



## RESEAU FRANÇAIS DE MECANOSYNTHESE

Lettre N°75

Juin 2001

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

Bureau du RFM : E. Gaffet (Président)  
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Full Congress Announcements  
Congress List (related to Nanomaterials)  
Cooperation (PhD, **new Post Doc proposal**, International Relationships)  
Books (related to Nanomaterials)  
Bibliography  
Technical Announcements (from M.B.N. srl & Fritsch)

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.

### 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

Lettre RFM N°75 - Juin 2001  
Corresp. : <mailto:Eric.Gaffet@utbm.fr>

The check has to be to the order : Réseau Français de Mécanosynthèse  
(Please do not use Eurocheck, the taxes do correspond to 40% of the amount of the check).

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## **FRITSCH: Your specialist in the processing laboratory**

**Contact :** Andrea Köhler

FRITSCH GMBH - Laborgerätebau-Industriestrasse 8 - D-55743 Idar-Oberstein - Germany

Tel.: 06784/70-0 - Fax: 06784/70-11

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FRITSCH is the world-wide leading manufacturer of instruments for sample preparation and particle size analysis in the laboratory. Since more than 80 years now, the company is concentrating on the three product groups and identified with the brand names of "pulverisette", "analysette" and "laborette" in research and industrial laboratories. The Fritsch name is synonymous with technical competence and economical efficiency in all matters in the field of

- milling
- particle sizing
- dividing

Because FRITSCH operates throughout the world, they have the unique advantage of vast experience in a wide variety of scientific disciplines which enables them to offer constructive solutions to ease problem solving. FRITSCH laboratory instruments ensure proven reliability and innovative technology in combination with simple operation.

Fritsch presents some of the products at the following exhibitions:

- **JRFM 2001 – 6èmes Journées du Réseau Français de Mécanosynthèse in Amiens, 21. + 22.05.2001**
- **E-MRS - European Materials Research Society Spring Meeting in Strassbourg, 6. + 7.06.2001**

Here a small glance of the products, which will be presented:

- **Vario-Planetary Mill "pulverisette 4"**  
Our "erlking" on the exhibitions: a Planetary Mill with absolutely new features of performance, specially developed for the material research, but also welcome in every High-tech laboratory. Power like a *Ferrari*, but with a lower price.
- **new generation of GTM – gas pressure and temperature measuring system**  
for the determination of gas pressure and temperature data during the grinding process in the grinding bowls
- **Additional lock-system**  
for tight fastening of grinding bowl and lid and is recommended strongly when grinding in an inert atmosphere, although when the gas is injected outside the mill:
- **Dry Dispersing Unit for "analysette 22" Laser-Particle-Sizer**  
Finally, here it is – lively and powerful like a *BMW*-motor. The new dry dispersing unit ensures the latest state of the art technology in the sample feeding and enlarges considerably the range of applications of the Laser-Particle-Sizers COMPACT and COMFORT.
- **Rotary Cone Sample Divider "laborette 27"**  
your most important partner for the sample division in the lab, because sample division is one of the most important laboratory operations in the processing of solids or suspensions

Drop in – you will be surprised.



# **EQUIPEMENTS SCIENTIFIQUES S.A**

## *Département BIO-TESTS*

**Contact : Daniel GUEZ - Directeur de département**  
127. RUE DE BUZENVAL - BP 26 - 92380 GARCHES (FRANCE)

Société anonyme au capital de 21.620.000 Francs R..C. Nanterre B 353 579 634 00018 code APE 516J

Téléphone : (33) 01 47 95 99 12 - Fax : (33) 01 47 01 16 22

e-mail : <mailto:bio@es-france.com> - Website: <http://www.es-france.com/>

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

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**ISMAMANAM2001**

University of Michigan, Ann Arbor, Michigan, USA  
Ann Arbor, Michigan, 24-29 June, 2001

<http://www.ners.engin.umich.edu/ISMAMANAM2001>

**Abstract deadline is March 1st, 2001.**

**Abstracts of 200-400 words should be submitted by e-mail to  
E-Mail : <mailto:ISMAMANAM-2001@umich.edu>**

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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)

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**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>**

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**"FUNDAMENTAL DASES of MECHANOCHEMICAL TECHNOLOGIES"**  
International Conference  
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/>

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**(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/>

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**(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>

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**"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>

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Workshops

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

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

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



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**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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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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**INTERNATIONAL CONFERENCE TRENDS IN MECHANICAL ALLOYING ;  
Science, Technology and Applications (TMA-2001)**

**By Dr. E. Ivanov (Tosoh) - report on the TMA'2001 meeting**

Under the aegis of Powder Metallurgy Association of India (PMAI), The Indian Institute of Metals (Metal Sciences Division) and the Institution of Engineers (RSC), Department of Metallurgical Engineering, Malaviya Regional Engineering College, Jaipur organised an International Conference on 'Trends in Mechanical Alloying: Science, Technology and Applications' on Feb. 21-23, 2001 (TMA-2001). Concurrently the 27<sup>th</sup> annual technical meeting(ATM) of PMAI was also held.

More than 100 delegates, comprising of academic & industrial personals from U.S.A., Japan, Spain, Korea, U.K. and India attended the conference. The conference was inaugurated by The chief guest Prof. P. Ramachandra Rao, Director, National Metallurgical Laboratory, Jamshedpur and was presided by world renowned powder metallurgist Prof. P. Ramakrishnan of I.I.T. Bombay. On this occasion Prof. S.C. Agarwal, Principal, Malaviya Regional Engineering College delivered the welcome speech and Dr. Ashok Mohan, President, Powder Metallurgy Association of India explained role of the association and the justification of coupling the ATM with this international conference. Dr. P.R. Soni, Convener of the event introduced the TMA-2001 and discussed the need of holding it. Prof. T.V. Rajan, Head, Department of Metallurgical Engineering delivered vote of thanks on the occasion. About 50 papers were presented in eight different technical sessions namely the process, characterisation and consolidation, surface engineering, amorphisation and quasicrystalline materials, nanocrystalline materials and special applications of MA materials. While keynote address Dr. E. Ivanov, Tosho SMD Inc., USA discussed potential applications of mechanochemical processing (MCP) and mechanical alloying (MA) for the development of various novel advanced materials like hydrogen storage materials, gas absorbers, fertilizers, catalyst, cosmetics and waste management. While delivering invited lecture Prof. C. Bansal of University of Hyderabad discussed formation, stability and phase transformation behaviour of nanocrystalline alloys in the  $Fe_3X$  alloy (X-Ge, Al, Sn, Sb, As, In, Si) with the help of Darken-Gurry plots of electronegativity Vs metallic radius. In another invited lecture Prof. K. Chattopadhyay of the Indian Institute of Sciences, Bangalore discussed nature of displacement reactions under the influence of mechanical actions and emphasized the suitability of MCP for the production of nanomaterials. Besides papers were presented on many novel aspects of mechanical alloying namely the reactive milling, MA by swift heavy ions, MA intermetallics, sinterability of MA powders, tribo MA materials, MA powder coatings, machining of MA heavy alloy, hard and soft magnetic materials, hydrogen storage materials, trace elements additions by MA, MA solder alloys.

The concluding session was jointly chaired by Dr. E. Ivanov and Prof. K. Chattopadhyay. All the session chairpersons; Dr. R. Sundaresan, Prof. M.M. Galkhindi, Prof. M.K. Bhargava, Prof. A.G. Escorial (Spain), Prof. Toshimi Yamane (Japan), Prof. P.N. Singh, Dr. P.B. Joshi, Prof. P.C. Angelo presented report of the respective session. It was concluded that MA technique which was developed originally for the production of oxide dispersion strengthened materials, the trend was towards nanocrystalline materials in the recent past but the same has now changed to mechanochemistry. The excitement towards MA technique is mainly due to well developed infrastructural P/M science and technology related to consolidation etc. However, to make the technique more adaptive to the industries standardization of high energy mills to improve their efficiency, development of the process models and softwares for prediction of the results is very essential. The technique is very versatile and getting grounds in development of many high-tech materials.



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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 tellurure de plomb"**

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

**Jury :**

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

L'objectif de cette thèse est de proposer un procédé d'élaboration de matériaux thermoélectriques destinés à être intégrés dans un thermogénérateur. Deux méthodes de synthèse sont abordées. La première, la solidification classique, s'effectue en tubes de silices scellés sous vide par trempe du matériau porté à fusion, la deuxième, la mécanosynthèse, a lieu dans un broyeur planétaire, et permet l'obtention des matériaux fins directement sous forme de poudre par broyage réactif. La mécanosynthèse permet de produire à température ambiante des poudres homogènes de granulométrie très fine et pourrait alors conduire à l'amélioration des propriétés thermoélectriques des matériaux tout en proposant un procédé moins onéreux et plus approprié pour une élaboration à l'échelle industrielle. Sa mise en œuvre facile devrait permettre un passage aisé du cadre du laboratoire à l'échelle industrielle.

L'étude présentée comporte deux parties:

- la première consiste à mettre au point la synthèse de poudres thermoélectriques à base de tellurure de plomb par mécanosynthèse et par solidification classique, une étude complète à été réalisée et les conditions de frittage ont été paramétrées.

- la deuxième consiste en une simulation des conditions du futur thermogénérateur à l'aide d'une maquette afin de tester le comportement des matériaux en situation réelle.

Les propriétés thermoélectriques des matériaux frittés issus des deux méthodes de synthèse utilisées de synthèse sont comparées. Cette étude comparative permet de dresser un bilan concernant les avantages et les inconvénients de la mécanosynthèse pour la production de matériaux thermoélectriques.



