

**RESEAU FRANÇAIS DE  
MECANOSYNTHESE**

**Lettre N°81**

**Décembre 2001**

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

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

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Rubrique Pages Sciences et Techniques pour l'Ingénieur (Rubrique Sciences)  
vous y trouverez les anciennes lettres du RFM (accessible par Adobe Acrobat)  
les statuts du RFM ainsi que les annonces concernant les JRFM'2001 et quelques éléments mis à jour régulièrement  
concernant les derniers résultats dans ce domaine.

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Les JRFM'2002 seront intégrées dans le cadre du Congrès  
**Matériaux 2002**

(Tours - France, du 21 au 25 Octobre 2002)

Symposium 1 :

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

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

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

**Sommaire**

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

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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/>

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Workshops

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

Plymouth, NH, USA June 30 - July 5 ,2002  
<http://sol.rutgers.edu/~shinbrot/gordon2002/gordon2002.html>

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**RQ11**

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

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**10th European Symposium on Comminution**

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

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International Symposium on  
Metastable, Mechanically Alloyed and Nanocrystalline Materials  
(ISMANAM-2002)

Seoul, Korea, 8-12 September, 2002.  
Web site : <http://anu.andong.ac.kr/~ismanam/>

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L. A. C. A. M. E – 2. 0. 0. 2  
EIGHTH LATIN AMERICAN CONFERENCE  
ON APPLICATIONS OF THE MÖSSBAUER EFFECT  
PANAMA, 22-27, SEPTEMBER, 2002.

E-mail: <mailto:lacame2000@fisica.ciens.ucv.ve>  
<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)



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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**

**Université de Grenoble - 13 Novembre 2001**

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

**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

La mécanosynthèse est une technique de choix pour la préparation de poudres nanocristallines. Dans le cas particulier de la synthèse de composés d'intercalation du graphite avec le lithium, l'ajout d'un agent liquide s'avère nécessaire afin de limiter l'agglomération du lithium sur les outils de broyage. Le dodécane a été choisi car celui-ci joue un rôle lubrifiant et dispersant efficace. Les conditions de température et de pression localement et momentanément atteintes à l'impact des billes permettent de former un composé superdense de stoechiométrie  $\text{LiC}_3$ . Ce composé, de composition voisine à celles des phases produites par compression isostatique (50 kbars), se distingue par sa grande stabilité. La mécanosynthèse est la première technique qui permet de préparer un composé stable dans les conditions ambiantes plus riche en lithium que le composé  $\text{LiC}_6$ . Le broyage de graphite seul dans le dodécane a également été étudié. En combinant l'action d'un broyeur planétaire et d'un liquide mouillant efficacement le graphite, la formation de graphite très anisométrique (facteur de forme de l'ordre de 100) est possible à partir d'un simple graphite naturel. Les graphites ainsi obtenus présentent d'intéressantes propriétés électrochimiques : le traitement permet en effet de réduire de façon significative la perte irréversible liée à la formation d'une couche de passivation.

Enfin, la préparation de nanoparticules de maghémite ( $\gamma \text{Fe}_2\text{O}_3$ ) par simple broyage de fer en milieu aqueux a été mise au point. Cette synthèse est remarquable en raison de la monodispersité de taille des particules obtenues, ce qui présente un intérêt évident dans le domaine du magnétisme. Les nanoparticules ont ensuite été dispersées sur du graphite anisotrope et les propriétés électrochimiques de ces composites ont été examinées. La taille nanométrique des grains de maghémite autorise une réversibilité partielle de la réaction électrochimique entre fer et maghémite.

**MOTS CLES**

Mécanosynthèse - Intercalation - Composés graphite /lithium - Maghémite - RMN de  $^7\text{Li}$  - Electrochimie

**Ball-milling in liquid media of superdense graphite-lithium compounds, of very anisometric graphite and of maghemite deposited or not on graphite**



The ball-milling is a very convenient technique to produce nanocrystalline powders. In the case of the synthesis of graphite intercalation compounds with lithium, the use of a liquid agent is necessary to avoid an important agglomeration of lithium on the milling tools. The dodecane was chosen because it allows a good lubrication and dispersion. The pressure and temperature temporarily induced by the shocks occurring during the milling lead to the formation of a superdense compound with a  $\text{LiC}_3$  stoichiometry. Its structure was determined by  $^7\text{Li}$  NMR at low temperature. This compound, with a composition close to those of the phases produced by isostatic compression (50 kbars), is characterised by its high stability. The ball-milling is the first technique, which allows to prepare a compound, stable under ambient conditions, with a higher lithium content than that of the  $\text{LiC}_6$  compound.

The milling of graphite into dodecane was also investigated. With the combination of the action of a planetary ball-mill and of a liquid medium wetting efficiently the graphite particles, the preparation of a very anisometric graphite (geometrical anisotropy around 100) is possible from a simple natural graphite. The electrochemical properties of the grounded graphites are very interesting : this treatment allows to widely reduce the irreversible capacity due to the formation of a passivating layer.

The synthesis of maghemite ( $\gamma\text{Fe}_2\text{O}_3$ ) nanoparticles by simple grinding of iron into water was also set up. This technique is remarkable due to the narrow size distribution of the obtained particles, which is very interesting in the field of magnetic applications. Then the nanoparticles were dispersed on anisometric graphite and the electrochemical properties of these composites were tested. The nanometric size of the maghemite particles allows a partial reversibility of the reaction between iron and maghemite and makes them a good candidate for anodic materials in lithium-ion batteries.

**Keywords**

Ball-milling – Intercalation – Graphite /lithium compounds – Maghemite –  $^7\text{Li}$  NMR – Electrochemistry

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**Thierry Girot**

Thèse INPL, 15 Octobre 2001

**"Cinétique et modélisation des transformations de phases induites par broyage à haute énergie dans  $\text{TiO}_2$  anatase "**

**Jury :**

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

Pour étudier les mécanismes fondamentaux mis en œuvre au cours des transformations polymorphiques induites par broyage à haute énergie, nous nous sommes intéressés à  $\text{TiO}_2$  de structure anatase. Au cours du broyage,  $\text{TiO}_2$  anatase se transforme en la phase rutile via une phase haute pression et/ou haute température du  $\text{TiO}_2$  nommée  $\text{TiO}_2$  II. Divers paramètres du procédé de broyage ont ainsi été modifiés et les résultats cinétiques ont été confrontés aux modèles développés sur ce procédé.

Les observations par microscopies électroniques à balayage et à transmission, l'analyse des diagrammes de diffraction des rayons X et les mesures granulométriques ont permis de proposer un mécanisme de transformation du grain monocristallin d'anatase au cours des premières minutes du broyage. Du fait de leurs propriétés mécaniques, les grains d'anatase ne subissent pas les phénomènes de fracture et soudage classiquement observés au cours de la mécanosynthèse et des grains nanométriques de  $\text{TiO}_2$  II se forment à la surface des particules d'anatase.

La confrontation des résultats cinétiques aux modèles de mécanosynthèse et l'analyse du mécanisme de la transformation nous ont permis de montrer que le paramètre pertinent pour décrire cette transformation est la puissance injectée par unité de volume de poudre piégée au cours de la collision.

Enfin, nous présentons une nouvelle approche prometteuse pour la compréhension des phénomènes mis en jeu à l'échelle des particules au cours du procédé de broyage : la simulation numérique par une méthode DEM. Les premiers résultats permettent d'expliquer quelques unes des observations faites au cours de cette étude.

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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**

**De L. 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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**From Dr. Dr. Deliang Zhang**

**Ph D Position**

**University of Waikato, New Zealand - 23 / 08/2001**

Titanium PhD ScholarShip - Department of Materials and Process Engineering-

The Department of Materials and Process Engineering at the University of Waikato is seeking a suitable candidate for the Titanium Ph.D Scholarship which has a value of up to NZ\$22,000 per year and is offered for three years. The recipient of the scholarship will be required to conduct research on a suitable topic in the area of processing, characterisation and development of titanium based materials. He/she will work within a dynamic team at Waikato University working on a large research project on processing and development of titanium based materials. The candidate must have a BE (Honours), BSc(Honours), or a Master degree in materials science and engineering or closely related subjects with good average grade

To apply, please send a copy of CV and undergraduate and postgraduate (if applicable) transcript to

Dr. Deliang Zhang, Department of Materials and Process Engineering,

The University of Waikato, Private Bag 3105, Hamilton, New Zealand,

Fax: 64-7-838 4835;

e-mail: [d.zhang@waikato.ac.nz](mailto:d.zhang@waikato.ac.nz).

The application process will remain open until a suitable candidate is identified.

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

**Ph D Position**

**Stuttgart - 21/08/2001**

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 has been selected a Marie Curie Training Site

and offers a one year position on Nanostructured Materials: Atomic Transport Properties for the Synthesis and Characterization of Novel Soft and Hard Magnets

The Ph.D. student will receive a monthly payment of 1200 Euro plus additional 100 Euro per month travel allowance. Post-Docs should ask us for further details.

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. The research interest of the candidates should be in at least one of the following fields: solid state physics, materials science including synthesis and characterization of materials, mechanical and magnetic properties of advanced materials, and structural studies.



Lettre RFM N°81 - Décembre 2001  
Corresp. : <mailto:Eric.Gaffet@utbm.fr>

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.

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

Prof. H.-E. Schaefer  
e-mail: [mailto:schaefer@itap.physik.uni-stuttgart.de](mailto:mailto:schaefer@itap.physik.uni-stuttgart.de)  
phone: +49-711-685-5261  
FAX: +49-711-685-5271

Dr. W. Sprengel  
e-mail: [mailto:sprengel@itap.physik.uni-stuttgart.de](mailto:mailto:sprengel@itap.physik.uni-stuttgart.de)  
phone: +49-711-685-5192  
FAX: +49-711-685-5271

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

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### Post Doc Position

#### Dijon/ France (10/07/2001) – From F. Bernard

The research group (fine grain materials) from the Research Lab UMR 5613 (Laboratoire de Recherche sur la Réactivité des Solides) is seeking for a post doctoral associate with experience in X – ray Diffraction (experimental and numerical approaches, computer simulation, Monte Carlo ...).

The candidate (he or she) has to demonstrate the ability to work independently, contribute to innovative numerical approach, and develop new projects in this area.

The work will be performed in collaboration between three french labs (Dijon – F. Bernard, Belfort – E. Gaffet, Vitry - Y. Champion).

**Scientific Field :** In spite of a lot a research effort, the mechanism of phase formation during MA is not well understood. It is most often proposed that the process of MA introduces a variety of defects (vacancies, dislocations, grain boundaries, stacking fault,...) which raise the free energy of the system making it possible to produce metastable phases. But there are very few investigations that deal with the characterization and quantification of the defects produced in mechanically alloyed powders. As a primary investigation, the effect of the mechanical activation mode (i.e. the friction or direct shock ones, at least the component ratio of both components) can be assumed on analysing the microstructure of post-mortem milled powders. XRD is really a valuable technique for a characterisation in terms of size and morphology of crystallites and imperfections (microstrains, dislocation, stacking faults,...). Indeed, the ball milling of metals or alloys induces extended variations in the intensity distribution of XRD diagrams and, in particular, in the line profile. Knowledge of the stacking fault density and the twin-fault density is essential to understand the nanomaterials behaviour.

