



**RESEAU FRANÇAIS DE  
MECANOSYNTHESE**

**Lettre N°85**

**Avril 2002**

**186 Groupes de Recherche  
(dont 112 à l'étranger / 34 Pays)**

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

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=====  
**Le RFM fête ses 7 années d'existence !!!**

**The RFM is 7 year old !!!**

**As for children, is it now a question of maturity ??**

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

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

Eric GAFFET

CNRS UMR5060 « Métallurgies et Cultures »

Nanomaterials Research Group

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

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

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 **20 Euros (20€)**

The check has to be to the order to : Réseau Français de Mécanosynthèse

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Le site web du RFM est :

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

Rubrique Pages Sciences et Techniques pour l'Ingénieur (Rubrique Sciences)

⇒ vous y trouverez les anciennes lettres du RFM (accessible par Adobe Acrobat), les statuts du RFM ainsi que les annonces concernant les JRFM'2001 et quelques éléments mis à jour régulièrement concernant les derniers résultats dans ce domaine.

Lettre RFM N°85 - Avril 2002

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

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## Congress and School Announcements

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### Nano 2002

16 - 21 Juin 2002  
Orlando, Florida - USA  
Website : <http://www.nano2002.com/>

### NANO-7 / ECOSS-21

#### 7th International Conference on Nanometer-Scale Science and Technology & 21st European Conference on Surface Science

June 24-28,  
in Malmö, Sweden.  
Website : <http://www.malmo-congress.com/nano-ecoss.html>  
(Deadline for abstract submission is **February 17**)

### 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>

### ICSTR

INTERNATIONAL CONFERENCE ON SOLVO-THERMAL REACTIONS  
July 22-26, 2002 - Hilton East Brunswick / East Brunswick, New Jersey,  
More information on this meeting can be found at <http://www.ICSTR.rutgers.edu/>  
or by contacting Professor Richard E. Riman at Rutgers University via  
[riman@email.rci.rutgers.edu](mailto:riman@email.rci.rutgers.edu)/732-445-4946(v)/732-445-6262 (f).

### RQ11

Rapidly Quenched and Metastable Materials  
25-30 August 2002  
Department of Materials, University of Oxford, UK  
Contact: RQ11 Conference Organiser, Beggars Roost, Channels End Road,  
Comworth Bedford MK44 2NS, U.K.  
Tel: +44 (0) 1234 378862  
Fax: +44 (0) 1234 376219  
E-mail: [mailto:rq11@materials.ox.ac.uk](mailto:mailto:rq11@materials.ox.ac.uk)  
Website: <http://www.materials.ox.ac.uk/rq11>

### 10th European Symposium on Comminution

Heidelberg from 2-5 September 2002.  
Org. European Federation of Chemical Engineering  
Full information available at <http://www.comminution2002.de>

### 8th ICCPS

8th International Conference on Ceramic Processing Science  
Hamburg - Sept. 2<sup>nd</sup> - 5, 2002.

The conference will focus on novel processing of advanced structural and functional ceramics and ceramic composites. The program will favor the most recent developments in this presented in only 10 topical sessions:

1. New Concepts for Economic Production of Powders of High Purity, Reactivity and Ease of Handling
2. Novel Powder Processing and Non-Conventional Shaping (Nanoprocessing, Cellular Structures, etc.)
3. Solution Processing (Thin Film Deposition, Soft Solution, Polymer-Derived, etc.)
4. Biomimetic Structuring (Biotemplates, Biomineralization, etc.)
5. Novel Reaction Forming (Controlled SHS, Reactive Casting, in situ Processing, etc.)
6. Computer-Controlled Shaping and Structuring (Rapid Prototyping, Solid Free-Forming, Controlled Heterogeneities, etc.)
7. Tailoring of Synergy Ceramic Microstructures (LTCC, Self-Sensing Devices, Smart Structures, MEMS, etc.)
8. Grain Boundary Engineering (Grain-Boundary-Free Microstructures, Directed Eutectics, Advanced Electroceramics, etc.)
9. Micromechanics of Composite Synthesis (Transient and Residual Stresses, Constrained Sintering, etc.)
10. COST 528: Chemical Solution Deposition of Thin Films

website : <http://www.tu-harburg.de/gk/8th-ICCPS>

### ISMANAM-2002

International Symposium on  
Metastable, Mechanically Alloyed and Nanocrystalline Materials  
Seoul, Korea, 8-12 September, 2002.  
Web site : <http://anu.andong.ac.kr/~ismanam/>



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L. A. C. A. M. E – 2. 0. 0. 2  
EIGHTH LATIN AMERICAN CONFERENCE  
ON APPLICATIONS OF THE MÖSSBAUER EFFECT  
PANAMA, 22-27, SEPTEMBER, 2002.  
E-mail: <mailto:lacame2000@fisica.ciens.ucv.ve>  
<http://www.up.ac.pa/Eventos/lacame2002/inicio.htm>  
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**Matériaux 2002**

Tours - France

21- 25 Octobre 2002

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

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

Les JRFM'2002 seront intégrées dans le cadre du Congrès

**Matériaux 2002** (Tours – France, du 21 au 25 Octobre 2002)

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

Symposium 1 :

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



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

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

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

**Jury:**

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

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

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**F. Dore**

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

Université de Grenoble - 13 Novembre 2001

**Jury :**

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

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**Raphaël JANOT**

Université de Nancy I – 24 octobre 2001

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

**Jury :**

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

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**Thierry Girot**

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

Thèse INPL, 15 Octobre 2001

**Jury :**

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

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**Sébastien Lehnard**

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

Université de Metz - 5 octobre 2001-08-23



**Jury :**

R. Schwarzer (Rapp.), E. Gaffet (Rapp.), JP Morniroli, V Skrotzi, R. Baccino, A. Hazotte,  
F. Wagner (Dir. Thèse), Th. Grosdidier (Co. Dir. Thèse)

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**Nathalie Bouad**

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

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

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

**Jury :**

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

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**Cooperative Research on Related Areas**

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

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

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

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

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

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

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

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



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

### Proposal from 11/03/2002

Announcing Ph.D. and postdoc positions:

A Ph.D. and a postdoc position is available in a joint project of the Fritz-Haber-Institut der Max-Planck-Gesellschaft (Matthias Scheffler, <http://www.fhi-berlin.mpg.de/th/th.html>) and The Pennsylvania State University (Henry C. Foley, <http://fenske.che.psu.edu/Faculty/Foley/index.html> and Kristen Fichthorn, <http://fenske.che.psu.edu/Faculty/Fichthorn/index.html>).

#### Theme:

The role of nano-porous carbon in dehydrogenation and oxidation catalysis

#### Project summary:

This is a highly interdisciplinary project involving, e.g. extensive density-functional theory calculations and Statistical Mechanics simulations (with DFT derived parameters).

The catalytic production of styrene is one of the most important processes in chemical industry (a key process for making most plastics). Recently it could be shown that the typically employed iron-oxide catalyst is in fact not the active material, but the true catalyst is formed during the induction period: The material that is actually doing the catalysis apparently is "nano porous carbon". This consists of strained and twisted graphite sheets that have a lot of defects (in particular five-fold rings). [http://www.fhi-berlin.mpg.de/th/Slides/Scheffler\\_transparencies\\_pdf/npc-2002.pdf](http://www.fhi-berlin.mpg.de/th/Slides/Scheffler_transparencies_pdf/npc-2002.pdf) summarizes some aspects of our recent work.

The planned research may start with an analysis of the chemical reactivity of nanotubes of different diameter and of the different regions of nano porous carbon. At a later step it is planned to model the dynamics of the flow of steam + ethylbenzene at such carbon structures, and the process of ethylbenzene dehydrogenation.

#### Where:

The student/postdoc will spend some time in Berlin and some time in the US. Details will be decided along the progress of the work.

We are looking for computational physicists, chemists, or chemical engineers. Good background in electronic structure theory, thermodynamics, and statistical mechanics is important

#### Please send your application material to:

Matthias Scheffler  
Fritz-Haber-Institut phone : ++49-30-8413 4711  
der Max-Planck-Gesellschaft fax : ++49-30-8413 4701  
Faradayweg 4-6 e-mail: [scheffler@fhi-berlin.mpg.de](mailto:scheffler@fhi-berlin.mpg.de)  
D-14 195 Berlin-Dahlem / Germany  
WWW: <http://www.fhi-berlin.mpg.de/th/th.html>

### Proposal from 8/03/2002

The Laboratory for Neutron Scattering (ETH Zurich & PSI Villigen) has an open position for a scientist to work in the field of powder neutron diffraction. The position is on a contractual basis and has a duration of 2-3 years with an option for prolongation. The starting date is November 1, 2002.

Research Scientists (Physicists, Chemists, Crystallographers) are invited to apply for this position

#### • Your tasks:

- Responsibility for the operation and further development of a powder neutron diffractometer at the spallation source SINQ at PSI Villigen.
- Co-operation with guest scientists in their experiments at SINQ.
- Performance of your own research projects.

#### • Your profile:

- You are a graduated research scientist (PhD) with some years' experience in the field of neutron scattering, particularly with neutron diffraction.
- You have some practical knowledge of computing and cryogenics.
- You are willing to work in a team and to communicate (establishing a professional relationship with guest scientists) as well as to work flexible hours.

#### • For further information

please contact Prof. Dr. A. Furrer, phone: +41-56-3102088, fax: +41-56-3102939, e-mail: [albert.furrer@psi.ch](mailto:albert.furrer@psi.ch)  
Please send applications with C.V., a list of publications and the names of two academic referees no later than by April 30, 2002, to: Prof. Dr. A. Furrer, Laboratory for Neutron Scattering, CH-5232 Villigen PSI, Switzerland.