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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**

**SPAIN (03/04/2001)- From J.J. Suñol (joanjosep.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 began 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:joanjosep.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(IRE), 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



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Corresp. : <mailto:Eric.Gaffet@utbm.fr>

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 Laboratorium, 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.metallimaterials.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>

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**From Dr. Jürgen Eckert Allemagbe (11/2000**

**Ph D or Post Doc Position**

We are looking for a PhD candidate / Postdoc to start as soon as possible in the framework of an European RTN network on bulk metallic glasses and nanostructured materials.

Dr. Jürgen Eckert

IFW Dresden - Institut für Metallische Werkstoffe

Postfach 27 00 16 - D-01171 Dresden -Germany

>Tel.: +49 (351) 4659-602/-324

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## Bibliographie Récente

### Livres ou "Special Issues"

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

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

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

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

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

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

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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)

[file://http /// www.elsevier.nl/inca/publication](http://www.elsevier.nl/inca/publication)

#### 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.

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**(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

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**DISPERSION STRENGTHENED ALUMINIUM PREPARED BY MECHANICAL ALLOYING**, by M Besterçi - <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-Al4C3 material)  
**4. Static and dynamic mechanical properties** (mechanical properties at elevated temperatures; mechanical properties at 20 °C; effect of interface on the mechanical properties; superplastic properties of the system; thermal stability of the system; creep characteristics; creep-fatigue characteristics)

References - ISBN 189832655X, 90 pages, 234x156 mm, soft laminated cover, £25.00, 1999

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

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

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

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

A. Inoue

Materials Science Foundation Vol. 4 - Trans Tech Publications : <http://www.sciencen.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**

Michal Besterçi, 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

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**1. Characteristics of dispersion-strengthened systems**  
**2. Mechanical alloying** (kinetics and mechanism of preparation of the Al-C system by mechanical alloying; compaction of powders and heat treatment of compacts;  
**3. Microstructure and quantitative evaluation of parameters of dispersion-strengthened materials** (definition and properties of interparticle distance; experimental possibilities of determination of structural objects; models of heterogeneous structures and their evaluation; simulation of model structures; analysis of the spatial distribution of particles in the Al-Al4C3 material)  
**4. Static and dynamic mechanical properties** (mechanical properties at elevated temperatures; mechanical properties at 20°C; effect of interface on the mechanical properties; superplastic properties of the system; thermal stability of the system; creep characteristics; creep-fatigue characteristics)

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

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**"Mechanical Alloying"**



Lettre RFM N°75 - Juin 2001  
Corresp. : <mailto:Eric.Gaffet@utbm.fr>

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

Kluwer Academic Publishers

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

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

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

Materials Research Society, Symposium Proceedings Volume 501, 1998

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

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**"Nanomatériaux"**

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

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

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

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

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

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

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**CHEMISTRY FOR SUSTAINABLE DEVELOPMENT**

Vol. 6, No. 2-3, MARCH-JUNE 1998

Proceedings of 2d International Conference on Mechanochemistry

(INCOME-2), which was held in Novosibirsk in 1997.

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

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**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.

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**BIBLIOGRAPHY ON MECHANICAL ALLOYING AND MILLING**

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

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

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**Proceeding du Congrès "Mechanically Alloyed, Metastable and Nanocrystalline Materials"- Barcelone (1997)**

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



## Périodiques

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

### [50] MECHANICALLY ALLOYING AND HIGH PRESSURE PULSED CURRENT SINTERING OF SI ADDED TI POWDERS [JAPANESE]

Kobayashi K. Nishio T. Matsumoto A. Ozaki K. Sugiyama A. - Journal of the Japan Institute of Metals. 65(3):179-182, 2001

Ti-1 at%Si, Ti-5 at%Si, Ti-10 at %Si and Ti-20 at%Si powders were synthesized by mechanically alloying (MA) of pure elemental Ti and Si powders using a planetary ball milling for 360 ks. Si addition was effective to produce large amount of Ti-based MA powder without adhesion to the vial wall and the surface of the milling balls. After milling for 360 ks, the amount of recoverable powder increased with increasing Si content in the MA powder. The formation of an amorphous phase was observed at Ti-5 at%Si and Ti-10 at%Si powders after 360 ks milling. Ti-10 at%Si powder prepared by milling for 360 ks, which consisted of amorphous phase and fine Ti particles, was consolidated using a pulsed current sintering (PCS) apparatus under a high pressure of 1568 MPa. The compact consolidated at 733 K was almost densified with the retention of non-equilibrium structure. The Ti-10 at%Si powder milled for 360 ks with a hardness of 378 Hv was suitable for a consolidation under a high pressure with a deformation of powder

### [49] EFFECT OF MECHANICAL ALLOYING ON COMBUSTION SYNTHESIS IN THE NI-AL-B SYSTEM [JAPANESE]

Ayabe K. Okabe T. - Journal of the Japan Institute of Metals. 65(3):195-198, 2001

Mechanical alloying (MA) of powder mixtures of Ni-33 mol%Al-x mol%B(x = 0-2.0) was performed for various milling times, and the effect of MA on combustion synthesis under air and vacuum heating conditions was investigated for the MA powders. The nature of combustion synthesis changed dramatically with R IA conditions (milling duration, quantity of B-doping, etc.), and suitably produced MA powders induced combustion synthesis in air (room temperature) and vacuum heating (near 673 K). The addition of B inhibited the combustion of the MA powders. By X-ray diffraction (XRD) analysis, it was found that intermetallic compounds such as NiAl, Ni<sub>3</sub>Al and Al<sub>3</sub>Ni<sub>2</sub> were formed. The increase in relative intensity of XRD peaks for the intermetallic compounds was greater for vacuum heating than for air. It is suggested that combustion in air for the MA powders in the present study is led by the heat of formation from the oxidation reaction between mechanochemically activated fine Al powders and oxygen. That is, the heat becomes a trigger for combustion synthesis between reactant species of Ni and Al. The above mechanism in air differs from that in vacuum heating where the MA powders themselves directly cause the exothermic reaction and combustion synthesis

### [48] PROCESSING OF A NB-18AL-24VALLOY BY MECHANICAL ALLOYING

Authors Dymek S. Dollar M. Wrobel M. - Archives of Metallurgy. 45(4):421-433, 2000.

The synthesis of an alloy with the composition of 58% Nb, 24% V i 18% Al (atomic %) has been performed. The mechanical alloying of elemental powders was selected as a method of synthesis. During the course of mechanical alloying the splitting of X-ray peaks on two components was observed. Each component corresponded to the niobium solid solution (Nb-I and Nb-II) with different lattice parameters and different chemical composition. The Nb-I solid solution and the pure niobium had alike lattice parameters. The intensity of peaks corresponding to the Nb-I decreased with the time of milling. The peaks disappeared completely after 180 hours of milling. The lattice parameter of the Nb-II solid solution decreased with milling time and the intensity of corresponding peaks increased. Two brittle phases: Nb<sub>3</sub>Al-base with A15 crystal structure and Nb<sub>2</sub>Al-base sigma phase were observed in solid (consolidated) material. No phase with B2 crystal structure was observed

### [47] SYNTHESIS OF ULTRADISPERSE BARIUM TUNGSTATES AND MOLYBDATES

Authors Grigor'eva TF. Vorsina IA. Korchagin MA. Barinova AP. Lyakhov NZ. - Russian Journal of Applied Chemistry. 73(11):1871-1874, 2000

IR spectroscopy, X-ray phase analysis, and electron microscopy were applied to study the reaction of tungsten and molybdenum oxides (IV and VI) with barium peroxide under conditions of mechanochemical and self-propagating high-temperature synthesis

### [46] EFFECTS OF ALUMINA TYPE AND ATTRITOR BALL SIZE ON THE PROCESSING OF RBAO-ZRO<sub>2</sub> CERAMICS

Kim IS. Lee SJ. - Materials Letters. 48(3-4):247-251, 2001

In RBAO (reaction bonded aluminum oxide)-ZrO<sub>2</sub> ceramics system, the effects of the type of alumina powder and the size of milling media on grinding and sintering behaviors were examined. The starting powder containing fused alumina was attrition-milled more effectively by using the single-size ball, and its sintered microstructure was homogeneous and dense, showing about 10% sintering shrinkage.

### [45] STUDY OF THE NANO-STRUCTURED NICKEL-BASED BRAZING FILLER SYNTHESIZED BY MECHANICAL ALLOYING

Jang JSC. Shih HP. - Materials Chemistry & Physics. 70(2):217-222, 2001

A series of BNi-2 nickel-based brazing fillers were synthesized by mechanical alloying (MA) the appropriate proportion of pure elements in a high-energy ball mill. The results of DTA show an exothermic peak around 700 K, which is presumed to be a metastable phase, for the alloy after 4 tt milling. After 10h milling, the final particle size of the powder can reach about 20 μm and exhibits a quite even equiaxed shape. The structural evolution with milling time by X-ray diffractometry shows the following transformation: mixture of pure elemental powders --> microcrystalline f.c.c. solid solution --> nanocrystalline f.c.c. solid solution. The crystal size calculated by X-ray diffraction was about 5 nm for the powder after 10h milling. This is in very good agreement with the evidence of nanocrystalline phase obtained by TEM observation. In addition, the evaluation of the brazing properties of the BNi-2 powder synthesized by MA is similar to the gas atomized BNi-2 powder.