A new line profile analysis method is proposed by Ustinov et al. [123], in order to take into account the dependence of the crystallite size, of the residual strains as well as of the planar defects, on the line profile broadening that may be observed on ball-milled materials. Such a method will allow to understand the influence of ball-milling parameters and for controlling the synthesis of nanostructured materials

**Financial Support :** Regional Financial Support from Burgundy Region in France

Interested candidates should send **correspondence** to:

BERNARD Frédéric - Université de Bourgogne - UFR Sciences et Techniques  
9, Avenue Alain Savary - Laboratoire de Recherches sur la Réactivité des Solides,  
UMR 5613 CNRS / Université de Bourgogne - Equipe "Matériaux à grains Fins"  
B.P. 47870 - 21078 DIJON CEDEX

fax : 33.3.80.39.61.67 - e-mail : [fbernard@u-bourgogne.fr](mailto:fbernard@u-bourgogne.fr)

*Please note that this proposal is opened for french students.*

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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.



Lettre RFM N°81 - Décembre 2001  
Corresp. : [mailto:Eric.Gaffet@utbm.fr](mailto:mailto:Eric.Gaffet@utbm.fr)

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/ssp>

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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](mailto:koehler@fritsch.de))

(7/07/2000) - **From Victor Rieckensky Publisher**

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

**MACROMOLECULAR MECHANOCHEMISTRY**

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

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

Macromolecular Mechanochemistry presents from theoretical and experimental point of view the main problems of this field, including the results obtained in more than a century of research. It is organised in two volumes: Polymer Mechanochemistry and Polymers with Chemomechanical Functions, respectively. The present volume deals with: Chained Polystage Character of Mechanochemical Process (1), Mechanochemistry of Polymers Deformation (2); Mechanochemistry of Polymer Fracture (including also the Fracture of Composite Materials) (3), and Mechanochemical Processes for Energy Conversion (4). In this frame, the theoretical and experimental material is organised in correlation to the reaction mechanism, the type of mechanical sollicitation, 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



Lettre RFM N°81 - Décembre 2001  
Corresp. : <mailto:Eric.Gaffet@utbm.fr>

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 Besterici - <http://www.demon.co.uk/cambsci/book51.htm>

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

3. Microstructure and quantitative evaluation of parameters of dispersion-strengthened materials (definition and properties of interparticle distance; experimental possibilities of determination of structural objects; models of heterogeneous structures and their evaluation; simulation of model structures; analysis of the spatial distribution of particles in the Al-Al4C3 material)

4. Static and dynamic mechanical properties (mechanical properties at elevated temperatures; mechanical properties at 20 °C; effect of interface on the mechanical properties; superplastic properties of the system; thermal stability of the system; creep characteristics; creep-fatigue characteristics)

References - ISBN 189832655X, 90 pages, 234 156 mm, soft laminated cover, £25.00, 1999

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

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

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



## "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 États 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.

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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

### [70] STRUCTURAL TRANSITIONS OF MECHANICALLY ALLOYED FE100-XCUX SYSTEMS STUDIED BY X-RAY ABSORPTION FINE STRUCTURE

Wei SQ. Yan WS. Li YZ. Liu WH. Fan JW. Zhang XY. - Physica B. 305(2):135-142, 2001

The local structures of the immiscible Fe100-xCux alloys (x = 0, 10, 20 40. 60, 80 and 100) produced by mechanical alloying have been investigated by X-ray absorption fine structure technique. For the Fe100-xCux solid solutions with x greater than or equal to 40, the local environment around the Fe atoms changes from bee to fee structure and the Cu atoms maintain the original coordination geometry after milling for 160h. In contrast, the local structures around the Cu atoms in both Fe80Cu20 and Fe90Cu10 alloys exhibit a transition from fee to bee structure. Furthermore, we found that the coordination numbers N in the first shell of the Fe and Cu atoms were largely deviated from the stoichiometric composition for the Fe100-xCux solid solutions with x greater than or equal to 40. The Debye-waller factor sigma of the fee Fe-Cu phase is larger than that of the bcc Fe-Cu phase, and the sigma (0.099 Angstrom) around Fe atoms is larger than that around Cu atoms (0.089 Angstrom) in the Fe100-xCux solid solutions with x greater than or equal to 40. This indicates that the mechanically alloyed Fe100-xCux supersaturated solid solutions with x greater than or equal to 40 is not a homogeneous alloy, but consists of fee Fe-rich and fcc Cu-rich regions. However, In Fe100-xCux solid solutions with x less than or equal to 20, the Cu atoms were almost homogeneously solved into the bee Fe-Cu phase. A possible mechanism for bee-to-fee and fee-to-bee changes in Fe100-xCux solid solutions is discussed in relation to the interdiffusion and the transition induced by the ball milling

### [69] A NOVEL METHOD FOR THE SYNTHESIS OF ZNS FOR USE IN THE PREPARATION OF PHOSPHORS FOR CRT DEVICES

Davies DA. Silver J. Vecht A. Marsh PJ. Rose JA. - Journal of the Electrochemical Society. 148(10):H143-H148, 2001

The decomposition of thiourea dioxide in aqueous solution at elevated temperatures, in the presence of zinc acetate, has been used to precipitate ZnS. The important reaction pathway for the formation of sulfide ions has been elucidated, and some common ZnS phosphors have been prepared from the precipitate. The preparation of ZnS by this method is extremely simple and does not yield large amounts of liquid or gas containing volatile sulfur species. Thus, this method has been shown to be an excellent method for the preparation of ZnS phosphors, particularly copper-activated materials, requiring no purification of the reagents, with Hale production of sulfur-containing waste species, and resulting in small particle size powders without postproduction milling or separation of the powders. These phosphors have been shown to have exceptional luminescent properties compared to standard commercial materials

### [68] PARTICULATE-REINFORCED AL-BASED COMPOSITE MATERIAL FOR ANODE IN LITHIUM SECONDARY BATTERIES

Jeong GJ. Kim YU. Sohn HJ. Kang T. - Journal of Power Sources. 101(2):201-205, 2001

Particulate-reinforced Al/SiC composite materials are prepared by ball-milling technique to be used as an anode material for lithium secondary battery. The microstructure of the composite powders show that the SiC particles are embedded homogeneously in the Al matrix. This feature is distinctively different from any other active/inactive composite anode materials reported recently. The cycle performance of these composite electrodes is superior to that of an unreinforced aluminium electrode. This improved cyclability may be due to an enhanced mechanical stability of the electrode

### [67] PREPARATION OF POROUS SILICA FROM MECHANICALLY ACTIVATED KAOLINITE

Temuujin J. Burmaa G. Amgalan J. Okada K. Jadambaa T. MacKenzie KJD. - Journal of Porous Materials. 8(3):233-238, 2001

Mesoporous silica has been prepared by leaching of the Al<sub>2</sub>O<sub>3</sub> component from mechanically amorphized kaolinite. The kaolinite was amorphized by grinding in a planetary ball mill for 1 h. After grinding the amorphized kaolinite was chemically treated with dilute sulfuric acid at 90 degreesC for varying times. The influence of the leaching time on the porous properties and structure of the silica was studied by XRD, XRF, FTIR and BET adsorption methods. The specific surface areas of the leached samples were found to vary from 312 m<sup>2</sup>/g to 284 m<sup>2</sup>/g. The pore size distribution, calculated



by the BJH method based on N<sub>2</sub> gas isotherms, showed a unimodal pore size distribution with an average pore size of about 3.8 nm. The total pore volume of the porous silica varied from 0.28 ml/g to 0.312 ml/g, with a uniform pore size distribution in the mesopore regions. New applications exploiting the characteristic pore size of this material are to be expected

**[66] X-RAY DIFFRACTION STUDY ON PRESSURE-INDUCED PHASE TRANSFORMATIONS IN NANOCRYSTALLINE ANATASE/RUTILE (TiO<sub>2</sub>)**

Wang ZW. Saxena SK. Pischedda V. Liermann HP. Zha CS. - Journal of Physics-Condensed Matter. 13(36):8317-8323, 2001

An in situ x-ray diffraction study was conducted to study the pressure-induced phase transformation in nanocrystalline anatase/rutile (TiO<sub>2</sub>) to 35.1 GPa. The nano-anatase phase remains stable to approximately 16.4 GPa, and then transforms to an amorphous phase, which is returned upon release of pressure. The nano-rutile phase starts to transform to the baddeleyite (ZrO<sub>2</sub>) structure at similar to 8.7 GPa, and the transformation is complete at approximately 16.4 GPa. On release of pressure the ZrO<sub>2</sub> structure transforms to the alpha -PbO<sub>2</sub> structure. The results are compared to previous work on phase changes in TiO<sub>2</sub> with different particle sizes

**[65] EFFECT OF CALCIUM SALTS ON ISOSYNTHESIS OVER ZrO<sub>2</sub> CATALYSTS**

Li YW. He DH. Cheng ZX. Su CL. Li JR. Zhu QM. - Journal of Molecular Catalysis A-Chemical. 175(1-2):267-275, 2001

The promoting effects of various calcium salts on the activity and selectivity of ZrO<sub>2</sub> in isosynthesis were studied in this work. Calcium salts were added into zirconia by mechanical mixing methods. Catalytic tests were performed under relatively mild operation conditions (673 K, 650 h<sup>-1</sup>, 5.0 MPa). CaF<sub>2</sub> and CaSO<sub>4</sub> were found to be effective additives, which could remarkably enhance the i-C<sub>4</sub> selectivity in total hydrocarbons while maintaining the activity of pure ZrO<sub>2</sub> when being added into zirconia. However, Ca(NO<sub>3</sub>)<sub>2</sub>, Ca(BO<sub>2</sub>)<sub>2</sub> and CaCl<sub>2</sub> wholly changed the distribution of hydrocarbons to favor the methanation. The results of temperature-programmed desorption (TPD) of NH<sub>3</sub> and CO<sub>2</sub> indicated that the performance of the catalysts depended on the acid-base properties of the catalysts. The appropriate amount of acid and base and the ratio of the basic to acidic sites on the catalysts are significant for the synthesis of i-C<sub>4</sub> hydrocarbons from CO hydrogenation

**[64] SUB-MICRON SIZED AL<sub>2</sub>TiO<sub>5</sub> POWDERS PREPARED BY HIGH-ENERGY BALL MILLING**

Uribe R. Baudin C. Mazerolles L. Michel D. - Journal of Materials Science. 36(21):5105-5113, 2001

High energy ball milling to obtain ultrafine aluminium titanate particles has been investigated. Tempered steel has been selected as material for the containers and balls because the desirable properties of aluminium titanate are not degraded by small amounts of Fe<sub>2</sub>O<sub>3</sub>. The starting powders have been milled during different periods (1-60 h) and the evolution of the morphology and crystallinity of the treated powders as well as the extent of contamination from the milling media have been characterised. Different experimental techniques, X-ray diffraction, BET-analysis, chemical analysis, scanning electron microscopy and low and high resolution transmission electron microscopy have been used. High energy ball milling has been proved to be an efficient route to obtain submicron sized (50-100 nm) aluminium titanate powders, but further milling of the powders is accompanied by contamination from the milling media and the formation of hard agglomerates

**[63] GROWTH OF MULTI-WALLED CARBON NANOTUBES ON MECHANICAL ALLOYING-DERIVED AL<sub>2</sub>O<sub>3</sub>-Ni NANOCOMPOSITE POWDER**

Liu BH. Zhong ZY. Ding J. Lin JY. Shi Y. Si L. - Journal of Materials Chemistry. 11(10):2523-2528, 2001.