### Proposal from 28/01/2002

The Laboratory for Neutron Scattering (Paul Scherrer Institute and ETH Zuerich) announces three openings for research scientists (physicists, chemists, crystallographers) at the Swiss Spallation Neutron Source 'SINQ' (<http://sinq.web.psi.ch/>). The posts represent excellent opportunities for postdoctoral scientists to develop their expertise, broaden experience and interact with scientists from many countries. We are looking for

A - Responsible for the new SANS-II facility (joint venture between PSI and Risoe National Lab.)

(<http://sinq.web.psi.ch/sinq/instr/sans2.html>)

(at your earliest convenience)

Reference Number: 3302A

B - Co-responsible for the new triple-axis spectrometer Rita-I (joint venture between PSI and Risoe National Lab.)

(<http://sinq.web.psi.ch/sinq/instr/rita1.html>)



(from 01/07/2001)

Reference Number: 3302B

C - Co-responsible for the triple-axis spectrometer TASP (<http://sinq.web.psi.ch/sinq/instr/tasp.html>)

(from 01/10/2002)

Reference Number: 3303A

Your tasks would be:

- Responsibility for the installation/operation and further development of the instruments, in particular co-operation with guest scientists in their experiments at SINQ
- Performance of neutron scattering experiments at SINQ for your own research projects
- Development and implementation of analytical software for the instruments

For further information (but not applications) please contact for

(A) Dr. S. Janssen, phone: +41-56-310-2875, e-mail: <mailto:stefan.janssen@psi.ch>,

(B) Dr. J. Mesot, phone: +41-56-310-4029, e-mail: <mailto:joel.mesot@psi.ch>

(C) Dr. P. Allenspach, phone: +41-56-310-4029-2527, e-mail: <mailto:peter.allenspach@psi.ch>

Information about the Laboratory for Neutron Scattering and about SINQ can be obtained from the following web pages:

<http://lns.web.psi.ch/>

<http://sinq.web.psi.ch/>

Please send applications with C.V., a list of publications and the names of two academic referees quoting reference 3302A, 3302B or 3303A, no later than by March 15, 2002 to: PAUL SCHERRER INSTITUT, Human Resources, CH-5232 Villigen PSI, Switzerland.

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**From Prof. H.E. Schaefer**

**(22/01/2002)**

In the framework of the 5th European Research and Development Program, the Institut fuer Theoretische und Angewandte Physik, Stuttgart University, Research Group of Prof. H.-E. Schaefer

(<http://www.itap.physik.uni-stuttgart.de/~gsweb/english/index.html>)

has been selected a Marie Curie Training Site and offers a one year position :

**Marie Curie Training Site**  
**Doctoral Training / Post-Doc Positions**  
at  
Stuttgart University, Germany  
Institute für Theoretische und Angewandte Physik  
Research Group Prof. H.-E. Schaefer

in the field of

Nanostructured Materials :  
Atomic Transport Properties for the Synthesis and  
Characterization of Novel Soft and Hard Magnets

(Contract No.: HPMT-CT-2001-00224)

Applicants are invited for a 12 month term as a research fellow supported by individual fellowships of the Marie Curie fellowship scheme. The successful candidates will be involved in the synthesis and processing of novel nanostructured materials and composites for soft magnets (Finemet-type) and hard magnets (FeNdB-type), as well as with the investigation of their microstructure, magnetic, and diffusional properties. The gas-phase condensation technique with subsequent compaction under high pressure is used for the production of highly dense nanocrystalline materials. Basic material characterisation will be carried out by x-ray diffraction, differential scanning calorimetry, optical microscopy, and atomic resolution electron microscopy (HRTEM). In addition, several instruments for characterisation of magnetic materials are available. These experimental techniques allow the investigation of a number of phenomena, including: order-disorder transformations, transformation kinetics, phase transitions, and relaxation processes. Furthermore, diffusion studies using the radioactive tracer technique are carried out in order to study the atomic transport properties in nanocrystalline structures.

The candidates have to satisfy the basic criteria of the training scheme as outlined on the Marie Curie Host Fellowship Web site <http://www.cordis.lu/improving/fellowships/home.htm>. As the fellowship forms part of a higher degree project, the candidates should be registered as full-time Ph.D. research students in a well recognized institution of the EC. The research interest of the candidates should be in at least one of the following fields: solid state physics, materials science including synthesis and characterisation of materials, mechanical and magnetic properties of advanced materials, and structural studies.

The group closely cooperates with the Max-Planck-Institut für Metallforschung, Stuttgart. This collaborative character of the research training provides an additional international profile to the education of the fellows increasing their interaction and eventually their active collaboration with research institutions in different European countries.



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

Applicants, also Post-Docs, should contact us for further information:

e-mail Prof. H.-E. Schaefer <mailto:schaefer@itap.physik.uni-stuttgart.de>  
Dr. W. Sprengel <mailto:sprengel@itap.physik.uni-stuttgart.de>  
phone: +49-711-685-5261 +49-711-685-5192  
FAX +49-711-685-5271 +49-711-685-5271  
<http://www.itap.physik.uni-stuttgart.de/~gsweb/english/index.html>

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**De L. Aymard CNRS / Amiens (France)**

**1/10/2001**

**Pour la Rentrée Universitaire 2001 - 2002**

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

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

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

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

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

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## Bibliographie Récente

### Livres ou "Special Issues"

(21/06/2001)

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

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

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

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

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

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

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

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

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

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

Volume 59 until 60 [Interfaces and Plasticity] and

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

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

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(07/06/2001)

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

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

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

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

(28/05/2001)

**Advanced Ceramic Materials**

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

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

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

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

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

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

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

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

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



Lettre RFM N°85 - Avril 2002  
Corresp. : <mailto:Eric.Gaffet@utbm.fr>

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

## MACROMOLECULAR MECHANOCHEMISTRY

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

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

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

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

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

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

(9/06/2000)

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

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

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

Preface

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

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

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

(3/02/2000)

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

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

Contents

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

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

Energy transfer and energy maps;

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

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

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



Lettre RFM N°85 - Avril 2002  
Corresp. : <mailto:Eric.Gaffet@utbm.fr>

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

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

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

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

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

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

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

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

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

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



## Patent / Brevet

### ONE STEP SYNTHESIS AND CONSOLIDATION OF NANOPHASE MATERIALS

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

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

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

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

ou encore pour la version brevet d'application

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

## Périodiques

### [53] INVESTIGATIONS OF THE FORMATION MECHANISM OF NANOSTRUCTURED NbAl<sub>3</sub> VIA MASHS REACTION

V. Gauthier, F. Bernard, E. Gaffet, D. Vrel, M. Gailhanou, J.P. Larpin - Intermetallics, 10 (2002) 377 - 389

The nanostructured NbAl<sub>3</sub> intermetallic compound was synthesized using the mechanically - activated self propagating high temperature synthesis (MASHS) technique. This process results from the combination of two steps : a short duration ball milling of a pure elemental Nb + 3 Al powder mixture followed by a self propagating high temperature synthesis (SHS) reaction induced by the Nb + 3Al reaction exothermicity. Synchrotron time - resolved XRD coupled with a 2D infrared camera were used to investigate the structural and thermal evolutions during the SHS reaction, and to study in situ the mechanism of NbAl<sub>3</sub> formation. The influence of the incoming heat flux and the mechanical activation effect on the phase transformation kinetics induced by the SHS process were studied. Owing to the temporal resolution of 100 and 200 ms between two consecutive diffraction patterns and IR images, respectively, it was shown that solid niobium reacts with aluminium via heterogeneous nucleation reaction, to form the NbAl<sub>3</sub> compound by successive combustion fronts.

### [52] THE MECHANICALLY ACTIVATED COMBUSTION REACTION IN THE Fe - Si SYSTEM : IN SITU TIME - RESOLVED SYNCHROTRON INVESTIGATIONS

C. Gras, N. Bernsten, F. Bernard, E. Gaffet - Intermetallics, 10 (2002) 271 - 282

Mechanical high energy ball milling of Fe + 2Si elemental powder mixtures was used to activate self sustaining combustion reaction in the case of iron disilicide synthesis. The reaction path as well as the influence of the microstructural parameters on phase transformation have been investigated in details. Time - resolved X - ray diffraction (TRXRD) using the fast recording kinetics offered by the synchrotron radiation was coupled to an infrared camera in order to study the internal structure of the combustion wave. The crystallite size and the amount of mechanical induced phases play an important role during the combustion : the reaction path and the end product composition mainly depend on the degree of mechanical activation (i.e. shock power and ball milling duration). Beta - FeSi<sub>2</sub> is formed during a slow diffusion process in the post combustion zone. The polyinterfaces created at a nanometric scale during the mechanical activation stage are responsible for this peculiar behavior.

