# RESEAU FRANÇAIS DE MECANOSYNTHESE
# Lettre N°63

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## Juin 2000

**178 (+2) Groupes de Recherche**

**(dont 105 (+2) à l'étranger / 36 Pays)**



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**SOMMAIRE**



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**3 Nouvelles Adhésions**
**V. Iplikci** - Roucaire - France
**J. Kano** - Tohoku University - Japon
**N. Minkova** - Faculty of Chemistry - Bulgarie

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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'99 et quelques éléments mis a jour régulièrement concernant les derniers résultats dans ce domaine.

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## ANNONCE DE CONGRES ET / OU ECOLES
## CONGRESS AND SCHOOL ANNOUNCEMENTS

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### PM2 TEC2000

**2000 International Conference on Powder Metallurgy & Particulate Materials**
31 Mai - 3 Juin 2000 - New York - USA
Contact : MPIF

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### 4th EUROMECH

26 - 30 Juin 2000 - Metz - France
E_Mail : euromech@lpmm.univ-metz.fr
WebSite : http://www.lpmm.univ-metz.fr/euromech

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### ISMANAM 2000

International Symposium on Metastable Mechanically Alloyed and Nanocrystalline Materials
9 - 14 Juillet 2000 - St Catherines College - Oxford UK
**E_Mail :** ismanam2000@materials.ox.ac.uk
**website :** http://www.materials.ox.ac.uk/OCAMAC/ISMANAM/ismanam2000.html

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### NCM8

**8th International Conference on the Structure of Non - Crystalline Solid**
6 - 11 Aout 2000
Website : http://www.sgt.org

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### XIVth International Symposium on the Reactivity of Solids

Budapest, Hungary through 27-31 August 2000
http://www.jate.u-szeged.hu/~isrs14.

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### Solid State Chemistry 2000

**Prague, Czech Republic, September 3 - 8,2000**

**and**

### 3rd INCOME

**International Conference on Mechanochemistry and Mechanical Alloying**
Prague, Czech Republic, September 4 - 8,2000
Organised by the Institute of Inorganic Chemistry (UACH), Czech Republic
**WebSite :** http:://www.iic.cas.cz/INCOME.htm

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### Inorganic Materials Conference

University of California Santa Barbara, USA
13 - 16 September 2000 http://www.elsevier.com/locate/im2000

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### Congrès de la Société Française de Chimie

Univ. Rennes I
18 - 22 Septembre 2000
**website** : http://www.sfc.fr

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### MATERIALS WEEK
### International Congress on Advanced Materials, their Processes and Applications

25 - 28 September 2000
ICM – International Congress Centre Munich in Conjunction with the Exhibition Materialica
website : www.materialsweek.org

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### J2IM 2000

**“Joints Intergranulaires et Interphases dans les Matériaux“**
Fontainebleau - EDF (Site des Renardières)
4 - 6 Octobre 2000
http://perso.club-internet.fr/adjr/j2im.html

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### Journées d’Automne 2000 / SF2M

Maison de la Chimie à Paris
du 17 au 19 octobre 2000
e_mail : sfm@wanadoo.fr
Website : www.sf2m.asso.fr

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## Nanomatériaux :
## Vers les Applications Industrielles
## Nanomaterials :
## Towards Engineering Applications

**Colloque** : France - Etats Unis - Canada
22 - 25 Octobre 2000 - Montréal, Canada
Contacts : Champion@glvt-cnrs.fr et/ou Eric.Gaffet@utbm.fr

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## PM 2000

**Powder Metallurgy World**
**Congress& Exhibition**
12 - 16 Novembre 2000 Kyoto - Japon
Contact : Fax : 81 - 3 - 3423 - 1600

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## The 1st International Conference on Advanced Materials Processing
## Rotorua, New Zealand, 19-23 November 2000.