Mechanical alloying was employed to produce the nanocomposite Al<sub>2</sub>O<sub>3</sub>-Ni. It was found that the mechanical alloying of a mixture of NiO and alpha -Al<sub>2</sub>O<sub>3</sub> generated a highly disordered structure. Reduction under a hydrogen atmosphere led to formation of nano-sized Ni crystallites in the Al<sub>2</sub>O<sub>3</sub> matrix. Relatively high BET specific surface areas indicate that the sub-micron nanocomposite particles have a porous structure. In comparison with the co-precipitated powder, the mechanical alloying-derived powder shows smaller particle/agglomerate size and much higher Ni reducibility. Multi-walled carbon nanotubes with a high production yield were successfully synthesized by using the mechanical alloying-derived nanocomposite as the catalyst

**[62] STRONG UNIDIRECTIONAL ANISOTROPY IN MECHANICALLY ALLOYED SPINEL FERRITES**

Shi Y. Ding J. - Journal of Applied Physics. 90(8):4078-4084, 2001

Cluster glass and relatively high coercivity at low temperatures were found in disordered ultrafine nickel ferrite powders. High-energy mechanical milling of spinel NiFe<sub>2</sub>O<sub>4</sub> led to formation of a wustitelike structure. Our investigation suggested that ferrimagnetic clusters formed in an antiferromagnetic matrix. The strong ferri/antiferromagnetic exchange coupling resulted in a strong unidirectional anisotropy and a coercivity of over 10 kOe at 4.2 K.

**[61] THERMAL EVOLUTION OF THE MAGNETIZATION IN NANOCRYSTALLINE FE PARTICLES INVESTIGATED BY ELECTRON HOLOGRAPHY**

Bonetti E. Del Bianco L. Pasquini L. Matteucci G. Beeli C. Signoretti S. - Journal of Applied Physics. 90(8):4152-4158, 2001 Oct 15.

Abbreviated Source J. Appl. Phys. 90(8):4152-4158, 2001

Micrometric, irregularly shaped Fe particles with a nanocrystalline structure have been prepared by mechanical attrition through ball-milling. Electron holography has been employed to visualize the stray field emerging from isolated Fe particles, both at 300 K and at selected temperatures T less than or equal to 1200 K, from which indirect information on the magnetic domain configuration has been inferred. By complementary x-ray diffraction and transmission electron microscopy investigations a relationship has been established between the changes of the leakage field and of the microstructure upon annealing: it indicates that the structural evolution is accompanied by strong modifications in the interior magnetization



pattern. This relationship finds explanation in the framework of the random anisotropy model, including temperature-induced reversible variations in the exchange correlation length and saturation magnetization. Moreover, the role played by the overall geometrical features of the particles in the determination of the actual domain configuration has been investigated.

**[60] STUDY OF THE PHASE COMPOSITION AND HOMOGENEITY OF FE-CU ALLOYS PREPARED BY MECHANOACTIVATION UNDER PRESSURE**

Chernyshev EG. Pilyugin VP. Patselov AM. Serikov VV. Kleinerman NM.- Physics of Metals & Metallography (English Translation of Fizika Metallov i Metallovedenie). 92(2):179-184, 2001

The mechanical alloying of iron-copper heterogeneous powder mixtures was studied in a wide range of compositions. Plastic deformation under pressure was performed at room temperature. The degree of homogeneity was estimated, and the concentration boundaries of the single-phase states of nonequilibrium solid solutions based on bcc iron and fcc copper were determined. Various aspects of the homogenization of iron-copper alloys and the formation of their phase compositions upon mechanoactivation by methods such as shear under pressure and ball milling are discussed

**[59] TEXTURE AND MICROSTRUCTURE IN VERY FINE GRAIN AL-AL<sub>3</sub>Ti ALLOYS OBTAINED BY EXTRUSION OF MECHANICALLY ALLOYED POWDERS**

Lenhard S. Helming K. Chang CP. Wagner F. Grosdidier T. - Materials Science & Technology. 17(9):1169-1173, 2001

Texture has been investigated in warm extruded Al - Al<sub>3</sub>Ti bars obtained from mechanically alloyed powders. The effects of various high volume fractions of Al<sub>3</sub>Ti phase (9, 18, and 27 vol-%) and the very fine grain sizes that can be obtained via the mechanical alloying processing route are described. Increasing the volume fraction of Al<sub>3</sub>Ti phase tends to decrease the anisotropy of the extruded product. The 'conventional' aluminium texture characterised by a predominant < 111 > (Al) fibre usually present in extruded bars is replaced at high volume fraction of Al<sub>3</sub>Ti particles (27%) and small size of aluminium grains (300 nm) by a weak and broad < 441 > (Al) fibre. This is suggested to be associated with a modification of the predominant deformation mechanism, changing from dislocation slip towards grain boundary sliding at small grain size

**[58] MANUFACTURING AND MICROSTRUCTURAL EVOLUTION OF MECHANICALLY ALLOYED OXIDE DISPERSION STRENGTHENED SUPERALLOYS [REVIEW]**

Capdevila C. Bhadeshia HKDH. - Advanced Engineering Materials. 3(9):647-656, 2001

Mechanical alloying is a process in which mixtures of powders are severely deformed until they form atomic solutions. Inert oxides can also be introduced to form a dispersion of fine particles which help strengthen the consolidated product. Significant quantities of iron and nickel-base alloys, with unusual properties, are produced commercially using this process. The total true strain during mechanical alloying can be as large as 9; there is proof that this leads to mixing on all atomic scale and to the development of a uniform grain structure which is sub-micrometer in size. Following mechanical alloying, the particles are consolidated using standard powder metallurgical techniques. The consolidated metal has a large stored energy, approaching 1 Jg(-1). This ought to make it easy to induce recrystallisation, but in practice the alloys fail to recrystallise except at very high temperatures close to melting. On the other hand, the recrystallisation temperature can be reduced dramatically by slightly deforming the consolidated product prior to heat treatment. It is in this context that the solution formation, microstructure and mechanical properties of such alloys are reviewed here

**[57] SUPERPLASTICITY EXTENSIBILITY AND DEFORMATION MECHANISM OF A NANOCRYSTALLINE COPPER SAMPLE**

Lu L. Sui ML. Lu K. - Advanced Engineering Materials. 3(9):663-667, 2001

By means of the electrodeposition technique, a bulk sample of nanocrystalline (nc) copper was prepared with high purity and high density. An extreme extensibility (elongation > 5000 %) without a strain hardening effect was observed when the nc Cu sample was rolled at room temperature. A detailed study on the microstructure evolution of the nc Cu during the cold-rolling process was examined by means of X-ray diffraction (XRD) analysis, transmission electron microscopy (TEM), and thermal analysis. It was indicated that the deformation process in the tic Cu sample is dominated by the grain boundary activity rather than lattice dislocation. This phenomenon agrees well with the observed mechanical behavior of the nc Cu sample.

**[56] CREEP PROPERTIES OF BALL-MILLED AND HIPED PURE TANTALUM**

Xu Q. Hayes RW. Lavernia EJ. - Scripta Materialia. 45(4):447-454, 2001

**[55] MICROSTRUCTURE OF CUO AFTER SHOCK-LOADING AND MILLING**

Gizhevsky BA. Kozlov EA. Ermakov AE. Lukin NV. Naumov SV. Samokhvalov AA. Arbuzov VL. Shalnov KV. Degtyarev MV. - Physics of Metals & Metallography (English Translation of Fizika Metallov i Metallovedenie). 92(2):153-157, 2001

X-ray diffraction and scanning tunneling microscopy were used to study the structure of polycrystalline CuO loaded by spherical shock isentropic waves and of CuO powder obtained by ball milling. Grain sizes along various directions were determined from the broadening of ((2) over bar 02), (020), and ((1) over bar 13) diffraction peaks for a number of samples cut from different layers along the radius of a compressed ball and for the nanopowder obtained by ball milling. It is shown that a dense strong single-phase CuO nanoceramics was obtained after explosive loading

**[54] DECOMPOSITION OF THE ND<sub>2</sub>FE<sub>14</sub>B INTERMETALLIC COMPOUND UPON SEVERE PLASTIC DEFORMATION BY SHEAR UNDER PRESSURE**

Gaviko VS. Popov AG. Ermolenko AS. Shchegoleva NN. Stolyarov VV. Gunderov DV. - Physics of Metals & Metallography (English Translation of Fizika Metallov i Metallovedenie). 92(2):158-166, 2001



The effect of severe plastic deformation by shear under high pressure in Bridgman-type anvil setup on the intermetallic compound Nd<sub>2</sub>Fe<sub>14</sub>B was studied by structural (X-ray and electron-microscopic) and magnetic methods. Alloys with a different content of this phase were investigated. It was found that under heavy plastic deformation the major part of the compound decomposes into the Fe-enriched bcc and Nd-enriched amorphous phases. With increasing deformation, the volume of the decomposed phase increases. Other phases influence only the rate of Nd<sub>2</sub>Fe<sub>14</sub>B compound decomposition. Decomposition is slower in hyperstoichiometric Nd<sub>20</sub>Fe<sub>70</sub>B<sub>10</sub> and Nd<sub>20</sub>Fe<sub>77.5</sub>B<sub>2.5</sub> alloys and is faster in hypostoichiometric Nd<sub>9</sub>Fe<sub>84</sub>B<sub>7</sub> alloy than in nearly stoichiometric Nd<sub>11.7</sub>Fe<sub>82.4</sub>B<sub>5.9</sub> alloy. The initial phase composition of the deformed samples was restored by fast heating to T > 600 degreesC and subsequent annealing at this temperature for 10-20 minutes. Stepped annealing or slow heating did not result in complete restoration of the initial phase composition. Possible factors that cause the different influence of these anneals on the phase changes are discussed

**[53] STUDY OF THE PHASE COMPOSITION AND HOMOGENEITY OF FE-CU ALLOYS PREPARED BY MECHANOACTIVATION UNDER PRESSURE**

Chernyshev EG. Pilyugin VP. Patselov AM. Serikov VV. Kleinerman NM. - Physics of Metals & Metallography (English Translation of Fizika Metallov i Metallovedenie). 92(2):179-184, 2001

The mechanical alloying of iron-copper heterogeneous powder mixtures was studied in a wide range of compositions. Plastic deformation under pressure was performed at room temperature. The degree of homogeneity was estimated, and the concentration boundaries of the single-phase states of nonequilibrium solid solutions based on bcc iron and fcc copper were determined. Various aspects of the homogenization of iron-copper alloys and the formation of their phase compositions upon mechanoactivation by methods such as shear under pressure and ball milling are discussed.

**[52] THE EFFECT OF ULTRASOUND IRRADIATION ON POLYCRYSTALLINE MOO<sub>3</sub>**

Jeevanandam P. Diamant Y. Motiei M. Gedanken A. - Physical Chemistry Chemical Physics. 3(18):4107-4112, 2001

The effect of ultrasound irradiation on polycrystalline molybdenum trioxide suspended in n-decane has been investigated. The changes in the physicochemical properties of MoO<sub>3</sub> have been investigated using techniques such as powder X-ray diffraction, UV-VIS and IR spectroscopies, BET surface area measurements, scanning electron microscopy, and EPR. Evidence for the formation of Mo(v) sites and shear defects has been presented. The physicochemical changes of MoO<sub>3</sub> due to ultrasound irradiation have been attributed to the formation of radicals and also the mechanical effects that can be created by ultrasound, such as shear forces, micro jets, and shock-waves. The mechanical effects are the results of the sonochemical cavity collapse onto molybdenum trioxide particles.

