RESEAU FRANÇAIS DE MECANOSYNTHESE

Lettre N°80

Novembre 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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Le site web du RFM est :
http://www.bls.fr/amatech
Rubrique Pages Sciences et Techniques pour l'Ingénieur (Rubrique Sciences)
vous y trouverez les anciennes lettres du RFM (accessible par Adobe Acrobat)
les statuts du RFM ainsi que les annonces concernant les JRFM'2001 et quelques éléments mis a 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 21au 25 Octobre 2002)
Symposium 1 :
Poudres et Matériaux Nanostructurés,
du fondamental aux applications industrielles
Website : http://www.materiaux2002.net
E_mail : materiaux@materiaux2002.net

Attention :
la date limite pour les propositions de communications
est le 9 Novembre 2001

Sommaire

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

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Congress and School Announcements

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Nano 2002

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

Workshops
Gordon Research Conference on Granular and Granular-Fluid Flow
Plymouth, NH, USA June 30 - July 5 ,2002
http://sol.rutgers.edu/~shinbrot/gordon2002/gordon2002.html

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

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

L. A. C. A. M. E – 2. 0. 0. 2
EIGTH 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

Matériaux 2002

Tours - France
21- 25 Octobre 2002
Website : http://www.materiaux2002.net
E_mail : materiaux@materiaux2002.net

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SOUTENANCES DE THESE
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Sophie Soiron

Influence de la mécanochimie 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 Poitier),* M. Edmond Payen (*Ecole de chimie de Lill)*
Examinateurs: 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 Compiègnse),* 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 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 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 (γ Fe_2O_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

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

Mécanosynthèse - Intercalation - Composés graphite /lithium - Maghémite - RMN de ^{7}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 LiC_3 stoechiometry. Its structure was determined by ^{7}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 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 (γ Fe_2O_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}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 TiO2 anatase "

Jury :
J. Foct, 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 TiO2 de structure anatase. Au cours du broyage, TiO2 anatase se transforme en la phase rutile via une phase haute pression et/ou haute température du TiO2 nommée TiO2 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 TiO2 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 tellurure de plomb"

Montpellier, Université Montpellier II, 10 mai 2001

Jury :

J. Foct, J.C. Niepce, H. Scherrer, R. Griot, A.M. Bouchardy, J. Delallée, Y. Lacrouts-Cazenave, M. Ribes, J.C. Tédenac, 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 etrangers 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 sejour 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. Jack Harrowfield - Autralie
Job Opportunities
23/08/2001

Advanced Nano Technologies Pty Ltd

Nanotechnology.....

Advanced Nano Technologies Pty Ltd is a $15 million joint venture between Advanced Powder Technology Pty Ltd and Samsung Corning Co Ltd, established to commercialise a patented mechanochemical process used to manufacture NanoPowders.

ANT has recently been awarded a $2.8 million R&D Start Grant from theAustralian Federal Government and as a result, we are currently expanding our Perth-based Research and Development team. Exciting opportunities exist for highly qualified and motivated scientists and engineers to join our R&D team and assist in developing a leading, globally-focused nanotechnology company.

Positions are available in the following areas:

Research Opportunities

Research Scientist -Surfactants/Coatings: To undertake research and development of dispersants for ANT's nanopowders. You will have a PhD or equivalent experience in surface or colloid chemistry. A good understanding of dispersion science and technology is essential.

Research Scientist - Particle Coatings : To undertake research and development of particle surface coatings for ANT's nanopowders to improve chemical stability and facilitate incorporation into various polymers and solvents. You will have a PhD or equivalent experience in surface, colloid and/or polymer chemistry.

Materials Scientist/Engineer - Nanopowder Synthesis: To undertake research and development of new nanopowders manufactured by the mechanochemical process technology. Your will have a PhD or equivalent experience in Materials Science/Engineering, Solid State materials Chemistry or Solid State Physics.

Product Engineer - Process Optimisation : To undertake product development, with particular focus on the optimisation of the manufacturing process for specific nanopowders. You will work with research and production personnel to achieve targeted results. A PhD or equivalent experience in Materials Science/Engineering is required.

Interested applicants should send their CV, with a cover letter explaining how their background and experience will assist them in tackling the challenges of performing research in a newly emerging, rapidly changing industry.

Send To: Human Resources, Advanced Nano Technologies,
112 Radium St Welshpool, 6106, Western Australia.
For further information contact Brian: info@ant-powders.com
ph (08) 9380 3077, fax (08) 9380 1116
Closing date: Sep 14th

www.ant-powders.com

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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.
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 synthesisand 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 productionof 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.

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 activecollaboration 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
phone: +49-711-685-5261
FAX +49-711-685-5271

Dr. W. Sprengel
e-mail 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, staking 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
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.
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

(07/06/2001)

« Strategic and Technological Watch on Nanomaterials »
by **E. Gaffet** (1998 – 2000) – 4 CD reports (6.000 analysed references)
Editeur : Innovation 128 - 24 Rue du Quatre Septembre - 75002 Paris - France - Fax : 33 1 42 65 47 76
Website : http://www.innovation128.fr/

(28/05/2001)
Advanced Ceramic Materials
***** Key Engineering Materials, Volume 122 until 124 *****
In spite of the very great progress made in ceramic science, and the elegance and excitement of the research which has been performed, the real driving force for developments in ceramics remains their potential applications. The opportunity for dramatic scientific advances was certainly one reason for the "ceramic fever" of a decade ago, but there is also no doubt that the
prediction of an annual market for fine ceramics, amounting to 6 billion Yen played a role The challenge is to ensure that ceramics can be successfully introduced into the full breadth of applications where their properties have long made them so appealing. The present volume takes a refreshing and firm step towards the realization of this aim. The publication of a book which sets out to present ceramics from the specific point of view of applications is an event greatly to be welcomed. Systematic organization into various types of application ensures that the reader can fully appreciate the outstanding opportunities offered; and the present limitations. Armed with such a survey, the engineer and scientist will be fully alert to possibilities for progress whenever these arise 1. Introduction. 2. Electrical and Electronic Functions. 3. Magnetic Functions. 4. Chemical and Physical Functions. 5. Mechanical and Thermal Functions. 6. Biological Functions. 7. Nuclear Applications. 8. Ceramic Coatings. 9. Selected Ceramics with Multi-Applications.
http://www.scientific.net/kem