### [51] REACTIVE MILLING AND MECHANICAL PROPERTIES OF NiAl COMPOSITE WITH HfC DISPERSOIDS

Yang FB. Guo JT. Zhou JY. - Journal of Materials Science & Technology. 18(1):59-62, 2002

A NiAl-based composite with HfB<sub>2</sub> dispersed particles has been synthesized by mechanical alloying of Ni, Al, Hf and C powders. The formation mechanism of NiAl-HfC during milling can be attributed to two chemical reactions: Ni+Al->NiAl+DeltaH; Hf+C->HfC+DeltaH, induced by mechanical collision in a certain period of time, which results in an abrupt exothermic reaction. Hot pressing (HP) and hot isostatic pressing (HIP) have been used to make the NiAl-10HfC compacts near fully dense. Compressive testing from room temperature to 1000degreesC indicated that the yield stress of NiAl-10HfC composite is 3similar to4 times higher than that of cast NiAl and correspond to the MA NiAl-10TiB(2) composite. In the meantime, yield strength at high temperature is dependent on strain rate, and deformation is controlled by diffusion mechanism

### [50] THE ANNEALING BEHAVIOR OF NANOCRYSTALLINE FERRITE FABRICATED BY BALL MILLING PURE FE [CHINESE]

Yin J. Zhou CR. Wang JQ. Hu ZQ. - Chin Shu Hsueh Pao: Acta Metallurgica Sinica. 38(1):23-26, 2002

The annealing behavior of nanocrystalline ferrite in pure Fe (0.004%C) has been studied through morphology observation and microhardness measurements. A relatively high thermal stability of the nano-ferrite formed by ball milling was observed. The irregular grain boundaries of nano-ferrite annealed at high temperature were attributed to that the grain growth of nanocrystalline ferrite took place by coalescence of neighboring grains. Although the microhardness of nano-ferrite decreased with increasing annealing temperature, a much higher hardness than that of the recrystallized work-hardened structure was still maintained under the same annealing condition. This may be attributed to the smaller grain size in the prior nano-ferrite region



**[49] MICROSTRUCTURAL EVOLUTION ON BALL-MILLING ELEMENTAL BLENDS OF NI, AL AND TI BY RIETVELD'S METHOD**

Dutta H. Pradhan SK. De M. - Materials Chemistry & Physics. 74(2):167-176, 2002

The nickel-base intermetallics with Al and Ti have been extensively studied in the recent years due to their promising engineering applications specifically in the field of high temperature due to high corrosion resistance and excellent high temperature strength. The most serious negative features of such ordered intermetallics are their grain boundary brittleness. Ductility of these materials can be improved by several methods. One of them is by reducing long-range ordering or introducing disordered phases in these materials. In the present work, Ni-base ternary Ni-Ti-Al system has been prepared with an intention to produce a composite intermetallic with disordered Ni-Ti-Al alloy (ductile) and Ni-Ti and Ni-Al ordered intermetallics. After several hours of milling of constituent metal powders in a planetary ball mill, the expected phases have finally appeared in the nanocrystalline form. The structural modifications at different stages of milling have been characterized in terms of several microstructure parameters like, particle size, microstrains, stacking faults, dislocation density, etc. and quantitative estimations of individual phase have been made by employing Rietveld's powder structure refinement methodology

**[48] HYDROXYAPATITE NANO SOL PREPARED VIA A MECHANOCHEMICAL ROUTE**

Nakamura S. Isobe T. Senna M. - Journal of Nanoparticle Research. 3(1):57-61, 2001

Well-dispersed sol with crystalline hydroxyapatite (HAp) was obtained directly by milling a mixture comprising Ca(OH)<sub>2</sub>, an aqueous solution of H<sub>3</sub>PO<sub>4</sub> and a dispersant, an ammonium salt of polyacrylic acid. The average crystallite size of HAp was below 20 nm. Ca/P molar ratio of the product was 1.51 +/- 0.04, i.e. Ca deficient from stoichiometric HAp. Minimum apparent viscosity was attained at a dispersant concentration 0.92wt% of sol. An as-milled sol was diluted by a factor 10-2.61 solid wt% to give a Newtonian fluid of 2 mPa s. From the diluted sol, we obtained a few mum thick dense film of HAp by dip coating on the slide glass precoated by chitosan

**[47] THE MICROSTRUCTURE AND SUPERPLASTIC PROPERTIES OF MECHANICALLY MILLED Ti-6Al-4V+0.5% BORON**

TMT Godfrey, A Wisbey, A Brown, R Brydson, C Hammond - TITANIUM ALLOYS AT ELEVATED TEMPERATURE: STRUCTURAL DEVELOPMENT AND SERVICE BEHAVIOUR (Series: MICROSTRUCTURE OF HIGH TEMPERATURE MATERIALS), 2001, Vol 755, Iss 4, pp 165-175

Superplastic forming of conventional titanium alloy sheet is limited commercially by the relatively long cycle times imposed by the high temperatures and slow strain rates required for forming. In order to minimise cycle times material with a fine grain size is required to enable increases in the forming rate and/or reductions in the deformation temperature. This study details an investigation of the production of Ti-6Al-4V + 0.5%B with a nanocrystalline grain size which was produced by mechanical milling. The material was consolidated by hot isostatic pressing at a range of temperatures during which TiB was formed by an in situ reaction between the titanium and the boron. The aim of (lie TiB was to pin the fine grain size of the titanium produced by mechanical milling. The consolidated material was hot tensile tested at a range of temperatures and strain rates. A superplastic elongation of 360% was achieved when testing at 900 C at a strain rate of 6 x 10(-2)s(-1) compared with 220% for conventional Ti-6Al-4V Sheet.

**[46] X-RAY DIFFRACTION STUDY OF PRESSURE-INDUCED AMORPHIZATION IN Lu-2(WO<sub>4</sub>)<sub>3</sub>**

Liu H. Secco RA. Imanaka N. Adachi G. - Solid State Communications. 121(4):177-180, 2002.

The effect of pressure on the crystal structure of Lu-2(WO<sub>4</sub>)<sub>3</sub> has been studied up to 8.0 GPa using X-ray powder diffraction. The recovered samples showed that the diffracted intensity decreases as the pressure increases. The first evidence of amorphization occurs at similar to 5.0 GPa and total amorphization is completed at 8.0 GPa. The bending of Lu-O-W bonds and the tilting of polyhedra in the framework structure are suggested as the mechanism of amorphization. The correlation of pressure-induced amorphization in materials that have a negative thermal expansion coefficient is further supported by structural and thermal expansion behavior in Lu-2(WO<sub>4</sub>)<sub>3</sub>

**[45] THE EFFECT OF BALL MILLING ON THE MICROSTRUCTURE OF CERAMIC AlN**

Yang ZQ. He LL. Ye HQ. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 323(1-2):354-357, 2002

Ceramic AlN was ball milled at room temperature. TEM and HREM studies were carried out on the ball-milled AlN particles/grains. AlN particles were plastically deformed by ball milling at room temperature via the generation and motion of dislocations. Four slip systems were observed in the ball-milled AlN particles. Therefore, the deformation behavior of ceramic AlN during the process of ball milling is similar to that of ordinary metals. Ball milling at low temperature induced amorphous nucleation in the surface layer of AlN particle.

**[44] EFFECT OF PRESSING TEMPERATURE ON MICRO STRUCTURE AND TENSILE BEHAVIOR OF LOW CARBON STEELS PROCESSED BY EQUAL CHANNEL ANGULAR PRESSING**

Shin DH. Pak JJ. Kim YK. Park KT. Kim YS. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 323(1-2):409-415, 2002

Two grades of low carbon steels, one containing vanadium and the other without vanadium, were subjected to equal channel angular pressing at a temperature range of 623-873 K. For steel without vanadium, the equal channel angular pressing at 623 K resulted in ultrafine ( similar to 0.3 mum) ferrite grains with high angle boundaries. At higher pressing temperature, coarser grains with low angle boundaries were formed, which indicates that the recovery occurs at a significant rate during the pressing. For the steel containing vanadium, submicrometer order ferrite grains and high dislocation density were



preserved up to pressing temperature of 873 K. The enhanced thermal stability of the steel containing vanadium was attributed to its peculiar microstructure consisted with fine ferrite grains with uniformly distributed nano-sized cementite particles. In addition, the tensile behaviors of the equal channel angular pressed steels were characterized and discussed based on the microstructure

**[43] FORMATION OF FCC TITANIUM DURING HEATING HIGH ENERGY BALL MILLED AL-TI POWDERS**

Zhang DL. Ying DY. - Materials Letters. 52(4-5):329-333, 2002

Phase transformation during high energy milling Al-25 at.% Ti and Ti-25 at.% Al powders and during heating the ball milled powders of the same compositions was studied. It was found that a small amount of Ti underwent an allotropic transformation from hcp Ti to fcc Ti during milling. This transformation also occurred during heating the properly milled Al-25 at.% Ti and Ti-25 at.% Al powders. The transformation was endothermic, and the onset temperature of this transformation was 321 degreesC. It is likely that only thin Ti layers which have a nanometer scale thickness and are embedded in Al matrix can undergo this transformation.

**[42] PREPARATION AND CHARACTERIZATION OF PLZT (8/65/35) CERAMICS VIA REACTION SINTERING FROM BALL MILLED POWDERS**

Kong LB. Ma J. Zhu W. Tan OK. - Materials Letters. 52(4-5):378-387, 2002

Lead lanthanum zirconate titanate ceramics (PLZT8/65/35) were successfully prepared via reaction sintering of partially reacted oxide mixtures derived from a high-energy ball milling process. The powders milled for different times were characterized by XRD, SEM and particle size analysis. The sintering behavior of the milled powders was also investigated by a dilatometer. A volumetric expansion observed indicates sintering reaction has occurred before the densification of the powders. Dielectric and ferroelectric measurement results of the sintered PLZT ceramics were comparable with the values reported in the literature for the same composition. The simple high-energy ball milling process has shown to be a comparable technique compared with the conventional solid-state reaction and most of the wet-chemistry-based processes.

