in the nanocrystalline form of Ni<sub>3</sub>Fe than in the bulk Ni<sub>3</sub>Fe

#### [55] APPLICATION OF EFFECTIVE POTENTIAL FORMALISM TO MECHANICAL ALLOYING IN AG-CU AND CU-FE SYSTEMS

Ravishankar N. Abinandanan TA. Chattopadhyay K. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):413-417, 2001

We have used the competing dynamics model and the effective potential formalism for studying phase stability during mechanical milling of Ag-Cu and Cu-Fe systems. We first present a method for extracting an effective potential for systems in which the atomic mobility may depend on alloy composition. Using an estimate for the typical ballistic diffusivity, we apply this formalism to calculate the dynamical phase diagram for the Ag-Cu and Cu-Fe binary systems under mechanical milling. For the Ag-Cu system, the dynamical phase diagram exhibits a miscibility gap that closes in on itself at low temperatures, due to the exponentially decreasing thermal diffusivity (and hence, increasing influence of ballistic diffusivity) with decreasing temperature. In the Cu-Fe system, in which the effective potential for both the bcc and fcc phases need to be taken into account, the fcc phase is shown to be stabilized at low temperatures for all the alloys, except the almost pure iron (which is stabilized in its bcc form). These results are critically compared with available experimental results

#### [54] OXIDATION AND REDUCTION IN COPPER/ZINC OXIDES BY MECHANICAL MILLING

Castricum HL. Bakker H. Poels EK. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):418-423, 2001



When zinc oxide is milled under vacuum or in the presence of oxygen, creation of various types of defects results in an increased amorphous fraction, as well as higher surface area. Mechanochemical reactions occur when copper and copper oxides are milled together with zinc oxide: oxidation of copper and copper oxides take place in the presence of oxygen, whereas reduction takes place under vacuum. These reactions are promoted by the presence of ZnO. Formation of a Cu<sub>2</sub>O-like intermediate is suggested, which is not observed when milled without ZnO. The various resulting copper species reduce at different temperatures in H<sub>2</sub> atmosphere. Both Cu-O specific surfaces and BET surfaces are substantially increased for all milled Cu/ZnO samples, making this method an interesting alternative for the preparation of promoted heterogeneous catalysts.

**[53] A METASTABLE ALLOTROPIC TRANSFORMATION IN Nb INDUCED BY PLANETARY BALL MILLING**

Chattopadhyay PP. Pabi SK. Manna I. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):424-428, 2001

The present investigation concerns planetary ball milling of elemental Nb in hardened steel and WC-coated media. Continued milling for about 20 h reduces the grain size below 10 nm with a concomitant lattice expansion. The final product obtained after 30-40 h milling is a pure f.c.c. phase. A detailed micro structural/compositional characterization by X-ray diffraction, resistivity measurements and wet chemical analysis proves that the said f.c.c. phase is neither NbN nor NbC. Instead, it is suggested that elemental Nb undergoes an irreversible b.c.c. → f.c.c. allotropic transformation when the grain size reduces to below 10 nm during high energy ball milling. Furthermore, this f.c.c. phase possibly undergoes an impurity stabilized ordering transformation on annealing at 900 degrees C for 2 h. Finally, the role of impurity in causing this allotropic change is examined

**[52] PREPARATION OF SOME METAL PHOSPHIDES BY BALL MILLING**

Takacs L. Mandal SK. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):429-433, 2001

Mechanochemical reactions were investigated in the Ni-P and Al-P systems. At the Ni<sub>80</sub>P<sub>20</sub> composition, the product is either an amorphous alloy or a mixture of crystalline phosphides, depending on the milling intensity. The reaction turns into a self-sustaining process between 25 and 40 at.% phosphorus. Surprisingly the much more exothermic formation of AlP takes place as a gradual process

**[51] REACTION BALL MILLING OF SYSTEMS INVOLVING IONIC BONDS**

Varghese V. Sharma A. Chattopadhyay K. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):434-437, 2001

The paper reports an attempt to understand the mechanism of mechanochemical synthesis by mechanical milling. Towards this goal, a systematic investigation of the mechanochemistry of the ionic compounds has been carried out. We have studied the nature of replacement reactions in solid CuSO<sub>4</sub>. 5H<sub>2</sub>O with Fe, Mg and Sn. The study focuses on structural characterization at different stages of milling to gain insight into the process of synthesis leading to the formation of nanocrystalline copper.

**[50] SYNTHESIS OF NANOCRYSTALLINE COPPER-TUNGSTEN ALLOYS BY MECHANICAL ALLOYING**

Raghu T. Sundaresan R. Ramakrishnan P. Mohan TRR. Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):438-441, 2001

Copper-tungsten exhibits total absence of solubility in both solid and liquid state. Mechanical alloying (MA) as a solid state, nonequilibrium process can be beneficial to the processing of such an immiscible system with the added features of refinement of structure. A study was undertaken to synthesise various Cu-W alloys and develop an ultrafine microcomposite structure of tungsten in copper matrix by mechanical alloying. Elemental powders of copper and tungsten were milled in high energy ball mills. The milling behaviour was found to depend on the composition, milling time and milling atmosphere. The milled powders were characterised for their particle size, microstructure and lattice parameters. Metastable mutual solid solubility in the system was confirmed. Crystallite sizes were found to be in the nanocrystalline regime. The conversion of milling energy effectively to generate deformed surfaces, which in turn led to metastable solid solubility and nanocrystalline structure, was aided by the presence of oxygen in the milling atmosphere.

**[49] MECHANICAL ALLOYING OF Fe-B POWDERS**

Gupta R. Gupta A.- Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):442-445, 2001

The nature of the solid-state amorphization induced by mechanical alloying has been studied. The XRD and Mossbauer spectroscopy at room temperature of samples mechanically alloyed for different times indicate that the mechanical alloying induces the amorphization reaction in the compositions Fe<sub>60</sub>B<sub>40</sub> and Fe<sub>70</sub>B<sub>30</sub>. A detailed study of transformation kinetics suggests that amorphization occurs via one-dimensional diffusion-controlled growth

**[48] BINARY AND TERNARY AMORPHOUS PALLADIUM ALLOYS - CHARACTERIZATION, ACTIVITY AND SELECTIVITY IN HYDROGENATION OF DIENES**

Varga M. Mulas G. Cocco G. Molnar A. Lovas A. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):462-466, 2001

Pd<sub>25</sub>Zr<sub>75</sub>, Pd<sub>22</sub>Cu<sub>10</sub>Zr<sub>68</sub>, and Pd<sub>72</sub>Cu<sub>10</sub>Si<sub>18</sub> alloy ribbons prepared by the rapid quenching technique, and Pd<sub>25</sub>Zr<sub>75</sub>, Pd<sub>25</sub>Cu<sub>10</sub>Zr<sub>65</sub>, Pd<sub>30</sub>Si<sub>20</sub> and Pd<sub>72</sub>Cu<sub>10</sub>Si<sub>18</sub> alloy powders made by mechanical alloying proved to be amorphous by X-ray diffraction and differential scanning calorimetry. The powder samples after HF treatment have been found to exhibit high selectivity in the semihydrogenation of 1,3-cyclooctadiene: the pronounced drop in the hydrogen uptake after the complete consumption of the diene allows the selective synthesis of the corresponding monoene

**[47] EXTRUSION BEHAVIOR OF Al COMPOSITE POWDER MANUFACTURED BY A STONE MILL TYPE CRUSHER**

Son HT. Kim TS. Lee JH. Maeng DY. Hong SJ. Won CW. Cho SS. Chun BS. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):559-563, 2001

The aim of the present investigation is to predict surface cracking and reinforcement distribution during hot extrusion in Al 6061 and 5083 composite powder reinforced by hybrid TiC-Al<sub>2</sub>O<sub>3</sub> particles. The composite powders were manufactured by crushing in the newly developed stone mill crusher using twin rolled flakes. With increasing initial billet temperature,



surface cracking occurred during extrusion due to a decrease in damage criterion. It was enlightened to obtain an optimal distribution of ceramic particles in;U alloy matrix as a function of milling cycles. Optimal distribution of the reinforcement in the matrix was taken into consolidation with milling cycles

**[46] CHARACTERISTICS OF CERAMIC PARTICLE REINFORCED AL COMPOSITE POWDER MANUFACTURED BY A STONE MILL TYPE CRUSHER USING TWIN ROLL CAST AL ALLOY FLAKE: A MODEL ON THE FORMATION OF COMPOSITE POWDER**

Lee JH. Kim TS. Maeng DY. Son HT. Hong SJ. Won CW. Cho SS. Chun BS. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):632-636, 2001

5083 and 6061 aluminium composite powders have been manufactured by a newly designed stone mill type crusher using twin rolled 5083 and 6061 flakes and ceramic powders such as Al<sub>2</sub>O<sub>3</sub>-TiC and Al<sub>2</sub>O<sub>3</sub>-B<sub>4</sub>C synthesized by self-propagating high-temperature synthesis (SHS) process. This research reports on the effect of such processing parameters as a gap clearance between the disks and the number of on the formation of aluminium particle and distribution of the ceramic particles, respectively, The variation of the particle shape as a function of the gap clearance was simulated with commercial finite element method (FEM) package, Ceramic particles show a uniform distribution in the Al alloy powder at 0.3 mm of gap clearance and four cycles of milling. This economic composite-powder preparation method involving rapid solidification process may have a potential for producing good composite materials.