(11/2000) Information from Fritsch (A. Kohler)
The subject of the sixth forum part, Fritsch Forum Part VI scheduled for September 14/15th, 2000, will be "high-energy fine grinding". Research and Development demand general-purpose grinding processes which simultaneously exactly define the required energy and the type of stress. This is the only way that reliable results can be achieved when determining activation energies or the mechanical alloying. It must be possible to reproducibly adjust all of the grinding parameters affecting the grinding results.
Participants from research, development and industry will report on demands and novel technological solutions in developing innovative milling technologies. One of the highlights of the event will be FRITSCH's new Vario-planetary mill "pulverisette 4". This planetary ball mill can simulate ball mills of conventional construction, precisely copy the types of stresses that occur there, and thus reproduce or optimise grinding processes. Due to the great flexibility when selecting the grinding parameters, it is possible to produce results that are unattainable with other ball mills. It is the ideal mill for mechanical activation and alloying. The main applications are in the area of material research and naturally wherever a powerful, innovative laboratory planetary mill is needed.
An extensive report has been written about this event which details and makes readily available the relevant parts of the lectures and the extensive results of the discussions. Anyone interested can request a copy of the complete report for this forum part VI event on the topic "high-energy fine grinding". Please contact Andrea Köhler, FRITSCH GMBH, Industriestrasse 8, D-55743 Idar-Oberstein, (Phone: 0049/6784/7046, E-Mail: koehler@fritsch.de)

(7/07/2000) - From Victor Riecansky Publisher
Cambridge International Science Publishing http://www.demon.co.uk/cambsci/homepage.htm
MACROMOLECULAR MECHANOCHEMISTRY
Volume 1: Polymer Mechanochemistry - by Cleopatra Vasiliu OPREA & Florin DAN
Department of Macromolecules, Gh. Asachi, Technical University, 6600 Iasi, Romania
Macromolecular Mechanochemistry presents from theoretical and experimental point of view the main problems of this field, including the results obtained in more than a century of research. It is organised in two volumes: Polymer Mechanochemistry and Polymers with Chemomechanical Functions, respectively. The present volume deals with: Chained Polystage Character of Mechanochemical Process (1), Mechanochemistry of Polymers Deformation (2); Mechanochemistry of Polymer Fracture (including also the Fracture of Composite Materials) (3), and Mechanochemical Processes for Energy Conversion (4). In this frame, the theoretical and experimental material is organised in correlation to the reaction mechanism, the type of mechanical solicitation, and the nature of environmental medium. This book is addressed to professors, students, and researchers involved in the field of polymer science, to engineers from the industry of synthesis and processing of plastic materials, elastomers and fibres, as well as to specialists from all technical domains that exploit polymer-based materials. They will find in the book examination of the theoretical, experimental and applied problems and wide access to the basic literature in this field. Contents
1. Chained polystage mechanism of mechanochemical processes
2. Mechanochemistry of polymers deformation
3. Mechanochemistry of Polymer Fracture
4. Mechanochemical Processes for Energy Conversion

Volume 1 (ISBN 189832672X) will be published in September 2000, approx. 500 pages, cased,
approximate price £80.00; (volume 2 will be published at the end of - 2000)
Send your preliminary order to mailto:orders@cisp.demon.co.uk

(9/06/2000)
"Mechanical Alloying : FABRICATION OF ADVANCED MATERIALS AT ROOM TEMPERATURE" by M. Sherif El-Eskandarany

(ISBN: 977-299-089-7) Published by DAR AL-FIKR AL-ARABI, Cairo-Egypt.
The price of the book is $50, and a special discount (20%) is offered to all the RFM member.
Preface
Mechanical alloying (MA) process using ball-milling and/or rod-milling techniques, has received much attention as a powerful tool for fabrication of several advanced materials, including equilibrium, nonequilibrium (e.g., amorphous, quasicrystals, nanocrystalline, etc.), and composite materials., In addition, it has been employed for reducing some metallic oxides by milling the oxide powders with metallic reducing agents at room temperature. The MA is unique process in that a solid state reaction takes place between the fresh powder surfaces of the reactant materials at room temperature., Consequently, it can be used to produce alloys and compounds that are difficult or impossible to be obtained by the conventional melting and casting techniques.,
This book intended primarily to serve as an introduction to the MA process, including general description of the process, starting material requirements, the equipment, characterizations of the milled powders, and consolidation techniques, which used to compact the powder into fully-dense bulk materials.
The book contains several typical examples of selected advanced materials that have been fabricated by MA. This book is aimed at either senior undergraduate/post graduate students or materials scientists/metallurgists. - M. Sherif El-Eskandarany - April 2000 - Cairo - EgyptContents -

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(3/02/2000)
Two new books on mechanical alloying are now available from Cambridge International Science Publishing (infos fournies par Anne Porter - Publishing Manager - Cambridge International Science Publishing http://www.demon.co.uk/cambsci/homepage.htm)
1. MECHANICAL ALLOYING - FUNDAMENTALS AND APPLICATIONS http://www.demon.co.uk/cambsci/book52.htm
Contents
Introduction (history, benefits of mechanical alloying); Mechanical alloying (alloying mills, mills in practice, improved mills, the process, parameters);
Variations of mechanical alloying (reaction milling, cryomilling, repeated rolling, double mechanical alloying, repeated forging); Process control agents in mechanical alloying; Mechanical alloying mechanisms (ductile-ductile system, ductile-brittle system, brittle-brittle system, metastable phase formation, amorphisation, nanocrystallization, extension of solid solubility, activation of solid state chemical interaction);
Energy transfer and energy maps;
Consolidation of mechanically alloyed powders (consolidation techniques, thermomechanical treatment); Mechanical properties of mechanically alloyed materials (tensile properties, fracture, creep, stress corrosion cracking susceptibility);
Modelling mechanical alloying (mechanistic models, deformation, coalescence and fragmentation, evolution of particle size, milling time, powder heating, powder cooling, atomistic model, thermodynamic and kinetic model) Joining of mechanically alloyed materials; Rapid solidification and mechanical alloying; Applications (nickel-based superalloys, Al-based materials, supersaturated solutions, magnetic materials, mechanically alloyed powders for spray coatings, superplasticity, tribological materials, composites, amorphous solids, nanocrystalline materials, solid-state chemical reactions, etc). ISBN 1898326568, 160 pages 234 156 mm, cased, £45.00, 1999