**[41] FORMATION OF CU-ZR-NI AMORPHOUS POWDERS WITH SIGNIFICANT SUPERCOOLED LIQUID REGION BY MECHANICAL ALLOYING TECHNIQUE**

Hu CJ. Lee PY. - Materials Chemistry & Physics. 74(1):13-18, 2002

The amorphous Cu-Ni-Zr alloys were successfully synthesized by mechanical alloying mixtures of pure crystalline Cu, Ni and Zr powders with SPEX high-energy ball mill. These ranges are similar to those for amorphous alloys produced by melt-spinning techniques. The structure and thermal stability of these alloys were analyzed by X-ray diffraction and differential scanning calorimeter. Several amorphous alloy samples were found to exhibit a wide supercooled liquid region before crystallization. The temperature interval of the supercooled liquid region defined by the difference between T-g and T-x, i.e.  $\Delta T(x) (= T-g - T-x)$ , is 34 K for Cu<sub>10</sub>Zr<sub>60</sub>Ni<sub>30</sub>, 48 K for Cu<sub>20</sub>Zr<sub>60</sub>Ni<sub>20</sub>, 78 K for Cu<sub>40</sub>Zr<sub>55</sub>Ni<sub>5</sub>, 81 K for Cu<sub>50</sub>Zr<sub>35</sub>Ni<sub>15</sub>, 61 K for Cu<sub>50</sub>Zr<sub>40</sub>Ni<sub>10</sub> and 64 K for Cu<sub>50</sub>Zr<sub>45</sub>Ni<sub>5</sub>.

**[40] MECHANOCHEMICAL REACTIONS OF TELLURIC ACID WITH ALKALINE FLUORIDES**

Fernandez-Bertran J. Reguera E. Paneque A. Yee-Madeira H. Gordillo-Sol A. - Journal of Fluorine Chemistry. 113(1):93-95, 2002

The mechanochemical reactions of telluric acid, Te(OH)<sub>6</sub> with alkaline fluorides (Na and K) have been studied using IR and XRD techniques. The reactions lead to the formation of hydrogen-bonding complexes, NaF.Te(OH)<sub>6</sub> and 2KF.Te(OH)<sub>6</sub>. The reactions are free from side products such as alkali tellurates, alkali fluorotellurates or HF<sub>2</sub><sup>-</sup> salts.

**[39] MICROSTRUCTURE AND MAGNETIZATION REVERSAL IN NANOCOMPOSITE SMCO5/SM2CO17 MAGNETS**

Yan A. Bollero A. Gutfleisch O. Muller KH. - Journal of Applied Physics. 91(4):2192-2196, 2002

A comparative investigation has been made of the microstructure and magnetization reversal behavior of nanocomposite SmCo<sub>5</sub>/Sm<sub>2</sub>Co<sub>17</sub> powders prepared by intensive milling and subsequent annealing. It was found that the saturation magnetization increases monotonically with the increase of the volume fraction of Sm<sub>2</sub>Co<sub>17</sub> at the expense of the coercivity. However, the remanence and the energy product first increase and then decrease with further increase of the fraction of Sm<sub>2</sub>Co<sub>17</sub>. The highest remanence of 0.72 T and energy product of 78 kJ/m<sup>3</sup> are obtained in the powders with 80% and 60% Sm<sub>2</sub>Co<sub>17</sub>, respectively. All the hysteresis loops exhibit a magnetically single-phase behavior. X-ray diffraction results reveal that the coexistence of two phases is found in the mixture of SmCo<sub>5</sub>/Sm<sub>2</sub>Co<sub>17</sub> powders. The demagnetization processes of the SmCo<sub>5</sub>/Sm<sub>2</sub>Co<sub>17</sub> powders are similar to those of nanocomposites consisting of hard and soft phases, in which the exchange-spring magnet behavior was observed. Positive as well as negative deviations in Wohlfarth's remanence analysis ( $\Delta M$ -plot) have been observed, indicating complex magnetic interactions in these materials

**[38] THERMODYNAMIC MECHANISMS OF MECHANICAL CRYSTALLIZATION OF AMORPHOUS FE-N ALLOY**

Liu L. Lun S. Liu SE. Zhao XD. Yao B. Su WH. - Journal of Alloys & Compounds. 333(1-2):202-206, 2002

An amorphous Fe-N alloy is prepared by ball milling a mixture of Fe and hexagonal boron nitride. Its crystallization processes were studied upon mechanical milling and annealing under normal and high pressure. The crystallization product of the amorphous Fe-N alloy induced by mechanical milling and annealing at temperatures between 690 and 800 K under pressures of 3-4 GPa is the epsilon-Fe<sub>x</sub>N phase, while the thermal crystallization product under normal pressure is gamma'-Fe<sub>4</sub>N. The difference in crystallization product between mechanical crystallization and thermal crystallization is attributed to the effects of local pressure and local temperature produced by ball collisions on crystallization of amorphous Fe-N alloy

**[37] HYDROGEN STORAGE PROPERTIES OF THE MG-NI-CRCL3 NANOCOMPOSITE**

Yu ZX. Liu ZY. Wang ED. - Journal of Alloys & Compounds. 333(1-2):207-214, 2002



The effects of the addition of the transition metal chloride CrCl<sub>3</sub> on the hydriding and dehydriding behavior of Mg-3 wt.% Ni hydrogen storage material were investigated. The characteristics of the ball-milled nanocomposite Mg-3 wt.% Ni-CrCl<sub>3</sub> such as hydriding capacity in the ball-milling process and the kinetics of hydriding/dehydriding were examined. The hydrogen absorption capacity of the composite is greatly increased in the ball-milling process under hydrogen pressure (0.5 MPa). The absorption rate of the composite is fast and the hydrogen storage capacity is more than that of the sample without CrCl<sub>3</sub>.

**[36] FORMATION OF STABLE AND METASTABLE PHASES IN NI-AL-NB AND NI-AL-ME-C (ME=TI, NB OR V) POWDER SYSTEMS DURING MECHANICAL ALLOYING AND THERMAL TREATMENT**

Krivoroutchko KA. Kulik T. Fadeeva VI. Portnoy VK. - Journal of Alloys & Compounds. 333(1-2):225-230, 2002

A series of quaternary Ni-Al-Me-C (Me=Ti, Nb or V) alloys and two Ni-Al-Nb alloys from various areas of the Ni-Al phase diagram were prepared by the mechanical alloying (MA) method starting from pure powder mixtures. X-ray diffraction and differential scanning calorimetry were used to investigate the powder alloys. It was found, that MA results in the formation of partially ordered metastable B2-type solid solutions in the case of carbon-free systems. On the other hand, the presence of carbon in the powder alloy results in the formation of ordered intermetallic phase and carbide during the thermal treatment. The grain size of carbide and intermetallic phase were kept within the nanosize range even after heating up to 700degreesC

**[35] COMPRESSIVE DEFORMATION BEHAVIOUR OF NANOCRYSTALLINE AL-5 AT.% TI ALLOYS PREPARED BY REACTIVE BALL MILLING IN H-2 AND ULTRA HIGH-PRESSURE HOT PRESSING**

Moon KI. Lee KS. - Journal of Alloys & Compounds. 333(1-2):249-259, 2002

Bulk nanocrystalline Al-5 at.% Ti alloys have been prepared by using an ultra high-pressure hot pressing (UHP-HP) method and their mechanical properties have been investigated through compression tests at room temperature and high temperatures (300, 400 and 500degreesC). TiH<sub>2</sub> and Al<sub>3</sub>Ti acted as effective dispersoids to prevent grain growth during the consolidation process. A full density was reached within 250 s by UHP-HP at 120degreesC under 4.8 GPa, in the specimen A 120 and its grain size was <50 nm. The consolidation temperature of 120degreesC is about 300-400degreesC lower than conventional temperatures. Abnormal grain growth was observed in the specimen A300 prepared by UHP-HP at 300degreesC, which was over one half of the absolute melting temperature of Al. At grains grew to >500 nm in this specimen. The mechanical properties and the deformation behaviours of the A120 and A300 specimens were very different in the compression tests. The compressive stress of the A120 specimen was 10 10 MPa and that of the A300 specimen was 467 MPa at room temperature. However, the strength of the former decreased greatly with increasing testing temperature. While the former specimens had a nanocomposite type microstructure, the latter showed very small change in ductility and strength with temperature. The optimum properties have been obtained in the specimens prepared by UHP-HP at 300degreesC as they have high ductilities and high strength values at both room temperature and high temperatures

**[34] INVESTIGATIONS ON THE SYNTHESIS, STRUCTURAL AND MICROSTRUCTURAL CHARACTERIZATIONS OF MG-BASED K2PTCL6 TYPE (MG2FEH6) HYDROGEN STORAGE MATERIAL PREPARED BY MECHANICAL ALLOYING**

Raman SSS. Davidson DJ. Bobet JL. Srivastava ON. - Journal of Alloys & Compounds. 333(1-2):282-290, 2002