**[51] SYNTHESIS, SINTERING AND MICROSTRUCTURE OF BETA-TRICALCIUM PHOSPHATE FOR PROSTHETIC APPLICATIONS**

Sinha MK. Sen PS. Basu D. - Journal of the Indian Chemical Society. 78(8):386-388, 2001

A method is described for preparing dense, polycrystalline beta-tricalcium phosphate. The phase composition of the precipitate made by mixing Ca(OH)<sub>2</sub> and H<sub>3</sub>PO<sub>4</sub> Solutions with subsequent drying and calcining has been studied as a function of initial mixing Ca/P molar ratio. Pure beta-tricalcium phosphate is prepared with initial Ca/P = 1.2 and without any pH control. Effects of milling time of calcined powder on the sintering behavior and microstructural development examined. Grinding for a longer time helps in achieving a relatively high density and fine-grain microstructure at 1200 degrees. The synthesized powder of beta-TCP shows good biocompatibility and no cytotoxicity.

**[50] INFLUENCE OF ALUMINUM SALT ADDITION ON IN SITU SINTERING OF ELECTROLYTE MATRICES FOR MOLTEN CARBONATE FUEL CELLS**

Lee IS. Kim W. Moon YJ. Lis H. - Journal of Power Sources. 101(1):90-95, 2001

Three aluminum salts are investigated as a sintering aid for the in situ sintering of electrolyte matrices for molten carbonate fuel cells (MCFCs). Only aluminum acetylacetonate shows a potential, At or above 420 degreesC, aluminum acetylacetonate changes to Al<sub>2</sub>O<sub>3</sub> and reacts with Li<sub>2</sub>CO<sub>3</sub> in the electrolyte to produce gamma -LiAlO<sub>2</sub>. This reaction product forms necks between matrix particles. Necks grow with increasing sintering time and correspondingly, the mechanical strength of the electrolyte matrix shows an abrupt increase, starting at a sintering time of about 100 h until it levels off at about 250 h. The porosity of the matrices fabricated with aluminum acetylacetonate is in the range acceptable for use in MCFCs.

**[49] STUDY OF THE ACTIVATION PROCESS OF MG-BASED HYDROGEN STORAGE MATERIALS MODIFIED BY GRAPHITE AND OTHER CARBONACEOUS COMPOUNDS**

Bouaricha S. Dodelet JP. Guay D. - Journal of Materials Research. 16(10):2893-2905, 2001

A nanocomposite (Mg-V)(nano) made of 90 wt% Mg and 10 wt% V was prepared by high-energy ball-milling during 40 h. The activation characteristics of (Mg-V)(nano). are rather poor, the hydrogen content [H] reaching 4 wt% after more than 100 h (t(4w%)) following the initial exposure of the material to H<sub>2</sub>. Adding 9 wt% graphite to (Mg-V)(nano) and resuming, the milling operation for 30 min leads to the formation Of (Mg-V)(nano)/G, which exhibits a t(4w%) value of only 10 min. The addition of more than 9 wt% graphite to (Mg-V)(nano) does not lead to any significant reduction of the t(4w%) value. However, extending the milling period with graphite over 30 min leads to a steady increase in t(4wt%) and, thus, to a deterioration of the activation characteristics. Comparison of the behavior of graphite with other C-based compounds revealed that perylene (C<sub>20</sub>H<sub>12</sub>) and pentacene (C<sub>22</sub>H<sub>14</sub>), which are made of linked benzene rings, and thus have a 2D structure similar to that of the graphene sheet, are as effective as graphite in improving the activation characteristics of (Mg-V)(nano). A structural investigation of (Mg-V)(nano)/G as a function of the milling time through both C 1s core-level x-ray photoelectron spectroscopy and C K edge x-ray absorption near-edge spectroscopy has shown that the integrity of graphite is progressively lost as the milling period is extended over 30 min. On the basis of these results, it is hypothesized that the



adsorption of graphene layer on freshly created Mg surfaces and the formation of highly reactive C species during milling prevents the re-formation of the surface oxide layer responsible for the poor activation characteristics of untreated

**[48] MECHANOCHEMICAL MODIFICATION OF DEOXYPEGANINE HYDROCHLORIDE BY POLYAMPOLITE**

Takhtaganova DB. Pak TS. Kristallovich EL. Aripov KN. Tashpulatov YT. - Chemistry of Natural Compounds. 37(1):65-68, 2001

The solubility and dialysis of deoxypeganine hydrochloride and natural polyampholite were studied by IR spectra and x-ray diffraction analysis. Inclusion complexes form during mechanical treatment of a 1:1 mixture of deoxypeganine hydrochloride and natural polyampholite

**[47] REDUCING THE DANGER OF EXPLOSIONS IN THE PRODUCTION OF POWDERS OF IRON AND ITS ALLOYS BY METHODS THAT INVOLVE MECHANICAL COMMINATION**

Vasil'eva GI. Neikov OD. Tokhtuev VG. - Powder Metallurgy & Metal Ceramics. 40(1-2):90-95, 2001

A method is described for producing powders, granules, and briquets of chemically active metals and alloys. The method is based on mechanical interlaminar-pulverization in an inertial crusher in the presence of a film-forming compound. The pulverization process is accompanied by microencapsulation of the powders. The method does not create any danger of explosion. The production operations are waste-free and non-polluting. The microencapsulated powders are less combustible and less explosive than the initial powders. Microencapsulation of the powders keeps dust from being formed during the production process. Results are examined from experimental studies of the combustibility, and explosiveness characteristics of powders of iron, ferroalloys, and high-speed steels. This information can be used as the initial data in developing recommendations on explosion-proofing

**[46] MECHANOCHEMICAL PROCESSING OF GOLD-BEARING SULPHIDES**

Welham NJ. - Minerals Engineering. 14(9):1119, (2001)

**[45] PROCESSING, MICROSTRUCTURE AND MECHANICAL PROPERTIES OF YTTRIA STABILIZED ZIRCONIA REINFORCED HYDROXYAPATITE COATINGS**

Fu L. Khor KA. Lim JP. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 316(1-2):46-51, 2001

It has been proved that adding yttria stabilized zirconia (YSZ) to hydroxyapatite (HA) could improve the mechanical properties of hydroxyapatite coatings and reduce the formation of calcium oxide which is brittle and not desirable in HA coatings. In this work, processing, microstructure and mechanical properties of yttria stabilized zirconia reinforced HA coatings have been studied. 30 wt.% yttria stabilized zirconia was added to HA. Two processes were applied. One included ball-milling the mixture of YSZ and HA powders, then plasma spheroidizing ball-milled powders (BMSP) and spraying of the coating. Another included ball-milling YSZ powders, blending with HA as purchased, plasma spheroidizing blended powders (BSP) and spraying of the coating. Experimental results showed that mechanical properties of the BMSP and BSP coatings were improved significantly, comparing to the pure HA coating. HA/YSZ solid solution formed and played a very important role in mechanical properties of yttria stabilized zirconia reinforced HA coatings. HA/YSZ solid solution in BSP coating was more than that in BMSP coating, and the properties of BSP coating were better than that of BMSP coating. Tensile tests showed that unmelted YSZ particles were the potential weakness of the yttria stabilized zirconia reinforced HA coatings.

**[44] THE PROCESSING OF KAOLIN POWDER COMPACT**

Chen CY. Tuan WH. - Ceramics International. 27(7):795-800, 2001.

Powder processing is crucial to the final properties of porcelain bodies. In the present study, a kaolin powder was used as the starting material, water or organic solvent was employed as the milling medium, and subsequently dried and formed by the die-pressing technique. The final phase, mullite, is obtained by sintering kaolin powder compacts. By using an organic solvent instead of water, the density, microstructure and mechanical properties show a better performance due to the avoidance of agglomerates formation.

**[43] MECHANOCHEMICAL SYNTHESIS AND ELECTROCHEMICAL CHARACTERISTICS OF Mg<sub>2</sub>Sn AS AN ANODE MATERIAL FOR LI-ION BATTERIES**

Kim H. Kim YJ. Kim DG. Sohn HJ. Kang T. - Solid State Ionics. 144(1-2):41-49, 2001

Mg<sub>2</sub>Sn prepared by mechanochemical process was examined as an alternative anode material for Li-ion batteries. Electrochemical tests demonstrated that the initial charge and discharge capacity of Mg<sub>2</sub>Sn was 556 and 460 mAh/g, respectively. Ex-situ XRD and differential capacity plots showed that lithium inserted into the Mg<sub>2</sub>Sn lattice first followed by alloying with Sn. Contrary to the isostructural Mg-based intermetallic compound, Mg<sub>2</sub>Si, alloying reaction between Li and Mg was not observed during lithiation of Mg<sub>2</sub>Sn. Mg<sub>2</sub>Sn showed better capacity retention characteristic than that of Mg<sub>2</sub>Si. It is thought that this may be attributed to that Mg formed at Mg<sub>2</sub>Sn electrode did not react with lithium, and also active materials of Mg<sub>2</sub>Sn electrode changed from Mg<sub>2</sub>Sn to Sn with the increase of cycles. Also Mg<sub>2</sub>Sn showed improved cycle performance under restricted voltage range due to prevention Sn from aggregation into larger clusters

**[42] EXPERIMENTAL INVESTIGATION OF BAND STRUCTURE MODIFICATION IN SILICON NANOCRYSTALS**

Pawlak BJ. Gregorkiewicz T. Ammerlaan CAJ. Takkenberg W. Tichelaar FD. Alkemade PFA. - Physical Review B. 6411(11):5308+, 2001

Experimental studies of size-related effects in silicon nanocrystals are reported. We present investigations carried out on nanocrystals prepared from single-crystal Si:P wafer by ball milling. The average final grain dimension varied depending on the way of preparation in the range between 70 and 230 nm. The ball milling was followed by sedimentation and selection of



the smallest grains. The initial grain size distribution was measured by scanning electron microscopy. Further reduction in size was achieved by oxidation at 1000 degreesC which creates a silicon dioxide layer around a silicon core. The oxidation process was monitored by transmission electron microscopy and the growth speed of SiO<sub>2</sub> was estimated in order to model the grain size of nanocrystals. Crystallinity of silicon grains was confirmed by x-ray diffraction and by transmission electron microscopy using a bright/dark field method and selected area diffraction pattern. In the silicon nanocrystals the electron energy levels are shifted which was observed separately for conduction band, valence band and energy band gap. Electron paramagnetic resonance was applied to investigate variation of the conduction band minimum by monitoring its influence on the hyperfine interaction of phosphorus shallow donor. On the basis of these results an explicit expression for conduction band upshift as a function of average grain size has been derived. Information about the downshift of the valence band was obtained from measurements on a photoluminescence band related to a deep to shallow level transition. A perturbation of a few meV for grain sizes of the order about 100 nm has been observed. Internal consistency of these findings has been examined by investigation of the photoluminescence band due to an electron-hole recombination whose energy is directly related to the band gap of silicon

**[41] PROCESSING, MICROSTRUCTURE AND MECHANICAL PROPERTIES OF YTTRIA STABILIZED ZIRCONIA REINFORCED HYDROXYAPATITE COATINGS**

Fu L. Khor KA. Lim JP. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 316(1-2):46-51, 2001

It has been proved that adding yttria stabilized zirconia (YSZ) to hydroxyapatite (HA) could improve the mechanical properties of hydroxyapatite coatings and reduce the formation of calcium oxide which is brittle and not desirable in HA coatings. In this work, processing, microstructure and mechanical properties of yttria stabilized zirconia reinforced HA coatings have been studied. 30 wt.% yttria stabilized zirconia was added to HA. Two processes were applied. One included ball-milling the mixture of YSZ and HA powders, then plasma spheroidizing ball-milled powders (BMSP) and spraying of the coating. Another included ball-milling YSZ powders, blending with HA as purchased, plasma spheroidizing blended powders (BSP) and spraying of the coating. Experimental results showed that mechanical properties of the BMSP and BSP coatings were improved significantly, comparing to the pure HA coating. HA/YSZ solid solution formed and played a very important role in mechanical properties of yttria stabilized zirconia reinforced HA coatings. HA/YSZ solid solution in BSP coating was more than that in BMSP coating, and the properties of BSP coating were better than that of BMSP coating. Tensile tests showed that unmelted YSZ particles were the potential weakness of the yttria stabilized zirconia reinforced HA coatings.