**[45] STRUCTURAL EVOLUTION DURING HEAT TREATMENT OF MECHANICALLY ALLOYED AL-CU-FE-(SI) ALLOYS**

Kim KB. Kim SH. Kim WT. Kim DH. Hong KT. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):822-829, 2001

Structural evolution during mechanical alloying and subsequent heat treatment of Al<sub>65</sub>Cu<sub>20</sub>Fe<sub>15</sub> and Al<sub>65</sub>Cu<sub>20</sub>Fe<sub>10</sub>Si<sub>5</sub> was studied by X-ray diffractometry (XRD), transmission electron microscopy (TEM) and differential scanning calorimetry (DSC). The Al<sub>65</sub>Cu<sub>20</sub>Fe<sub>15</sub> and Al<sub>65</sub>Cu<sub>20</sub>Fe<sub>10</sub>Si<sub>5</sub> powders milled for 10 h hewed a layered structure consisting of Al-, Cu- and Fe-rich layers. DSC traces obtained from the powder during heating, up to 600 degreesC, showed two exothermic peaks with peak temperatures of 330 and 440 degreesC in Al<sub>65</sub>Cu<sub>20</sub>Fe<sub>15</sub> and 330 and 500 degreesC in Al<sub>65</sub>Cu<sub>20</sub>Fe<sub>10</sub>Si<sub>5</sub> powders. The lower exothermic peaks correspond to the formation of Al<sub>2</sub>Cu and Al<sub>7</sub>Cu<sub>2</sub>Fe phases from the layered structure. The second high temperature exotherms correspond to the formation of Al(Fe,Cu) and 1/1 cubic approximant in the Al<sub>65</sub>Cu<sub>20</sub>Fe<sub>15</sub> powder and to the formation of Al<sub>13</sub>Fe<sub>9</sub> and 1/1 cubic approximant in the Al<sub>65</sub>Cu<sub>20</sub>Fe<sub>10</sub>Si<sub>5</sub> powder. The Al<sub>65</sub>Cu<sub>20</sub>Fe<sub>15</sub> and Al<sub>65</sub>Cu<sub>20</sub>Fe<sub>10</sub>Si<sub>5</sub> powders annealed for 5 h at 750 degreesC showed microstructure consisting of Al(Cu,Fe) and Al<sub>13</sub>Fe<sub>4</sub> phases, and Al<sub>13</sub>Fe<sub>4</sub>, icosahedral and new approximant phases, respectively. The partial substitution of Fe by Si increased the stability of the 1/1 cubic approximant and icosahedral phases

**[44] FORMATION OF NANO-SIZED Ti<sub>68</sub>Fe<sub>26</sub>Si<sub>6</sub> ICOSAHEDRAL QUASICRYSTALLINE PHASE BY RAPID QUENCHING AND MECHANICAL ALLOYING**

Lee JH. Kim KB. Lee JS. Kim DH. Kim WT. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):849-854, 2001

Investigation using rapid quenching (RQ) and mechanical alloying (MA) were conducted to produce Ti<sub>68</sub>Fe<sub>26</sub>Si<sub>6</sub> icosahedral quasicrystals. Subsequent heat treatment was performed on both rapidly quenched ribbons and mechanically alloyed powders in a vacuum furnace under various heat treatment conditions of temperature ranging from 440 to 900 degreesC with a constant heating rate of 10 degreesC/min and holding time up to 30 h. The amorphous phase of Ti<sub>68</sub>Fe<sub>26</sub>Si<sub>6</sub> ribbon was successively achieved by RQ for the first time from the present investigation. During the heat treatment, the icosahedral quasicrystalline phase was transformed from the melt-spun amorphous ribbon at the glass transition temperature of 430 degreesC and remained up to approximately 520 degreesC. The quasicrystalline phase, however was somewhat difficult to be obtained from heat treatment of the Ti<sub>68</sub>Fe<sub>26</sub>Si<sub>6</sub> powder milled for 5 h under the present experimental conditions

**[43] MECHANICAL ALLOYING OF AL-CU-FE ELEMENTAL POWDERS**

Barua P. Murty BS. Srinivas V. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):863-866, 2001

The formation of quasicrystalline powders by mechanical alloying of crystalline elemental powders has been investigated for Al<sub>70-x</sub>(Cu,Fe)<sub>(30+x)</sub> (x = 0, 5, 7) alloy systems. The alloying process during milling and additional annealing at elevated temperature has been characterized by X-ray diffraction and transmission electron microscopy. It is observed that as x increases, tendency of i-phase formation decreases in these mechanically alloyed powder compositions. The cubic solid solution identified as beta -phase is observed to be present as one of the major phases in all the above alloy compositions

**[42] CO-EXISTING MAGNETIC PHASES OF NANO-SCALED GRANULAR CO<sub>20</sub>CU<sub>80</sub> ALLOY SYSTEM**

Yoo YG. Yu SC. Kim WT. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):928-931, 2001

Nano-scaled granular Co<sub>20</sub>Cu<sub>80</sub> alloy was prepared by mechanical alloying technique and the resulting powder was consolidated by hot pressing. Structural evolution and magnetization behavior were examined by using X-ray diffractometry and magnetization measurement by VSM and SQUID magnetometer. XRD results and variation of magnetization with processing time show the formation of a supersaturated solid solution. Magnetization of the alloy could be interpreted as a mixture of ferromagnetic and superparamagnetic components, resulting from the non-uniform size distribution of magnetic clusters in the Cu matrix. With increasing temperature superparamagnetic component becomes the dominant contributor Maximum MR ratio of the hot pressed specimen was about 3.3% at room temperature, under a maximum magnetic field of 10 kOe

**[41] APPEARANCE OF FERROMAGNETISM IN F.C.C. SOLID SOLUTIONS OF BINARY AND TERNARY FE-CU-BASED SYSTEMS PREPARED BY MECHANICAL ALLOYING TECHNIQUE**

Ino H. Hayashi K. Otsuka T. Isobe D. Tokumitsu K. Oda K. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):972-974, 2001

The ferromagnetic f.c.c. solid solutions are formed by mechanical alloying in the Fe-Cu binary systems containing less than 60% of iron. The Curie temperature of the alloy decreases with decreasing iron concentration. The expansion of average



atomic volume of the alloys is about 1.6% of pure copper and 5.3% of f.c.c, gamma -iron regardless of copper content except for dilute iron alloys. It is suggested from the present work that the ferromagnetism in the Fe-Cu alloys originates when the atomic volume is expanded by a certain value (5,3% of gamma -iron is enough), and when a certain number of neighboring iron atoms exist to percolate the ferromagnetic interaction and possibly ti, generate the magnetic moment of iron. In the studies of ternary systems silver atoms hardly dissolved in Fe-Cu solution, whereas gold atoms formed solid solution.

**[40] THE INFLUENCE OF FE<sup>3+</sup> IONS AT TETRAHEDRAL SITES ON THE MAGNETIC PROPERTIES OF NANOCRYSTALLINE ZNFE<sub>2</sub>O<sub>4</sub>**

Chinnasamy CN. Narayanasamy A. Ponpandian N. Chattopadhyay K. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):983-987, 2001

A systematic study on the Variation of Mossbauer hyperfine parameters with grain size in nanocrystalline zinc ferrite is lacking. In the present study, nanocrystalline ZnFe<sub>2</sub>O<sub>4</sub> ferrites with different grain sizes were prepared by ball-milling technique and characterised by X-ray, EDAX, magnetisation and Mossbauer studies. The grain size decreases with increasing milling time and lattice parameter is found to be slightly higher than the bulk value. Magnetisation at room temperature (RT) and at 77 K could not be saturated with a magnetic field of 7 kOe and the observed magnetisation at these temperatures can be explained on the basis of deviation of cation distribution from normal spinel structure. The Mossbauer spectra were recorded at different temperatures between RT and 16 K. The values of quadrupole splitting at RT are higher for the milled samples indicating the disordering of ZnFe<sub>2</sub>O<sub>4</sub> on milling. The strength of the magnetic hyperfine interactions increases with grain size reduction and this can be explained on the basis of the distribution of Fe<sup>3+</sup> ions at both tetrahedral and octahedral sites

**[39] THERMODYNAMIC AND MAGNETIC PROPERTIES OF MULTICOMPONENT (FE, NI)<sub>70</sub>ZR<sub>10</sub>B<sub>20</sub> AMORPHOUS ALLOY POWDERS MADE BY MECHANICAL ALLOYING**

Liu YJ. Chang ITH. Lees MR. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):992-996, 2001

The correlation between the thermodynamic and magnetic properties of mechanically alloyed (MAed) (Fe<sub>1-x</sub>Ni<sub>x</sub>)<sub>70</sub>Zr<sub>10</sub>B<sub>20</sub> (x = 0.1-0.4) amorphous alloy powders was investigated. The activation energy (E<sub>a</sub>) for primary crystallization of the b.c.c. alpha -Fe: phase decreased significantly: from 356.7 to 139.7 kJ/mol with increasing Ni/Fe ratio from 0.11 to 0.43 and then increased to 304 kJ/mol for the alloy with a Ni/Fe ratio of 0.67. The Curie temperatures of the (Fe<sub>1-x</sub>Ni<sub>x</sub>)<sub>70</sub>Zr<sub>10</sub>B<sub>20</sub> (x = 0.1-0.4) alloy powders milled at 18h were found to be in the range 425-625 K. The room temperature coercivity of the as-milled amorphous powders was in the range 37-44 G. However, they were dramatically reduced to values in the range 11-24 G after annealing at temperatures from 250 to 575 K. The minimum coercivities obtained after in situ annealing of amorphous powder were 8 G for (Fe<sub>0.6</sub>Ni<sub>0.4</sub>)<sub>70</sub>Zr<sub>10</sub>B<sub>20</sub> and 10 G for (Fe<sub>0.8</sub>Ni<sub>0.2</sub>)<sub>70</sub>Zr<sub>10</sub>B<sub>20</sub>, respectively.