This paper deals with the formation of new ternary hydride Mg<sub>2</sub>FeH<sub>6</sub> (K<sub>2</sub>PtCl<sub>6</sub> type) in a single-step procedure following the process of mechanical alloying of initial stoichiometric ingredients Mg and Fe under hydrogen. The optimum yield of formation of single phase Mg<sub>2</sub>FeH<sub>6</sub> was achieved by hydrogen (similar to 10 atm.) milling of constituent elements at a speed of 400 rpm for various milling durations. The structural characterization of the ball-milled (2 Mg+Fe) powder was carried out using Philips X-ray diffractometer by taking samples from the attritor mill at regular intervals of time. It was found that the Mg<sub>2</sub>FeH<sub>6</sub> phase starts forming at a milling duration of 14 h and the optimum Mg<sub>2</sub>FeH<sub>6</sub> phase formation was obtained at 20 h. The proportion of this phase was estimated by employing Rietveld refinement analysis of the X-ray powder diffraction data and it was found to be 63%. This is the highest phase proportion reported so far for the Mg<sub>2</sub>FeH<sub>6</sub> phase when formed from elemental Mg and Fe following the route of mechanical alloying. Together with the Mg<sub>2</sub>FeH<sub>6</sub> phase, some quantity of Fe (about 37%) is also present. Fe, being a magnetic impurity, can be removed leaving the Mg<sub>2</sub>FeH<sub>6</sub>, content to be nearly 90-100%. However, such purification was not done in the present investigation. We also investigated synthesis of the material obtained by longer milling durations of 25, 28 and 30 h. The XRD patterns for the 25, 28 and 30 h ball-milled materials revealed that the intensity of Mg<sub>2</sub>FeH<sub>6</sub> peaks is reduced in comparison to the Fe peaks. This implies that beyond 20 h, there is no further increase in the phase proportion and the amorphization starts taking place. The post-sintering process of these mechanically alloyed samples did not improve the Mg<sub>2</sub>FeH<sub>6</sub> phase proportion and yield as evidenced by XRD. The X-ray structural characterizations revealed that the as-milled Mg, FeH, material (milling duration of 20 h; under H, pressure similar to 10 atm., speed similar to 400 rpm) corresponds to the known face centered cubic structure with lattice parameter a=0.6446(2) nm. The elemental (chemical) compositional analysis was carried out for the mechanically alloyed Mg<sub>2</sub>FeH<sub>6</sub> materials using the EDAX technique. The results confirm the correct stoichiometric ratio of the initial mixture (2Mg+Fe). The surface morphologies of the (2Mg+Fe) mixture before and after mechanical alloying are performed using scanning electron microscopic technique. The SEM explorations reveal the spongy like feature of Mg<sub>2</sub>FeH<sub>6</sub> agglomerates.

**[33] ONE-STEP SYNTHESIS OF MAGHEMITE NANOMETRIC POWDERS BY BALL-MILLING**

Janot R. Guerard D. - Journal of Alloys & Compounds. 333(1-2):302-307, 2002

Iron powder was milled within water for different duration using a planetary ball mill equipped with stainless steel vials. The in-situ production of hydrogen hinders the hematite formation during the grinding. X-ray diffraction, chemical analysis, high resolution transmission electron microscopy (HRTEM) and Mossbauer spectroscopy reveal that the obtained nanostructured



powders consist of maghemite. Direct synthesis of maghemite nanoparticles from iron powder is so realised. Particles of about 15 nanometers are obtained after 48 h of milling.

Author e-mail Address bokhonov@solid.nsk.su

**[32] THE FORMATION OF GRAPHITE ENCAPSULATED METAL NANOPARTICLES DURING MECHANICAL ACTIVATION AND ANNEALING OF SOOT WITH IRON AND NICKEL**

Bokhonov B. Korchagin M. - Journal of Alloys & Compounds. 333(1-2):308-320, 2002

The investigation of morphological and structural changes during high energy ball milling and thermal annealing of the mixtures soot-iron and soot-nickel demonstrated that the activation is accompanied by the formation of nano-sized metal particles (10-50 nm) distributed over the amorphous carbon matrix. Prolonged mechanical activation of the amorphous soot-iron system (for more than 3-5 min) leads to the formation of nano-sized cementite Fe<sub>3</sub>C phase. Moreover, mechanical activation of the soot-metal compositions causes a substantial decrease in graphitization temperature of the amorphous carbon: for the soot-iron system, the temperature at which the amorphous carbon starts to crystallize is 250-300degreesC while for the soot-nickel system, the minimal temperature at which the crystallization of the amorphous carbon was observed exceeded 600degreesC. Morphological characteristics of the annealed, mechanically activated soot-metal samples depend on the time of preliminary mechanical activation. The annealing of soot-metal samples obtained after short-time mechanical activation ( 1-3 min) causes a crystallization of the amorphous carbon as onion-like graphite-metal structures. Annealing of the soot/metal samples after mechanical treatment for more than 5 min leads to the formation of metal nanoparticles (40-50 nm) encapsulated by graphite. The longer preliminary mechanical activation, the smaller the size of encapsulated particles. In-situ electron microscopic studies of the interaction of metal particles with amorphous carbon thin film showed that the interaction starts in these systems at temperatures about 600degreesC. The interaction in the systems iron-amorphous carbon film and nickel-amorphous carbon film proceeds via the formation of the carbide phases Fe<sub>3</sub>C and Ni<sub>3</sub>C; their decomposition results in the formation of crystal carbon and metal nanoparticles.

**[31] MECHANOCHEMICAL SYNTHESIS OF POLY(BUTADIENE-B-ACRYLIC ACID)**

Popa M. Daranga M. Riess G. - European Polymer Journal. 38(3):407-412, 2002

The preparation of block copolymer with polybutadiene, respectively poly(acrylic acid) sequences, using mechanochemical methods was investigated. The synthesis involves the generating of polybutadiene macroradicals, by ultra-high speed stirring of the polymer in toluene solution. The macroradicals are capable to initiate the polymerization of acrylic acid present in the reaction medium, leading to block copolymer formation. The influence of different parameters, such as temperature, duration, acrylic acid/polybutadiene molar ratio, on the yield and composition of the synthesized copolymer was studied.

**[30] CHARACTERIZATION OF THE MICROSTRUCTURE CHANGES OF POLYPROPYLENE INDUCED BY PAN-MILLING**

Lu CH. Wang Q. - Chinese Journal of Structural Chemistry. 21(1):7-12, 2002.

The microstructure changes of polypropylene induced by a complex combination of shearing, friction, compression and stretching actions during pan-milling were revealed by spectroscopic techniques. X-ray diffraction analysis showed that the structure of polypropylene transferred from crystal into amorphous after undergoing enough milling operation. No transformation between crystal forms was observed. The study of the high-frequency region of the Raman spectrum between 2800 and 3100cm<sup>-1</sup> of polypropylene indicated that molecular motion and chain deformation of PP led to amorphization and deterioration of packing regularity during pan-milling. By co-panmilling PP with bis-(2,2,6,6-tetramethyl-4-piperidinyl) sebacate under ambient condition, ESR signals of free radicals formed by mechanochemical scission of main chain were observed, and an increase of ESR intensity with milling was detected.

**[29] THE EFFECT OF BALL MILLING ON THE MICROSTRUCTURE OF CERAMIC ALN**

Yang ZQ. He LL. Ye HQ. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 323(1-2):354-357, 2002

Ceramic AlN was ball milled at room temperature. TEM and HREM studies were carried Out on the ball-milled AlN particles/grains. AlN particles were plastically deformed by ball milling at room temperature via the generation and motion of dislocations. Four slip systems were observed in the ball-milled AlN particles. Therefore, the deformation behavior of ceramic AlN during the process of ball milling is similar to that of ordinary metals. Ball milling at low temperature induced amorphous nucleation in the surface layer of AlN particle

**[28] EFFECT OF PRESSING TEMPERATURE ON MICRO STRUCTURE AND TENSILE BEHAVIOR OF LOW CARBON STEELS PROCESSED BY EQUAL CHANNEL ANGULAR PRESSING**

Shin DH. Pak JJ. Kim YK. Park KT. Kim YS. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 323(1-2):409-415, 2002

Two grades of low carbon steels, one containing vanadium and the other without vanadium, were subjected to equal channel angular pressing at a temperature range of 623-873 K. For steel without vanadium, the equal channel angular pressing at 623 K resulted in ultrafine ( similar to 0.3 μm) ferrite grains with high angle boundaries. At higher pressing temperature, coarser grains with low angle boundaries were formed, which indicates that the recovery occurs at a significant rate during the pressing. For the steel containing vanadium, submicrometer order ferrite grains and high dislocation density were preserved up to pressing temperature of 873 K. The enhanced thermal stability of the steel containing vanadium was attributed to its peculiar microstructure consisted with fine ferrite grains with uniformly distributed nano-sized cementite particles. In addition, the tensile behaviors of the equal channel angular pressed steels were characterized and discussed based on the microstructure



**[27] FORMATION OF FCC TITANIUM DURING HEATING HIGH ENERGY BALL MILLED AL-TI POWDERS**

Zhang DL. Ying DY. - Materials Letters. 52(4-5):329-333, 2002

Phase transformation during high energy milling Al-25 at.% Ti and Ti-25 at.% Al powders and during heating the ball milled powders of the same compositions was studied. It was found that a small amount of Ti underwent an allotropic transformation from hcp Ti to fcc Ti during milling. This transformation also occurred during heating the properly milled Al-25 at.% Ti and Ti-25 at.% Al powders. The transformation was endothermic, and the onset temperature of this transformation was 321 degreesC. It is likely that only thin Ti layers which have a nanometer scale thickness and are embedded in Al matrix can undergo this transformation

**[26] PREPARATION AND CHARACTERIZATION OF PLZT (8/65/35) CERAMICS VIA REACTION SINTERING FROM BALL MILLED POWDERS**

Kong LB. Ma J. Zhu W. Tan OK. - Materials Letters. 52(4-5):378-387, 2002

Lead lanthanum zirconate titanate ceramics (PLZT8/65/35) were successfully prepared via reaction sintering of partially reacted oxide mixtures derived from a high-energy ball milling process. The powders milled for different times were characterized by XRD, SEM and particle size analysis. The sintering behavior of the milled powders was also investigated by a dilatometer. A volumetric expansion observed indicates sintering reaction has occurred before the densification of the powders. Dielectric and ferroelectric measurement results of the sintered PLZT ceramics were comparable with the values reported in the literature for the same composition. The simple high-energy ball milling process has shown to be a comparable technique compared with the conventional solid-state reaction and most of the wet-chemistry-based processes.