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DISPERSION STRENGTHENED ALUMINIUM PREPARED BY MECHANICAL ALLOYING, by M Besterci - 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/cambsi/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 nanométres. A ces dimensions, les propriétés des matériaux changent radicalement.
Au début des années 90, les japonais ont été les premiers a lancé d'ambitieux programmes de R & D puisque le MITI a consacré aux nanomatériaux près de 200 millions de dollars pour la période 1990 - 2000 et que la Science & Technology Foundation a investi presque la même somme pour co - financer des projets de laboratoires publics et privés. Les Etats Unis puis les pays européens ont investi plus tardivement mais déjà ont obtenu des résultats prometteurs (••••••) Certaines

applications existent déjà au niveau international, quelque 400 sociétés se partagent aujourd'hui un marché voisin de 1 milliard de dollars mais qui devrait tripler, voire quintupler à l'horizon 2001.(••••••)
(•••) Pour aider les industriels concernés à imaginer les applications qu'ils pourraient s'approprier et identifier les acteurs internationaux, la présente étude dresse un état de l'art complet des nanomatériaux en décrivant leurs procédés d'élaboration actuels ou envisagés et en détaillant leurs différentes propriétés physico - chimiques et les géométries que l'on peut obtenir.
Enfin l'étude permet de cerner les applications actuelles et potentielles•••

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

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 Jouel heating sufficient to intitiate the reaction to form the reaction product while simultaneously applying pressure to the reacting body to densify it to a density approaching the theoritical density of the pure product. Surprinsingly, this process results in a reaction product that retains the nanocrystalline structure of the starting materials, despite the fact that a reaction has occured 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://l2.espacenet.com/dips/bnsviewer?CY=ep&LG=en&DB=EPD&PN=WO0112366&ID=WO+++0112366A1+I+

Périodiques

[37] SUPERFLUIDITY OF AQUEOUS SOLUTIONS OF MECHANICALLY ACTIVATED QUARTZ IN CAPILLARY POROUS BODIES
Red'kina NL. Khodakov GS. - Theoretical Foundations of Chemical Engineering (English Translation of Teoreticheskie Osnovy Khimicheskoi Tekhnologii). 35(4):341-346, 2001
Water permeation through porous beds of ground quartz is not explicable in terms of the classical filtration theory: as the filtration rate is reduced, the water permeability of these beds grows monotonically to a doubled value, while the theory predicts that it must be invariable in the range of filtration rates examined. This phenomenon is a direct consequence of the mechanical activation (solubilization) of quartz during grinding. The superfluidity phenomenon is also observed with specially prepared aqueous solutions of mechanically activated quartz and porous bodies formed from chemically inactive powders. It is demonstrated that the superfluidity of water in beds of mechanically activated powders is due to the formation of such solutions during filtration

[36] PHASE FORMATION AND PROPERTIES OF MECHANICALLY ALLOYED AMORPHOUS AL85Y8NI5CO2
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 Al85Y8Ni5Co2 prepared by mechanical alloying and consolidation

[35] EFFECT OF HIGH-TEMPERATURE FIBERIZATION ON THE CHEMICAL STRUCTURE OF SOFTWOOD
Widsten P. Laine JE. Qvintus-Leino P. Tuominen S. - Journal of Wood Chemistry & Technology. 21(3):227-245, 2001.
Softwoods fiberized at high temperatures (above 170 degreesC) were subjected to bulk and surface chemical analyses. It was found that the frequency of lignin beta -O-4 linkages declined while that of phenolic hydroxyl groups increased with an increase in fiberization temperature. The amount of water extractable aromatic compounds increased with increasing temperature of fiberization, which was associated with cleavage of lignin ether linkages. The water extractable material generated was enriched in hemicelluloses and contained aromatic compounds rich in phenolic hydroxyl groups and low in beta -O-4 linkages. The amount and hemicellulose content of the water extracts increased with increasing fiberization temperature. Lipophilic extractives covered most of the fiber surfaces while the surface lignin content of extractives-free fibers roughly doubled their bulk lignin content.

[34] 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.

[33] 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.

[32] LOCALIZED AMORPHIZATION IN SIC INDUCED BY BALL MILLING
Yang XY. Wu YK. Ye HQ. - Journal of Materials Science Letters. 20(16):1517-1518, 2001.

[31] FORMATION OF EUTECTIC RUAL/RU NANOCOMPOSITE BY MECHANICAL ALLOYING AND SUBSEQUENT ANNEALING
Liu KW. Mucklich F. Birringer R. - Journal of Materials Research. 16(9):2459-2462, 2001
No abrupt reaction was observed during mechanical alloying (MA) of Ru and At powder mixtures with an eutectic composition (Ru70Al30). As-milled powders constitute mainly a Ru(Al) solid solution and/or mixture (matrix), and a very small quantity of RuAl. The complete reaction between Ru and At during MA was speculated to be hampered by excess Ru in Ru60Al30. No exothermic heat release was detected in differential scanning calorimetry for as-milled powders. Precipitation of RuAl from as-milled Ru(Al) matrix was observed after annealing at various temperatures. The phase fraction of Ru and RuAl reaches art approximately equilibrium value after annealing at 1173 K

[30] THERMOCHEMISTRY OF RARE-EARTH ORTHOPHOSPHATES

Ushakov SV. Helean KB. Navrotsky A. Boatner LA. - Journal of Materials Research. 16(9):2623-2633, 2001
The enthalpies of formation for the compounds (RE (3+))PO4, (where RE = Sc, Y, La-Nd, Sm-Lu) were determined by oxide-melt solution calorimetry. Calorimetric measurements were performed in a Calvet-type twin microcalorimeter in sodium molybdate (3Na(2)O . 4MoO(3)) and lead borate (2PbO . 2B(2)O(3)) solvents at 975 K. The experiments were carried out using both powdered single crystals grown by a flux technique and powders synthesized by precipitation. Formation enthalpies were derived from the drop-solution enthalpies for (RE)PO4, RE oxides, and P2O5. Enthalpies of formation for the (RE)PO4 compounds with respect to the oxides at 298 K become more negative with increasing RE3+ ionic radius; i.e., in going from ScPO4 (-209.8 +/- 1.0 kJ/mol), to LuPO4 (-263.9 +/- 1.9 kJ/mol), to LaPO4 (-321.4 +/- 1.6 kJ/mol). From structural considerations, a similar trend is expected for the isostructural RE vanadates and arsenates, as well as for the tetravalent actinide orthosilicates