### [44] MECHANOCHEMICAL SYNTHESIS OF LANTHANUM OXYFLUORIDE FROM LANTHANUM OXIDE AND LANTHANUM FLUORIDE

Lee J. Zhang Q. Saito F. - Journal of the American Ceramic Society. 84(4):863-865, 2001

A powder mixture of lanthanum oxide (La<sub>2</sub>O<sub>3</sub>) and lanthanum fluoride (LaF<sub>3</sub>) was ground by a planetary ball-mill to investigate the mechanochemical reaction forming lanthanum oxyfluoride (LaOF) at room temperature. The grinding enables us to form LaOF monophase, and the reaction proceeds with an increase in grinding time, whereas the crystallite size



of LaOF formed is about 15-20 nm irrespective of the grinding time. Other rare-earth oxyfluorides (R-OF, R = Pr, Nd, Sm, Gd) can be synthesized by grinding the constituent components (R<sub>2</sub>O<sub>3</sub> and RF<sub>3</sub>)

**[43] ON THE FORMATION OF PURE AND PT-DOPED IRON SILICIDES USING BALL MILLING**

Desimoni J. Sanchez FH. - Journal of Physics-Condensed Matter. 13(11):2737-2747, 2001

The sequence of phase formations for elemental powders of stoichiometric FeSi<sub>2</sub> and Fe<sub>1-x</sub>PtxSi<sub>2</sub> (0.03 less than or equal to x less than or equal to 0.50) mixtures prepared by mechanical alloying at room temperature in an Ar atmosphere in a horizontal mill is presented. Sample evolution was followed by means of Mossbauer spectroscopy and x-ray diffraction. After the milling, the results indicate the formation of different disordered iron silicides, depending on milling time. The kinetics of iron silicide formation was also studied; a diffusion-controlled process with decreasing nucleation rate was found. In the case of Pt-doped samples, the segregation of Pt silicides was observed even for the smallest concentration, and no noticeable incorporation of Pt into the beta -FeSi<sub>2</sub> lattice could be inferred. Samples were also subjected to annealing at 1123 K for 4 h to produce ordering in the structures

**[42] LOCAL STRUCTURES OF MECHANICALLY ALLOYED FE<sub>100-X</sub>CUX SOLID SOLUTIONS STUDIED BY X-RAY ABSORPTION FINE STRUCTURE [CHINESE]**

Yan WS. Fan JW. Li YZ. Cui HB. Liu WH. Zhang XY. Wei SQ. - Acta Physica Sinica. 50(4):758-764, 2001

The local structures of the immiscible Fe<sub>100-x</sub>Cux alloys (x = 0,10,20,40,60,80 and 100) produced by mechanical alloying have been investigated by XAFS. For the Fe<sub>100-x</sub>Cux (x greater than or equal to 40) alloys, the local structures around Fe atoms change from bcc structure to fee one, and the Cu atoms maintain the original coordination geometry after milling for 160 h. On the contrary, the local structures around Cu atoms in both Fe<sub>80</sub>Cu<sub>20</sub> and Fe<sub>90</sub>Cu<sub>10</sub> alloys appears a transition from fee to bcc structure. We found that the disorder factor sigma (0.0099nm) of fee Fe-Cu phase is larger than that (0.0081nm) of bcc Fe-Cu phase, and the sigma (0.0099nm) around Fe atoms is larger than that (0.0089nm) of Cu in the Fe<sub>100-x</sub>Cux (x greater than or equal to 40) alloys. This suggests that the mechanically alloyed Fe<sub>100-x</sub>Cux alloys is not a homogeneous supersaturated solid solution, but consists of Fe-rich and Cu-rich regions for various compositions. A possible mechanism for bcc-to-fee and fcc-to-bcc changes in Fe<sub>100-x</sub>Cux alloys is discussed in relation to the interdiffusion and transition induced by the ball milling

**[41] FABRICATION OF NITI INTERMETALLIC COMPOUND COATING MADE BY LASER PLASMA HYBRID SPRAYING OF MECHANICALLY ALLOYED POWDERS**

Hiraga H. Inoue T. Kamado S. Kojima Y. Matsunawa A. Shimura H. - Surface & Coatings Technology. 139(1):93-100, 2001

NiTi intermetallic compounds not only have shape memory effects but also high erosion resistance. Therefore, applying this material as a coating is an effective method for preventing erosion. In this study, a mixture of Ti and Ni powders was subjected to a mechanical alloying process. Then, the mechanical and structural properties of the coating fabricated by vacuum plasma spraying and laser plasma hybrid spraying were examined. The results of this study are summarized as follows: (1) Mechanically alloyed powders suitable for plasma spraying were obtained under the conditions of 2 mass% methyl alcohol as a process control agent and 72 ks alloying duration. (2) A NiTi intermetallic compound coating was directly fabricated by vacuum plasma spraying. This means that nickel and titanium were alloyed while they were passed through a plasma flame. The erosion resistance of this coating is more than 20 times as high as that of the coating made from a simple mixture of nickel and titanium powders. (3) The erosion resistance of the coating made by laser plasma hybrid spraying of mechanically alloyed powder was approximately 40 times as high as that of titanium alloy. This high erosion resistance was obtained at low relative fluence compared with that of the coating made from the simple mixture. This coating can prevent degradation of the interface, because a high fluence induces the formation of a brittle intermetallic compound at the interface

**[40] A TRICKLING FILTER APPLICATION FOR THE TREATMENT OF A GOLD MILLING EFFLUENT**

Evangelho MR. Goncalves MMM. Sant'Anna GL. Boas RCV. - International Journal of Mineral Processing. 62(1-4 Special Issue SD):279-292, 2001

Some industrial processes, such as plating and gold mining, utilise cyanide and related compounds in their operations. The resulting effluents contain cyanide, thiocyanate, and other toxic ions, which are noxious to the environment and require removal before effluent discharging. Several methods are available for cyanide removal and/or detoxification. Natural degradation, alkaline chlorination, and oxidation with hydrogen peroxide are the most frequently used methods in full-scale operations. However, there are technical and economical concerns related to these methods, which make biological treatment processes a feasible alternative. The use of a trickling filter is suitable for treating wastewaters with load variation. It is a simple system, is easy to operate, and presents low energy requirements. In this type of bioreactor, it is possible to retain microorganisms with a slow growth rate, such as those responsible for cyanide degradation. The present work reports the results obtained in a fixed bed bioreactor in which were evaluated: (a) the influence of the recirculation ratio (0 to 0.75), (b) the role of a biomass in cyanide removal, and (c) the biomass characteristics. The experiments were carried out in a continuous pilot scale trickling filter, 7.4 m high, packed with plastic rings of polypropylene. The experimental set-up consisted of two identical units, one of which was used for tests without biomass (abiotic tests). The reactors were fed with a mixture of synthetic gold milling effluent and sewage, and the treatment efficiency was evaluated through the monitoring of the following parameters: chemical oxygen demand (COD), free cyanide, thiocyanate, copper, iron and zinc concentrations. The results indicated that it was possible to remove more than 90% of the free cyanide, thiocyanate, copper and zinc originally found in the influent. These removal efficiencies were obtained when the pilot bioreactor was operated without recirculation, and moreover, higher recirculation ratio brought about a decreasing in the pH of the influent, lowering the efficiency of zinc removal. It was observed that the microbial activity was found to be responsible for thiocyanate degradation and copper removal. The tests carried out in the reactor without biomass showed that the percentage of free cyanide removed by volatilisation was low (22.6%), and even lower when recirculation did not take place (7.7%). This fact confirms the important role, which the biomass plays in the degradation of this pollutant



**[39] APPLICATION OF ECAP - TECHNOLOGY FOR PRODUCING NANO- AND MICROCRYSTALLINE MATERIALS**

VI Kopylov - INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION (Series: NATO SCIENCE SERIES, PARTNERSHIP SUB - SERIES 3: HIGH TECHNOLOGY), 2000, Vol 80, pp 23-27 - NATO ADVANCED RESEARCH WORKSHOP ON INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION; MOSCOW, RUSSIA. AUGUST 2-7, 1999

Conditions for producing nano- and microcrystalline structures (NMC) by ECAP technology are described. The concept of "clear ECAP" for describing uniform ECAP deformation is introduced. A model of grain refinement and temperature-strain rate conditions of effective refinement is described.

**[38] SYNTHESIS AND CHARACTERIZATION OF NANOCRYSTALLINE TIAL BASED ALLOYS**

ON Senkov, FH Froes - INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION (Series: NATO SCIENCE SERIES, PARTNERSHIP SUB - SERIES 3: HIGH TECHNOLOGY), 2000, Vol 80, pp 43-48 - NATO ADVANCED RESEARCH WORKSHOP ON INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION; MOSCOW, RUSSIA. AUGUST 2-7, 1999

Gamma TiAl based alloys and a TiAl/Ti<sub>5</sub>Si<sub>3</sub> composite with nanocrystalline and submicrocrystalline structures were produced by mechanical alloying (MA'ing) and hot isostatic pressing (HIP'ing). MA'ing in a high-energy mixer mill led to severe plastic deformation and amorphization of the powder particles. The gamma -TiAl alloys produced from the amorphous powders had a nanocrystalline structure. The grain size increased when the HIP temperature and time increased. Grain growth in the TiAl alloys during annealing for up to 800 hours in the temperature range of 725 degreesC to 1200 degreesC was studied. No effect of the Ti<sub>5</sub>Si<sub>3</sub> phase particles on grain growth kinetics was found.