**[38] MAGNETIC PROPERTIES OF AMORPHOUS CO<sub>2</sub>-XFEXGE ALLOYS SYNTHESIZED BY MECHANICAL ALLOYING**

Bhowmik RN. Ranganathan R. Sarkar S. Bansal C. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):1014-1018, 2001

Amorphous Co<sub>2-x</sub>Fe<sub>x</sub>Ge (0.0 less than or equal to x less than or equal to 0.30) alloys were synthesized by direct mechanical alloying of the elemental powders in a SPEX 8000 miser mill, and their micro-structure and magnetic properties were investigated using X-ray diffraction (XRD), Mossbauer spectroscopy and de magnetization measurements. The as-milled alloys showed a maximum in magnetization at substantially high temperatures of about 200-240 K and field dependent irreversibility effects, which could be attributed to a superparamagnetic blocking of uncoupled magnetic clusters in a non-magnetic matrix. When the samples were subjected to a heat treatment at 440 degreesC for 1 h, the maxima shifted to very low temperatures in the region of 40-45 K. A broadening of the transition as well as shift to lower temperature was observed with increase in the field. This behavior was consistent with a spin glass-like freezing taking place at this temperature, and in agreement with the observations of Zhou and Bakker who synthesized amorphous Co<sub>2</sub>Ge by mechanical milling of crystalline Co<sub>2</sub>Ge for long durations of about 240 h. There is also a systematic increase in the spin glass freezing temperature with Fe concentration. Our results imply that the as-milled alloys are superparamagnetic, the heat treatment carried out on the as-milled samples is resulting in homogenous amorphous alloys, and the magnetic behavior is changing from superparamagnetic to spin glass

**[37] CU-MG POWDERS AND RIBBONS - CHARACTERIZATION AND CATALYTIC TESTS REACTIONS**

Molnar A. Varga M. Mulas G. Mohai M. Bertoti I. Lovas A. Cocco G. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 304(Special Issue SI):1078-1082, 2001

Cu<sub>15</sub>Mg<sub>85</sub> and Cu<sub>3</sub>Mg<sub>97</sub> alloy powders, prepared by mechanical alloying of the constituent elements and Cu<sub>78</sub>Mg<sub>22</sub> and Cu<sub>14.5</sub>Mg<sub>85.5</sub> ribbons made by rapid quenching were characterized by physical methods (DSC, XRD, and XPS). Carbon and oxygen impurities and Mg (mainly as oxide) were detected by XPS of the as-received samples, and only the Cu<sub>78</sub>Mg<sub>22</sub> ribbon was shown to have copper on the surface. The powders, in turn, are the only samples possessing Cu<sub>0</sub> measured by the N<sub>2</sub>O titration method. The catalytic properties of the specimens were tested in the dehydrogenation of 2-propanol and in the one-step synthesis of methyl isobutyl ketene (MIBK) from acetone. The Cu<sub>15</sub>Mg<sub>85</sub> powder, is the only alloy exhibiting satisfactory catalytic performance: MIBK could be isolated in a yield of 35%. The data indicate that copper species determined by N<sub>2</sub>O titration are involved in the catalytic reactions.

**[36] FORMATION OF SOLID SOLUTION OF CARBON IN BCC IRON BY COLD DEFORMATION**

Shabashov VA. Mukoseev AG. Sagaradze VV. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 307(1-2):91-97, 2001

The solid solution of carbon in BCC Fe was studied by the Mossbauer method. Samples were synthesized from a powder mixture of armco-iron and thermal black by shear under compression between Bridgman anvils at room temperature. In addition to the formation of a solid solution with carbon concentration at iron interstices of approximate to 22 at.%, samples deformed under compression to a shear of epsilon = 6.7 contained a nonequilibrium nanosized mechanical mixture of carbon and iron. This mixture formed approximate to 50% cementite at 870 K (1 h). The structural component (sab-spectrum) B



was resolved in spectra of synthesized samples of iron with carbon. It is possibly connected with the lattice dilatation and localization of interatomic bonds near grain boundaries and interstitial carbon atoms.

**[35] STRUCTURAL ANALYSIS OF MODIFIED HYDROXYAPATITE POWDERS**

Fanovich MA. Castro MS. Lopez JMP. - Materials Research Bulletin. 36(3-4):487-496, 2001

In this work, the presence of structural defects in modified hydroxyapatite (HA) samples were studied. The behavior of HA samples with different chemical compositions was compared. Structural alterations were produced by thermal treatments and high-energy milling. Electron paramagnetic resonance spectroscopy (EPR) was used in order to identify the presence of structural defects. Samples were analyzed at room temperature and the signals observed by electron paramagnetic resonance at  $g = 1.999$  were assigned to oxygen vacancies in the apatitic structure. The determination of vacancy concentration ( $V_{\text{o}}(\cdot)$ ) in each sample was related with the characteristic parameters of the signal and the experimental conditions. XRD and SEM were used as additional characterization techniques. The production and concentration of these vacancies was correlated to the modifications introduced during the processing of the materials. The results were also related with the chemical composition, particle size and crystallinity of the samples.

**[34] FABRICATION OF TUBULAR ELECTROLYTES FOR SOLID OXIDE FUEL CELLS USING STRONTIUM- AND MAGNESIUM-DOPED LAGAO<sub>3</sub> MATERIALS**

Du YH. Sammes NM. - Journal of the European Ceramic Society. 21(6):727-735,

Long, straight, dense, and even-shaped tubular electrolytes (200-300 mm in length, 2.4-2.5 mm inside diameter and 0.3-0.4 mm wall thickness) were successfully fabricated from strontium- and magnesium-doped  $\text{ZrGaO}_3$  materials by way of extrusion. An economic and practical process was developed to extrude the small tubes using water-based and organic-based additives and optimized process parameters. Particle size distribution and specific surface area of the synthesized powder were modified by calcination and ball milling. Obtaining workable pastes played an important role in achieving smooth, linear, even and dense green tubes. The final products showed a dense microstructure and improved mechanical strength over pressing routes. Modulus of rupture of the extruded materials was found to be  $180 \pm 16$  MPa at room temperature and  $113 \pm 11$  MPa at 800 degreesC.

**[33] MECHANICAL PROPERTIES AND MICROSTRUCTURES OF AL<sub>2</sub>O<sub>3</sub>-5 VOL.% YAG COMPOSITES**

Wang HZ. Gao L. Shen ZJ. Nygren M. - Journal of the European Ceramic Society. 21(6):779-783, 2001

$\text{Al}_2\text{O}_3$ -5 vol.% YAG powder mixtures were prepared by three different methods: (i) Calcination of co-precipitated Al- and Y-hydroxides; (ii) precipitation of  $\text{Al}(\text{OH})_3$  in a slurry containing nano-sized YAG particles, followed by calcination; (iii) ball milling of  $\text{Al}_2\text{O}_3$  and YAG particles. Almost fully dense  $\text{Al}_2\text{O}_3$ -5 vol. % YAG compacts were obtained by hot-pressing these powders at a temperature equal to or exceeding 1550 degreesC. Mechanical tests performed at room temperature showed that the bending strength of the  $\text{Al}_2\text{O}_3$ -5 vol.% YAG composite prepared from powder (i) was 604 MPa, and the fracture toughness 5.0 MPa  $\text{m}^{1/2}$ , whereas compacts of the other two powders had lower bending strength and fracture toughness values. Microstructure investigations revealed a homogeneous distribution of YAG in the  $\text{Al}_2\text{O}_3$  matrix in the compacts prepared from the powders (i) and (ii), although some nano-sized pores were found within the  $\text{Al}_2\text{O}_3$  matrix prepared from powder (i).

**[32] FLAKE CU-SN ALLOYS AS NEGATIVE ELECTRODE MATERIALS FOR RECHARGEABLE LITHIUM BATTERIES**

Xia YY. Sakai T. Fujieda T. Wada M. Yoshinaga H. - Journal of the Electrochemical Society. 148(5):A471-A481, 2001

We have prepared the intermetallic compound  $\text{Cu}_6\text{Sn}_5$  using mechanical-alloying, gas-atomizing, and melt-spinning techniques. The electrochemical performance of the compound is critically dependent on its morphology due to different preparation methods. The  $\text{Cu}_6\text{Sn}_5$  alloy created by mechanical alloying, consisting of  $<1 \mu\text{m}>$  thick flake powder, has the best battery performance of all compounds. It delivers a rechargeable capacity of 200 mAh/g (2000 Ah/L) over 50 cycles when the cycled voltage range is restricted to 0.2-1.5 V. The effect of the mechanical-alloying time and Cu/Sn ratio on its battery performance was further investigated. The presence of excess Cu in alloy, relative to  $\text{Cu}_6\text{Sn}_5$ , showed improved cyclability at the expense of capacity, whereas an excess of Sn resulted in poor cyclability. A lithium-ion cell based on a flaked Cu-Sn microcomposite alloy negative electrode and a 5 V  $\text{LiNi}_x\text{Mn}_{2-x}\text{O}_4$  positive electrode was assembled. The cell showed an average working voltage at 4.0 V and cycled well with a reversible capacity of ca. 200 mAh/g based on the pure Cu-Sn alloy when a cell was cycled between 3.5 and 4.6 V

**[31] EFFECT OF WHISKER ASPECT RATIO ON THE DENSITY AND FRACTURE TOUGHNESS OF SIC WHISKER-REINFORCED Si<sub>3</sub>N<sub>4</sub>**

Sneary PR. Yeh Z. Crimp MJ. - Journal of Materials Science. 36(10):2529-2534, 2001.