**[25] FORMATION OF CU-ZR-NI AMORPHOUS POWDERS WITH SIGNIFICANT SUPERCOOLED LIQUID REGION BY MECHANICAL ALLOYING TECHNIQUE**

Hu CJ. Lee PY. - Materials Chemistry & Physics. 74(1):13-18, 2002

The amorphous Cu-Ni-Zr alloys were successfully synthesized by mechanical alloying mixtures of pure crystalline Cu, Ni and Zr powders with SPEX high-energy ball mill. These ranges are similar to those for amorphous alloys produced by melt-spinning techniques. The structure and thermal stability of these alloys were analyzed by X-ray diffraction and differential scanning calorimeter. Several amorphous alloy samples were found to exhibit a wide supercooled liquid region before crystallization. The temperature interval of the supercooled liquid region defined by the difference between T-g and T-x, i.e.  $\Delta T(x) (= T-g - T-x)$ , is 34 K for Cu<sub>10</sub>Zr<sub>60</sub>Ni<sub>30</sub>, 48 K for Cu<sub>20</sub>Zr<sub>60</sub>Ni<sub>20</sub>, 78 K for Cu<sub>40</sub>Zr<sub>55</sub>Ni<sub>5</sub>, 81 K for Cu<sub>50</sub>Zr<sub>35</sub>Ni<sub>15</sub>, 61 K for Cu<sub>50</sub>Zr<sub>40</sub>Ni<sub>10</sub> and 64 K for Cu<sub>50</sub>Zr<sub>45</sub>Ni<sub>5</sub>

**[24] CHANGES OF SURFACE AND VOLUME PROPERTIES OF CALCITE DURING A BATCH WET GRINDING PROCESS**

Garcia F. Le Bolay N. Frances C. - Chemical Engineering Journal. 85(2-3):177-187, 2002

An experimental study on the ultra-fine wet grinding of calcite in a batch stirred bead mill is reported. The evolution of the size distribution and of the specific surface area of the particles versus grinding time was measured revealing that an agglomeration phenomenon takes place during the comminution process. The X-ray diffraction profiles versus milling time did not show any polymorphic phase transformation but an intensity reduction and a broadening of diffraction peaks. The size distributions can be properly fitted by a linear combination of statistical log-normal laws, taking the agglomeration of particles into account. The analysis of rheological properties of the ground suspensions revealed a plastic thinning behaviour which can be described by the Hershel-Bulkley equation.

**[23] THE COMPETING CRYSTALLINE AND AMORPHOUS SOLID SOLUTIONS IN THE AG-CU SYSTEM**

Sheng HW. Wilde G. Ma E. - Acta Materialia. 50(3):475-488, 2002

Upon nonequilibrium processing using vapor quenching or mechanical alloying, the supersaturated fcc solid solution predominates over the amorphous solution in the Ag-Cu system. The thermodynamic and kinetic origins of this phase selection are explored. The enthalpy of formation of both solutions has been determined as a function of composition using calorimetry measurements and molecular dynamics (MD) simulations. The enthalpy of the fcc solution is found to be lower than that of the competing amorphous phase. The preference for the fcc crystalline state is enhanced by the low kinetic barrier to crystallization of the amorphous solution, which occurred during quenching even when high cooling rates were employed or during annealing at low temperatures. Consequently, the retention of an amorphous Ag-Cu solution required kinetic trapping using ultrahigh quenching rates achievable only under stringent vapor deposition conditions or in MD simulations. However, transmission electron microscopy revealed the presence of local regions of amorphous Ag-Cu after cold rolling of multilayers, of elemental Ag and Cu foils at room temperature. This result of partial amorphization demonstrates the possibility of mechanically driven solid-state amorphization in a system with a positive heat of mixing.

**[22] FINE GRINDING OF AGGREGATED POWDERS**

Hogg R. Dynys AJ. Cho H. - Powder Technology. 122(2-3 Special Issue SI):122-128, 2002

In many cases, including natural ores as well as synthetic powders, fine grinding involves the breakage of bound aggregates rather than solid particles. The characteristics of breakage in such systems have been investigated by experimental studies of grinding kinetics, in a model system of partially sintered alumina particles, ground in a laboratory centrifugal ball mill. The effects of aggregate strength (extent of sintering) and energy input (mill speed) on the breakage rates and breakage distributions have been evaluated. Breakage appears to occur primarily through splitting of the aggregated mass into two or three smaller aggregates accompanied by release of the primary particles, leading to strongly bimodal breakage distributions.

**[21] MECHANOCHEMICAL SYNTHESIS OF LACRO3 BY GRINDING CONSTITUENT OXIDES**

Zhang QW. Lu JF. Saito F. - Powder Technology. 122(2-3 Special Issue SI):145-149, 2002



Hydrous amorphous chromium oxide ( $\text{Cr}_2\text{O}_3 \cdot n\text{H}_2\text{O}$ ) and crystalline one ( $\text{Cr}_2\text{O}_3$ ) powders prepared by heating  $\text{Cr}(\text{OH})_3 \cdot n\text{H}_2\text{O}$  at different temperatures were separately ground with  $\text{La}_2\text{O}_3$  powder using a planetary ball mill at room temperature to investigate mechanochemical synthesis of  $\text{LaCrO}_3$ . Hydrous amorphous chromium oxide prepared reacts with  $\text{La}_2\text{O}_3$  to form  $\text{LaCrO}_3$  and the reaction proceeds with an increase in grinding time. No reaction takes place in the case of crystalline  $\text{Cr}_2\text{O}_3$ . The ground sample consists of agglomerates and large particles with size up to around 100  $\mu\text{m}$  and shows low specific surface area of about 5  $\text{m}^2/\text{g}$ . The mechanochemical method can be also used to synthesize other chromium rare earth complex oxides by grinding the mixture of hydrous chromium oxide and rare earth (Pr, Nd and Sm) oxides

**[20] HYDROTHERMAL REACTIONS UNDER MECHANOCHEMICAL ACTION**

Boldyrev VV. - Powder Technology. 122(2-3 Special Issue SI):247-254, 2002

The possibility for hydrothermal processes to take place under the action of mechanical pulses that arise in mechanical activators is considered for the system: a solid substance-water. Experimental data are presented on the mechanochemical synthesis of gidenbergite, tobermorite and other hydrosilicates that are usually produced according to the autoclave technology.

**[19] STRUCTURAL AND CATHODOLUMINESCENCE STUDY OF MECHANICALLY MILLED SILICON**

Diaz-Guerra C. Montone A. Piqueras J. Cardellini F. - Semiconductor Science & Technology. 17(1):77-82, 2002

The structural and luminescent properties of nanocrystalline silicon produced by high-energy ball milling of Si single crystals have been investigated using transmission electron microscopy (TEM), x-ray diffraction (XRD) and cathodoluminescence (CL) in a scanning electron microscope. XRD measurements show that the average size of the nanocrystals in the milled samples is about 30 nm but TEM reveals a wide range of size distribution including crystallites with the dimension of few nanometres. Ball milling causes the appearance of a visible luminescence band at 1.61 eV, attributed to the presence of nanocrystals, and a near-infrared band peaked at about 0.79 eV which is suggested to be related to the high density of extended defects formed during the mechanical treatment. These bands, attributed to processes in Si, are not observed in the cathodoluminescent spectra of untreated and ball-milled  $\text{SiO}_2$  powder.

**[18] GRAIN GROWTH BEHAVIOR OF CRYOMILLED INCONEL 625 POWDER DURING ISOTHERMAL HEAT TREATMENT**

Chung KH. Lee J. Rodriguez R. Lavernia EJ. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 33(1):125-134, 2002

Nanocrystalline INCONEL 625 powders were fabricated via cryomilling (mechanical alloying under a liquid nitrogen environment), and their grain growth behavior during isothermal heat treatment was investigated in detail. The grain size after milling for 8 hours was approximately 22 nm, measured by transmission electron microscopy (TEM) observations and X-ray diffraction (XRD). Along with this refined structure, the NiO and  $\text{Cr}_2\text{O}_3$  oxide particles were distributed in the cryomilled material with average size of 3 nm. Following heat treatment at 800 degreesC, correspond to  $T/T_m = 0.65$ , for 4 hours, the grain size was approximately 240 nm, which represents an improved grain stability compared to that of conventional INCONEL 625 and cryomilled pure Ni. The improved grain stability of cryomilled INCONEL 625 is originated from a particle pinning effect by the oxide particles in addition to solute drag. The grain stability of the cryomilled powders at 900 degreesC was better than that at lower temperatures. This behavior was attributed to the formation of two types of secondary particles that precipitated at this temperature, which were identified as spherical NbC carbides and cylindrical-shaped  $\text{Ni}_3\text{Nb}$  intermetallic precipitates. These precipitates promote grain growth resistance at this particular temperature via a grain-boundary pinning effect. Contribution of 30 pct Nb solute atoms in alloy on the forming precipitates on grain boundary, the grain growth will be restricted to approximately 2100 nm, on the basis of a Zener mechanism. This calculation is in qualitative agreement with the experimental results. The observation that precipitation kinetics were accelerated over those of conventional INCONEL 625 was rationalized on the basis of the shortened diffusion paths and more nucleation sites available in the nanocrystalline materials

**[17] SOLID-STATE CRYSTALLINE-GLASSY CYCLIC PHASE TRANSFORMATIONS OF MECHANICALLY ALLOYED CU33ZR67 POWDERS**

El-Eskandarany MS. Inoue A. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 33(1):135-143, 2002

Cyclic crystalline-glassy-crystal line phase transformations have been investigated during high-energy ball milling of elemental  $\text{Cu}_{33}\text{Zr}_{67}$  powders under an argon gas atmosphere. The results show that the metallic glass, which is obtained after 86 ks of milling time, transforms into a new metastable phase of nanocrystalline  $\text{CuZr}_2$  upon milling for 173 ks. It subsequently transforms to the same glassy phase after 259 ks of the milling time. Such cyclic phase transformations are observed several times during the milling. The present work shows that this phenomenon does not have any obvious analogues with the periodic redox reactions or with diffusive-reactive phenomena known in chemistry. On the basis of our results, the destabilizing effect of the defects created by the milling media (balls), which leads to the cyclic transformations, depends not only on the milling time but also on the milling rotation speed.