**[37] X-RAY STUDIES AND COMPUTER SIMULATION OF NANOSTRUCTURED SPD METALS**

IV Alexandrov - INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION (Series: NATO SCIENCE SERIES, PARTNERSHIP SUB - SERIES 3: HIGH TECHNOLOGY), 2000, Vol 80, pp 103-108 - NATO ADVANCED RESEARCH WORKSHOP ON INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION; MOSCOW, RUSSIA. AUGUST 2-7, 1999

The results of investigations of structure peculiarities in metals subjected to severe plastic deformation (SPD) are presented. Special attention is paid to X-ray investigations of microstructure evolution in bulk nanostructured samples of pure Cu obtained by SPD processing by torsion under high applied pressure and equal channel angular (ECA) pressing. Computer simulation is used to analyze the X-ray results.

**[36] STRUCTURAL CHANGES INDUCED BY SEVERE PLASTIC DEFORMATION OF FE- AND CO-BASED AMORPHOUS ALLOYS**

N Noskova, L Korshunov, A Potapov, N Tchernenko - INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION (Series: NATO SCIENCE SERIES, PARTNERSHIP SUB - SERIES 3: HIGH TECHNOLOGY), 2000, Vol 80, pp 115-120 - NATO ADVANCED RESEARCH WORKSHOP ON INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION; MOSCOW, RUSSIA. AUGUST 2-7, 1999

The amorphous alloys Fe<sub>81</sub>Si<sub>7</sub>B<sub>12</sub>, Fe<sub>81</sub>Si<sub>4</sub>B<sub>13</sub>C<sub>2</sub>, Fe<sub>64</sub>Co<sub>21</sub>B<sub>15</sub>, Fe<sub>73.5</sub>Cu<sub>1</sub>Nb<sub>3</sub>Si<sub>13.5</sub>B<sub>9</sub>, and Fe<sub>5</sub>Co<sub>70</sub>Si<sub>15</sub>B<sub>10</sub> prepared as strips by fast melt quenching on the rotating Cu disc were examined. The strips were 6-12 mm wide and 25-40 μm thick. The structure, coercivity and friction coefficient of these amorphous alloys after plastic deformation by tension, rolling, shear pressure, and dry sliding friction were analyzed. The samples underwent mechanical loading at 293 K in air at rates excluding heating of the material. Coercivity was shown to increase by a factor of ten or more at all types of deformation. The electron microscopic study of the structure of deformed strips revealed the presence of crystalline precipitates 2 to 50 nm in size having a relatively equiaxed shape in the amorphous matrix of the Fe-based alloys and a nearly plate shape in the Co-based alloys. The phases were identified from electron microdiffraction patterns of the alloys.

**[35] CALCULATION OF ENERGY INTENSITY AND TEMPERATURE OF MECHANOACTIVATION PROCESS IN PLANETARY BALL MILL BY COMPUTER SIMULATION**

EV Shelekhov, VV Tcherdyntsev, LY Pustov, SD Kaloshkin, IA Tomilin - INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION (Series: NATO SCIENCE SERIES, PARTNERSHIP SUB - SERIES 3: HIGH TECHNOLOGY), 2000, Vol 80, pp 139-145 - NATO ADVANCED RESEARCH WORKSHOP ON INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION; MOSCOW, RUSSIA. AUGUST 2-7, 1999

Mechanical alloying (MA) is one of the novel processes of alloy formation by severe plastic deformation of metallic components in high-energy ball mills. Analysis of the energy intensity and average temperature of the milling process in planetary ball mill was carried out by computer simulation. The dependences of energy dissipation and average temperature in a vial on the fill fraction of the Vial by balls, on the elasticity of ball's collision and their friction coefficient are determined. The results of computer simulation were compared with ones calculated using the analytical formula. The obtained results allow one to choose optimal modes of ball milling with regard to the specific character of concrete tasks.

**[34] NANOSTRUCTURE STATE AS NONEQUILIBRIUM TRANSITION IN GRAIN BOUNDARY DEFECTS IN SPD CONDITION**

OB Naimark - INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION (Series: NATO SCIENCE SERIES, PARTNERSHIP SUB - SERIES 3: HIGH TECHNOLOGY), 2000, Vol 80, pp 179-187 - NATO ADVANCED RESEARCH WORKSHOP ON INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION; MOSCOW, RUSSIA. AUGUST 2-7, 1999



**[33] TENSILE SUPERPLASTICITY IN NANOCRYSTALLINE MATERIALS PRODUCED BY SEVERE PLASTIC DEFORMATION**

RS Mishra, SX McFadden, AK Mukkerjee - INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION (Series: NATO SCIENCE SERIES, PARTNERSHIP SUB - SERIES 3: HIGH TECHNOLOGY), 2000, Vol 80, pp 231-240 - NATO ADVANCED RESEARCH WORKSHOP ON INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION; MOSCOW, RUSSIA. AUGUST 2-7, 1999

Tensile superplasticity has been observed in a number of severe plastic deformation (SPD) processed alloys with nanocrystalline microstructure. The observations of superplasticity in nanocrystalline materials are briefly reviewed with emphasis on the aspects that are different from superplasticity in microcrystalline materials. The temperature for onset of superplastic elongation coincides with microstructural instability. The important features include, high strain rate superplasticity in an aluminum alloy, low temperature superplasticity, extensive strain hardening and high flow stresses. A comparison of the experimental results with existing models shows the difference in superplastic deformation kinetics. The deformation mechanisms for microcrystalline materials are not simply scaleable to nanocrystalline range. It is difficult to establish the parameters for deformation mechanism because of grain growth. The observations of low temperature and high strain rate superplasticity in nanocrystalline materials with some unique features opens up new possibilities for scientific and technological advancements.

**[32] PLASTICITY AND WORK-HARDENING AT 300-4.2 K OF NANOSTRUCTURED COPPER AND NICKEL PROCESSED BY SEVERE PLASTIC DEFORMATION**

VZ Bengus, ED Tabachnikova, RZ Valiev, IV Alexandrov, VD Natsik - INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION (Series: NATO SCIENCE SERIES, PARTNERSHIP SUB - SERIES 3: HIGH TECHNOLOGY), 2000, Vol 80, pp 255-260 - NATO ADVANCED RESEARCH WORKSHOP ON INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION; MOSCOW, RUSSIA. AUGUST 2-7, 1999

Mechanical properties of bulk nanostructured Cu and Ni with 200 nm grains (manufactured by the equal channel angular pressing) are measured under uniaxial compression at 300, 77 and 4.2 K. Measured mechanical characteristics exceed several times those of coarse grained polycrystalline Cu and Ni.

**[31] COPPER GRAIN BOUNDARY DIFFUSION AND DIFFUSION INDUCED CREEP IN NANOSTRUCTURED NICKEL**

WR Kolobov, GP Grabovetskaya, MB Ivanov, RZ Valiev, TC Lowe - INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION (Series: NATO SCIENCE SERIES, PARTNERSHIP SUB - SERIES 3: HIGH TECHNOLOGY), 2000, Vol 80, pp 261-265 - NATO ADVANCED RESEARCH WORKSHOP ON INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION; MOSCOW, RUSSIA. AUGUST 2-7, 1999

**[30] STRUCTURE AND DEFORMATION BEHAVIOR OF SPD CU-BASED NANOCOMPOSITE**

W Buchgraber, RK Islamgaliev, YR Kolobov, NM Amirkhanov - INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION (Series: NATO SCIENCE SERIES, PARTNERSHIP SUB - SERIES 3: HIGH TECHNOLOGY), 2000, Vol 80, pp 267-272 - NATO ADVANCED RESEARCH WORKSHOP ON INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION; MOSCOW, RUSSIA. AUGUST 2-7, 1999

Severe plastic torsion straining was used in the present work for processing of the metal matrix Cu + 0.5%Al<sub>2</sub>O<sub>3</sub> nanocomposite. The mean grain size, particle size distribution and elastic strain were studied by TEM and XRD. It is shown that high ultimate strength (680 MPa) and microhardness (2300 MPa) as well as high thermal stability and electrical conductivity are the features of the nanocomposite samples. The decrease of the creep rate by an order magnitude and the increase of the time to failure by a factor of 4-5 is revealed in nanocomposite as compared the extruded sample.

**[29] SUPERPLASTICITY OF MECHANICALLY ALLOYED NANOCRYSTALLINE AND AMORPHOUS MATERIALS**

OM Smirnov, J Seung, IV Poustovalova - INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION (Series: NATO SCIENCE SERIES, PARTNERSHIP SUB - SERIES 3: HIGH TECHNOLOGY), 2000, Vol 80, pp 327-332 - NATO ADVANCED RESEARCH WORKSHOP ON INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION; MOSCOW, RUSSIA. AUGUST 2-7, 1999

**[28] INFLUENCE OF GRAIN BOUNDARY DIFFUSION FLUXES OF ALUMINUM ON STRENGTH PROPERTIES AND CREEP OF COPPER AND CU-0.9% VOL AL<sub>2</sub>O<sub>3</sub> NANOCOMPOSITE**

YR Kolobov, KV Ivanov, GP Grabovetskaya - INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION (Series: NATO SCIENCE SERIES, PARTNERSHIP SUB - SERIES 3: HIGH TECHNOLOGY), 2000, Vol 80, pp 339-344 - NATO ADVANCED RESEARCH WORKSHOP ON INVESTIGATIONS AND APPLICATIONS OF SEVERE PLASTIC DEFORMATION; MOSCOW, RUSSIA. AUGUST 2-7, 1999

**[27] SYNTHESIS AND CHARACTERIZATION OF NANOCOMPOSITE COATINGS**

J He, M Ice, EJ Lavernia - NANOSTRUCTURED FILMS AND COATINGS (Series: NATO SCIENCE SERIES, PARTNERSHIP SUB - SERIES 3: HIGH TECHNOLOGY), 2000, Vol 78, pp 131-148 - NATO ADVANCED RESEARCH WORKSHOP ON NANOSTRUCTURED FILMS AND COATINGS; SANTORINI, GREECE. JUNE 28-30, 1999

The synthesis of nanocomposite coatings is described in this paper. The nanocomposite feedstock powders are synthesized using mechanical milling, and the characteristics of the milled powders, i.e., morphology, agglomeration behavior, powder size, grain size and structural evolution during milling, are analyzed using X-ray diffraction, SEM and TEM. Using high velocity oxygen fuel (HVOF) spraying, the nanocomposite coatings are sprayed, and the microstructures and properties of the resulting coatings are characterized.