Controlling the suspension properties prior to slip casting optimizes the homogeneity, density and fracture toughness of silicon carbide whisker reinforced silicon nitride ( $\text{SiC}_w/\text{Si}_3\text{N}_4$ ). Further improvements in the mechanical properties are realized by combining ball milling with ultrasonic dispersion of the composite suspension. Ball milling reduces the  $\text{SiC}_w$  aspect ratio from 25 to 15 which in turn increases the dispersion of the whiskers within the suspension, resulting in increases in the green and sintered density, along with the fracture toughness. In a binderless process, 20 volume% reduced aspect ratio ( $r = 15$ )  $\text{SiC}_w/\text{Si}_3\text{N}_4$  can be densified to 95% theoretical density by pressureless sintering using 8%  $\text{Y}_2\text{O}_3$  and 2%  $\text{Al}_2\text{O}_3$  by weight as sintering aids. These composites had measured values of fracture toughness from 9-10.5 MPa  $\cdot \text{m}^{1/2}$ , representing an average increase of approximately 30% over the fracture toughness for monolithic  $\text{Si}_3\text{N}_4$  processed under identical conditions.

**[30] STRUCTURE, MAGNETIC PROPERTIES, AND COERCIVITY MECHANISM OF NANOCOMPOSITE SMC<sub>05</sub>/ALPHA-Fe MAGNETS PREPARED BY MECHANICAL MILLING**

Zhang J. Zhang SY. Zhang HW. Shen BG. - Journal of Applied Physics. 89(10):5601-5605, 2001

Nanocomposite  $\text{SmCo}_{5+x}$  wt %alpha -Fe ( $x=0, 20, 30, \text{ and } 35$ ) powders were prepared by mechanical milling and subsequent annealing. X-ray analyses show that hard phases can be 1:7, 1:5, or 2:7 phase with the increase of alpha -Fe content in as-milled powders annealed at 550 degreesC for 30 min. The high remanence and maximum energy product (BH)(max) were obtained by this method. The single-phase behavior of some powders was discussed according to the exchange spring model of Kneller and Hawig. For as-milled  $\text{SmCo}_{5+x}$  wt %alpha -Fe ( $x=0, 20, 30, \text{ and } 35$ ) powders annealed at 550 degreesC for 30 min, analyses of their initial magnetization curves and the coercivities of minor hysteresis loops in dependence on the applied field reveal that coercivities of these powders are all controlled mainly by domain wall



pinning. The addition of alpha -Fe can not change the coercivity mechanism of powders, although it affects strongly their magnetic properties

**[29] SYNTHESIS OF SILVER POWDER USING A MECHANOCHEMICAL PROCESS**

Keskinen J. Ruuskanen P. Karttunen M. Hannula SP. - Applied Organometallic Chemistry. 15(5):393-395, 2001

Keywords Silver. Mechanical alloying. Mechanochemical processing. Displacement reaction. X-ray diffraction.

Abstract Fine silver powder was synthesized in a mechanochemical process by inducing a solid-state displacement reaction between AgCl and sodium. The process employed was ball milling in a planetary-type ball mill. The reaction products were elemental silver and NaCl in powder form. The silver particles were separated out by washing the NaCl by-product from the milled powder mixture. The milled powders were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The XRD determination showed that the reaction between AgCl and sodium was complete in almost all the experiments carried out. In some cases a minor quantity of Ag<sub>2</sub>Na was formed. SEM and TER I examinations revealed that, depending on the milling parameters employed, the size of the particles in the synthesized metallic silver powder was in the range 50-1000 nm

**[28] ON PREDICTING ROLLER MILLING PERFORMANCE PART I: THE BREAKAGE EQUATION**

Campbell GM. Webb C. - Powder Technology. 115(3):234-242, 2001

The mathematical relationship (breakage equation) between the inlet and outlet particle-size distributions of a roller milling operation is described, and the breakage function linking the two is defined. The forms of the breakage equation and the breakage function are different for roller milling than for other comminution operations, such as hammer milling or ball milling. The breakage equation is discretised to give a matrix form, from which it is demonstrated that during roller milling of wheat, particles break independently of one another. This is an important assumption of breakage equations for many comminution operations and experimental results are presented, which confirm its applicability to the roller milling of wheat grains. Breakage matrices are successfully used to predict the outlet particle-size distributions from First Break milling of wheat. Later papers in this series consider the form of the breakage function

**[27] ON PREDICTING ROLLER MILLING PERFORMANCE PART II. THE BREAKAGE FUNCTION**

Campbell GM. Bunn PJ. Webb C. Hook SCW. - Powder Technology. 115(3):243-255, 2001

Paper I of this series showed that the relationship between the inlet and outlet particle-size distributions in a roller milling operation can be found by integrating, over the range of input particles, the breakage of each individual particle as a function of its physical characteristics. This is possible because particles break independently during roller milling. The pattern of breakage for individual particles is called the breakage function. This paper considers the form of the breakage function and determines it experimentally for roller milling of wheat. The breakage function is shown to depend critically on the ratio of roll gap to input particle size (the milling ratio). For a given ratio, the breakage function for wheat grains is linear with respect to output particle size, over a wide range. This is quite different from the particle-size distribution produced by, e.g., hammer milling. It perhaps explains why roller milling is so suited to milling of wheat to produce flour: the broad and even distribution of particle sizes produced allows effective separation of bran and efficient recovery of white flour. Breakage functions depend on wheat variety and physical characteristics and on the design and operation of the roller mill. Single-kernel testing is becoming widespread in wheat quality testing; distributions of individual kernel parameters, such as size, mass, hardness and moisture content, are measured. The breakage function approach potentially provides a link between single-kernel testing and milling performance.

**[26] EFFECT OF FE<sub>2</sub>O<sub>3</sub> CRYSTALLITE SIZE ON ITS MECHANOCHEMICAL REACTION WITH LA<sub>2</sub>O<sub>3</sub> TO FORM LAFeO<sub>3</sub>**

Zhang QW. Saito F. - Journal of Materials Science. 36(9):2287-2290, 2001.

Fe<sub>2</sub>O<sub>3</sub> powders with different crystallite sizes prepared by heating FeOOH at various temperatures were ground with La<sub>2</sub>O<sub>3</sub> powder using a planetary ball mill to investigate the effect of crystallite size on mechanochemical synthesis of LaFeO<sub>3</sub>. Fe<sub>2</sub>O<sub>3</sub> powder with smaller crystallite size obtained by heating at lower temperature reacts more easily with La<sub>2</sub>O<sub>3</sub> than that with larger size. The mechanochemical reaction proceeds with an increase in grinding time. Specific surface area of the LaFeO<sub>3</sub> powder synthesized has a large value of over 11 m<sup>2</sup>/g. The mechanochemical process can be also applied to synthesize other iron complex oxides with rare earth elements such as Pr, Nd and Sm

**[25] ELEVATED TEMPERATURE DEFORMATION BEHAVIOR OF DISPERSION-STRENGTHENED AL AND AL-LI-MG ALLOYS**

Minay J. Dashwood R. McShane H. - Journal of Materials Engineering & Performance. 10(2):136-142, 2001

A model describing the behavior of dispersion-strengthened aluminum alloys, when subjected to elevated temperature plastic deformation, is presented. The aims are twofold: to use the model for extrapolation of laboratory data to predict behavior under service conditions where the strain rate is extremely low (< 10<sup>-9</sup> s<sup>-1</sup>); and to design and fabricate materials having specific elevated temperature properties based on microstructural predictions from the model. The results of constant strain-rate compression tests covering a range of temperatures from 250 to 550 degreesC and strain rates of 5 x 10<sup>-5</sup> to 10<sup>-1</sup> s<sup>-1</sup> are presented in conjunction with microstructural investigations using transmission electron microscopy (TEM) and x-ray diffraction. Materials mechanically alloyed with (a) no dispersoids, (b) 23 nm radius TiO<sub>2</sub> dispersoids, and (c) 10 nm diameter Al<sub>2</sub>O<sub>3</sub> dispersoids have been studied. The effect of varying the volume fraction of the TiO<sub>2</sub> dispersoids and adding alloying additions of Mg and Li to the matrix Al have been investigated. In addition, the TiO<sub>2</sub> particles are shown to have reacted to form Al<sub>2</sub>Ti. An adaptation to the detachment model of Rosler and Arzt has been proposed to account for the behavior of these types of materials and to enable accurate prediction of deformation behavior at elevated temperatures and low strain rates

**[24] BEHAVIOR OF Mg<sub>2</sub>Ni HYDROGEN-ABSORBING ALLOY IN AN ALKALINE SOLUTION [JAPANESE]**

Nakano H. Kuji T. Aizawa T. - Electrochemistry. 69(4):259-263, 2001

Two specific phenomena were observed during electrochemical charging and discharging processes for Mg<sub>2</sub>Ni alloy electrode at the first cycle, i.e., the decrease of the absorbed hydrogen content (difference between the charged quantity of electricity and the hydrogen gas volume converted to the quantity of electricity) in an alloy during charging process and the discharge capacity larger than the absorbed hydrogen content. In order to investigate these phenomena, the electrochemical behavior of Mg<sub>2</sub>Ni alloy powders prepared by casting and bulk mechanical alloying(BMA) was studied in 6M KOH solution



and water. It was consequently found that  $Mg(OH)_2$  was formed and at the same time atomic hydrogen was also formed in the solution so that the hydrogen was absorbed into  $Mg_2Ni$  alloy. Hydrogen gas was also generated from the alloy surface after hydrogen absorbed was almost saturated in the alloy. Consequently, it was clearly concluded that the specific phenomenon of the former is due to the generation of gaseous hydrogen when  $Mg_2Ni$  alloy reacted with  $H_2O$ . The latter is also because of the discharge of the hydrogen absorbed into  $Mg_2Ni$  by forming  $Mg(OH)_2$ , except for discharge of the hydrogen absorbed electrochemically.