[29] DEVELOPMENT OF NANOCRYSTALLINE STRUCTURE DURING CRYOMILLING OF INCONEL 625
He JH. Lavernia EJ. - Journal of Materials Research. 16(9):2724-2732, 2001
Nanocrystalline Inconel 625 alloy, with a uniform distribution of grains, was synthesized using cryogenic mechanical milling. Microstructures of the powder, cryomilled for different times, were investigated using transmission electron microscopy (TEM), scanning electron microscopy, and x-ray diffraction. The results indicated that both the average powder particle size and average grain size approached constant values as cryomilling time increased to 8 h. The TEM observations indicated that grains in the cryomilled powder were deformed into elongated grains with a high density of deformation faults and then fractured via cyclic impact loading in random directions. The fractured fragments from the elongated coarse grains formed nanoscale grains. The occurrence of the elongated grains, from development to disappearance during intermediate stages of milling, suggested that repeated strain fatigue and fracture, caused by the cyclic impact loading in random directions, and cold welding were responsible for the formation of a nanocrystalline structure. A high density of mechanical nanotwins on {111} planes was observed in as-cryomilled Inconel 625 powders cryomilled, as well as in Inconel 625 powder milled at room temperature, Ni20Cr powder milled at room temperature, and cryomilled pure Al.

[28] MECHANICAL BEHAVIOR OF SUBMICRON-GRAINED GAMMA-TIAL-BASED ALLOYS AT ELEVATED TEMPERATURES
Bohn R. Klassen T. Bormann R. - Intermetallics. 9(7):559-569, 2001
Submicron-grained intertmetallic compounds based on gamma -TiAl were prepared by high-energy milling and hot isostatic pressing. At temperatures above 500 degreesC, the flow stress is strongly reduced and work-hardening completely disappears. Compression tests performed at temperatures between 500 and 900 degreesC reveal a marked strain rate sensitivity of the flow stress, suggesting superplasticity to occur. This could be confirmed by tensile straining of Ti-45Al-2.4Si samples, resulting in elongations of up to 175% at 800 degreesC. Small silicide particles (d approximate to 100 nm) of the type Ti-5(Si,Al)(3), embedded in the grain boundaries of the gamma -TiAl matrix, impede a coarsening of the microstructure. However, at strain rates above (epsilon)over dot = 10(-3) s(-1), these dispersoids are suggested to promote the formation of voids and to reduce the overall deformability. At 800 degreesC, an apparent activation energy of Q(app) = 351 kJ/mol can be derived. Superplastic behavior at 800 degreesC is accomplished by grain boundary sliding accommodated by diffusional processes inside the gamma -TiAl phase. Thus, the high temperature deformation mode is similar to the mechanisms found for more conventionally grained TiAl alloys at deformation temperatures greater than or equal to 1000 degreesC.

[27] SYNTHESIS OF NANOCRYSTALLINE NBAL3 BY MECHANICAL AND FIELD ACTIVATION
Gauthier V. Bernard F. Gaffet E. Munir ZA. Larpin JP. - Intermetallics. 9(7):571-580, 2001
The mechanically-activated, field-activated, and pressure-assisted synthesis (MAFAPAS) process, which combines the simultaneous synthesis and densification of nanophase materials, was utilized to produce nanocrystalline NbAl3 material from Nb+3Al mechanically activated powders. Nb+3Al elemental powders were co-milled for a short time in a specially designed planetary ball mill to obtain nanoscale distributed reactants but to avoid the formation of any product phases. These were then subjected to high AC currents (1500-1650 A) and uniaxial pressures (56-84 MPa). Under these conditions, a reaction is initiated by field activation and completed within a short period of tithe (3-6 min). Using XRD analyses, the MAFAPAS end-product was identified as NbAl3. Back-scattered electron SEM observations coupled with EDXS analyses showed the presence of small amounts of alumina precipitates together with unreacted niobium in the NbAl3 matrix. The end-product relative density ranged from 85 to 96%. The NbAl3 crystallite size, determined by XRD line-broadening analysis using the Langford method, was in the range of 57-150 um

[26] NANOSTRUCTURED MULTI-COMPONENT MATERIALS BY MECHANICAL ALLOYING
Politis C. Spiliotis AD. Kapaklis V. Baskoutas S. - Chinese Physics. 10(Suppl S):S27-S30, 2001
We have shown that nanostructured multi-component materials like TaC, Nb78Ge22, Mo70Si20B10, Mo60Os20B20, Ru52Zr6B42, (NbC)(20)Co-80 and (WC)(80)-(Ti90Cu10)(20) with an effective length size reduced to atomic level (a few nanometers) can be produced by mechanical alloying of the elemental and/or alloy powders in a high-energy ball mill. All process steps and samples preparation for characterization were done under glove box conditions

[25] NANOSTRUCTURED AND AMORPHOUS MATERIALS BY MECHANICAL ALLOYING
Politis C. - Chinese Physics. 10(Suppl S):S31-S35, 2001
Mechanical Alloying is particularly attractive because it can be used to process structurally uniform and isotropic bulk quantities of powdered materials that have a fine length scale. Furthermore, it appears to be applicable to many combinations of elements. Here experimental results regarding the synthesis and characterization of several binary and ternary

nanocrystalline and amorphous alloys of Ti, Zr, Hf, V, Nb and Fe with other transition elements and with B, C and Si are reported. The nanocrystalline and amorphous powders, with effective particle size between 7 and 30nm, were prepared by mechanical alloying in a high-energy ball-mill using commercial elemental powders and/or alloy powder.

[24] BALL MILLING: A NEW ROUTE FOR THE SYNTHESIS OF SUPERDENSE LITHIUM GICS

Janot R. Conard J. Guerard D. - Carbon. 39(12):1931-1934, 2001.