**[26] SOLID STATE LITHIUM SECONDARY BATTERIES USING AN AMORPHOUS SOLID ELECTROLYTE IN THE SYSTEM (100-X)(0.6LI(2)S CENTER DOT 0.4SIS(2)) CENTER DOT XLI(4)SIO(4) OBTAINED BY MECHANOCHEMICAL SYNTHESIS**

Authors Komiya R. Hayashi A. Morimoto H. Tatsumisago M. Minami T. - Solid State Ionics. 140(1-2):83-87, 2001  
Electrochemical cells were constructed using amorphous materials in the system  $(100 - x)(0.6\text{Li}(2)\text{S} \cdot 0.4\text{SiS}(2)) \cdot x\text{Li}(4)\text{SiO}(4)$ , obtained by mechanochemical synthesis, as an electrolyte,  $\text{LiCoO}_2$  as a positive electrode and indium as a negative electrode. Charge and discharge behaviors of the cells at a constant current were investigated to see the possibility for utilization as secondary batteries. Charge-discharge efficiency at the 1st cycle was more than 75% in the cells using the solid electrolytes synthesized by mechanical milling (MM) for more than 5 h. Charge-discharge curves of the cells using the amorphous materials milled for more than 10 h, were similar to those of the cells using the corresponding melt-quenched glass samples. The charge-discharge capacity decreased gradually from 90 to 70 mA h/g till about the 10th cycle, and became stable after the 10th cycle. The Coulombic efficiency of the cell showed almost 100%, except for the 1st and 2nd cycles. The amorphous materials synthesized by MM were concluded to work as the electrolyte for solid state lithium secondary batteries

**[25] RAMAN SPECTROSCOPIC STUDY ON PRESSURE-INDUCED AMORPHIZATION IN NANOCRYSTALLINE ANATASE (TiO<sub>2</sub>)**

Wang ZW. Saxena SK. - Solid State Communications. 118(2):75-78, 2001.  
A raman spectroscopic investigation was carried out to study the pressure-induced phase transformation in nanocrystalline anatase ( $\text{TiO}_2$ ) up to 37 GPa. As compared to a macrocrystalline solid which transforms to the  $\alpha$ - $\text{PbO}_2$  structure at 2.6-4.5 GPa, we found that the nano-anatase phase remains stable to pressures as high as similar to 24 GPa, and then transforms to an amorphous phase. The new amorphous phase is quenchable upon release of pressure to ambient condition. An increased surface energy may stabilize the nano-anatase, suppressing the appearance of anatase-to- $\alpha$ - $\text{PbO}_2$  phase transformation (2.6-4.5 GPa in bulk anatase), and then leads to the amorphization of nano-anatase under strong compression

**[24] SYNTHESIS OF ULTRAFINE ZIRCONIA POWDERS BY MECHANOCHEMICAL PROCESSING**

Dodd AC. Raviprasad K. McCormick PG. - Scripta Materialia. 44(4):689-694, 2001

**[23] FORMATION OF TERNARY CARBIDE Fe<sub>3</sub>Mo<sub>3</sub>C BY MECHANICAL ACTIVATION AND SUBSEQUENT HEAT TREATMENT**

Tsuchida T. - Journal of Materials Science. 36(7):1735-1740, 2001  
Ternary carbide,  $\text{Fe}_3\text{Mo}_3\text{C}$  was prepared from the powder mixture of  $\text{Fe}/\text{Mo}/\text{C} = 1/1/1$  which was ground for 3 h in a planetary ball mill and subsequently heated at a temperature as low as 700 degreesC, its amount increased with heating temperature. In contrast, when the 1 h-ground and unground samples were heated at 700-1000 degreesC,  $\text{Mo}_2\text{C}$  formed. From the results obtained about the effect of mixing ratio, grinding time and heating temperature of  $\text{Fe}/\text{Mo}/\text{C}$  samples on the formation of  $\text{Fe}_3\text{Mo}_3\text{C}$ , it was found that the formation of  $\text{Fe}_3\text{Mo}_3\text{C}$  strongly depends on the mixing homogeneity and the activated state of the particles of Fe, Mo and C components induced by mechanical grinding.  $\text{Fe}_3\text{Mo}_3\text{C}$  obtained belongs to a hard magnet, having saturation magnetization of 0.4 emu g<sup>-1</sup>, remanence of 0.13 emu g<sup>-1</sup> and coercivity of 200 Oe

**[22] MECHANICAL AND MAGNETIC PROPERTIES OF NI-CO DISPERSED Al<sub>2</sub>O<sub>3</sub> NANOCOMPOSITES**

Oh ST. Sando M. Niihara K. - Journal of Materials Science. 36(7):1817-1821, 2001  
Effects of the fabrication processing on the microstructure and properties of composites were investigated. High-density Ni-Co dispersed- $\text{Al}_2\text{O}_3$  ( $\text{Al}_2\text{O}_3/\text{Ni-Co}$ ) composites were obtained by hydrogen reduction and consolidated using hot pressing and pulse electric current sintering (PECS) of  $\text{Al}_2\text{O}_3$ ,  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  and  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  powder mixtures. Microstructural investigations of the hot-pressed composite fabricated using again wet/dry ball-milled powder mixture after calcination revealed that fine Ni-Co particles, about 145 nm in diameter, dispersed homogeneously at the matrix grain boundaries. In particular, fine microstructure of dispersion with the average size of 90 nm was realized for the specimen consolidated by PECS method. High strength of over 1 GPa and hardness of 19 GPa were measured for the nanocomposites prepared from the again ball-milled powder mixture. The ferromagnetism of nano-sized Ni-Co contributes to the magnetic properties of the composites. A change in the coercive force with dispersion size was observed. Also, the extent of magnetic response by an applied stress was strongly influenced by the size of Ni-Co particles. The relations between microstructure and mechanical as well as magnetic properties are discussed

**[21] COMBUSTION SYNTHESIS OF MECHANICALLY ACTIVATED POWDERS IN THE TI-SI SYSTEM**

Maglia F. Anselmi-Tamburini U. Cocco G. Monagheddu M. Bertolino N. Munir ZA. - Journal of Materials Research. 16(4):1074-1082, 2001

The effect of the mechanical activation of the reactants on the self-propagating high-temperature synthesis (SHS) of titanium silicides was investigated. SHS experiments were performed on reactant powders that were milled for different times. Mechanical activation was shown to have a large influence on the combustion characteristics, particularly on wave speed. A much weaker effect was observed on the products phase composition. Single-phase products were obtained only from  $\text{Ti}:\text{Si} = 1:2$  and  $\text{Ti}:\text{Si} = 5:3$  starting compositions. Observation of microstructural evolution in quenched reactions of  $\text{Ti}:\text{Si} = 1:2$  mixtures milled for relatively long times revealed that the combustion reaction was primarily a solid-state process restricted to a surface layer of the large Ti grains. A secondary process involving a solid-liquid interaction between solid Ti and melted Si was dominant in the post front region. The mechanical activation in this case took the role of increasing the contact surface between the reactants. A single reaction coalescence mechanism involving only liquid phases was proposed for the  $\text{Ti}:\text{Si} = 5:3$  composition. For this composition the apparent activation energy for the overall combustion process was determined (155 kJ mol<sup>-1</sup>) and was shown to be independent on the degree of mechanical activation of the reactants.

**[20] SYNTHESIS OF Nd<sub>2</sub>Fe<sub>14</sub>B POWDERS BY SPRAY-DRYING AND REDUCTION-DIFFUSION PROCESSES**

Dong XL. Kim BK. Choi CJ. Park KS. Zhang ZD. - Journal of Materials Research. 16(4):1083-1089, 2001  
The magnetic Nd-Fe-B powders were prepared by a mechanochemical method, including the processes of spray drying, debinding, milling, H<sub>2</sub> reduction, Ca reduction, and washing. The liquid solution dissolved with various metal salts was first spray-dried to prepare the precursor powders having uniformly dispersed Nd, Fe, and B components. The precursor



powders in turn were subjected to the subsequent processes. The particle size of the resultant Nd-Fe-B powders was about 1  $\mu\text{m}$ . Effects of the process parameters on phases, morphologies, microstructures, compositions, and thermal properties of the powders were investigated