**[16] THE STRUCTURE AND ELECTROCHEMICAL PROPERTIES OF BALL-MILLED  $\text{Ti}_{0.9}\text{Zr}_{0.2}\text{Mn}_{1.6}\text{Ni}_{0.2}\text{V}_{0.2}$  ALLOYS**

Xu YH. Chen CP. Wang XL. Wang QD. - Solid State Ionics. 146(1-2):157-161, 2002

The high-energy ball-milling technique is generally used to modify the surface structure and texture of materials in order to improve their physical or chemical properties. In the conditions of thermodynamic equilibrium, the hydrogen-storage



capacity of Ti<sub>0.9</sub>Zr<sub>0.2</sub>Mn<sub>1.6</sub>Ni<sub>0.2</sub>V<sub>0.2</sub> alloy can reach 240.9 ml/g, from which it can be calculated that its theoretical capacity is 537.3 mA h/g. However, its discharge capacity in KOH solution is only less than 40 mA h/g. In order to improve this capacity, the effect of ball milling with Ni powders on the electrochemical and structural properties of Ti<sub>0.9</sub>Zr<sub>0.2</sub>Mn<sub>1.6</sub>Ni<sub>0.2</sub>V<sub>0.2</sub> alloy was investigated. The results show that proper ball milling with Ni powders is very effective to improve the discharge capacity of the Ti<sub>0.9</sub>Zr<sub>0.2</sub>Mn<sub>1.6</sub>Ni<sub>0.2</sub>V<sub>0.2</sub> alloy by increasing the surface electrocatalytic ability.

**[15] METASTABLE AND TRANSIENT STATES OF CHEMICAL ORDERING IN FE-V NANOCRYSTALLINE ALLOYS - ART. NO. 024204**

Ziller T. Le Caer G. Isnard O. Cenedese P. Fultz B. - Physical Review B. 6502(2):4204-+, 2002

Chemical ordering of the disordered alloys Fe<sub>0.78</sub>V<sub>0.22</sub>, Fe<sub>0.53</sub>V<sub>0.47</sub>, Fe<sub>0.39</sub>V<sub>0.61</sub>, and Fe<sub>0.37</sub>V<sub>0.63</sub> was performed by annealing at temperatures from 723 to 973 K. The initial state of chemical disorder was produced by high-energy ball milling, and the evolution of order was measured by neutron diffractometry and by Fe-57 Mossbauer spectrometry. The hyperfine magnetic field distributions obtained from the Mossbauer spectra provided quantitative measurements of the number of antisite Fe atoms in the partially ordered alloys. The long-range order parameters in steady state after long annealing times were used as states of metastable equilibrium for a generally successful comparison with the metastable Fe-V phase diagram calculated by Sanchez et al. [Phys. Rev. B 54, 8958 (1996)]. For the metastable equilibrium state of order in Fe<sub>0.53</sub>V<sub>0.47</sub> at low temperatures, the order parameters were smaller than expected. This corresponded to an abundance of antisite atoms, which were not removed effectively by annealing at the lower temperatures

**[14] SIMULTANEOUS DTA-TG STUDY OF MONTMORILLONITE MECHANOCHEMICALLY TREATED WITH CRYSTAL-VIOLET**

Lapides I. Yariv S. Golodnitsky D. - Journal of Thermal Analysis & Calorimetry. 67(1):99-112, 2002.

The mechanochemical solid-state adsorption of the cationic dye crystal violet (CV) by montmorillonite was investigated by XRD and simultaneous DTA-TG. Solid CV was ground with the clay for 5 min and four different varieties of CV mechanochemically treated clay were investigated. X-ray and DTA data were compared with those of CV-montmorillonite obtained from an aqueous suspension. X-ray and DTA studies of a ground mixture and a ground mixture heated at 110degreesC suggest that the mechanochemical adsorption of organic cations takes place on the external surfaces of the clay. The study of a ground mixture washed with water, and washed with water and acetone reveal that water is essential for the penetration of CV into the interlayer space

**[13] NANOSIZED ZINC-OXIDE PARTICLES DERIVED FROM MECHANICAL ACTIVATION OF ZN-5(NO3)2(OH)8)CENTER DOT 2H(2)O IN SODIUM CHLORIDE**

Fah CP. Xue JM. Wang J. - Journal of the American Ceramic Society. 85(1):273-275, 2002

Nanosized ZnO particles are successfully synthesized via mechanical activation of a zinc nitrate hydroxide hydrate (Zn-5(NO3)2(OH)8)2H(2)O precursor in NaCl matrix for 15 h. The ZnO particles obtained are in the nanosize range of similar to 20 nm, with a well-established hexagonal morphology. They compare favorably with those derived from conventional calcination of the precursor. The decomposition of Zn-5(NO3)2(OH)8)2H(2)O precursor and formation of nanocrystalline ZnO cannot be completed by mechanical activation in the absence of NaCl, which acts as both an effective dispersing matrix and drying agent although it remains chemically inert during mechanical activation. The powder derived from calcination at 400degreesC does not possess powder characteristics comparable to that of the powder derived from the mechanical activation in NaCl, because of the extensive particle coarsening and aggregation at the calcination temperature

**[12] EVIDENCE OF SMALL CRYSTALLITES IN MILLED FE/CO ALLOY OBSERVED BY MOSSBAUER SPECTROSCOPY**

Passamani EC. Larica C. Nascimento VP. - Journal of Materials Science. 37(4):819-823, 2002

Structural and magnetic properties of nanocrystalline Fe<sub>0.67</sub>Co<sub>0.33</sub> alloy prepared by high energy ball milling have been studied by x-ray diffraction and Mossbauer spectroscopy. The x-ray diffraction pattern of the sample milled for 160 hours indicates the existence of a single bcc phase. On the other hand, Mossbauer measurements, at different temperatures, show that the milled sample has two magnetic components with the same average hyperfine parameters. One sextet component is associated with large crystallites of bcc Fe<sub>0.67</sub>Co<sub>0.33</sub> alloy, stable in vacuum up to 825 K and the other component is attributed to small crystallites of Fe<sub>0.67</sub>Co<sub>0.33</sub> alloy having sizes in the range from 10 to 18 nm.

**[11] WEAR AND OXIDATION RESISTANCE OF AL2O3 PARTICLE DISPERSED AL3Ti COMPOSITE WITH A NANOSTRUCTURE PREPARED BY PULSED ELECTRIC CURRENT SINTERING OF MECHANICALLY ALLOYED POWDERS**

Uenishi K. Matsubara T. Shibutani T. Kobayashi KF. - Intermetallics. 10(1):105-111, 2002

Al<sub>3</sub>Ti-matrix composite layers containing Al<sub>2</sub>O<sub>3</sub> particles were formed on Ti substrate by pulsed electric current sintering (PECS) of mechanically alloyed (MA) powders to improve the wear and oxidation properties of the Ti substrate. Reducing the grain size of each element by MA makes the combustion synthesis of Al<sub>3</sub>Ti possible at a lower temperature. The grain size formed by the combustion synthesis of Al-Ti-Al<sub>2</sub>O<sub>3</sub> powder mechanically alloyed for 720 ks was about 10 nm and its growth during sintering was suppressed by the existence of Al<sub>2</sub>O<sub>3</sub>. The densification behavior of the powder was investigated quantitatively. The obtained Al<sub>3</sub>Ti/Al<sub>2</sub>O<sub>3</sub> composite layer showed better wear and oxidation resistance than the monolithic Al<sub>3</sub>Ti layer

**[10] COBALT SILICIDE FORMATION INSIDE SURFACE DEFECTS OF A SILICON SUBSTRATE**

Belousov I. Grib A. Linzen S. Seidel P. - Nuclear Instruments & Methods in Physics Research Section B-Beam Interactions with Materials & Atoms. 186:61-65, 2002



The formation process of a CoSi<sub>2</sub> layer after vacuum Co film deposition on an Si single crystal surface was investigated by means of scanning tunneling microscope (STM), SEM and RHEED. The pinholes which cause different kinds of film roughness show marks of melting processes, It is shown that in the limits of the theory of mechanochemical activity the activation energy of the silicide formation is lower inside the defect than in the bulk silicon. Therefore, the exothermic reaction of silicidation starts earlier which leads to a heating of the CoSi product. We calculated the time dependencies of the temperature inside the defect for different areas of its surface using the layer growth diffusion model and proved that the temperature increases up to the melting point at a certain critical area.