[23] THE EFFECT OF MECHANICAL ACTIVATION ON THE PREPARATION OF SrTiO3 AND Sr2TiO4 CERAMICS FROM THE SOLID STATE SYSTEM SrCO3 ^ TiO2 "

V. Berbenni, A. Marini and G. Bruni- Journal of Alloys and Compounds -vol. 329(1-2) p. 230-238 (2001).

By thermoanalysis (TGA and DSC) and X-ray powder diffraction it has been shown that the compounds SrTiO3 and Sr2TiO4 can be prepared by mechanical activation of respectively 1:1 and 2:1 SrCO3 - TiO2 (rutile) mixtures followed by annealing of 12 hours at 800 - 850°C. These compounds could not be obtained by heating the physical mixtures up to temperatures as high as 1000°C . Moreover the enthalpies of the reactions leading from Sr carbonate and rutile to the formation of these compounds have been determined. By combining these data with the enthalpy of SrCO3 decomposition, also obtained in this work, the

enthalpies of formation of both SrTiO3 and Sr2TiO4 have been calculated. On the contrary no Sr3Ti2O7 has been shown to form, by the same annealing procedure, also when starting from a mechanically activated mixture. DSC and XRD results agree in indicating that a mixture of Sr2TiO4 and SrTiO3 forms instead.

[22] IN-LINE PARTICLE SIZE MEASUREMENT FOR CONTROL OF JET MILLING

Ma ZH. van der Veen H. Merkus HG. Scarlett B. - Particle & Particle Systems Characterization. 18(2):99-106, 2001

Jet milling is suitable for the production of very fine powders with a well-defined particle size distribution (PSD). The major disadvantage of the process is the poor energy efficiency. In practice jet mills have to be operated below their maximum capacity in order to avoid the production of off-specification material, due to operation disturbances. Therefore, it is important to automate operation with a realtime PSD instrument for quality control. In this paper, a study for in-line particle size measurement using laser diffraction is presented to improve operation control. This measurement system uses a novel CMOS pixel array, which has a robust and flexible feature for the application. It also includes an in-line cell system, where direct measurement is implemented in the product particle stream coming from the mill and an air purge for the cell window allows continued operation. Experiments were conducted on a 10-inch spiral jet mill pilot plant with alpha-AL(2)O(3) as test material. The milling process is investigated by varying the operational conditions. The measurement results are useful to build a grinding model for control purpose

[21] ISAMILL MEDIUM COMPETENCY AND ITS EFFECT ON MILLING PERFORMANCE

Gao M. Young MF. Cronin B. Harbort G. - Minerals & Metallurgical Processing. 18(2):117-120, 2001

Traditional fine-grinding mills in mineral processing rely on the use of steel medium for particle size reduction. The IsaMill (a horizontal stirred mill with a 3,000-L net chamber volume and a rated motor power of 1,120 kW) has successfully adopted a novel product-separator design, which has led to a major revision of medium use in fine grinding. The IsaMill separator has successfully operated with a wide variety of medium types, such as silica sands, river gravel, smelter slag, run-of-mine ore fractions and steel shots, with sizes ranging from 0.5 to 10 mm. Great flexibility in the IsaMill operations has been achieved for processing feed as coarse as 120 pn to a product as fine as 5 mum. This paper discusses the performance of two inert grinding media in the IsaMills that are used at Mount Isa Mines Ltd. for the processing of galena- and sphalerite-bearing ores to below 10,um. They are the copper reverberatory furnace slag (CRFS) and the heavy-medium plant rejects (HMPR). A methodology was also demonstrated for comparing the medium performance and for scaling up the IsaMills from a 1.5-L laboratory batch mill to a full-scale 3,000-L mill.

[20] PAN-MILLING MIXING - A NOVEL APPROACH TO FORMING POLYMER BLENDS AND CONTROLLING THEIR MORPHOLOGY

Chen Z. Wang Q. - Polymer International. 50(9):966-972, 2001

A novel technique (pan-milling mixing) was developed to control the morphology and thus enhance the mechanical properties of polypropylenelpolyamide 6 (PP/PA6) systems. Through pan-milling at ambient temperature, PP/PA6 pellets of particle size 2-4mm can be effectively pulverized to well-mixed micrometre fine powders in the solid state. During pan-milling of mixtures of PP and PA6, the polymer molecules undergo chain scission and form copolymers that compatibilize the two polymers in situ. By press moulding the finely mixed PP/PA6 powder obtained at a temperature between the melting points of PA6 and PP (for example 200 degreesC), a blend can be obtained in which the PA6 powder, retained throughout the process in the solid state, is well dispersed in the PP matrix. The mechanical properties of the system are much better than that of PP/PA6 blends prepared by common twin screw extrusion mixing and injection moulding. Tensile strengths of the fine PA6 particle filled PP/PA6 (70/30) blend is 29.3 MPa, which is 6.1 MPa higher than that of a conventionally prepared PP/PA6 blend. The Izod notched impact strength of a fine PA6 particle-filled PP/PA6 (70/30) blend is 6.34kJ m(-2), which is 1.72 kJ m(-2) higher than that of a conventionally prepared PP/PA6 blend. Morphological analysis shows that the domain size of PA6 in the system is much smaller than that of the PP/PA6 blend, and can be controlled by the processing conditions such as temperature

[19] NONEQUILIBRIUM MAGNETIC DYNAMICS IN MECHANICALLY ALLOYED MATERIALS - ART. NO. 094438

De Toro JA. de la Torre MAL. Arranz MA. Riveiro JM. Martinez JL. Palade P. Filoti G. - Physical Review B. 6409(9):4438-+, 2001

The magnetically glassy behavior of mechanically alloyed Fe61RC30Cr9 is reported in detail, including a static and dynamic study of the freezing process, the observation of aging in a mechanically alloyed sample, Mossbauer analysis, and annealing experiments. Despite the clear collective character of the low-temperature change of regime, no thermodynamical spin-glass mean-field transition could be proved. On the other hand, the careful comparison of the magnetic behavior with that reported in strongly interacting fine particles systems hinted towards the presence of that kind of particles in our samples. Structural considerations based on XRD, Mossbauer, and the evolution of the ae susceptibility peaks upon annealing pointed to the existence of very fine Fc-rich clusters able to support a magnetic moment. confirming the diagnosis extracted from the magnetic dynamics analysis. The argument is strengthened by the study of the effects of milling on the freezing temperature in a second sample showing a similar behavior: Fe35Al50B15. The explanation can be extended naturally to previously reported mechanically alloyed, spin-glass-like samples, which hints towards the generalization of our interpretation