**[19] STRUCTURE AND PHASE CHARACTERISTICS OF AMORPHOUS BORON-CARBON-NITROGEN UNDER HIGH PRESSURE AND HIGH TEMPERATURE**

Huang JY. Zhu YT. Mori H. - Journal of Materials Research. 16(4):1178-1184, 2001

An amorphous boron-carbon-nitrogen (a-BCN) phase was synthesized by ball milling of a mixture of hexagonal BN (h-BN) and graphite with a nominal composition of (BN)<sub>0.5</sub>C<sub>0.5</sub> in atomic ratio. Electron energy-loss spectroscopy studies indicated that the bonding of the a-BCN is in an sp<sup>2</sup> configuration and the mixing between the BN and the C species was achieved at a nanometer scale, but the a-BCN phase was more likely a mechanical mixture rather than a chemical mixture. High-pressure and high-temperature (HPHT) treatment at 7.7 GPa and 2300 degreesC of the a-BCN phase resulted in complete segregation of the carbon and BN species, forming a nanocrystalline composite material comprising cubic BN (c-BN), amorphous carbon, and turbostratic graphite. The grain size of the c-BN phase was about 70 nm. No mutual solubilities between c-BN and carbon were found, and the two different species (C and BN) were well separated. An epitaxial relationship, i.e., the (0002) planes of graphite being parallel to the (111) planes of c-BN, was also found. The formation of ternary BCN phases was never found in the present experiment. Our experimental results also suggest the possibility of synthesizing c-BN grains encapsulated with graphite under controlled HPHT conditions

**[18] MECHANOCHEMICAL REACTIONS IN MIXTURES OF LEAD OXIDES**

Zyryanov VV. Gusev AA. - Inorganic Materials. 37(3):257-263, 2001

The mechanochemical processes, including mechanochemical synthesis of mixed-valence lead oxides, in mixtures of PbO, Pb<sub>3</sub>O<sub>4</sub>, and PbO<sub>2</sub> were studied by x-ray diffraction. The results demonstrate that the reduction of Pb<sup>4+</sup> is accompanied by the formation of Pb<sub>2</sub>O<sub>3</sub>, Pb<sub>3</sub>O<sub>4</sub>, and possibly PbO<sub>1.37</sub>. Phases of variable composition close in structure to minium, Pb<sub>3</sub>O<sub>4</sub>, are shown to coexist during mechanical processing. The structure of the metastable oxide Pb<sub>3</sub>O<sub>4-x</sub> with Pb<sup>2+</sup> : Pb<sup>4+</sup> > 2(Pb<sup>2+</sup><sub>x2</sub>+Pb<sup>1-x4</sup>+O<sub>4-2x</sub>) is tentatively determined. The sequence of the observed transformations is interpreted in terms of the model for the reaction zone, with consideration for primary and secondary interactions

**[17] MECHANOCHEMICAL SYNTHESIS AND STRUCTURE OF NEW PHASES IN THE PB-V-O SYSTEM**

Zyryanov VV. Lapina OB. - Inorganic Materials. 37(3):264-270, 2001

The products of mechanochemical synthesis in the Pb-V-O system were characterized by x-ray diffraction and magnetic resonance spectroscopy techniques. The mechanical processing was found to yield only new crystalline compounds, Pb<sub>2.67</sub>V<sub>1.33</sub>O<sub>5.96</sub> and Pb<sub>3.5</sub>V<sub>4.5</sub>O<sub>14.75</sub>. Structural analysis of these phases revealed a low atomic density, mixed-valent states of the constituent cations, the presence of three of four types of vanadium polyhedra, and splitting of crystallographic sites, in line with the views on the mechanisms of mechanochemical reactions developed in the model for the reaction zone.

**[16] PHOTOLUMINESCENCE OF NANOSTRUCTURED PBTiO<sub>3</sub> PROCESSED BY HIGH-ENERGY MECHANICAL MILLING**

Leite ER. Santos LPS. Carreno NLV. Longo E. Paskocimas CA. Varela JA. Lanciotti F. Campos CEM. Pizani PS. - Applied Physics Letters. 78(15):2148-2150, 2001

This letter reports on a process to prepare nanostructured PbTiO<sub>3</sub> (PT) at room temperature with photoluminescence (PL) emission in the visible range. This process is based on the high-energy mechanical milling of ultrafine PbTiO<sub>3</sub> powder. The results suggest that high-energy mechanical milling modifies the particle's structure, resulting in localized states in an interfacial region between the crystalline PT and the amorphous PT. These localized states are believed to be responsible for the PL obtained with short milling times. When long milling times are employed, the amorphous phase that is formed causes PL behavior. An alternative method to process nanostructured wide-band-gap semiconductors with active optical properties such as PL is described in this letter.

**[15] CR(VI) GENERATION DURING MILLING**

Beukes JP. Guest RN. - Minerals Engineering. 14(4):423-426, 2001

An investigation of the dry milling of chromite at a ferroalloy producer revealed high Cr(VI) concentrations in the milled ore. This prompted further investigation into the possible generation of Cr(VI) during milling. Experiments conducted in the laboratory proved that Cr(VI) is generated during dry milling. This fact implies that the health of workers on and around such milling installations could be compromised. Leaching of Cr(VI) from settled dust originating from dry mills could also lead to soil and groundwater contamination. Due to the vast differences in toxicity of Cr(III) and Cr(VI) chemical analysis is often required to distinguish between these two oxidation states. However, pulverising or milling of samples during sample preparation, which is common practise for solids, could lead to misleading analytical results. This investigation proved that even material that initially contained no Cr(VI) could be regarded as toxic if treated incorrectly

**[14] AN EVALUATION OF THE FLOW BEHAVIOR DURING HIGH STRAIN RATE SUPERPLASTICITY IN AN AL-MG-Sc ALLOY**

Komura S. Horita Z. Furukawa M. Nemoto M. Langdon TG. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 32(3A):707-716, 2001

An Al-3 pct Mg-0.2 pct Sc alloy was fabricated by casting and subjected to equal-channel angular pressing to reduce the grain size to similar to 0.2  $\mu\text{m}$ . Very high tensile elongations were achieved in this alloy at temperatures over the range from 573 to 723 K, with elongations up to >2000 pct at temperatures of 673 and 723 K and strain rates at and above 10(-2) s(-1). By contrast, samples of the same alloy subjected to cold rolling (CR) yielded elongations to failure of <400 pct at 673 K. An analysis of the experimental data for the equal-channel angular (ECA)-pressed samples shows consistency with conventional superplasticity including an activation energy for superplastic flow which is within the range anticipated for grain boundary diffusion in pure Al and interdiffusion in Al-Mg solid solution alloys



**[13] EFFECTS OF SI AND NI POWDER REFINING ON THE FORMATION MECHANISM OF NICKEL SILICIDES INDUCED BY MECHANICAL ALLOYING**

Lee WH. Byun CS. Lee SH. Kim DK. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 32(3A):805-810, 2001

The synthesis of the Ni<sub>2</sub>Si, Ni<sub>5</sub>Si<sub>2</sub>, and NiSi phases has been investigated by mechanical alloying (MA) of Ni-33.3 at. pct Si, Ni-28.6 at. pct Si, and Ni-50 at. pct Si powder mixtures. As-received and 60-minute premilled elemental powders were subjected to MA. The average surface area of the premilled Ni powder particles, which had a flaky shape, was 3.5 times larger than that of the as received Ni powder particles, which had a spherical shape. The as-received Si powder was angular in shape and the mean particle size was 19.1 μm, whereas the mean particle size of the premilled Si powder was 10 μm. A self-propagating high-temperature synthesis (SHS) reaction, followed by a slow solid-state diffusion reaction, was observed to produce Ni silicide phases during MA of the elemental powders. The reactants and the product, however, coexisted for a long period of MA time. On the other hand, only the SHS reaction was observed to produce Ni silicides during MA of the premilled elemental powders, indicating that Ni silicides formed rather abruptly in a short period of MA time. The mechanisms and reaction rates for the formation of Ni silicides via MA appeared to be influenced by the elemental powder particle size and shape as well as the heat of formation of the products.

**[12] MECHANICAL MILLING OF GAS-ATOMIZED AL-NI-MM (MM = MISCH METAL) ALLOY POWDERS**

Hong SJ. Kim TS. Suryanarayana C. Chun BS. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 32(3A):821-829, 2001

Al-14Ni-14Mm (Mm = misch metal) alloy powders rapidly solidified by the gas atomization method were subjected to mechanical milling (MM). The microstructure, hardness, and thermal stability of the powders were investigated as a function of milling time using X-ray diffraction (XRD), transmission electron microscopy (TEM), and differential scanning calorimetry (DSC) methods. In the early stages of milling, a cold-welded layer with a fine microstructure formed along the edge of the milled powder (zone A). The interior of the powder remained unworked (zone B), resulting in a two-zone microstructure, reminiscent of the microstructures in rapidly solidified ribbons containing zones A and B. With increasing milling time, the crystallite size decreased gradually reaching a size of about 10 to 15 nm and the lattice strain increased reaching a maximum value of about 0.7 pct for a milling time of 200 hours. The microhardness of the mechanically milled powder was 132 kg/mm<sup>2</sup> after milling for 72 hours and it increased to 290 kg/mm<sup>2</sup> after milling for 200 hours. This increase in microhardness is attributed to a significant refinement of microstructure, presence of lattice strain, and presence of a mixture of phases in the alloy. Details of the microstructural development as a function of milling time and its effect on the microhardness of the alloy are discussed

**[11] ZIRCONIA-SPINEL COMPOSITES. PART I: SYNTHESIS OF POWDERS AND DENSE MATERIALS**

Quenard O. Laurent C. Peigney A. Rousset A. - Materials Research Bulletin. 35(12):1967-1977, 2000