**[40] STRAIN HETEROGENEITY AND THE PRODUCTION OF COARSE GRAINS IN MECHANICALLY ALLOYED IRON-BASED PM2000 ALLOY**

Capdevila C. Miller U. Jelenak H. Bhadeshia HKDH. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 316(1-2):161-165, 2001

Mechanically alloyed iron-based ODS alloys have the potential for application in heat exchangers for biomass processing, with gas operating temperatures and pressures of approximately 1100 degreesC and 15-30 bar. The yttria dispersion in such alloys improves the high-temperature creep and stress rupture life. The elevated temperature strength is enhanced by the development of a coarse-grained microstructure during recrystallisation. Factors controlling the evolution of this desirable microstructure are explored in this work, focusing specifically on PM2000, which is a yttria dispersion strengthened, mechanically alloyed material. The microstructure following mechanical alloying and consolidation is fine and uniform, making it difficult to nucleate recrystallisation. Therefore, the introduction of a strain heterogeneity in the microstructure is found to promote the nucleation of recrystallisation. In contrast, uniform deformation reduces the chances of nucleation and hence leads to the development of a coarser microstructure.

**[39] DISSOLUTION OF CARBON IN NI-1AT.% FE UPON STRONG COLD DEFORMATION**

Mukoseev AG. Shabashov VA. Sagaradze VV. Sagaradze IV. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 316(1-2):174-181, 2001

Smelted Ni-C alloys (containing the Fe-57 isotope for Mossbauer studies) and powder mixtures of Ni + C (thermal black), Ni + Fe<sub>3</sub>C and Ni + VC were cold-deformed by compression and shear in Bridgman anvils to produce interstitial solid solutions of carbon in nickel. Mossbauer spectroscopic and X-ray structure analyses showed that the carbon concentration of the Ni-C solid solution was 1.5-2 at.%, which was several times smaller than the carbon concentration of Fe-Ni-C alloys produced under similar conditions. The type of carbon-containing particles determined the kinetics but had little effect on the limiting concentration of the synthesized Ni-C solid solutions. Ni-C was formed at a higher degree of deformation than Fe-C.

**[38] MECHANISTIC ASPECTS AND ATOMIC-LEVEL CONSEQUENCES OF ELASTIC INSTABILITIES IN HOMOGENEOUS CRYSTALS**

Yip S. Li J. Tang MJ. Wang JG. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 317(1-2 Special Issue SI):236-240, 2001

Elastic stability criteria, derived for a homogenous lattice under arbitrary external load, provide an upper bound on the critical stress or strain which a crystalline solid can withstand. Since the onset of a structural instability is load dependent, the corresponding theoretical strength can be given similar interpretation. When combined with molecular dynamics simulation, such results allow a systematic discussion of competing structural transitions, as illustrated here in a case study of pressure-induced polymorphic and crystal-to-amorphous transitions and in some remarks on the thermoelastic mechanism for homogeneous (mechanical) melting, a process which defines an upper limit of metastability.



**[37] DIRECT REDUCTION OF MECHANICALLY ACTIVATED GALENA AND SPHALERITE WITH HYDROGEN**

Balaz P. Briancin J. - Journal of Thermal Analysis. 65(3):769-776, 2001.

In this paper the reduction of lead and zinc sulphide by hydrogen is described. It has been found that the rate of formation of elemental lead or zinc is favourably affected by mechanical activation of PbS and ZnS produced by intensive grinding. This effect was observed in the region 678-1048 K for galena and in the region 851-1023 K for sphalerite. It has appeared that disordering in the structure of both minerals results in the decrease in experimental activation energy

**[36] REDUCTION PROCESSES IN THE COURSE OF MECHANOCHEMICAL SYNTHESIS OF  $\text{Li}_{1+x}\text{V}_3\text{O}_8$**

Kosova NV. Vosel SV. Anufrienko VF. Vasenin NT. Devyatkina ET. - Journal of Solid State Chemistry. 160(2):444-449, 2001

Mechanical activation (MA) of the  $\text{LiOH} + \text{V}_2\text{O}_5$  and  $\text{Li}_2\text{CO}_3 + \text{V}_2\text{O}_5$  mixtures followed by brief heating at 673 K was used to prepare dispersed  $\text{Li}_{1+x}\text{V}_3\text{O}_8$ . It was shown that structural transformations during MA are accompanied by reduction processes. EPR spectra of  $\text{Li}_{1+x}\text{V}_3\text{O}_8$  are attributed to vanadyl  $\text{VO}_2^+$  ions with weak exchange interaction. The interaction of localized electrons ( $\text{V}^{4+}$  ions) with electron gas (delocalized electrons), which is exhibited through the dependence of EPR line width of vanadium ions versus measurement temperature (C-S-C relaxation), is revealed. It is shown that C-S-C relaxation is different for intermediate and final products. The properties of mechanochemically prepared  $\text{Li}_{1+x}\text{V}_3\text{O}_8$  are compared with those of HT- $\text{Li}_{1+x}\text{V}_3\text{O}_8$ , obtained by conventional solid state reaction. Mechanochemically prepared  $\text{Li}_{1+x}\text{V}_3\text{O}_8$  is characterized by a similar amount of vanadium ions, producing electron gas, but a higher specific surface area

**[35] MECHANOCHEMICAL SYNTHESIS OF  $\text{LaOX}$  ( $X = \text{Cl}, \text{Br}$ ) AND THEIR SOLID STATE SOLUTIONS**

Lee J. Zhang QW. Saito F. - Journal of Solid State Chemistry. 160(2):469-473, 2001

A mixture of lanthanum oxide ( $\text{La}_2\text{O}_3$ ), chloride ( $\text{LaCl}_3$ ), and bromide ( $\text{LaBr}_3$ ) was ground in air by a planetary ball mill to investigate synthesis of lanthanum oxychloride ( $\text{LaOCl}$ ), oxybromide ( $\text{LaOBr}$ ), and their solid solutions,  $\text{LaOCl}_{1-x}\text{Br}_x$  (0 less than or equal to x less than or equal to 1,  $\Delta x = 0.25$ ). The synthesizing reactions proceed with an increase in grinding time. Unit cell dimensions, a, c, and lattice volume of the solutions evolve linearly with an increase in x in the  $\text{LaOCl}_{1-x}\text{Br}_x$  series. Comparing unit cell dimensions of  $\text{LaOX}$  synthesized by mechanochemical reaction to those of  $\text{LaOX}$  synthesized by solid-state reaction at high temperature, there is no difference in the length of c, while a is shortened slightly. This may be attributed to the complex cation layer of  $(\text{LaO})(n)(n+)$ , with a close relationship to a of the cell dimensions, being affected by the intensive grinding.

**[34] NMR INVESTIGATIONS ON ION DYNAMICS AND STRUCTURE IN NANOCRYSTALLINE AND POLYCRYSTALLINE  $\text{LiNbO}_3$**

Bork D. Heitjans P. - Journal of Physical Chemistry B. 105(38):9162-9170, 2001

Nanocrystalline (n-)  $\text{LiNbO}_3$  samples with average grain sizes between 16 and 105 nm were prepared from polycrystalline (p-) material with an average grain size of the order of one micrometer by high-energy ball milling. NMR investigations of (i) the Li-7 spin-lattice relaxation (SLR) rate  $T_1^{-1}$  in the laboratory and  $T_1^{-1e}$  in the pulsed rotating reference frame and of (ii) Li-7 spectra, in particular line shapes and motional narrowing (MN) of the central line, were performed in the temperature range from 300 K to a maximum of 1400 K in the case of p- $\text{LiNbO}_3$  and from 140 to 460 K in the case of n- $\text{LiNbO}_3$ . The following results were obtained. (1) The SLR rate measurements yield an apparent activation energy of the Li diffusion in n- $\text{LiNbO}_3$  that is about 1/3 of the value obtained for the p-material. (2) The frequency dependence of the SLR rate according to  $T_1^{-1e}$  is proportional to  $\nu^{-\beta}$  with  $\beta$  in the range from 1.1 to 1.5 as well as the asymmetry of the diffusion-induced peak in the  $\log T_1^{-1e}$  vs  $T_1^{-1}$  diagram of p- $\text{LiNbO}_3$  are proving non-BPP behavior for both samples. (3) In n- $\text{LiNbO}_3$  MN starts already at 250 K, i.e., about 400 K lower than in p- $\text{LiNbO}_3$ , and reflects an apparent activation energy that is approximately 1/3 of the value found for the p-material. (4) In contrast to p- $\text{LiNbO}_3$ , with increasing temperature the Li-7 NMR spectra of n- $\text{LiNbO}_3$  are revealing a characteristic structure of the central line, namely a superposition of two contributions. This is regarded as a consequence of the different dynamic properties of atoms in the interfacial regions (IR) and in the grains. From the spectrum at 450 K the fraction of atoms belonging to IR can be estimated. (5) The intensities of the quadrupole satellites showing different temperature dependencies in the p- and n-samples are indicating some exchange between the two spin reservoirs 'IR' and 'grains'. This leads to the hypothesis that n-ceramics cannot simply be regarded as heterogeneous materials where the two types of zones, i.e., IR and grains, are independent and closed

**[33] PHASE TRANSFORMATION AND MAGNETIC PROPERTIES OF  $\text{SmCo}_7\text{-xBx}$  ALLOYS PREPARED BY MECHANICAL ALLOYING**

You CY. Zhang ZD. Sun XK. Liu W. Zhao XG. Geng DY. - Journal of Magnetism & Magnetic Materials. 234(3):395-400, 2001

The phase transformation and magnetic properties of  $\text{SmCo}_7\text{-xBx}$  ( $x = 0, 0.2, 0.5$  and 1) alloys prepared by mechanical alloying have been investigated systematically. The coercivities of the alloys without B increase with increasing annealing temperature, as a consequence of complete crystallization of TbCu<sub>7</sub>-type phase. The substitution of B for Co is favorable to the formation of Th<sub>2</sub>Zn<sub>17</sub>- and CaCu<sub>5</sub>-type phases when annealed at 650 degreesC, accompanied by the enhancement of the coercivities. Increasing the annealing temperature causes the formation of soft magnetic phase SM<sub>2</sub>Co<sub>14</sub>B in the B-substituted alloys. In the alloys with  $x = 0.5$  and 1 annealed at 850 degreesC, the major phase is SM<sub>2</sub>Co<sub>14</sub>B, which degrades the magnetic properties sharply. A remanence enhancement has been observed in the  $\text{SmCo}_7\text{-xBx}$  alloys due to the exchange coupling of the nanoscale structure