**[9] MECHANICAL MILLING OF MAGNESIUM POWDER**

Hwang S. Nishimura C. McCormick PG. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 318(1-2):22-33, 2001

The evolution of microstructure during mechanical milling of magnesium powder has been studied. Commercially pure (99.6%, - 325 mesh) magnesium powder was mechanically milled in a modified SPEX 8000 shaker mill in an inert atmosphere. The optical microscopy of the powder in the early stage of milling showed deformation by twinning and re-twinning within the grains developing sub-grain boundaries, which eventually defined nanometre-sized grains. The grain size reduction examined using XRD revealed a rapid decrease and then saturation of the grain size at approximately 42 nm. A relatively large final grain size compared to other mechanically milled metals was obtained due to the high recovery rate of magnesium. The corresponding internal strain was also observed to be low, confirming that enhanced recovery had occurred during milling. The internal strain during milling showed inverse grain size dependence. Moire fringe patterns of TEM micrographs showed absence of dislocations within the grains of as milled magnesium powder

**[8] NANOSIZED ZINC-OXIDE PARTICLES DERIVED FROM MECHANICAL ACTIVATION OF ZN-5(NO<sub>3</sub>)<sub>2</sub>(OH)<sub>8</sub>CENTER DOT 2H<sub>2</sub>O IN SODIUM CHLORIDE**

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**[6] PROCESSING AND CHARACTERIZATION OF CLUSTER DIAMOND DISPERSED AL-SI-CU-MG COMPOSITE**

Hanada K. Mayuzumi M. Nakayama N. Sano T. - Journal of Materials Processing Technology. 119(1-3 Special Issue SI):216-221, 2001

Cluster diamond (CD), which consists of ultra-fine diamond particles with a mean particle size of 5 nm, shows excellent lubricating ability. By dispersing CD in metals, metal matrix composites that have low friction and good wear properties can be produced with self-lubricating ability. In the present study, CD-dispersed Al-Si-Cu-Mg alloy-based matrix composites were fabricated by the powder metallurgy method. The mixing of CD and Al-Si-Cu-Mg alloy powders was carried out by the mechanical milling method. The amount of CD added was varied from 0 to 10% by volume. The mixed composite powders were consolidated at 773 K by hot pressing in a vacuum. X-ray diffraction analysis showed that the added CD had not changed in structure after the consolidation process. Friction measurements were performed at 1.7 mm/s and 0.2 N by the pin-on-disc method to measure the friction coefficient and wear width of consolidated Al-Si-Cu-Mg/CD composite powders. The results showed that the addition of 1 vol.% CD to Al-Si-Cu-Mg alloy significantly improved the friction coefficient compared to that of Al-Si-Cu-Mg alloy. Furthermore, the addition of graphite CD (GCD) achieved a friction coefficient of 0.14. However, the friction coefficient and wear width deteriorated with an increase in the volume fraction of CD or GCD.

**[5] WEAR AND OXIDATION RESISTANCE OF AL<sub>2</sub>O<sub>3</sub> PARTICLE DISPERSED AL<sub>3</sub>Ti COMPOSITE WITH A NANOSTRUCTURE PREPARED BY PULSED ELECTRIC CURRENT SINTERING OF MECHANICALLY ALLOYED POWDERS**

Uenishi K. Matsubara T. Shibutani T. Kobayashi KF. - Intermetallics. 10(1):105-111, 2002

Al<sub>3</sub>Ti-matrix composite layers containing Al<sub>2</sub>O<sub>3</sub> particles were formed on Ti substrate by pulsed electric current sintering (PECS) of mechanically alloyed (MA) powders to improve the wear and oxidation properties of the Ti substrate. Reducing the grain size of each element by MA makes the combustion synthesis of Al<sub>3</sub>Ti possible at a lower temperature. The grain size formed by the combustion synthesis of Al-Ti-Al<sub>2</sub>O<sub>3</sub> powder mechanically alloyed for 720 ks was about 10 nm and its growth during sintering was suppressed by the existence of Al<sub>2</sub>O<sub>3</sub>. The densification behavior of the powder was



investigated quantitatively. The obtained Al<sub>3</sub>Ti/Al<sub>2</sub>O<sub>3</sub> composite layer showed better wear and oxidation resistance than the monolithic Al<sub>3</sub>Ti layer

**[4] MICROSTRUCTURE AND ELECTRICAL PROPERTIES OF THIN FILMS OF RE<sub>51.75</sub> PRODUCED BY CO-SPUTTERING**

Kuwabara K. Inui H. Yamaguchi M. - Intermetallics. 10(2):129-138, 2002

Microstructural evolution and changes in electrical resistivity of ReSi<sub>1.75</sub> thin films produced by co-sputtering has been investigated as a function of annealing temperature. Crystallization of amorphous ReSi<sub>1.75</sub> thin films occurs at 600°C without forming any metastable phases. The crystallization temperature for ReSi<sub>1.75</sub> is considerably higher than those observed for other transition-metal disilicides. The crystal structure as well as the domain (twinned) structure observed for crystallites in ReSi<sub>1.75</sub> thin films annealed above 600°C are essentially the same as those observed in bulk crystals of ReSi<sub>1.75</sub>. Although the grain size of crystallites increases with the increase in annealing temperature, thin films annealed below 650°C exhibit a nano-crystalline structure. Thin films of amorphous and crystalline ReSi<sub>1.75</sub> and bulk polycrystalline ReSi<sub>1.75</sub> all exhibit electrical resistivity values decreasing with the increase in temperature, indicating the semiconducting nature. Values of electrical resistivity for crystallized thin films are systematically higher than those for amorphous ReSi<sub>1.75</sub> thin films and bulk polycrystalline ReSi<sub>1.75</sub> and increase with the decrease in annealing temperature, exhibiting a peak at just above the crystallization temperature. The high values of electrical resistivity around the crystallization temperature are discussed in terms of the formation of the nano-crystalline structure in thin films, for which the effective medium approximation is not valid.

**[3] A STUDY ON THE MICROSTRUCTURE OF D0(23) AL<sub>3</sub>Zr AND L1(2) (Al+12.5 at. % Cu)<sub>3</sub>Zr INTERMETALLIC COMPOUNDS SYNTHESIZED BY PBM AND SPS**

Moon KI. Kim SC. Lee KS. - Intermetallics. 10(2):185-194, 2002

The spark plasma sintering (SPS) of L1(2) phase Al<sub>3</sub>Zr and (Al+ 12.5 at.% Cu)<sub>3</sub>Zr powders with a nanocrystalline microstructure has been studied to produce bulk intermetallic compounds which maintain metastable structures such as L1(2), structure and nanocrystalline microstructure. The powders were prepared by 10 It planetary ball milling (PBM). Full-density L1(2)(Al + 12.5 at.% Cu)<sub>3</sub>Zr intermetallic compounds were obtained by SPS for 0 min at 600°C. The specimens prepared with a longer holding time than 0 min at 600°C or a higher temperature than 600°C had local melting areas where micro-cracks were found. They had a lower relative density than the specimen SPS sintered at 600°C for 0 min. The smallest grain size was obtained in the specimen prepared at 600°C for 0 min, which was 20-30 nm as confirmed by TEM observation. This was the smallest grain size ever reported in the trialuminide specimens processed by various consolidations of nanocrystalline powders. Accordingly, the highest micro-hardness, 989.5 H-v, was obtained in the specimen and this value was three times higher than those of the specimens with micro grain sizes. Full density Al<sub>3</sub>Zr intermetallics were prepared by SPS at 700°C for 0 min. However, their crystal structure was D0(23) and microhardness was 778.1 H-v. By Using SPS, the sintering time can be reduced within 10 min. It was thought that the decrease in sintering temperature for the PBM Al<sub>3</sub>Zr and (Al + 12.5 at.% Cu)<sub>3</sub>Zr powders by 200-300°C compared with the conventional sintering temperature resulted in the refinement of microstructure to the nano-size level

**[2] MILLS AS MECHANO-REACTORS [GERMAN]**

Heegn H. - Chemie Ingenieur Technik. 73(12):1529-1539, 2001

In addition to size reduction, other phenomena observed in mills and which have been exhaustively examined by scientific methods are attracting increasing technological attention. These include changes of materials and reactions going beyond dispersion and which are known by their terms mechanical activation, mechanochemistry, mechanical alloying, mechanofusion, etc. The present article assesses the suitability of certain types of mills for use as mechanoreactors. The significance of the nature, speed, and energy of the forces acting and of the derived quantities power and energy input is discussed. A methodological basis is given for upscaling from laboratory and pilot studies to industrial production and commercialisation. Recent results concerning mechanical activation, mechanical alloying, and mechanochemistry of various material systems are presented

**[1] SYNTHESIS OF MAGNESIA - CALCIUM ZIRCONATE - DICALCIUM SILICATE MATERIALS BY REACTION SINTERING OF DOLOMITE - ZIRCON MIXTURES. PROCESSING STUDY [SPANISH]**

Rodriguez JL. Castro PP. - Boletín de la Sociedad Espanola de Ceramica y Vidrio. 40(6):463-471, 2001

The interdependence of green density and particle size and their influence on the reaction sintering behaviour of zircon dolomite powder mixtures were investigated. Powder size was controlled by attrition-milling for different times and was defined as the amperage size obtained by laser particle size analysis. Green compact density was shown to be related to the powder particle size distribution for identical consolidation conditions. Both the green density and the degree of powder agglomeration affect the sinter behaviour over the entire process. The experimental results showed that compacts with similar green density and which contained fewer coarse particles had a better sintering behaviour. The most important controlling parameters are characteristics of the raw materials as particle or agglomerate particle size and particle size distribution, homogeneity and compaction of the raw materials and sintering temperature.



Correspondants du Réseau Français de Mécanosynthèse  
186 Laboratoires ou Groupes de Recherche (34 Pays)  
Bureau : E. Gaffet (Président), G. Le Caër (Secrétaire Général), A.R. Yavari (Trésorier)

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