MgAl<sub>2</sub>O<sub>4</sub> and x wt% ZrO<sub>2</sub>-MgAl<sub>2</sub>O<sub>4</sub> (1 less than or equal to x less than or equal to 30) composite powders were prepared by the urea combustion route. The powders were further ground by either ball milling or attrition to refine the grain size. Zirconia was found solely in the tetragonal form. Dense materials were prepared by hot pressing. The spinel matrix grains were submicronic in size. The ZrO<sub>2</sub> particles were homogeneously dispersed at the grain junctions of the matrix, and their average size increased from less than 0.20 μm to ca. 0.45 μm with increasing content of ZrO<sub>2</sub>. The formation of monoclinic ZrO<sub>2</sub> particles proceeded accordingly, being easier in materials with a finer matrix grain size (ex-attrition)

**[10] FAST AMORPHIZATION REACTION IN ZrNi SYSTEM PREPARED BY MECHANICAL ALLOYING**

Al-Hajry A. - Materials Research Bulletin. 35(12):1989-1998, 2000

The amorphous phase of ZrNi system was obtained by mechanical alloying (MA) of elemental crystalline powders in an inert atmosphere. The time required to achieve the amorphous state was 5 h. In contrast with all previous reports on this system, this amorphization time is considerably shorter. X-ray diffraction and differential scanning calorimetry (DSC) measurements were used to assess the amorphous state. Moreover, the comparison of the ZrNi alloy produced by MA with another prepared by fast quenching (FQ) confirmed that the final structure of the MA sample is fully amorphous. The reduced radial distribution function G(r) obtained by a Fourier transform of the structure factor S(Q) showed that a single amorphous phase formed at the end of the amorphization reaction. Further MA results in the recrystallization of the ZrNi alloy. Recrystallization takes place at a milling time even shorter than any reported milling time to obtain an amorphous phase in this system

**[9] MECHANICAL ALLOYING OF SM AND FE MIXTURES HAVING THE MOLAR RATIO OF 1/2 AND 1/3**

Thongmee S. Tang IM. Methasiri T. - Materials Research Bulletin. 35(13):2183-2188, 2000

The mechanical alloying of Sm-Fe mixtures of molar ratios 1/2 and 1/3 were carried out. The milled mixtures were subjected to a two-step annealing process; first annealing for 1 h at 900 degreesC in argon gas, followed by a second annealing for 4 h at temperatures between 300 and 450 degreesC (T-a2) in nitrogen gas. Both the X-ray diffraction (XRD) and Mossbauer studies indicate that heat treatment of both mixtures results in the growth of the 2-17 (Sm<sub>2</sub>Fe<sub>17</sub>N<sub>x</sub>) intermetallic. The coercivity of both milled mixtures increases as T-a2 is raised, while the specific magnetization of both are seen to decrease initially and then increase as T-a2 is raised further.

**[8] STUDY OF THE MECHANO-CHEMICAL TRANSFORMATION OF GOETHITE TO HEMATITE BY TEM AND XRD**

Gonzalez G. Sagarzazu A. Villalba R. - Materials Research Bulletin. 35(14-15):2295-2308, 2000

A study of the mechano-chemical transformation of goethite to hematite was carried out using transmission electron microscopy (TEM) and X-ray diffraction (XRD). Synthetic goethite was dry-ground in air for different times up to 104 h and



characterized with the aim of understanding the mechanisms taking part in the transformation, the phases formed, and particle distribution, morphology and size. It was found that the transformation is topotactic, without the formation of intermediate phases, and takes place by fragmentation of crystals, creation of twins, dehydration of goethite particles, and formation of voids. The voids are arranged in almost parallel striations, and are associated with the loss of hydroxyl groups and formation of hematite in small domains of equiaxial crystallites. The hematite crystal size is about 10 nm at the initial stages and after prolonged periods of grinding remains constant at about 19 nm.

**[7] PREPARATION OF FE-MO-C TERNARY CARBIDE BY MECHANICAL ALLOYING**

Zhu JJ. Jiang JH. Jacobsen CJH. Lin XP. - Journal of Materials Chemistry. 11(3):864-868, 2001.

The ternary carbide  $\eta$ -Fe<sub>3</sub>Mo<sub>3</sub>C was synthesized by mechanical alloying of elemental Fe, Mo, and graphite and subsequent heat treatment. The alloying process in the Fe-Mo-C system and the thermal stability of the ball-milled sample have been studied by X-ray powder diffraction and Mossbauer spectroscopy. It is found that alloying occurs in the Fe-Mo-C system during ball-milling. Initially, the milling process reduces the grain sizes of the pure elements and Fe-C alloying occurs to form an amorphous Fe<sub>3</sub>C-type phase. With increasing milling times, Mo diffuses into the Fe-C alloys, which accelerates the formation of the non-magnetic amorphous Mo-Fe-C alloy. The content of Mo in the amorphous Mo-Fe-C alloy increases with milling time. After 60 to 90 hours ball-milling, a crystallization reaction of the amorphous Mo-Fe-C alloy into  $\eta$ -Fe<sub>3</sub>Mo<sub>3</sub>C and Mo<sub>2</sub>C occurs. The ball-milled samples are composed of  $\eta$ -Fe<sub>3</sub>Mo<sub>3</sub>C, Mo<sub>2</sub>C, and residual amorphous Mo-Fe-C alloy together with a slight contamination from the WC balls. It is found that the reaction between Mo<sub>2</sub>C and the residual amorphous Mo-Fe-C phase occurs at low temperatures while WC reacts with  $\eta$ -Fe<sub>3</sub>Mo<sub>3</sub>C to form Fe<sub>3</sub>W<sub>3</sub>C at high temperatures. The samples annealed at high temperatures (> 1073 K) are composed of crystalline  $\eta$ -Fe<sub>3</sub>Mo<sub>3</sub>C-type phases ( $\eta$ -Fe<sub>3</sub>Mo<sub>3</sub>C with an isomorphous substitution of W for Mo) with a lattice constant of 11.117(8) Angstrom and an isomer shift of -0.21(1) mm s<sup>-1</sup>

**[6] THE INFLUENCE OF ADDITIVES DURING WET ULTRA-FINE GRINDING IN AGITATOR BEAD MILLS PART I: GENERAL PRINCIPLES AND EXPERIMENTAL**

Reinisch E. Bernhardt C. Husemann K. - Cfi, Ceramic Forum International/Berichte der Dkg (Deutsche Keramische Gesellschaft). 78(3):E38-E42, 2001

Additives that increase the repulsive interactions between particles can improve the flowability of a suspension. Thus, at constant energy 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 walls 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 particle interactions, rheological properties and comminution parameters are of interest, primarily in grinding techniques in nano-technology, since only through the addition of appropriate additives can a very large percentage of particle sizes in the range  $<1 \mu\text{m}$  be achieved.

**[5] DRY AND LUBRICATED WEAR RESISTANCE OF MECHANICALLY-ALLOYED ALUMINIUM-BASE SINTERED COMPOSITES**

Bermudez MD. Martinez-Nicolas G. Carrion FJ. Martinez-Mateo I. Rodriguez JA. Herrera EJ. - Wear. 248(1-2):178-186, 2001

The friction and wear behaviour of three mechanically alloyed aluminium-base materials, consolidated by an alternative powder-metallurgy process, has been studied with a pin-on-disk tribometer, sliding against AISI 52100 steel pins. Their tribological properties have been compared, using simple sintered aluminium, as standard material. Volume loss, under dry wear conditions, is very dependent on the measurement method. The more regular wear tracks, achieved under lubrication, allows the track-width method to be used with better accuracy. In general, unreinforced Al shows the lowest wear resistance, while composites with the highest hardness and second-phase Volume content are the most wear resistant, especially at high load. Unreinforced Al suffers a transition in wear mode, from mild to severe wear, at load 4.90 N<sup>-2</sup> in lubricated tests. The wear mechanism is of the adhesive/abrasive type

**[4] RAPID SOLIDIFICATION OF Ti-25 MOL%AL ALLOY BY PLASMA SPRAYING**

Uenishi K. Murase M. Kobayashi KF. - Materials Transactions Jim. 42(2):269-274, 2001

Mechanically alloyed Ti-25 mol%Al powders were low pressure plasma sprayed in order to produce nanostructured cut intermetallic compound on mild steel. Rapidly quenched sprayed layers with various cooling rates were formed by changing the substrate temperature and spray distance. Mechanically alloyed powders with convoluted structure of pure Al and Ti melted in the plasma flame and completely reacted to form  $\alpha$  (2) intermetallic compound. The relative density of the sprayed layer increased with the substrate temperature and an almost 100% dense layer was obtained in the case of a substrate temperature over 650 K. The microstructure of the sprayed layer consisted of equiaxed nano grains, of which size increased with substrate temperature as well. The grain size was reduced down to a minimum of about 200 nm at the substrate temperature of about 500 K. Effect of cooling rate on the grain size was estimated using Boswell's model, which agreed well with the experimental results.



**[3] THE EFFECT OF MECHANICAL ACTIVATION ON THE EXOELECTRON EMISSION PROPERTIES OF ACTIVATED CHARCOAL**

Klyuev VA. Kutuzova OA. Revina ES. Toporov YP. - Technical Physics Letters. 27(3):187-188, 2001.

The results of experiments indicate that the mechanical activation of a material leads to an increase in the total exoelectron emission current and in the number of emission peaks excited by heating the activated sample

**[2] ELECTRICAL PROPERTIES OF NEW ORGANIC COMPOSITES OBTAINED BY MECHANOCHEMICAL SYNTHESIS**

Smirani I. Lipiec R. Brau A. Farges JP. Graja A. - Journal of Materials Science. 36(5):1227-1230, 2001

We present new results on organic semiconductive and metallic composites obtained by direct solid-solid charge-transfer (CT) reaction. By this method, samples of arbitrarily large size are readily achievable. In the present study, we consider composites formed from the reaction between the following pairs of donors and acceptors: TTF and iodine, BEDT-TTF and TCNQ, BEDT-TTF and TCNE, BEDT-TTF and AuI as well as BEDT-TTF and AuI<sub>3</sub>. Most of the composites show semiconducting properties only. Two of them, however, (BEDT-TTF)/(AuI) and (BEDT-TTF)<sub>2</sub>/(AuI<sub>3</sub>), exhibit a metallic behavior.