[18] DYNAMIC DEFORMATION BEHAVIOR OF AN OXIDE-DISPERSED TUNGSTEN HEAVY ALLOY FABRICATED BY MECHANICAL ALLOYING

Park S. Kim DK. Lee S. Ryu HJ. Hong SH. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 32(8):2011-2020, 2001

The objective of this study is to investigate the dynamic deformation and fracture behavior of an oxide-dispersed (OD) tungsten heavy alloy fabricated by mechanical alloying (MA). The tungsten alloy was processed by adding 0.1 Wt pct Y2O3 powders during MA, in order to form fine oxides at triple junctions of tungsten particles or at tungsten/matrix interfaces. Dynamic torsion tests were conducted for this alloy, and the test data were compared with those of a conventional liquid-phase sintered I(LPS) specimen. A refinement in tungsten particle size could be obtained after MA and multistep heat treatment without an increase in the interfacial area fraction between tungsten particles. The dynamic test results indicated that interfacial debonding between tungsten particles occurred over broad deformed areas in this alloy, suggesting the possibility of adiabatic shear-band formation. Also, oxide dispersion was effective in promoting interfacial debonding, since the fine oxides acted as initiation site for interfacial debonding. These findings suggest that the idea of forming fine oxides would be useful for improving self-sharpening and penetration performance in tungsten heavy alloys.

[17] REDUCTION OF POROSITY IN OXIDE DISPERSION-STRENGTHENED ALLOYS PRODUCED BY POWDER METALLURGY

Chen YL. Jones AR. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 32(8):2077-2085, 2001

In this work, the porosity in a iron-based oxide dispersion-strengthened (ODS) alloy, namely, PM2000, has been studied together with its recrystallization behavior. It has been found that pores are only found in coarse secondary recrystallized grains formed at the early stage of recrystallization. It is suggested that a lack of fast diffusion paths, particularly grain boundaries, will prevent gas trapped in the materials during mechanical alloying (MA) from diffusing away, and porosity is formed as a result. Thus, it is proposed that an extended anneal below the secondary recrystallization temperature will help to reduce the subsequent evolution of porosity in these materials, and the method has been demonstrated to work successfully in PM2000

[16] ALTERATION OF BAND STRUCTURE IN SI NANOCRYSTALS

Pawlak BJ. Gregorkiewicz T. Ammerlaan CAJ. - Materials Science & Engineering C: Biomimetic Materials, Sensors & Systems. 15(1-2 Special Issue SI):273-275, 2001

The photoluminescence of Si nanocrystals with dimensions ranging from 70 to 250 nm was investigated. Grains were prepared by mechanical ball-milling and subsequent sedimentation steps in order to segregate them in size. This was followed by heat treatment in argon atmosphere at 1000 degreesC to reduce the density of dislocations introduced during milling. At the same temperature, an oxidation process in air was carried out in order to create a stable SiO2/Si interface and reduce the dimensions of the grains. Crystallinity of the samples was checked by X-ray diffraction and transmission electron microscopy-related techniques. Two emission bands have been determined. One was identified with the DI center usually assigned to dislocations. The second one is identified as being due to an excitonic recombination and shows phonon replicas characteristic for bulk silicon. This band is reported here for the first time. In the fraction of the smallest silicon grains, a gradual upshift of the excitonic line was observed with diminishing average grain diameter. This was associated with band structure perturbation due to size -confinement

[15] LEAD ZIRCONATE TITANATE CERAMICS DERIVED FROM OXIDE MIXTURE TREATED BY A HIGH-ENERGY BALL MILLING PROCESS

Kong LB. Ma J. Huang HT. Zhu W. Tan OK. - Materials Letters. 50(2-3):129-133, 2001

Lead zirconate titanate (Pb(Zr0.52Ti0.48)O-3 or PZT) ceramics were directly prepared from their oxide mixture, which was treated by a high-energy ball milling process. X-ray diffraction (XRD) result indicated that there was no reaction between the component oxides during the milling process. The particle size of the mixture was greatly reduced to nanometer scale by the milling process. Sintering behavior of the mixture demonstrated a distinguished volumetric expansion before densification, clearly showing that a reaction sintering occurred at about 770 degreesC. PZT ceramics sintered at 1000 degreesC for 4 h exhibited a dielectric constant of 1156 and dielectric loss of 0.03, a remnant polarization of 29 muC/cm(2) and a coercive field of 18.4 kV/cm. Piezoelectric parameters of the 1000 degreesC-sintered PZT ceramics were k(33) of 40.4%, k(31) of 40.9%, d(33) of 175 pC/N and d(31) of -148 pC/N, respectively. These results have shown that preparation of PZT ceramics in this way is practical.

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

Zhang DL. Ying DY. - Materials Letters. 50(2-3):149-153, 2001
Phase transformation during high-energy milling Al-25 at.%Ti and Ti-25 at.%Al powders and during heating of ball-milled powders of the same compositions was studied. It was found that a small amount of Ti underwent an allotropic transformation from hcp Ti to fcc Ti during milling. This transformation also occurred during heating the properly milled Al-25 at.%Ti and Ti-25 at.%Al powders. The transformation was endothermic, and the onset temperature of this transformation was 321 degreesC. It is likely that only thin Ti layers which have a nanometer-scale thickness and are embedded in Al matrix can undergo this transformation

[13] INFLUENCE OF POWDER PRE-TREATMENTS ON DISPERSION ABILITY OF AQUEOUS SILICON NITRIDE-BASED SUSPENSIONS

Oliveira MILL. Chen KX. Ferreira JMF. - Journal of the European Ceramic Society. 21(13):2413-2421, 2001
The rheological properties and the dispersion stability of highly concentrated aqueous silicon nitride powder suspensions in the presence of an anionic polyelectrolyte dispersant have been investigated. Silicon nitride-based suspensions with solids loading as high as 60 vol. % could be prepared following a slow process of increasing the solids volume fraction in steps of 5 vol. %, starting from 45 vol.%. After each increment of solids, ball milling was conducted for periods of 24 h, which then increased up to 48 h with solids loading increasing. Slips were prepared either from the as received or the pre-treated powders. Zeta potential measurements were conducted on Si3N4, Al2O3 and Y2O3 powders submitted to different pre-treatments, in absence and in the presence of dispersant, to evaluate the influence of these experimental variables on surface-charge properties of the particles. It was concluded that both aqueous ball milling and calcining enhance the repulsive interaction forces between. particles, like the presence of the dispersant. On the other hand, rheological and particle size analysis revealed that ball milling and the ageing time enhance the dispersing ability of the powders and decrease the viscosity (for a given solids loading) and the shear-thinning character of the suspensions.