Secretary, ICAMP 2000,
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
Or visit the conference web site:
http://mape.waikato.ac.nz/conferences/amp.htm

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## "Scattering Studies of Mesoscopic Scale Structure and Dynamics in Soft Matter"

Messina Italy
22th to 25th of November 2000
(for details see LRFM60)

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## 2000 MRS Fall Meeting

27 Nov - 1 Dec. 2000
Boston - Massachusetts - USA
(for details see LRFM60)

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## Science et Technologie des Poudres

Nancy - France
3 - 5 Avril 2001
website : http://www.inpl-nancy.fr/stpoudres3.html
e_Mail : stpoudres@inpl-nancy.fr

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## PM2 TEC2001

**2001 International Conference on Powder Metallurgy**
**& Particulate Materials**
13 - 17 May 2001 - New Orleans - USA
Contact : MPIF

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## 7th International Symposium on Agglomeration

29, 30, 31 May 2001
Albi - France
Website : http://www.univ-inpt.fr/~agglom
or http://www.enstimac.fr

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# SOUTENANCE DE THESE

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**Nouveau**

**C. Goujon**

**"Elaboration par cryobroyage et métallurgie des poudres de nanocomposites à matrice d'alliage d'aluminium renforcée par des particules de nitrure d'aluminium"**

**25 Mai 2000**

**Jury :**

P. Goeuriot (Dir. Thèse), G. Le Caer (Rapp.), D. Michel (Rapp.), & F. Bernard, Y. Laurent, M. Suery, F. Thévenot, S. Vicens,

*Résumé non disponible à la date de publication de LRFM63*

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**J. Joardar**

**"Synthesis of nanocrystalline aluminides in Al - Ni - Fe system by Mechanical Alloying"**

Avril 2000

Thesis Supervisor : B.S. Murty et S.K. Pabi (IIT, Kharagpur)

Thesis Examiners : C.C Koch (North Carolina State University) , P. Ramakrishnan (IIT, Bombay)

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**Christine Barbeau**

(Laboratoire de Métallurgie Physique - Futuroscope)

**Sructure dans les matériaux élaborés sous HIP : cas des alliages à base tungtène par frittage et du carbure de titane par combustion auto-propagée**

13 mars 2000

**Thèse de Doctorat de l'Université de Poitiers**

**JURY:** A. TRAVERSE, Directeur de Recherche, LURE Orsay, Rapporteur - F. NARDOU, Professeur, Université de Limoges, Rapporteur, D. VREL, Chargé de Recherche, Université de Villetaneuse - Examinateur, M.F. BEAUFORT - Chargé de Recherche CNRS, LMP Poitiers - Examinateur, M. GROSBRAS - Chargé de Recherche CNRS, LMP Poitiers, Examinateur - J. MIMAULT, Professeur, Université de Poitiers, Examinateur et Directeur de Thèse

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**Hugues GUÉRAULT**

**PROPRIÉTÉS STRUCTURALES ET MAGNÉTIQUES DE POUDRES DE FLUORURES NANOSTRUCTURÉES MF3 (M=Fe, Ga) OBTENUES PAR BROYAGE MÉCANIQUE**

*28 Janvier 2000*

**THèSE DE DOCTORAT** - Université du Maine - **Physique des Matériaux et des Surfaces**

**Jury :** Gérard Le CAER, Directeur de Recherche, Ecole des Mines - Nancy (Rapporteur), Marc NOGUES, Chargé de Recherche, Université de Versailles (Rapporteur), Jean-Franÿois BÉRAR, Ingénieur de Recherche, CNRS - Grenoble, Frédéric BERNARD, Maître de Conférence, Université de Bourgogne - Dijon, Jean-Yves BUZARÉ, Professeur, Université du Maine - Le Mans, Marc LEBLANC, Professeur, Université du Maine Le Mans, Jean-Marc GRENèCHE, Directeur de Recherche, Université du Maine (Directeur de thèse)

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**Cyril Lenain**

**APPLICATION DE LA MECANOCHIMIE A LA PREPARATION D'ALLIAGES HYDRURABLES NANOCRISTALLINS AB5, MG-NI, AB2 (M) ET DE COMPOSITES M-C, M-Cu : ETUDE DE LEURS PROPRIETES ELECTROCHIMIQUES.**

**THESE DE DOCTORAT** - Specialite: sciences des materiaux presentee a l'Universite de Picardie Jules Verne

**Jury :** M. le Prof. J. Etourneau (Universite Bordeaux) M. D. Fruchart (DR, Lab. Cristallographie, Grenoble) Mme A. Percheron - Guegan (DR, LCMTR, Thiais) M. le Prof. L. Schlapbach (Universite de Fribourg) M. le Prof. J-M. Tarascon (Universite de Picardie) M. L. Aymard (Universite de Picardie)

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**F.Charlot**

**Etude et compréhension des réactions auto-entretenues activées mécaniquement. Elaboration du composé FeAl nanostructuré.**

6 Déc. 1999 - Université de Technologie de Belfort - Montbéliard

Nanomatériaux : Elaboration et Transitions de Phases Hors Equilibre, UPR 806 CNRS, UTBM - Sévenans.