**[23] EFFECT OF EXTENDED GRINDING ON THE DISSOLUTION OF A TA/NB CONCENTRATE**

Welham NJ. - Canadian Metallurgical Quarterly. 40(2):143-154, 2001

A tantalum/niobium concentrate was ball milled for up to 100 hours in a laboratory mill without any evidence of a phase change other than a decrease in unit cell symmetry. After 2 hours of milling, the dry milled powders had passed through their maximum BET surface areas and milling for longer times resulted in a decreased surface area. The surface area of wet milled powders continued to increase with milling time. These differences were attributed to the presence of water hindering, impact induced rewelding of the particles. The extent of dissolution in a mixture of hydrochloric acid and sodium fluoride increased with milling time until over 86% of the concentrate was solubilised in 24 hours. It was found that a powder milled for 50 hours dissolved 4500 times more rapidly than a powder milled for 2 hours. Both strong acid and high fluoride were required for the maximum rate. The dissolution was shown to be two stages: an initial, rapid solution diffusion controlled reaction followed by a slower chemically controlled step. The dissolution associated with the fast step increased with milling time.

**[22] NOVEL PROCESS FOR ENHANCED LUNAR OXYGEN RECOVERY**

Welham NJ. - Journal of Materials Science. 36(9):2343-2348, 2001

The generally accepted method for recovering oxygen on an extraterrestrial body is by thermal reduction of indigenous minerals, the most amenable of which is ilmenite,  $FeTiO_3$ . Thermodynamic modelling shows that carbon is a more effective reductant than hydrogen. In this paper the effect of extended ball milling on the carbothermic reduction of a terrestrial beach sand derived ilmenite is examined. The rate of oxygen recovery into the gas phase is substantially faster for powders milled together and a concomitant lowering of onset temperature is also noted. XRD examination of the final powders indicate that reduction of ilmenite proceeds via elemental iron and rutile which is then further reduced to sub-oxides. The presence of nitrogen, or excess carbon, leads to vastly greater oxygen recovery due to the formation of titanium nitride or carbide with complete release of oxygen from the mineral achieved in 1 h at 1200 degreesC.

**[21] INFLUENCE OF THE MECHANICAL GRINDING ON THE MAGNETIC PROPERTIES OF  $GdMn_2$**

Chevalier B. Bobet JL. Nakhil M. Etourneau J. - Journal of Alloys & Compounds. 320(1):33-39, 2001

The cubic  $C15$  phase  $GdMn_2$  was submitted to high-energy ball milling. The resulting products were investigated by X-ray powder diffraction, scanning electron microscopy, ac-magnetic susceptibility and dc-magnetization measurements. With increasing speed of the milling treatment, amorphization of the sample appears, as shown by X-ray diffraction analysis. Furthermore, this treatment induces a magnetic transition from antiferromagnetism (unmilled sample) to ferromagnetism below  $T_c$  congruent to 10(5) K (milled sample at high speed). This effect is compared with that of the application of hydrostatic pressure on  $GdMn_2$ .

**[20] HYDROGEN STORAGE PROPERTIES OF THE MECHANICALLY ALLOYED  $LaNi_5$ -BASED MATERIALS**

Authors Liang G. Huot J. Schulz R. - Journal of Alloys & Compounds. 320(1):133-139, 2001

Mechanical alloying has been used to synthesize  $LaNi_5$ -based hydrogen storage alloys. Mechanical milling of the La and Ni powder blend results in the direct formation of nanocrystalline  $AB_5$  phase. Hydrogen storage measurements show that this as-milled  $LaNi_5$  compound does not absorb much hydrogen reversibly. Annealing leads to grain growth, release of microstrain, and to an increase of storage capacity. Substitution of La or Ni by a third element can easily be achieved by mechanical alloying. The structure and hydrogen storage properties of these  $LaNi_5$ -based alloys prepared by mechanical alloying and annealing show no big difference with those of melt casting alloys.

**[19] PREPARATION OF PLATELIKE NANO ALPHA ALUMINA PARTICLES**

Wu YQ. Zhang YF. Huang XX. Guo JK. - Ceramics International. 27(3):265-268, 2001.

A novel synthesis process has been developed for producing high purity nonagglomerate nano platelike alpha - $Al_2O_3$  particles. The process mainly utilizes a seed-effect of fine alpha - $Al_2O_3$  grains, worn from the milling mediums and uniformly mixed with the hydrous alumina during grinding, and also utilizes  $ZnF_2$  additive to reduce the transformation temperature and modify the alumina particle shape. The aspect ratio and the average size of  $Al_2O_3$  particles prepared at 900 degreesC for 1 h is 2-4 and 40 nm, respectively.

**[18] SYNTHESIS OF  $Al_2O_3$ -NB COMPOSITE BY REACTIVE MILLING**

EMJA Pallone, DR Leiva, R Tomasi, WJ Botta - ADVANCED POWDER TECHNOLOGY II (Series: KEY ENGINEERING MATERIALS), 2001, Vol 189-1, pp 38-42 - 2ND INTERNATIONAL LATIN-AMERICAN CONFERENCE ON POWER TECHNOLOGY; FOZ DO IGUAÇU, BRAZIL. NOVEMBER, 1999

Synthesis of compounds or composites can be achieved by reactive milling, a process in which a powder mixture reacts due to mechanical activation given by high-energy ball milling. Different types of reaction can occur in reactive milling. Reactions of oxide reduction or displacement by a metal as reducing agent are often of the self-sustaining type. In the present work, we report on the microstructural and phase evolution during reactive milling synthesis of  $Nb+Al_2O_3$  composite from the powder mixtures of  $Nb_2O_5$  and Al. High-energy ball milling was performed in a SPEX 8000 shaker/mill apparatus with a ball/material mass ratio of 4:1. The reaction was monitored by a thermocouple fixed in the external surface of the vial allowing the detection of the temperature peak associated with the sudden reaction. For the studied reaction, the ignition occurs after 68 min of milling. Samples of powder mixtures were prepared by different milling time, to allow its characterisation at different conditions just before, during and after the reaction. The powder transformations during milling, the microstructural evolution and the completeness of the reaction were determined by X-ray diffraction, specific surface area measurements, scanning electron microscopy and differential scanning calorimetry.

**[17] EFFECT OF POWDER MILLING AND DOPANT ADDITION ON THE CURRENT-VOLTAGE CHARACTERISTICS OF  $SrO_2$ -BASED CERAMICS**



W Lacerda, WC Las, M Cilense, JA Varela - ADVANCED POWDER TECHNOLOGY II (Series: KEY ENGINEERING MATERIALS), 2001, Vol 189-1, pp 138-143 - 2ND INTERNATIONAL LATIN-AMERICAN CONFERENCE ON POWER TECHNOLOGY; FOZ DO IGUAÇU, BRAZIL. NOVEMBER, 1999

Non-linear electrical properties of SnO<sub>2</sub>-based ceramics were investigated as a function of powder agglomeration condition and as a function of dopant addition. All doped powders presented a single phase, cassiterite, as evidenced by X-ray diffraction analysis. The effect of milling was quite evident, with non-milled powder showing higher agglomerated particle size than milled powder. Cr addition seemed to increase the non-linear coefficient. Cu and Mn rendered dense ceramics, but alpha values for systems with Mn were higher than for systems with Cu.

**[16] PRODUCTION OF ALUMINA MATRIX NANOCOMPOSITES WITH INCLUSIONS OF TiC AND TiB<sub>2</sub>, VIA REACTIVE MILLING**

ASA Chinelatto, OA Contardi, EMJA Pallone, R Tomasi - ADVANCED POWDER TECHNOLOGY II (Series: KEY ENGINEERING MATERIALS), 2001, Vol 189-1, pp 208-213 - 2ND INTERNATIONAL LATIN-AMERICAN CONFERENCE ON POWER TECHNOLOGY; FOZ DO IGUAÇU, BRAZIL. NOVEMBER, 1999

One of the most recent alternatives on development of toughness in ceramic materials is the addition of nanometric particles of a second phase into a ceramic matrix. These systems are denominated ceramic matrix nanocomposites. In this work we describe the production of alumina matrix nanocomposites with inclusions of TiC and TiB<sub>2</sub> using as precursors, mixtures of nanometric powders prepared by reactive milling. The reactive milling was performed in a high-energy mill SPEX Mixer/Mill 8000, through the reactions:  $4Al + 3C + 3TiO_2$  and  $TiO_2 + B_2O_3 + 10/3Al$ , producing alumina with TiC and TiB<sub>2</sub>, respectively. The reaction products were formed as strong agglomerates of nanometric particles and conventional ball milling performed the deagglomeration. The dispersed powders were characterized by X-ray diffraction and differential scanning calorimetry. The reaction products were mixed with a commercial alumina (Sumitomo AKP-53) to produce final composites with 5-weight % and 10-weight % of inclusions by ball milling in ethyl alcohol. The alcohol suspensions were dried by heating at 70 degreesC. The powder mixtures were pressed and sintered at 1500 degreesC and 1600 degreesC in high-vacuum atmosphere for 1 hour. The sintered samples reached final relative density of 95-97%TD and the high resolution scanning electron microscopy microstructure analysis has shown the dispersion of the inclusions with a few remaining agglomerates from the synthesis process.