[12] GIANT MAGNETORESISTIVE PROPERTIES OF FEXAU100-X ALLOYS PRODUCED BY MECHANICAL ALLOYING

Socolovskya LM. Sanchez FH. Shingu PH. - Journal of Magnetism & Magnetic Materials. 226(Part 1 Special Issue SI):736-737, 2001
The FexAu100-x allovs were produced for the first time by mechanical alloying. Resistance of samples with iron concentrations of x = 15. 20. 25. and 30 at % were measured at 77 K under an applied field of 14 kOe. A maximum in magneto resistive ratio (Deltap,p) of 3.5% was obtained for Fe(25)AU(75). Samples were annealed in order to enhance magnetoresistive properties. These samples exhibit larger ratios. primarily due to the elimination of defects. X-ray diffraction Mossbauer spectroscopy and magnetoresistance measurements were performed, in order to correlate bulk and hyperfine magnetic properties with crystalline structure. X-ray diffractograms show an FCC structure, with no evidence for a BCC one.

[11] RECENT DEVELOPMENT OF MATERIALS DESIGN THROUGH A MECHANOCHEMICAL ROUTE

Senna M.- International Journal of Inorganic Materials. 3(6):509-514, 2001
A critical overview is given of the principle and practice of modem mechanochemical processes for the preparation and design of high value-added materials. Some fundamental principles of bridging bond formation under mechanical stressing are given. The importance of the low coordination states and effects of symmetry disturbance of crystal and ligand fields are emphasized. Several successful application examples are displayed, stemming from inorganic complex oxides for electronic and construction materials, bioceramic materials to metal organic coordination compounds

[10] REAL STRUCTURE AND CATALYTIC ACTIVITY OF LA1-XSRXCOO3 PEROVSKITES

Isupova LA. Alikina GA. Tsybulya SV. Boldyreva NN. Kryukova GN. Yakovleva IS. Isupov VP. Sadykov VA. - International Journal of Inorganic Materials. 3(6):559-562, 2001
Mechanoceramical synthesis of La1-xSrxCoO3 (0 less than or equal tox less than or equal to1) perovskites was made from simple oxides. Samples calcined at 900 and 1100 degreesC for 4 h are nearly monophase and well crystallized. Sr adding was found to cause a structure rearrangement from the hexagonal (at x less than or equal to0.4) to the cubic one (at 0.8>x>0.4) and back to the hexagonal at x>0.8. There are two maxima of the catalytic activity versus chemical composition: at x=0.3 and at x=0.8. TEM data for these samples were obtained and disordered surface layers were detected. There is a correlation between the catalytic activity and surface layers microstructure.

[9] ELECTRORHEOLOGICAL PROPERTIES OF MECHANICALLY ACTIVATED GIBBSITE

Kitamura M. Senna M. - International Journal of Inorganic Materials. 3(6):563-567, 2001
Change in the surface state during milling of gibbsite (GB) is elucidated by electrorheological properties of the suspension comprising GB and silicone oil. Under de electrical field up to 2 kV/mm, milling brings about remarkable increase in the yield stress, e.g., milling GB for 3 h increases 100 times the yield stress at 2 kV/mm. Amount of thermal dehydration below 200 degreesC, A(OH<200), increases by milling as a consequence of mechanochemical dehydration and readsorption. The yield stress increases quasi proportionally with A(OH<200). Thus, ER yield stress of GB serves as a quantitative measure of mechanochemical dehydration of GB

[8] TRIBOCHEMICAL MODIFICATION OF THE MICROSTRUCTURE OF V2O5

Su DS. Roddatis V. Willinger M. Weinberg G. Kitzelmann E. Schlogl R. Knozinger H. - Catalysis Letters. 74(3-4):169-175, 2001.
The tribochemical activation of vanadium pentoxide V2O5 is studied by means of X-ray diffraction, electron microscopy and electron energy-loss spectroscopy. The two-stage process can be described as crushing of large crystals into small ones (macroscopic process) followed by amorphisation and reagglomeration of the fragments (microscopic process). No milling

equilibrium state can be found. Energy-loss spectra reveal the reduction of vanadium via oxygen loss. The formation and distribution of V4+ or V3+ species depends on the history of milling

[7] SYNTHESIS AND CHARACTERIZATION OF NANOCRYSTALLINE NITI SHAPE-MEMORY ALLOY BY SHOCK-COMPRESSION

X Xu, NN Thadhani - FUNDAMENTAL ISSUES AND APPLICATIONS OF SHOCK - WAVE AND HIGH - STRAIN - RATE PHENOMENA, PROCEEDINGS, 2001, pp 297-304 - INTERNATIONAL CONFERENCE ON FUNDAMENTAL ISSUES AND APPLICATIONS OF SHOCK-WAVE AND HIGH-STRAIN-RATE PHENOMENA (EXPLOMET 2000); ALBUQUERQUE, NEW MEXICO. , 2000

Shock synthesis of shape-memory NiTi alloy was investigated in this study, starting with an elemental mixture of Ni+Ti (mixed in equiatomic ratio) as well as pre-alloyed nitinol (Ni50.88T49.12) powder. The powders were ball milled using a SPEX 8000 mixer/mill in an Ar atmosphere, to form an intimate mixture of reactants in the case of the Ni+Ti precursor powder, and an amorphous alloy in the case of the pre-alloyed nitinol powder. The ball-milled powders were subsequently shock-consolidated with a three-capsule, plate-impact fixture, using an 80-nm diameter single-stage gas-gun. In the case of the Ni+Ti powder mixture, the recovered compact showed formation of multiple phases including the NiTi B2- and R-phase, NiTi2, and a trace of Ni3Ti. The shock-compressed nitinol powder compacts showed partial crystallization in the as-consolidated state, and full crystallization to a nanocrystalline structure upon subsequent annealing. In this paper, the phase-formation mechanisms and shape-memory properties of the shock-synthesized NiTi alloy as a function of grain size, will be described.