Matériaux à Grains Fins, LRSS UMR 5613 CNRS-Université de Bourgogne.

**Jury (Provisoire) :** G. Bertrand, G.LeCaer (Rapp.) , F.Thévenot (Rapp.) , F.Bernard (Co - Dir.), E.Gaffet (Co - Dir.), J.C.Gachon, M.Bessière, M.Gailhanou

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**Frédéric BERNARD**

25 Novembre 1999 - Amphi de l'ESIREM - Dijon

**De l'introduction de " mécanique " dans l'élaboration de la poudre au massif nanométrique vers la maîtrise des propriétés thermomécaniques.**

**Jury :**D.LOUER, Directeur de Recherches CNRS(Université de Rennes II) rapporteur H. VAN DAMME, Professeur (Université d'Orléans) rapporteur J.C. TEDENAC, Professeur (Université de Montpellier II) Rapporteur J. FOCT, Professeur (Université de Lille) G. LE CAER , Directeur de recherches CNRS (Ecole des Mines, Nancy) G. BERTRAND, Professeur (Université de Bourgogne) A. NONAT, Directeur de Recherches CNRS (Université de Bourgogne) J.C. NIEPCE, Professeur (Université de Bourgogne)

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**Frédérique PERROT-SIPPLE**

17 Novembre 1999 - Université de Bourgogne - Dijon

**Maitrise de la taille de nanograins d'oxydes de structure perovskite**
**pour applications électrocéramiques:**
**- Synthèse par chimie douce,- Broyage par attrition.**

**Jury :** M. J.-M. HAUSSONNE Professeur, Ecole d'Ingénieurs de CherbourgM. A. ROUSSET Professeur, Université de Toulouse M BEAUGER Alain Ingénieur de Recherche et Développement TPC Saint ApollinaireM. BERTRAND Gilles Professeur, Université de Bourgogne M. CHARTIER Thierry Chargé de Recherche, ENSCI de Limoges M. HUGENTOBLER Denis Directeur Stratégie et Développement ligne céramique, TPC Saint ApollinaireM. MUTIN Jean-Claude Directeur de Recherche, Université de Bourgogne Mme RIEUX Nadine Ingénieur de Recherche Alstom-PERT, Massy **Directeurs de thèse:**M. D. AYMES Maître de Conférences, Université de Bourgogne M. P. PERRIAT Professeur, INSA de Lyon

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**H. SOUHA**

Thèse de Doctorat d'Etat Marocaine
Faculté des Sciences Dahr El Mehraz Fes.

**Elaboration par recuit et par réaction de combustion du composé Cu3Si à partir d'un mélange de poudres activées mécaniquement. Réactivité du composé Cu3Si vis à vis du chlorure cuivreux.`**

**Jury :**B. Gillot, G. Bertrand, F. Bernard (Co - Dir.), E. Gaffet (Co - Dir.)

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## O. Held

**"Etude des réactions de combustions solide-solide ou solide liquide auto-entretenues pour différents intermétalliques du système Al-Ni-Ti.**
**Elaboration de ces mêmes intermétalliques par broyage mécanique et étude de leur cinétique de cristallisation**

Nancy, Faculté des Sciences, le 5/11/99.

**Jury :** J. L. Jorda, J. M. Moreau, P. Satre, J. C. Gachon, F. A. Kuhnast, F. Bernard, J. F. Bérar, M. Bessière.

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## D. Cracco

**"Recherche de Nouveaux Alliages Hydrurables de Forte Capacité Massique**
**Utilisable comme Matériaux d'Electrode Négative d'Accumulateur Ni - MH"**

CNRS - Thiais - France - 25 Juin 1999

**Jury :** B. Darriet, L. Schlapbach, B. Knosp, R. Portier, A. Percheron - Guégan

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## A. Gentil - Sagot

**Amélioration de la tenue au fluage d'un alliage d'argent (AIC)**
**par introduction d'une dispersion d'oxydes.**
**Elaboration par Métallurgie des Poudres**
**Ecole des Mines - Paris - 17 Juin 1999**