**[32] PLASMOCHEMICAL PREPARATION OF NI-CONTAINING ZEOLITES**

Shermatov N. Normatov IS. Mirsaidov U. Rasulov UZ. - Inorganic Materials. 37(9):960-962, 2001

Ni-containing zeolite catalysts were prepared by impregnating a zeolite with a nickel chloride solution, followed by plasmochemical processing. The effects of crystallite size and mechanical-activation time on the activity of the resultant catalysts were studied

**[31] HIGH LITHIUM ION CONDUCTIVITY OF GLASS-CERAMICS DERIVED FROM MECHANICALLY MILLED GLASSY POWDERS**

Hayashi A. Hama S. Morimoto H. Tatsumisago M. Minami T. - Chemistry Letters. (9):872-873, 2001

Crystallization of the 80Li(2)S . 20P(2)S(5) (mol%) glassy powders derived from mechanical milling improved their conductivities. The obtained glass-ceramic exhibited high conductivity of approximately  $10^{-3}$  S cm<sup>-1</sup> at room temperature. The stabilization of the high-temperature phase of Li<sub>7</sub>PS<sub>6</sub> crystal in the glass matrix brought about those high conductivities

**[30] SELF-ASSEMBLY OF PHOTOLUMINESCENT SILICON FILMS: INFLUENCE OF DOPING ON THE PHYSICAL PROPERTIES**

Di Francia G. La Ferrara V. Morvillo P. Lettieri S. Maddalena P. - Applied Physics Letters. 79(14):2202-2204, 2001

Thin photoluminescent silicon films are fabricated by means of a purely wet-chemical process using, as a starting material, a fine powder obtained by ball milling p- and n-type silicon wafers. The reaction is characterized by a coalescence phenomenon and produces photoluminescent films whose physical properties depend on the material type. Samples fabricated by processing a mixture of p- and n-type powders exhibit different photoluminescent spectra, have lower reactivity towards oxidating environments, and show the longest emission lifetimes. In order to explain those properties, we propose that, as long as the reaction proceeds and consumes the silicon powders, nanostructures containing both p- and n-type silicon form. Suppression of the Auger recombination in such structures can account for the experimental findings

**[29] FORMATION OF SUPERSATURATED SINGLE-PHASE BCC SOLID SOLUTIONS IN FE-ZN BINARY SYSTEM BY MECHANICAL ALLOYING**

Zhou F. Chou YT. Lavernia EJ. - Materials Transactions. 42(8):1566-1570, 2001

Mechanical alloying (MA) was used in the investigation of the Fe-Zn binary system, which, in the equilibrium state, exhibits, negligible mutual solid solubility at room temperature. The formation of single-phase bcc solid solutions with a nanometer-scaled microstructure was achieved by MA in Fe<sub>100-x</sub>Zn<sub>x</sub> (x less than or equal to 65) powder mixtures. The nearest neighbor distances of these Fe-Zn solid solutions obey the linear relationship given by the Vegard's law for ideal solutions, suggesting that the solid solutions formed by MA are homogeneous. The average lattice volume expansion also linearly increases to about 13.4 % at 65% Zn. At elevated temperatures the nanostructured solid solutions undergo phase transformations yielding intermetallic compound. The mechanisms for the phase formation in the Fe-Zn system are discussed

**[28] THE LIQUID-ENHANCED PLASTICITY AND DEFORMATION BEHAVIOR OF CU-MG-TiC NANOCRYSTALLINE COMPOSITE**

Shen BL. Yamasaki T. Ogino Y. Kimura H. Inoue A. - Materials Transactions. 42(8):1582-1587, 2001

Cu-Mg-TiC bulk nanocrystalline composites were prepared by mechanical alloying, HIP process and hot-rolling. Tensile tests at constant crosshead speeds were carried out using a universal testing machine at several different temperatures under an argon atmosphere. The elongation drastically increased as the fraction of melt increased, and a maximum normal elongation of about 200 % was obtained at temperature where atomic fraction of liquid phase was about 0.5 and the strain rate sensitivity m was in the range of 0.8 to 1.0 for every composite. After the deformation, nanocrystalline structures with average grain sizes of about 30 nm were retained.

**[27] MG-BASED HYDROGEN STORAGE MATERIALS WITH IMPROVED HYDROGEN SORPTION**

Oelerich W. Klassen T. Bormann R. - Materials Transactions. 42(8):1588-1592, 2001

Nanocrystalline MgH<sub>2</sub>/MexO<sub>y</sub> composite powders were produced by high energy ball milling (MexO<sub>y</sub> = ScO<sub>3</sub>, TiO<sub>2</sub>, V<sub>2</sub>O<sub>5</sub>, Cr<sub>2</sub>O<sub>3</sub>, Mn<sub>2</sub>O<sub>3</sub>, Fe<sub>3</sub>O<sub>4</sub>, CuO, Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>). The hydrogen absorption and desorption kinetics were determined in view of a technical application. The addition of selected oxides lead to an enormous catalytic acceleration of hydrogen sorption compared to pure nanocrystalline hydrides. In absorption, the catalytic effect of TiO<sub>2</sub>, V<sub>2</sub>O<sub>5</sub>, Cr<sub>2</sub>O<sub>3</sub>, Mn<sub>2</sub>O<sub>3</sub>, Fe<sub>3</sub>O<sub>4</sub>, and CuO is comparable. Concerning desorption, the composite material containing Fe<sub>3</sub>O<sub>4</sub> shows the fastest kinetics followed by V<sub>2</sub>O<sub>5</sub>, Mn<sub>2</sub>O<sub>3</sub>, Cr<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub>. Only 0.2 mol% of the catalysts is sufficient to provide a fast sorption kinetics. Additionally, Mg absorbs hydrogen already at room temperature by the use of metal oxides as catalysts. Furthermore, it is demonstrated for the first time that MgH<sub>2</sub>/MexO<sub>y</sub>-powders release hydrogen at 200 degreesC.

**[26] MECHANICALLY ALLOYED NANOCRYSTALLINE HYDROGEN STORAGE MATERIALS**

Liang GX. Schulz R. - Materials Transactions. 42(8):1593-1598, 2001

Mechanical alloying and mechanical grinding have been used to synthesize or treat AB, AB(2), and AB(5) type alloys and Mg-based hydrogen storage materials. A nanocrystalline or an amorphous structure was obtained after milling, depending on the system and its composition. The structure and the hydrogen storage properties of various nanocrystalline alloys were investigated. It was found that the activation and kinetics of hydrogen absorption/desorption were improved. However, a severe loss of storage capacity was observed in the AB, AB(2) and AB(5) systems, but not much in Mg-based systems. The storage capacity can be recovered by thermal annealing at elevated temperatures. A preliminary explanation is given for the change of absorption/desorption kinetics and storage capacity.

**[25] SYNTHESIS AND RELATED KINETICS OF NANOCRYSTALLINE Ni BY HYDROGEN REDUCTION OF NiO**



Lee JS. Kim BS. - Materials Transactions. 42(8):1607-1612, 2001

The present study has attempted to elucidate the formation mechanism of nanocrystalline (nc) Ni by hydrogen reduction of fine NiO powder in terms of related kinetics aspects. So, the kinetics and related mechanism of hydrogen reduction of NiO were investigated on the basis of structure modification of the NiO powder during reaction. The ball-milled NiO agglomerate powder having 20 nm in grain size and a log-normal pore size distribution was used for study, The non-isothermal reduction study showed that the nano-agglomerate NiO underwent a two-step reduction process which is presumably due to a chemical reaction at lower temperatures and a diffusion controlled process at higher temperatures. The activation energy for the nano-agglomerate NiO was 85.4 kJ/mol for lower temperatures and 105.1 kJ/mol for higher temperatures. The value for lower temperatures is consistent with that of as-received NiO of 85.6 kJ/mol. Such higher activation energy for higher temperatures can be attributed to the retardation of the reduction process by the change in the reduction mechanism from the chemical reaction to the diffusion process. Conclusively, the structure change during the reduction is believed to be responsible for the change in the reduction mechanism

**[24] MECHANOCHEMICAL SYNTHESIS OF NIOBIUM PENTOXIDE NANOPARTICLES**

Tsuzuki T. McCormick PG. - Materials Transactions. 42(8):1623-1628, 2001

Synthesis of Nb<sub>2</sub>O<sub>5</sub> nanoparticles by mechanochemical processing has been investigated. The reactions 2NbCl(5) + 5Na(2)CO(3) --> Nb<sub>2</sub>O<sub>5</sub> + 10NaCl + 5CO(2)(g) and 2NbCl(5) + 5MgO --> Nb<sub>2</sub>O<sub>5</sub> + 5MgCl(2) led to the formation of Nb<sub>2</sub>O<sub>5</sub> aggregates of 100-1000 nm in size. Addition of NaCl in the starting mixture of 2NbCl(5) + 5Na(2)CO(3) prevented the aggregates forming. After heat treatment of the as-milled powder at 300 degreesC, amorphous Nb<sub>2</sub>O<sub>5</sub> having a high surface area of 196 m(2)/g was obtained. Heat treatment at 550 degreesC resulted in a powder consisting of 10-100 nm single crystal Nb<sub>2</sub>O<sub>5</sub> Particles along with a network of ultrafine amorphous particles. Annealing at higher temperature led to the formation of the Na<sub>2</sub>Nb<sub>2</sub>O<sub>11</sub> Phase

**[23] CRYSTALLIZATION AND THERMAL STABILITY OF MECHANICALLY ALLOYED W-NI-FE NONCRYSTALLINE MATERIALS**

He Z. Courtney TH. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 315(1-2):166-173, 2001

Tungsten-nickel noncrystalline alloys containing remnant nanocrystalline W particles have been synthesized by mechanical alloying (MA). Two initial compositions (50 and 75 at.% W) milled for various times have been investigated. The W content of the noncrystalline matrix depends on the MA (milling) time and the overall alloy W content. For 50 at.% W alloys, three exothermic reactions - monitored by differential scanning calorimetry - take place on continuous heating of the alloy. Crystalline W first precipitates from the noncrystalline matrix at about 825 K. This is followed by partial crystallization (to an fcc phase) of the noncrystalline matrix. At even higher temperatures, two intermetallics (NiW and, to a lesser extent, Ni<sub>3</sub>W) form from the remaining Ni-W noncrystalline matrix along with additional amounts of the fcc and W phases. Similar crystallization processes occur for 75 at.% W alloys milled for less than 15 h. However, when milled for times exceeding 15 h, only one exothermic reaction occurs in these alloys. The crystallization temperature is found to increase with increasing W content in the noncrystalline phase.

**[22] MECHANOCHEMICAL MODIFICATION OF POLYSTYRENE AND POLYMETHYLMETHACRYLATE**

Schmidt-Naake G. Frenzel A. Drache M. Janke G. - Chemical Engineering & Technology. 24(9):889-894, 2001

**[21] SIZE EFFECTS IN THE CHEMISTRY OF HETEROGENEOUS SYSTEMS [REVIEW] [RUSSIAN]**

Uvarov NF. Boldyrev VV. - Uspekhi Khimii. 70(4):307-329, 2001.