**[15] SYNTHESIS OF A SiC-SIALON COMPOSITE BY NITRIDATION OF A SiC-ALSI MIXTURE**

K Makuntuala, JC Bressiani - ADVANCED POWDER TECHNOLOGY II (Series: KEY ENGINEERING MATERIALS), 2001, Vol 189-1, pp 548-553 - 2ND INTERNATIONAL LATIN-AMERICAN CONFERENCE ON POWER TECHNOLOGY; FOZ DO IGUAÇU, BRAZIL. NOVEMBER, 1999

SiC-SiAlON composite was produced from a mixture of SiC powders with wide particle size distribution and Al-34Si alloy powder by nitridation. The aluminum-silicon alloy was produced by the mechanical alloying method. The composition studied was 75 wt% SiC and 25 wt% (Al-34Si). The powder was compacted by uniaxial and cold isostatic pressing, forming tablets 25 mm in diameter and 10 mm thick. These tablets were then nitridized in a graphite resistance furnace. The heating rate up to 1000 degreesC was 20 degreesC/min, and 10 degreesC/min up to 1400 degreesC. Dwelltime at 1400 degreesC was 8 hours. Thermodynamic analysis of the system was accomplished. Powders of raw materials were characterized by sedimentation analysis, chemical analysis, scanning electron microscopy, and X-ray diffraction. The material obtained after nitridation was characterized by scanning electron microscopy, energy dispersive spectroscopy, and X-ray diffraction. The porosity and Final density were determined by gas picnometry.

**[14] PRODUCTION OF AL-FE-NB-SI ALLOYS BY MECHANICAL ALLOYING AND HOT EXTRUSION**

RE Coelho, FA Filho - ADVANCED POWDER TECHNOLOGY II (Series: KEY ENGINEERING MATERIALS), 2001, Vol 189-1, pp 555-560 - 2ND INTERNATIONAL LATIN-AMERICAN CONFERENCE ON POWER TECHNOLOGY; FOZ DO IGUAÇU, BRAZIL. NOVEMBER, 1999

The present work shows the solid state interaction among Al, Fe, Nb and Si elemental powders, mixed by mechanical alloying (MA) and consolidated by hot extrusion. The MA powders and extruded materials were observed by scanning electron microscopy (SEM), energy dispersive spectrometry (EDS) and X-ray diffraction. The tensile tests and Vickers hardness were performed on the as-extruded material, as well as hydrostatic density measurement. Several kinds of dispersoids were detected in this alloying, suggesting that they are the main causes for the enhanced mechanical properties.

**CU-MO ALLOYS OBTAINED BY MECHANICAL ALLOYING**

J Cornejo, V Martinez, S Ordonez - ADVANCED POWDER TECHNOLOGY II (Series: KEY ENGINEERING MATERIALS), 2001, Vol 189-1, pp 561-566 - 2ND INTERNATIONAL LATIN-AMERICAN CONFERENCE ON POWER TECHNOLOGY; FOZ DO IGUAÇU, BRAZIL. NOVEMBER, 1999

In the present work the production and microstructural evolution of Cu based alloys, with amounts of Mo of 3, 5 y 10 wt%, obtained by mechanical alloying (MA) were studied. The MA process was carried out using a Spex 8000 high-energy ball mill during different milling times. The process was followed through X-ray diffraction and scanning electron microscopy. Crystallite sizes of the powder mechanically alloyed were calculated from X-ray diffraction patterns by Scherrer's formula and the results showed an exponential decrease with a minimum value of 12 nm for 50 hours of milling time. The Vickers hardness (HV) of mechanically alloyed powder increased with milling time and the Mo content reaching a value of 634 HV for Cu-20wt% Mo and 50 hours of milling time. The amount of Mo dissolved in the copper solid solution was obtained from the lattice parameter by means of Vegard's law. The Cu-20wt% Mo alloy reached the maximum value for 50 hours (0.36247 nm), for Cu-3wt% Mo and Cu-5wt% Mo the maximum value was obtained for 10 hours.

**[13] EFFECT OF THE HIGH-ENERGY BALL MILLING ON THE COMBUSTION SYNTHESIS OF NbAl<sub>3</sub>**

RML Neto, CJ daRocha - ADVANCED POWDER TECHNOLOGY II (Series: KEY ENGINEERING MATERIALS), 2001, Vol 189-1, pp 567- 2ND INTERNATIONAL LATIN-AMERICAN CONFERENCE ON POWER TECHNOLOGY; FOZ DO IGUAÇU, BRAZIL. NOVEMBER, 1999

For many years intermetallics and particularly aluminides have been synthesized by exothermal reactions. In this work, NbAl<sub>3</sub> was synthesized by simultaneous combustion from its elemental powders. A high-energy ball milling apparatus (SPEX mill) was used to accomplish the mixing of the powders promoting a highly refined dispersion. To get this, only the milling time was changed. For studying the influence of milling action on reaction synthesis, differential thermal analysis



(DTA) was conducted on loose powders of the Nb75Al milled mixtures up to 1100 degreesC at a fixed heating rate under argon. In order to get some information about intermediate reactions observed small samples of the mixtures were heated in the DTA apparatus up to 750 degreesC. X-ray diffraction analysis was performed in as-milled and partially reacted samples for phase identification. Preliminary results showed that as milling time is increased, the ignition temperature is decreased to temperatures below the melting point of aluminum. Also a variation in the reaction evolution is observed, changing from two-stage to single stage reaction.

#### [12] HIGH-ENERGY BALL MILLING OF AL-BASED ALLOYS

CAD Rodriguez, WJ Botta - ADVANCED POWDER TECHNOLOGY II (Series: KEY ENGINEERING MATERIALS), 2001, Vol 189-1, pp 573-577- 2ND INTERNATIONAL LATIN-AMERICAN CONFERENCE ON POWER TECHNOLOGY; FOZ DO IGUAÇU, BRAZIL. NOVEMBER, 1999

Nanocomposites with an amorphous matrix containing metallic nanocrystals can be obtained through the controlled crystallisation of amorphous alloys. The microstructural control and knowledge of phase transformations associated with the amorphous precursors are important stages for development of such nanocomposites and in the present work we performed high-energy milling to produce the following Al-based amorphous alloys: Al90Fe5Nb5, Al90Fe7Nb3, Al90Fe5Cr5 and Al90Fe7Zr3. Characterisation of the different mixtures as a function of milling time was accomplished by X-ray diffraction, scanning electron microscopy and transmission electron microscopy. Crystallite size variations after milling at different times were measured by X-ray peak broadening analysis and the crystallisation process or recrystallisation of the milled powders were followed by differential scanning calorimetry. Milling of all alloys, resulted in partial amorphisation and also in intermetallic compound formation. The crystallisation temperatures of the primary phase and intermetallic compounds were found to be very close for most alloys, what makes the production of nanocomposite unfeasible. However for Al90Fe7Zr3 milled alloy, crystallisation occurred in two distinct stages with significant differences in the crystallisation temperatures of the different phases presenting good perspectives for metallic nanocomposites development.

#### [11] MECHANOCHEMICAL MECHANISMS IN STRESS CORROSION

JJ Gilman - CHEMISTRY AND ELECTROCHEMISTRY OF CORROSION AND STRESS CORROSION CRACKING: A SYMPOSIUM HONORING THE CONTRIBUTIONS OF R.W. STAEBLE, 2001, pp 3-25- SYMPOSIUM ON CHEMISTRY AND ELECTROCHEMISTRY OF CORROSION AND STRESS CORROSION CRACKING - A SYMPOSIUM HONORING THE CONTRIBUTIONS OF RW STAEBLE; NEW ORLEANS, LOUISIANA. FEBRUARY 11-15, 2001

The effects of applied mechanical potentials on chemical reactions are discussed. Chemical reactivity is determined by chemical hardness. That is, by the gap in the bonding energy spectrum between the bonding and anti-bonding energy states. It always increases during a chemical reaction. Bond-bending (shear) has a large effect on the chemical hardness, whereas bond-stretching (dilation) has relatively little effect. This leads to specific mechanochemical mechanisms that are discussed; and to a characteristic rate law, based on electron tunneling, that is consistent with observations of stress corrosion fractures and with the very direct experiments called "hammer chemistry".

external molecular envelope.

#### [10] COMBUSTION SYNTHESIS OF A ALPHA ' ALFESI INTERMETALLIC

Murali S. Sriharan T. Hing P. - International Journal of Powder Metallurgy. 37(3):67-74, 2001

Processing parameters used in the combustion synthesis of intermetallics have an effect on the product and its microstructure. This study examines the production of an intermetallic (Al, Fe, Si) by combustion synthesis incorporating the effects of ball milling parameters, composition of the powder mixture, and Al particle size. Combustion synthesis of the stoichiometric mixture prepared by ball milling using fine Al particles (similar to 6 µm) produced a mono phase (Al, Fe, Si) intermetallic. X-ray diffraction traces showed mono phases in the products by the microstructures revealed traces of unreacted Al. Off-stoichiometric powder compositions in the range 6-0w/o Si and 31-34w/o Fe also led to the production of a mono phase product. The use of coarse Al particles (>67 µm) resulted in incomplete synthesis.

#### [9] EFFECT OF MILLING TIME ON MECHANICAL PROPERTIES OF AL2O3-NiAl COMPOSITES

Tuan WH. Chang ST. Chou WB. Pai YP. - British Ceramic Transactions. 100(1):35-37, 2001.