**[1] SURFACE ALTERATION OF (VO)<sub>2</sub>P<sub>2</sub>O<sub>7</sub> BY ALPHA-Sb<sub>2</sub>O<sub>4</sub> AS A ROUTE TO CONTROL THE N-BUTANE SELECTIVE OXIDATION**

Ait-Lachgar-Ben Abdelouabad K. Rouillet M. Brun M. Burrows A. Kiely CJ. Volta JC. Abon M. - Applied Catalysis A-General. 210(1-2):121-136, 2001

The catalytic properties of (VO)<sub>2</sub>P<sub>2</sub>O<sub>7</sub>/alpha -Sb<sub>2</sub>O<sub>4</sub> mixed oxides system for n-butane mild oxidation have been investigated on two mechanical mixtures (M1 and M2) of the same well crystallized (VO)<sub>2</sub>P<sub>2</sub>O<sub>7</sub> (reference vanadyl pyrophosphate) with two different morphologies of alpha -Sb<sub>2</sub>O<sub>4</sub>. The M1 mixture of (VO)<sub>2</sub>P<sub>2</sub>O<sub>7</sub> with alpha -Sb<sub>2</sub>O<sub>4</sub> (1), prepared by oxidation of Sb<sub>2</sub>O<sub>3</sub>, leads to the oxidative dehydrogenation (ODH) of n-butane, whereas the M2 mixture of (VO)<sub>2</sub>P<sub>2</sub>O<sub>7</sub> With a commercial alpha -Sb<sub>2</sub>O<sub>4</sub> (2) (Aldrich) with a different morphology improves the maleic anhydride selectivity as compared to the reference (VO)<sub>2</sub>P<sub>2</sub>O<sub>7</sub> catalyst (synergetic effect). After reaction, no ternary VPSbO phase is detected by XRD and DTA and it was controlled that the two alpha -Sb<sub>2</sub>O<sub>4</sub> oxides are catalytically inactive. The (VO)<sub>2</sub>P<sub>2</sub>O<sub>7</sub> reference catalyst which produced only maleic anhydride as mild oxidation product shows by XPS a slightly oxidized surface (14% V<sup>5+</sup>-86% V<sup>4+</sup>). Contamination of the (VO)<sub>2</sub>P<sub>2</sub>O<sub>7</sub> phase by migration of Sb species occurs after catalytic reaction in the case of the M1 mixture as shown by XPS, LEIS and TEM-EDX analysis. XPS showed that (VO)<sub>2</sub>P<sub>2</sub>O<sub>7</sub> is partially superficially reduced (86% V<sup>4+</sup>-14% V<sup>3+</sup>). This feature is consistent with the decrease of acidity as observed by pyridine adsorption-desorption. In opposition with the M1 mixture, no contamination of the (VO)<sub>2</sub>P<sub>2</sub>O<sub>7</sub> phase is observed after catalytic reaction in the case of the M2 mixture. The XPS study shows, in this case, that (VO)<sub>2</sub>P<sub>2</sub>O<sub>7</sub> is partially oxidized (30% V<sup>5+</sup>-70% V<sup>4+</sup>) at a higher level than for the reference (VO)<sub>2</sub>P<sub>2</sub>O<sub>7</sub> catalyst. This situation is associated with the increase of selectivity observed for maleic anhydride (synergetic effect). The difference in the catalytic results for the two M1 and M2 mixtures, as compared to the (VO)<sub>2</sub>P<sub>2</sub>O<sub>7</sub> reference catalyst, can be explained by the alteration of the surface composition of (VO)<sub>2</sub>P<sub>2</sub>O<sub>7</sub> and the distribution of vanadium oxidation state due to different interaction between Sb<sub>2</sub>O<sub>4</sub> and (VO)<sub>2</sub>P<sub>2</sub>O<sub>7</sub>, depending on the orientation of the alpha -Sb<sub>2</sub>O<sub>4</sub>, crystals.



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

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# JRFM'2001

21 et 22 Mai 2001 - Amiens - France

**Thème 2001 :**

Influence de la mécanosynthèse sur les propriétés physico - chimiques des matériaux

**Contact :**

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JRFM'2001

Laboratoire de Réactivité et Chimie des Solides

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## JRFM'2001 (Programme Provisoire)

### Conférences Invitées

1) E. Gaffet

Mécanosynthèse et Activation Mécanique"

CNRS UMR 5060 Université de Technologie de Belfort Montbeliard (UTBM) - 90010 Belfort Cedex

2) M. Boudina

synthèse réactive des matériaux pour des applications aéronautiques à base de Ti-Al-Nb en utilisant de l'hydrogène.

3) Fumio Saito

Title: Mechanochemical Dissociation of HBB

Authors: Qiwu Zhang, Hiroki Matsumoto, Fumio Saito and Michel Baron\*

Affiliations: IAMP, Tohoku University (Japan), \*Ecole des Mines d'Albi (France)

### Conférences orales

11) J. Focf, A. Legris, R.S. de Figueiredo

"Expériences virtuelles de mécanosynthèse : quand la simulation dévoile ce que les examens réels cachent"

Laboratoire de Métallurgie Physique et Génie des Matériaux - (L.M.P.G.M.) - UMR CNRS 8517 / Université des Sciences et Technologies de Lille

10) S. Galdeano\*, M-H. Mathon\*, L. Chaffron\*\* et C-H. de Novion\*

"Etude des corrélations entre les conditions de broyage et la nanostructure dans le composé magnéto-résistif Cu<sub>80</sub>(Fe<sub>0.3</sub>Co<sub>0.7</sub>)<sub>20</sub>"

\*Laboratoire Léon Brillouin (CEA/CNRS), CEN Saclay, 91191 Gif-sur-Yvette

\*\* DTA/DECM/SRMP, CEN Saclay, 91191 Gif-sur-Yvette

9) Sylvie Begin-Colin, T. Girot, G. Le Caër, F. Radjai, X. Devaux

"Mécanismes et modélisation de transformations de phase induites par broyage dans TiO<sub>2</sub>"

Laboratoire de Science et Génie des Matériaux Métalliques

Ecole des Mines - 54 042 Nancy Cedex

8) J.M. Le Breton, G. Khelifati, L. Aymard et J. Teillet

"Broyage réactif sous hydrogène d'alliages Nd-Fe-B : destruction et recombinaison de la phase Nd<sub>2</sub>Fe<sub>14</sub>B"

Laboratoire de Magnétisme et Applications, Groupe de Physique des Matériaux

UMR CNRS 6634, Faculté des Sciences de Rouen

76821 Mont St Aignan Cedex

7) A. Fnidicki, C. Lemoine, J. Teillet

Effets de la contamination en oxygène et en azote gazeux sur les propriétés structurales et magnétiques des alliages Fe-Cr obtenus par mécanosynthèse.

Magnétisme et Applications - UMR 6634 CNRS

Université de Rouen - 76521 Mont Saint Aignan Cédex

6) B. Chevalier, J-L. Bobet et J. Etourneau

Influence du broyage énergétique sur les propriétés magnétiques d'intermétalliques à base de gadolinium et de manganèse

B. Chevalier, J-L. Bobet et J. Etourneau.

ICMCB - CNRS [UPR 9048]

Groupe IV : Matériaux Magnétiques et Déterminations Structurales

Université Bordeaux I - Avenue du Dr. A. Schweitzer - 33608 Pessac (France)

5) Sophie Soiron, Cyrille Lenain, Luc Aymard, F. Chevallier

Graphite broyé sous hydrogène ou sous oxygène: propriétés électrochimiques

4) Michel Baron, Alain Chamayou, et Alexandre GIL

Centre Poudres et Procédés - Ecole des Mines d'Albi Carmaux

Campus Jarlard - Route de Teillet - 81013 ALBI CT Cedex 09

3) G. Saint-Ayès, L. Chaffron, G. LeCaër, G. Martin, J. Viet, G. André

"Usure des roues de TGV : une approche de type alliage forcé"

SRMP/DECM / Bât. 520

CEA Saclay

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2) Raphaël Janot et, Daniel Guérard

sur la synthèse de nanoparticules de maghémite par broyage mécanique

1) E Gaffet

"Nanomateriaux : Aspects Technico - Economiques"

CNRS, - Groupe "Nanomateriaux"

Université de Technologie de Belfort Montbeliard (UTBM)

### Posters

G. Bertrand, C. Meunier, S. Vives, E. Gaffet

Activation Mécanique de Précurseurs pour la Projection Thermique

(UTBM, CNRS, CREST/UFC)



Raphaël JANOT et, Daniel Guérard  
La préparation, par mécanosynthèse d'hydrures de rubidium et de césium



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**Technical Announcement**

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**VARIO-PLANETARY MILL "pulverisette 4"**

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.

This is the ideal mill for mechanical activation and alloying. The main applications are in the field of materials research and, of course, wherever a powerful, innovative planetary mill is required.

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
- Emulation of various ball mills
- Variably adjustable pressure on sample (friction and/or impact)
- Final fineness << 1 µm
- Simultaneous grinding in up to 4 small or 2 large grinding bowls
- Quick, secure fastening of the grinding bowls
- Ease of cleaning

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