Size effects in one and multi-component solid-phase systems are discussed. Specific features of the size effects in microcrystalline and nano-sized systems are noted. Methods for the mechanochemical synthesis of nanoparticles are considered. The chemical properties of nano-sized systems are analysed. Particular attention is devoted to the results of studies of the properties of nanocomposites and nanoparticles. The problems related to their stabilities and mechanochemical synthesis are discussed

**[20] CRYSTALLIZATION AND THERMAL STABILITY OF MECHANICALLY ALLOYED W-NI-FE NONCRYSTALLINE MATERIALS**

He Z. Courtney TH. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 315(1-2):166-173,

Tungsten-nickel noncrystalline alloys containing remnant nanocrystalline W particles have been synthesized by mechanical alloying (MA). Two initial compositions (50 and 75 at.% W) milled for various times have been investigated. The W content of the noncrystalline matrix depends on the MA (milling) time and the overall alloy W content. For 50 at.% W alloys, three exothermic reactions - monitored by differential scanning calorimetry - take place on continuous heating of the alloy. Crystalline W first precipitates from the noncrystalline matrix at about 825 K. This is followed by partial crystallization (to an fcc phase) of the noncrystalline matrix. At even higher temperatures, two intermetallics (NiW and, to a lesser extent, Ni<sub>3</sub>W) form from the remaining Ni-W noncrystalline matrix along with additional amounts of the fcc and W phases. Similar crystallization processes occur for 75 at.% W alloys milled for less than 15 h. However, when milled for times exceeding 15 h, only one exothermic reaction occurs in these alloys. The crystallization temperature is found to increase with increasing W content in the noncrystalline phase

**[19] CREEP PROPERTIES OF EUTECTIC SN-3.5AG SOLDER JOINTS REINFORCED WITH MECHANICALLY INCORPORATED NI PARTICLES**

Guo F. Lee J. Lucas JP. Subramanian KN. Bieler TR. - Journal of Electronic Materials. 30(9):1222-1227, 2001



The creep deformation behavior of eutectic Sn-3.5Ag based Ni particle reinforced composite solder joints was investigated. The Ni particle reinforced composite solder was prepared by mechanically dispersing 15 vol.% of Ni particles into eutectic Sn-3.5Ag solder paste. Static-loading creep tests were carried out on solder joint specimens at 25 degreesC, 65 degreesC, and 105 degreesC, representing homologous temperatures ranging from 0.6 to 0.78. A novel-design, miniature creep-testing frame was utilized in this study. Various creep parameters such as the global and localized creep strain, steady-state creep rate, onset of tertiary creep, and the activation energy for creep were quantified by mapping the distorted laser ablation pattern imprinted on the solder joint prior to testing. The Ni-reinforced composite solder joint showed improved creep resistance compared to the results previously reported for eutectic Sn-3.5Ag solder, Sn-4.0Ag-0.5Cu solder alloys, and for eutectic Sn-3.5Ag solder reinforced with Cu or Ag particle reinforcements. The activation energy for creep was similar to 0.52 eV for Sn-3.5Ag and Sn-4Ag-0.5Cu solder alloys. The activation energies ranged from 0.55-0.64 eV for Cu, Ag, and Ni reinforced composite solder joints, respectively. Most often, creep fracture occurred closer to one side of the solder joint within the solder matrix

**[18] THE EFFECT OF MECHANICAL ACTIVATION ON THE SYNTHESIS OF BIOCOMPATIBLE CA-10(PO<sub>4</sub>)(<sub>6</sub>)(OH)(<sub>2</sub>)**

Zakharov NA. Toporov YP. Klyuev VA. Orlovskii VP. - Technical Physics Letters. 27(9):746-748, 2001

The effect of mechanical activation on the course of reactions involved in the synthesis of calcium hydroxyapatite Ca-10(PO<sub>4</sub>)(<sub>6</sub>)(OH)(<sub>2</sub>) was studied; the composition, crystallographic parameters, spectroscopic characteristics, and dielectric properties of the products were determined. The role of the composition of initial components on the rate of synthesis is analyzed

**[17] PHASE FORMATION AND PROPERTIES OF MECHANICALLY ALLOYED AMORPHOUS AL<sub>85</sub>Y<sub>8</sub>NI<sub>5</sub>CO<sub>2</sub>**

Borner I. Eckert J. - Scripta Materialia. 45(2):237-244, 2001

Amorphous aluminum-based alloys with more than 80 at.% Al are promising new high-strength lightweight materials. Such alloys can be produced by quenching from the melt or by powder metallurgy. This paper focusses on phase formation and properties of Al<sub>85</sub>Y<sub>8</sub>Ni<sub>5</sub>Co<sub>2</sub> prepared by mechanical alloying and consolidation

**[16] THE CHARACTERISTICS OF MECHANICALLY ACTIVATED MIXTURES OF COPPER HYDROXOCARBONATE WITH ALUMINIUM**

Wieczorek-Ciurowa K. Shirokov JG. Parylo M. Gamrat K. - Journal of Thermal Analysis. 65(2):359-366, 2001.

The chemical and physical processes occurring during grinding of copper hydroxocarbonates mixtures with aluminium were studied. A planetary ball mill was used. A thermogravimetry and X-ray powder diffraction method allowed to determine the composition of solid products after mechanical activation. The amount of the Cu-2(OH)(<sub>2</sub>)CO<sub>3</sub> undecomposed and Al<sub>2</sub>O<sub>3</sub>. 3H(<sub>2</sub>)O, CuO, Al<sub>2</sub>O<sub>3</sub>, Cu-0, Cu<sub>x</sub>Al<sub>y</sub> alloys and remained Al-0 in the systems is strongly dependent on the duration of grinding and on the proportion of components. The comparative results are presented

**[15] SYNTHESIS OF TITANIUM CARBIDE AND TITANIUM DIBORIDE BY MECHANOCHEMICAL DISPLACEMENT REACTION**

Kudaka K. Iizumi K. Izumi H. Sasaki T. - Journal of Materials Science Letters. 20(17):1619-1622, 2001.

**[14] EFFECT OF SI POWDER REFINING ON THE SELF-PROPAGATING HIGH TEMPERATURE SYNTHESIS REACTION OF TITANIUM SILICIDE INDUCED BY MECHANICAL ALLOYING**

Lee WH. Reucroft PJ. Byun CS. Kim DK. - Journal of Materials Science Letters. 20(17):1647-1649, 2001.

**[13] SiO<sub>2</sub> MODIFIED CO-FERRITE WITH HIGH COERCIVITY**

Ding J. Gong H. Melaka R. Wang S. Shi Y. Chen YJ. Phuc NX. - Journal of Magnetism & Magnetic Materials. 226(Part 2 Special Issue SI):1382-1384,

Magnetic and Mossbauer measurements have shown that 1-2 wt% of SiO<sub>2</sub> were solved in the CoFe<sub>2</sub>O<sub>4</sub> structure after mechanical milling and subsequent heat treatment. Coercivity values up to 3.5 kOe were measured for CoFe<sub>2</sub>O<sub>4</sub>/SiO<sub>2</sub> powders. High coercivities were also achieved in SiO<sub>2</sub> doped Co-ferrite thin films prepared by sputtering technique. The Co-ferrite thin film deposited on silicon wafer using a 5 Wt%-SiO<sub>2</sub>/Co-ferrite target possessed a coercivity of 7.4 kOe, which is the highest value in Co-ferrite and spinel materials according to our knowledge

**[12] INFLUENCE OF MECHANICAL STRESS ON THE MICROSTRUCTURE OF AS-CAST FE-74(Nd<sub>1-x</sub>MM<sub>x</sub>)(<sub>20</sub>)B-6 ALLOYS, MM = BRAZILIAN MISCH METAL**

Alves CS. Cabral FAO. de Souza CP. Gama S. Ribeiro CA. Araujo RC. - Journal of Magnetism & Magnetic Materials. 226(Part 2 Special Issue SI):1455-1457, 2001

Partial replacement of neodymium by cerium-rich Brazilian misch metal, has been studied on Fe-74(Nd<sub>1-x</sub>MM<sub>x</sub>)(<sub>20</sub>)B-6 alloys by X-ray diffraction, thermomagnetic analysis, microprobe analysis and optical microscopy. Misch metal refers to a mixture of light rare-earth metals containing 56% Ce, 18% La, 13% Nd, 05% Pr, 02% Fe and 10% others rare-earths (wt%). Intermetallic compounds of Fe-74(Nd<sub>1-x</sub>MM<sub>x</sub>)(<sub>20</sub>)B-6 with x = 0.5, 0.7 and 0.9 were investigated. As-cast, pre-milled alloys present a binary microstructure with the Fe<sub>14</sub>Nd<sub>2</sub>B compound as matrix phase and Fe<sub>2</sub>MM. The amount of Fe<sub>2</sub>MM increases and the Curie temperature of the 14:2:1 compound decreases with increasing x. When these samples are submitted to 30 min of very high energy ball milling, the signal of the 14:2:1 phase disappears and the formation of a microstructure is observed with a new ferromagnetic stable phase with T<sub>c</sub> around 500 degreesC together with an increase of alpha -Fe in comparison for pre-milled samples

**[11] STRUCTURAL AND MAGNETIC STUDIES OF NANOCRYSTALLINE (Fe<sub>2</sub>CO)<sub>(30)</sub>CU-70 ALLOYS**

Nascimento VP. Passamani EC. Takeuchi AY. Larica C. Nunes E. Alves KMB. Journal of Magnetism & Magnetic Materials. 226(Part 2 Special Issue SI):1493-1495,



Structural and magnetic properties of nanocrystalline Fe<sub>2</sub>Co and (Fe<sub>2</sub>Co)<sub>30</sub>Cu-70 alloys prepared by ball milling have been studied by X-ray, Mossbauer and magnetization measurements. The X-ray and Mossbauer results of Fe<sub>2</sub>Co milled alloy indicate a formation of pure BCC phase with B-hf of 36 T. Samples of (Fe<sub>2</sub>Co)<sub>30</sub>Cu-70 alloys were prepared either by milling the mixture of Fe<sub>2</sub>Co alloy with pure Cu powder (series I) or by milling the elemental powder mixture (series II). The X-ray and magnetization results of the final material (series I and II) indicate the formation of alloy with T-C at about (420 +/- 1) K. Heat treatment of samples I and II induces precipitation of single-domain particles in Cu matrix

**[10] MILL PROCESSABILITY OF BROMINATED ISOBUTYLENE-CO-PARAMETHYLSTYRENE AND ITS BLENDS WITH EPDM**

Kumar B. De PP. De SK. Bhowmick AK. Peiffer DG. - Journal of Applied Polymer Science. 82(6):1483-1494, 2001  
Milling behavior of brominated isobutylene-co-paramethylstyrene (BIMS) and its blends with ethylene propylene diene terpolymer (EPDM) rubber, was investigated over a range of temperatures and friction ratios in a drop mill operation. BIMS showed striking changes, that is, from a loose nery band to a tight elastic band, as the temperature of the rolls was increased from 30 degreesC to 90 degreesC . For EPDM a loose band was observed at all temperatures and friction ratios studied. For the blends of BIMS and EPDM, the milling behavior changed from a tight elastic band to a loose bagging band on increasing the EPDM content. The critical nip gap (CNG), at which the front-to-back roll (F-B) transition occurred, was also measured. BIMS showed a much higher value of CNG than that of EPDM, indicating that the former had a significantly higher tendency for F-B transition than the latter material. For different blends of BIMS and EPDM, the CNG decreased on increasing the EPDM content, indicating a decrease in the tendency for F-B transition. The results were explained in terms of the rubber-to-metal adhesion and the viscosity of the polymers

**[9] THE EFFECT OF NI CONTENT ON THE ELECTROCHEMICAL AND SURFACE CHARACTERISTICS OF MG90-XTi10NiX (X=50, 55, 60) TERNARY HYDROGEN STORAGE ELECTRODE ALLOYS**

Zhang Y. Liao B. Chen LX. Lei YQ. Wang QD. - Journal of Alloys & Compounds. 327(1-2):195-200, 2001  
Mg-based ternary Mg<sub>90-x</sub>Ti<sub>10</sub>Ni<sub>x</sub> (x = 50, 55, 60) electrode alloys of different Ni content, were prepared by mechanical alloying (MA) in this work. The main phase of these alloys is amorphous according to the XRD analysis. The electrochemical test indicates that the cycling stability of the electrodes made of these alloys improves with increasing Ni content, while the initial discharge capacity decreases dramatically. XPS analysis reveals that the outmost surface layer on the Mg-Ti-Ni alloys is a Mg(OH), passive film, in which the content of Mg increases, and the degree of oxidation of Mg becomes higher as the cycling goes on. Below the Mg(OH)<sub>2</sub> film is a composite layer of several oxides including NiO, TiO<sub>2</sub> and Mg(OH)<sub>2</sub>, This layer is insoluble and compact, and helps to inhibit further corrosion of the fresh alloy surface underneath. In this work, it was also found that with increasing Ni content, the high-rate dischargeability (HRD) and the exchange current density of the ternary Mg-Ti-Ni alloys are significantly increased. Metallic Ni particles generated by reduction from NiO during charging are regarded as the cause for the improved electrochemical activation.