Alumina-NiAl composites were prepared by powder metallurgy processing for the evaluation of mechanical properties as a function of composite composition. Fine alumina powder and coarse angular NiAl particles were attrition milled for different time periods, using zirconia grinding media, resulting in the NiAl particles being squeezed into elongated flakes. Dense composites were prepared by hot pressing in graphite dies at 1450 degreesC and samples prepared for microstructure and properties characterisation. Increase in milling time was found to increase the aspect ratio of the NiAl flakes which aligned perpendicular to the hot pressing direction during fabrication. The strength and toughness of the composites were found to be higher than predicted by the rule of mixtures and this is ascribed both to the bridging effect of NiAl flakes between crack surfaces, which is enhanced by the increased aspect ratio from increased milling time, and microstructural refinement from grain growth inhibition

#### [8] A NOVEL CHEMICAL-MECHANICAL PLANARIZATION TECHNOLOGY USING PRE-THIN-SURFACE GRINDING IN ULSI MANUFACTURING PROCESS

Watanabe J. Etoh R. Hirano M. - PRECISION MACHINING OF ADVANCED MATERIALS. 196 PG. 25-30. 2001 - TRANS TECH PUBLICATIONS LTD, BRANDRAIN 6, CH-8707 ZURICH-UETIKON, SWITZERLAND. URL: [www.ttp.net](http://www.ttp.net)

The chemical-mechanical polishing (CMP) technology has been applied for ULSI planarization process. In this work, the novel process using pre-thin-surface grinding and CMP for highly efficient planarization are proposed. Patterned inter-layer-dielectric (ILD) film surface is ground or scratched with a thin diamond disk wheel. Since the surface has some roughed texture and is chemically activated through storing strain energy, the pre-thin-surface ground ILD film is selectively polished so that the superior planarization for the protruded parts could be completed by following conventional CMP. Practically, 2mm-square patterned ILD film which could not be previously planarized with conventional CMP process is perfectly planarized using the new process. The removal-rate enhancing factors are investigated with respect to decreasing activation energy induced by residual stress



**[7] MOSSBAUER SPECTROSCOPY AND MAGNETIC STUDIES OF NANOCRYSTALLINE IRON PRODUCED BY MILLING IN AN ARGON ATMOSPHERE**

El'sukov EP. Dorofeev GA. Ul'yanov AI. Zagainov AV. Maratkanova AN. - Physics of Metals & Metallography (English Translation of Fizika Metallov i Metallovedenie). 91(3):258-265, 2001

Mossbauer and Auger electron spectroscopies, X-ray diffraction analysis, and magnetic measurements were used to study the properties of nanocrystalline iron produced by milling in an inert atmosphere in a planetary ball mill. The transition to the nanocrystalline state with an average grain size of similar to 10 nm was found to be accompanied by neither changes in the specific magnetization saturation and temperature dependences of the magnetic susceptibility nor the formation of an additional sextet in the Mossbauer spectrum. Slight variations of the bcc-lattice parameter and the widths of the Mossbauer spectrum peaks (similar to 20%) are observed, whereas parameters such as the hyperfine magnetic field, isomeric shift, and quadrupole splitting remain unchanged. The coercive force was found to show nonmonotonic variations with changing time of milling

**[6] MOSSBAUER STUDY OF NANOSTRUCTURED GAMMA-NI-FE ALLOYS PREPARED BY HIGH ENERGY BALL MILLING**

Kim JG. Han KH. Jenog JY. Lee JS. Qin XY. Shin KH. - Journal of the Korean Physical Society. 38(4):384-387, 2001

The temperature dependence of the Mossbauer parameters of nanostructured gamma -Ni-Fe alloys prepared by high-energy ball milling was studied by using some theoretical models. The Mossbauer spectra were obtained by using a Mossbauer spectrometer within the temperature range from liquid-nitrogen temperature to 650 K. From the temperature dependence of the natural logarithmic absorption area of the Mossbauer spectrum, the Debye temperature were determined to be 277.198 K (sextet) and 183.847 K (central single line) for gamma -Ni-55Fe, and 289.043 K (sextet) and 211.127 K (central single line) for gamma -Ni-45Fe. The strength of the interaction between magnetic ions and the coefficients of the spin-wave for the samples were also determined by applying the spin-wave theory to the temperature dependence of the magnetic hyperfine field of the samples

**[5] METASTABLE STRUCTURES IN ALPHA-BETA ' BRASS**

Gialanella S. Lutterotti L. - Journal of Alloys & Compounds. 317:479-484, 2001

Rapid solidification and mechanical grinding were used to induce metastable structures in a Cu-Zn alloy with a composition falling in the two phase alpha-beta' (face centred cubic-B2 ordered) region of this binary system. Thermal treatments were also carried out to induce further changes and transformations to more stable states starting from the conditions attained with the mentioned non-equilibrium processes routes. In the rapidly solidified material, a body centred cubic structure was observed. At this stage the presence of long-range order could be inferred. Nonetheless, upon heat treating the material at 500 degreesC for 15 min the formation of the alpha-beta' mixture could be induced. A similar condition could also be achieved by grinding the starting melt-spun ribbons in a high energy ball-mill. The remarkable difference was that in this case the alpha -phase had a non-equilibrium composition, which is compatible with that of the parent beta'-phase. After heat treatment, the milled specimen has the same composition of the alpha -phase as the one found in the melt-spun heat treated materials. These results indicate a diffusionless transformation was induced by the milling process into the as-spun material from the parent beta'-phase into the alpha-beta' mixture. Phase evolution and compositions were evaluated using X-ray diffraction analyses.

**[4] HYDRIDING PROPERTIES OF MECHANICALLY ALLOYED ICOSAHEDRAL PHASE Ti45Zr38Ni17**

Konstanchuk IG. Ivanov EY. Bokhonov BB. Boldyrev VV. - Journal of Alloys & Compounds. 319(1-2):290-295, 2001

The interaction with hydrogen was investigated for mechanochemically synthesized icosahedral phase of the composition Ti45Zr38Ni17. It was demonstrated that, unlike icosahedral phases of the same composition formed as a result of rapid quenching from melt or the annealing of alloys, hydriding of mechanochemically synthesized icosahedral phase at 503 K is not preceded by an induction period and starts at a maximal rate even when hydrogen pressure is less than 0.1 MPa. A diagram of the composition of icosahedral phase versus equilibrium hydrogen pressure (P-T-C diagram) at 503 K was experimentally obtained. Based on published data and the data obtained in our experiments, an assumption was made on the existence of two types of hydrogen positions in the icosahedral structure of the hydrogenated phase. The first type is more energetically bound hydrogen occupying the positions within the structure-forming icosahedral Bergman clusters. The concentration of such hydrogen is approximately corresponding to the relation H/M=1.1. The second type is less bound hydrogen occupying the linkages between these clusters as solid solution.

**[3] STRUCTURAL INVESTIGATIONS OF THE Al50Fe25Ti25 POWDER MIXTURE MECHANICALLY ALLOYED UNDER VARIOUS CONDITIONS**

Krasnowski M. Matyja H. - Journal of Alloys & Compounds. 319(1-2):296-302, 2001

The aim of this work was to study the structural and phase transformations that take place during mechanical alloying of the ternary Al50Fe25Ti25 alloy in a high-energy planetary ball mill. Two experiments were performed: one with addition of ethanol and the other without any additional agent. The structural changes and the formation of new phases occurring in the material during mechanical alloying at various milling times were examined using X-ray diffractometry (XRD), differential scanning calorimetry, conventional transmission electron microscopy and high-resolution electron microscopy. The XRD patterns show that a b.c.c. Fe(Al) solid solution and an fcc phase with the lattice parameter  $a(0)=4.295$  Angstrom isomorphous with TiC are the final products of the process performed with addition of ethanol. Electron microscopy observations reveal that after the longest milling time, the structure of the powder particles becomes nanocrystalline. No phase changes were found in the XRD pattern of the final product of ethanol-added milling, annealed at 720 degreesC for 2 h. The milling process performed without addition of ethanol leads to an amorphous phase. Heating of this phase in a calorimeter causes crystallisation of the ternary tau (2) phase (Al2FeTi) and an Fe(Al,Ti) solid solution.

**[2] ADVANCES IN THE SYNTHESIS AND CHARACTERIZATION OF BORON NITRIDE**

Huang JY. Zhu YT. - Editors Fisher DJ - DEFECTS AND DIFFUSION IN CERAMICS : AN ANNUAL RETROSPECTIVE III. 186-1 PG. 1-32. - SCITEC PUBLICATIONS LTD, BRANDRAIN 6, CH-8707 UETIKON-ZUERICH, SWITZERLAND. URL: <http://www.ttp.net>  
Book Series Title DEFECT AND DIFFUSION FORUM