[6] ELECTRONIC AND OPTICAL PROPERTIES OF ISOSTRUCTURAL BETA-FESI2 AND OSSI2

Migas DB. Miglio L. Henrion W. Rebien M. Marabelli F. Cook BA. Shaposhnikov VL. Borisenko VE. - Physical Review B. 6407(7):5208-+, 2001

We present both theoretical and experimental investigations of electronic and optical properties of isostructural beta -FeSi2 and OsSi2 by means of a full-potential linear augmented plane wave method and optical measurements. We report also ellipsometric and reflectance measurements on samples of polycrystalline osmium disilicide prepared by mechanical alloying. From ab initio calculations these compounds are found to be indirect band-gap semiconductors with the fundamental gap of OsSi2 larger some 0.3-0.4 eV than the one of beta -FeSi2. In addition to that, a low value of the oscillator strength is predicted for the first direct transitions in both cases. Computed optical functions for these materials were compared to the ones deduced from optical measurements, indicating very good agreement and the presence of some anisotropic effects.

[5] THE EFFECT OF HIGH ENERGY BALL MILLING ON THE CRYSTAL STRUCTURE OF GDNI(5)

Stubicar M. Blazina Z. Tonejc A. Stubicar N. Krumes D. - Physica B. 304(1-4):304-308, 2001

X-ray powder diffraction was used to determine the effect of, dry, in air performed high energy ball milling, on the intermetallic compound GdNi5. It was found that the crystal structure of GdNi5 is not stable. At the early stage of milling (up to after 10 h of milling) the gadolinium component oxidises causing thus the decomposition of GdNi5 into monoclinic Gd2O3 and metallic nickel. Both. the crystallite (grain) size and the particle size of powder decrease during the early stage of milling. At the later stage of milling (up to 50 h) the nickel phase from the mixture of Gd2O3 and nanocrystalline nickel oxidises into nanocrystalline NiO. Therefore, the final product after 150 h of milling of GdNi5 is a mixture of oxides of the constituent metals. i.e., amorphous Gd2O3 and nanocrystalline NiO. Traces of contamination by alpha -SiO2 have been observed in the milled powder, being more pronounced as the milling process proceeds. This is ascribed to the wear effect of agate milling assembly

[4] MECHANOCHEMICAL REACTIONS IN CU/ZNO CATALYSTS INDUCED BY MECHANICAL MILLING

Castricum HL. Bakker H. van der Linden B. Poels EK. - Journal of Physical Chemistry B. 105(33):7928-7937

Preparation of Cu/ZnO catalysts is discussed in which mechanochemical reactions are involved. These reactions include oxidation of copper and copper oxides during milling in the presence of oxygen, reduction under vacuum, and formation of carbonates in CO2. Oxidation and reduction are promoted by the presence of ZnO. Formation Of Cu2O-like intermediates is suggested in oxidic mixtures, which are not observed when milling occurs without ZnO. This effect may be partly due to defect formation. The various resulting copper species reduce at different temperatures in H-2 atmosphere and this gives an indication for the intimacy of the Cu-Zn interaction. When milling is carried out in the presence Of CO2, mixed carbonates are formed. Intimate mixtures of Cu and Zn are thus obtained, exhibiting high surface areas after reduction that can be explained by evolved porosity. A linear relation between Cu-0 surface area and activity for methanol synthesis at 2 bar is observed. Irreversible deactivation is explained by over-reduction and sintering. Both Cu-0 specific surfaces and BET surfaces (determined after reduction in hydrogen) are substantially increased for all milled Cu/ZnO samples, but especially for samples that undergo mechanochemical reactions. This method thus allows investigation of mixed oxide catalyst precursors and provides an interesting alternative for the preparation of promoted heterogeneous catalysts

[3] STRUCTURE AND MAGNETIC PROPERTIES OF POWDERS PREPARED BY MECHANICALLY ALLOYING 50FE+25AL+25SI MIXTURES

Fadeeva VI. Sviridov IA. Nikitin SA. Ovtsenkova YA. - Inorganic Materials. 37(8):790-796, 2001

A 50Fe + 25Al + 25Si powder mixture was mechanically alloyed in a high-energy ball mill, and the products of the solid-state reactions were characterized by x-ray diffraction and magnetic measurements. The results show that the process involves the formation of a metastable bcc Fe < Al,Si > solid solution, which decomposes into the ordered phases FeAl1-xSix (B2 structure) and FeSi (B20) upon heating to 700 degreesC or long-term milling. The observed effect of milling on the magnetic properties of the powders indicates that the proportion of the ferromagnetic component in the alloy decreases with increasing milling time as a result of the ordering of the solid solution and the formation of the B2 and B20 paramagnetic phases.

[2] HIGH TEMPERATURE MECHANICAL BEHAVIOR OF POLYCRYSTALLINE ALUMINA FROM MIXED NANOMETER AND MICROMETER POWDERS

Goldsby JC. - Ceramics International. 27(6):701-703, 2001.

Sintered aluminum oxide materials were formed using commercial methods from mechanically mixed powders of nano- and micrometer alumina. The powders were consolidated at 1500 and 1600 degreesC with 3.2 and 7.2 ksi applied stress in argon. The conventional micrometer sized powders failed to consolidate. While 100% nanometer-sized alumina and its mixture with the micrometer powders achieved > 99% density. Preliminary high temperature creep behavior indicates no super-plastic strains. However high strains (> 0.651%)were generated in the nanometer powder, due to cracks and linked voids initiated by cavitation

[1] ON THE REACTION SEQUENCE OF WC-CO FORMATION USING AN INTEGRATED MECHANICAL AND THERMAL ACTIVATION PROCESS

Ban ZG. Shaw LL. - Acta Materialia. 49(15):2933-2939, 2001

A systematic study on the reaction sequence of WC-Co composite formation by annealing high energy ball milled WO3, CoO and graphite powder mixtures in a hydrogen atmosphere has been conducted. X-ray diffraction has been used as the main tool to analyze the phase transformation of the powder mixture during processing. It was observed that WO3 is reduced to W phase by passing through the intermediate W20O58 and WO2 phases and the subsequent carburization sequence appears as W --> Co6W6C --> Co3W3C -->W2C --> WC. The intermediate Co3W has been found in the reduction stage, which can be subsequently carburized at higher temperatures

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