**[8] LOCATION OF DEUTERIUM ATOMS ABSORBED IN NANOCRYSTALLINE GRAPHITE PREPARED BY MECHANICAL ALLOYING**

Fukunaga T. Itoh K. Orimo S. Aoki M. Fujii H. - Journal of Alloys & Compounds. 327(1-2):224-229, 2001  
Neutron diffraction is an important technique for studying the structure of hydride materials, H/D isotopic substitution was employed to observe the location of deuterium atoms in deuterated nano-graphite because the coherent scattering length of deuterium is large enough to be observed in comparison with that of the metal atoms forming hydrogen absorbing materials. The preparation of the nano-crystalline graphite and the absorption of deuterium atoms in the graphite were carried out simultaneously by mechanical alloying under D-2 gas atmosphere. The transformation from the hexagonal graphite as a starting material into amorphous-like carbon was observed by neutron diffraction. The conformation in the graphite was changed by the creation of dangling bonds and deuterium was absorbed by the solid-gas reaction when the milling proceeded. Two types of the deuterium coordinations were found in radial distribution function, RDF(r). One is the C-D covalent bond and the other is deuterium located between layers of the graphite

**[7] HYDROGEN SORPTION PROPERTIES OF AN MG-TI-V-FE NANOCOMPOSITE OBTAINED BY MECHANICAL ALLOYING**

Khrusanova M. Grigorova E. Mitov I. Radev D. Peshev P. - Journal of Alloys & Compounds. 327(1-2):230-234, 2001  
The absorption-desorption characteristics with respect to hydrogen of a magnesium-based nanocomposite obtained by high-energy ball milling have been investigated. The composite contains 5 wt.% (similar to 3at.%) Ti, 10 wt.% (similar to 5.5 at.%) V and 10 wt.% (similar to 5 at.%) Fe, of which the former two transition metals only form a binary hydride. It has been shown that at 623 K the composite may be hydrided up to a very high absorption capacity whose values remain appropriate for practical purposes even at much lower hydriding temperatures. Part of the iron present in the composite has been found to interact with magnesium and hydrogen under the hydriding conditions, the ternary hydride Mg<sub>2</sub>FeH<sub>6</sub> being formed. Its presence in the composite-hydrogen system has been assumed to be responsible for the reduced rate of hydrogen desorption from the particle surfaces and for some peculiarities of the composite behaviour during hydriding

**[6] MECHANICALLY ACTIVATED TRANSFORMATIONS IN THE COORDINATION SPHERE OF PLATINUM COMPLEXES INDUCED BY IMPACT GRINDING OF SOLID K<sub>2</sub>PTX<sub>6</sub> (X = CL, BR) AND K<sub>2</sub>PTCL<sub>4</sub> SALTS**

Mitchenko SA. Khomutov EV. Kovalenko VV. Popov AF. Beletskaya IP. - Inorganica Chimica Acta. 320(1-2):31-37, 2001

Mechanical treatment of solid K<sub>2</sub>PtX<sub>6</sub> (X = Cl, Br) salts under air or argon leads to the formation of paramagnetic platinum(III) complexes via homolytic cleavage of Pt-X bonds. Lewis acid sites (LASS) were also detected on the surface of



mechanically activated  $K_2PtCl_6$  using a paramagnetic probe. The latter species can be attributed to coordinatively unsaturated platinum(IV) complexes formed as a result of heterolysis of Pt-Cl bonds. Mechanical treatment of solid  $K_2PtCl_4$ , on the contrary, does not lead to homolytic Pt-Cl bond cleavage. In this case only heterolysis of the Pt-Cl bond takes place, leading to the formation of coordinatively unsaturated platinum(II) complexes.

**[5] METHYL IODIDE REACTIONS ON THE SURFACE OF MECHANICALLY PRE-ACTIVATED PLATINUM(II) SALT IN THE HETEROGENEOUS SYSTEM:  $K_2PtCl_4$  POWDER-MEI VAPOR**

Mitchenko SA. Khomutov EV. Vdovichenko AN. Zhikharev IV. Popov AF. Beletskaya IP. - *Inorganica Chimica Acta*. 320(1-2):38-46, 2001

Mechanically pre-activated  $K_2PtCl_4$  salt consumes methyl iodide producing methyl chloride at room temperature. The reaction mechanism includes the following steps sequence: oxidative addition of methyl iodide to platinum(II) complexes with intermediate formation of methyl platinum(IV) complexes and further decomposition of the latter in the course of inner-sphere reductive elimination yielding methyl chloride. The first step of the reaction proceeds owing to the assistance of active centers regenerated in the course of each event of MeI into MeCl transformation taking part in the chain halogen substitution process. It could be assumed that the role of active centers is played by coordinatively unsaturated platinum(II) complexes located on the surface. These species bearing a positive efficient charge can render electrophilic assistance for the nucleophilic substitution. The chain termination can be caused by recombination of coordinatively unsaturated platinum(II) complexes and interstitial chloride ions forming an inactive  $K_2PtCl_4$  complex

**[4] MAGNETIC PROPERTIES AND MECHANICAL STRENGTH OF MNZN FERRITE**

Matsuo Y. Ono K. Hashimoto T. Nakao F. - *IEEE Transactions on Magnetics*. 37(4 Part 1):2369-2372, 2001

From MnZn ferrite ("6H20" of FDK production), cores having grain size of about 8 similar to 10  $\mu m$  were produced by controlling the calcination temperature, after-calcination milling time, and sintering conditions. The ferrite powder production conditions and core microstructure were examined to determine their effects on the magnetic properties and mechanical strength of MnZn ferrite cores. A tendency of core loss to decline with a decrease in grain size was confirmed. Strength increased also with a decrease in grain size. Thus, a relation similar to that between strength and fracture toughness in the Equation of Griffith-Irwin was noted. By these findings, it has become possible to produce MnZn ferrite cores with lower loss and greater strength.

**[3] HIGHLY COERCIVE MILLED AND MELT-SPUN (PR, ND)Fe14B-TYPE MAGNETS AND THEIR HOT WORKABILITY**

Bollero A. Kirchner A. Gutfleisch O. Muller KH. Schultz L. - *IEEE Transactions on Magnetics*. 37(4 Part 1):2483-2485, 2001

Highly coercive isotropic  $(Pr_{1-x}Nd_x)_2Fe_{14}B$ -type magnets (0 less than or equal to x less than or equal to 1) have been processed using high energy ball milling and melt-spinning techniques, and an assessment of their hot deformation behavior has been carried out. A very high coercivity of  $\mu H_0(c) = 2.7$  T and a remanence of  $J(r) = 0.72$  T were found after annealing partly amorphous  $Pr_{15}DyFe_{75.9}B_{8}Zr_{0.1}$  ribbons. Samples containing Dy (1at.%) and Zr (0.1at.%) exhibit an improved stability against grain growth with increasing the annealing temperature. It has been found that the deformation forces necessary for texturing by hot deformation are the lowest for the as-milled Pr-containing alloys. As-milled  $Pr_{15}Fe_{77}B$  has been textured by die-upsetting at 750 degreesC obtaining magnetic properties as high as  $J(r) = 1.27$  T,  $\mu H_0(c) = 1.20$  T and  $(BH)_{max} = 307$  kJ/m<sup>3</sup>

**[2] MAGNETIC PROPERTIES OF MECHANICALLY-ALLOYED SM-CO NANOPHASE HARD MAGNETS**

Gallagher K. Venkatesan M. Coey JMD. - *IEEE Transactions on Magnetics*. 37(4 Part 1):2528-2530, 2001

Highly coercive  $Sm_{14}Co_{86}$  powders were prepared by mechanical alloying with a view to enhance the high temperature magnetic properties by optimizing the annealing conditions. The powders were annealed in quartz tubes with a continuous vacuum of similar to  $10^{-6}$  mbar at different temperatures. X-Ray diffraction studies confirmed the formation of a disordered 2:17 phase. DTA measurement on the as-milled amorphous powders revealed a single crystallization event at around 500 degreesC. Magnetic measurements showed room temperature coercivities of at least 1 T and Curie temperatures around 820 degreesC. The behavior of the initial magnetization curve lies in between those observed for low H-c 2:17 precipitation hardened magnets and  $SmCo_5$  or  $Nd_2Fe_{14}B$  magnets. The magnitude of coercivity is found to be sensitive to annealing temperature. The detailed comparison of magnetic properties with annealing conditions will be presented.

**[1] SIGNIFICANT DIELECTRIC ENHANCEMENT IN  $0.3BiFeO_3$ - $0.7SrBi_2Nb_2O_9$**

Gu HS. Xue JM. Wang J. - *Applied Physics Letters*. 79(13):2061-2063, 2001

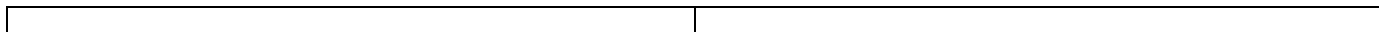
Significant dielectric enhancement is observed in  $0.3BiFeO_3$ - $0.7SrBi_2Nb_2O_9$ , when a single-phase layered perovskite structure was formed by sintering the mechanically activated oxide composition. The Curie point of  $xBiFeO_3$ - $(1-x)SrBi_2Nb_2O_9$  was shifted upward with an increase in the  $BiFeO_3$  content.  $0.3BiFeO_3$ - $0.7SrBi_2Nb_2O_9$  exhibits a dielectric constant of  $1.84 \times 10^5$  at the Curie point of 750 degreesC. The lattice dimensions of  $xBiFeO_3$ - $(1-x)SrBi_2Nb_2O_9$  decrease slightly with an increase in the content of  $BiFeO_3$  over the composition range of  $x=0-0.2$ , while 0.3 mol  $BiFeO_3$  in  $SrBi_2Nb_2O_9$  led to recovery in the lattice dimensions. The much enhanced dielectric properties observed in  $0.3BiFeO_3$ - $0.7SrBi_2Nb_2O_9$  are therefore due to the enlarged rattling space for both  $Nb^{5+}$  and in particular for smaller  $Fe^{3+}$ .



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