We review the progress in the experiments and understanding of high pressure and high temperature (HPHT) induced phase transformation in boron nitride. The HPHT induced phase transformation is significantly enhanced by refining the microstructure of the starting material, e.g. by ball-milling hexagonal boron nitride (h-BN) to a defective, nanocrystalline or even amorphous state. For example, cubic boron nitride (c-BN) forms from nanocrystalline or amorphous BN (a-BN) matrix at 900 degreesC and complete a-BN to c-BN phase transformation occurs at 1350 degreesC under 7.7 GPa. These temperatures and pressures are significantly lower than required to transform coarse-grained crystalline h-BN to c-BN. High resolution transmission electron microscopy (HRTEM) and electron energy loss spectroscopy (EELS) revealed that the c-BN phase nucleates directly from the sp(3) hybridized amorphous matrix, which is originally induced by ball milling and is therefore responsible for the lower HPHT requirements. This c-BN nucleation mechanism is completely different from the so-called diffusionless "puckering" mechanism that operates in the nucleation of c-BN from coarse-grained h-BN in HPHT experiments, but very similar to one of the proposed mechanisms involved in the chemical vapor deposition (CVD) of diamond and c-BN. HRTEM also shed new light on the phase transformation of slightly deformed coarse-grained h-BN under HPHT conditions. The interface structures among h-, w- and c-BN reveal that the phase transformation can proceed by different routes including h --> w --> h --> g --> c, h --> w --> 6H' --> c, h --> w --> 2H --> 2H' --> c, h --> am --> c and h --> c, where h, w, c, g, 6H', 2H' and am represent h-BN, w-BN, c-BN, g-BN, 6H'-BN, 2H'-BN and a-BN, respectively. Irrespective of these different transformation routes, all the phase transformations follow the same orientation relationships, i.e. [11(2) over bar 0](h)//[11(2) over bar 0](w)//[1(1) over bar 0](c) and (1(1) over bar 00)(h)//(1(1) over bar 00)(w)//(1 1 1)(c). The phase transformations also follow a general rule: at low temperatures, the formation of c-BN is always preceded by the formation of intermediate phases such as w-BN, 2H' and 6H', and the transformation is martensitic in nature; at higher temperatures, there is a direct h-BN to c-BN transformation, and the transformation is diffusional dominated. The transformation from w-BN to c-BN is achieved by introducing periodic stacking-faults (SFs) in w-BN. Based on the HRTEM images, three new BN polymorph phases i.e., g-BN, 2H'-BN and 6H'-BN, were proposed for the first time. HRTEM also revealed that ball-milling introduces significant defects such as SFs, twins, Frank dislocations, delamination, rotating and shearing of the sp(2) layers, and disordering in the h-BN lattice. These defects are found to promote the subsequent hexagonal to cubic transformation.

**[1] EVOLUTION OF MICROSTRUCTURE AND HARDENING, AND THE ROLE OF Al<sub>3</sub>Ti COARSENING, DURING EXTENDED THERMAL TREATMENT IN MECHANICALLY ALLOYED Al-Ti-O BASED MATERIALS**

Barlow IC, Jones H, Rainforth WM. - Acta Materialia. 49(7):1209-1224, 2001

Extruded rods of mechanically alloyed (MA) Al-0.25wt% C-10vol% TiO<sub>2</sub> (A1), Al-0.35wt% Li-1wt% Mg-0.25wt% C-10vol% TiO<sub>2</sub> (A2), and Al-0.35wt% Li-1wt% Mg-0.25wt% C-7.5vol% TiO<sub>2</sub> (A3) have been subjected to thermal treatments at 500 degreesC, 550 degreesC, 600 degreesC, and 655 degreesC for times up to 1500 h. At 550 degreesC and 600 degreesC, the materials displayed hardness increments, but the increase was highest for A1 (similar to 30 kg/mm<sup>2</sup>) due to fine Al<sub>3</sub>Ti precipitation. The starting grain size of similar to 0.5 μm in A1 showed virtually no increase even after 1500 h at 600 degreesC. XRD and TEM indicated transformation of TiO<sub>2</sub> and TiO to Al<sub>3</sub>Ti. Numerous "block-shaped" Al<sub>2</sub>O<sub>3</sub> particles (alpha, delta, eta, and gamma polymorphs) were formed in A2 at 600 degreesC. The alpha-Al<sub>2</sub>O<sub>3</sub> particles exhibited some lattice matching with the Al matrix. An initially rapid Al<sub>3</sub>Ti coarsening rate at 600 degreesC in A1 was reduced significantly between 336 h and 1500 h. Dispersion strengthening by Al<sub>3</sub>Ti and Al<sub>2</sub>O<sub>3</sub> was mainly responsible for the maximum strength of Al after heat treatment, while the Hall-Petch contribution was less significant, despite the fine grain size.



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### **COMMUNIQUE DE PRESSE : Un rhéomètre pour l'évaluation des propriétés d'écoulement de la poudre**

Ce rhéomètre, breveté en Grande-Bretagne, permet de résoudre bon nombre des problèmes associés au traitement de la poudre humide et sèche dans les domaines de la recherche, du développement et de la production.

Grâce à un principe des plus novateurs, le **rhéomètre à poudre FT3 de Freeman Technology** évalue les propriétés d'écoulement des poudres et des semi-solides. De fait, la fluidité se mesure à la quantité d'énergie requise pour induire un état d'écoulement dynamique. Ainsi, avec le FT3, les mesurages peuvent être répétés quasiment à l'infini et les procédures d'essai et d'analyse sont automatisées, d'où un gain de temps précieux et l'absence de toute intervention humaine.

Le traitement des poudres se révèle souvent être une opération ardue, en raison des nombreux facteurs qui influent sur les propriétés d'écoulement, comme la vitesse de coulée, la compaction, la ségrégation, l'attrition, l'adhésivité et la fluidification. Or, le rhéomètre à poudre FT3 est capable de classer les poudres en mesurant la dépendance de leur fluidité vis-à-vis de chacun de ces facteurs.

Le mesurage de la fluidité est généralement compliqué par les variations observées en termes de tassement. La solution apportée par le FT3 à ce problème est un procédé de conditionnement, qui produit une densité de tassement uniforme et reproductible préalablement aux essais de fluidité. Il est ainsi possible de comparer, de façon parfaitement fiable, des mesures prises à différents moments et en différents lieux.

Outre les essais programmés, des routines d'homogénéisation entièrement programmables peuvent être conçues en vue d'homogénéiser des poudres sèches ou des mélanges poudre-liquide, voire des matières plus complexes encore, telles la farine et l'eau. Grâce à ces routines, des programmes complexes peuvent être définis de bout en bout et, le cas échéant, répétés à volonté. Une analyse énergétique complète du programme d'homogénéisation est également possible.

Les applications du FT3 sont diverses, depuis les études de formulation en recherche et développement (R&D) jusqu'à l'évaluation des effets d'attrition dans un procédé de fabrication donné, en passant par la définition de critères de fluidité aux fins du contrôle qualité (CQ). Et ces applications présentent de grands avantages puisqu'elles permettent, entre autres, de réduire les délais d'élaboration des produits nouveaux, de limiter le nombre des arrêts de production et d'améliorer le contrôle de la qualité des matières premières comme des produits finis.



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The "pulverisette 4" vario-planetary mill is capable of emulating ball mills of conventional design, precisely simulating the types of stress entailed and thus reproducing or optimising grinding processes. Due to the high flexibility available for selecting the grinding parameters, it is possible to achieve results unattainable with any other ball mills.

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When particles < 10 mm are fed in, a final fineness up to 0.1 µm can be achieved. The useful capacity is between 2 x 30 ml in the case of 80 ml grinding bowls and 2 x 125 ml when 250 ml grinding bowl are used.

Method of operation:

With standard planetary ball mills the grinding bowls are rotating and mounted eccentrically on a rotating support disc. The rotational speed of the supporting disc can be selected at will; the grinding bowl rotates at a fixed transmission ratio.

Due to the overlapping of grinding bowls and supporting disc, the material to be ground and the grinding balls execute movements and trajectories in the grinding bowl, which are defined by the transmission ratio.

With the "pulverisette 4" vario-planetary mill the rotational speeds of grinding bowls and supporting disc can be adjusted completely independently of each other. By varying the transmission ratio it is possible to control the movements and trajectories of the grinding balls at will so that the balls strike the inner wall of the bowl vertically (high impact energy), approach each other tangentially (high friction) or just roll down the inner wall of the bowl (centrifugal mills).

All intermediate levels and combinations of frictional and impact pressures can be set as required. By changing the transmission ratio it is therefore possible for the first time to carry out mechanical activation as well as mechanical alloying.

Furthermore, it is also possible for the first time to optimally adjust a planetary ball mill to the material to be ground, the size of the grinding bowls and the grinding balls.

Features of performance:

- for the first time, all grinding parameters can be selected at will for optimal preparation of sample
- Programming of the grinding parameters by PC software as desired
- RS232 interface for programming and to transfer grinding parameters to the PC
- Real-time display of the speeds to monitor the grinding process
- Reversing option (direction of rotation reversed periodically) to improve the grinding results
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**Allemagne (6)**  
●●●●●●

**Angleterre (4)**  
●●●●●●

**Argentine (3)**  
●●●●●●

**Australie (8)**  
●●●●●●

**Belgique (1)**  
●●●●●●

**Brésil (5)**  
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**Bulgarie (1)**  
●●●●●●

**Canada (7)**  
●●●●●●

**Chine (7)**  
●●●●●●

**Corée du Sud (4)**  
●●●●●●

**Croatie (3)**  
●●●●●●

**Danemark (1)**  
●●●●●●

**Egypte**  
●●●●●●

**Espagne (2)**  
●●●●●●

**Grèce (1)**  
●●●●●●

**Hongrie (3)**  
●●●●●●

**Inde (2)**  
●●●●●●

**Israël (4)**  
●●●●●●

**Italie (8)**  
●●●●●●

**Japon (11)**  
●●●●●●

**Nouvelle - Zélande (1)**  
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**Pologne (3)**  
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**Portugal (1)**  
●●●●●●●

**Roumanie (2)**  
●●●●●●●

**Russie (8)**  
●●●●●●●

**Singapour (3)**  
●●●●●●●

**Slovaquie (3)**  
●●●●●●●

**Suède (3)**  
●●●●●●●

**Tunisie (1)**  
●●●●●●●

**U.S.A.(8)**  
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**Viet Nam (2)**  
●●●●●●●

**Yougoslavie (2)**  
●●●●●●●

**France**  
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