**Jury :** M. Grosbras, L. Charrin, S. Kleine, D. Havart, J. - L. Strudel, Y. Bienvenu

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## Cooperative Research on Related Areas
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### COREE du SUD (19/01/2000)
From Professor Soon H. Hong
Dept. of Materials Science and Engineering - Korea Advanced Institute of Science and Technology
373-1 Kusung-dong, Yusung-gu - Taejon, 305-701, Korea
E-mail : HYPERLINK mailto:shhong@sorak.kaist.ac.kr / shhong@sorak.kaist.ac.kr
Fax. : 82-42-869-3310 - Tel. : 82-42-869-3327
We are currently working on the mechanical alloying processes and the characterization of mechanical & thermal properties of nanocrystalline materials and composite materials, such as SiC/Al, WC/Co and W/Cu for structural or thermal management applications. We are very pleased to discuss for international cooperative research on related topics with Members of Mechanosynthese Group.

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## Ph D Position and Post Doc Position Requests - Proposals

### FRANCE (14 / 02 / 2000)
### Ph D Thesis Proposal
A partir de septembre 2000 (Bourse du Ministère)
**"Obtention par mécanosynthèse de mélanges composites à base de magnésium ayant des propriétés d'hydruration optimisées. Caractérisation de leurs propriétés structurales et physiques.**
En résumé, le sujet proposé portera sur les deux points suivants :
1 - Etude de mélanges composites Mg (ou Mg2Ni) + intermétalliques élaborés par mécanosynthèse (structure, composition chimique, capacités d,absorption d,hydrogène, morphologie, surface, granulométrie, **);**
2 - Réalisation d,électrodes négatives à partir de ces mélanges et études électrochimiques.
Les techniques utilisées au cours de ce travail seront* :
Préparation : - Broyeur planétaire - Four à lévitation - Four à arc - Banc d,hydruration (construction de courbe PCT)
Caractérisation : - Diffraction des rayons X sur poudres - Microsonde électronique - Microscopie électronique ( à balayage et en transmission) - Mesures de surfaces spécifiques (BET) - Granulométrie (diffraction Laser - "Mesures" électrochimiques - Mesures calorimétriques XPS, EPMA, ...
* ceci est une liste non exhaustive des différentes techniques que le candidat devra utilisé
**Contact :**
**Jean-Louis BOBET**
Associate professor - Institut de Chimie de la Matière Condensée de Bordeaux Avenue du Dr A. Schweitzer
33608 Pessac Cedex FRANCE
Tel : 33-(0)5-56-84-26-53 Fax : 33-(0)5-56-84-24-80 e mail : bobet@icmcb.u-bordeaux.fr
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### USA (8 / 02 / 2000)
Rutgers University is seeking a **postdoctoral associate** with demonstrated expertise in powder synthesis and processing (forming and sintering methods) to work on research focused on textured ceramic ferroelectric materials. The candidate must be able to work as part of a multidisciplinary team involving industry and academia focused on making transducer and actuator materials. The candidate should demonstrate the ability to work independently, publish in archival journals and present their work in a public forum. The candidate should send a curriculum vitae, three representative publications (preferably with the candidate as a first author) and the names, addresses, email and phone numbers of three references that can comment on the candidate's capabilities. The position is available immediately at a salary of $32,000 with health benefits included. The position is available immediately. Placment is preferred prior to August with priority given to a qualified candidate with earlier availability.
**Interested candidates should send correspondenc**e to: Professor Richard E. Riman, Rutgers University, Department of Ceramic and Materials Engineering, 607 Taylor Road, Piscataway, NJ 08854-8065, riman@alumina.rutgers.edu / 732-445-4946 / 732-445-6264
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### COREE du SUD (10 / 01 / 2000)
From Professor Soon H. Hong
Dept. of Materials Science and Engineering - Korea Advanced Institute of Science and Technology
373-1 Kusung-dong, Yusung-gu - Taejon, 305-701, Korea
E-mail : HYPERLINK mailto:shhong@sorak.kaist.ac.kr /shhong@sorak.kaist.ac.kr
Fax. : 82-42-869-3310 - Tel. : 82-42-869-3327
The Composite Materials Laboratory at Korea Advanced Institute of Science and Technology is looking for a postdoctoral position. The postdoctoral contract will be one year on the field of modeling and simulation of mechanical & thermal properties of composite materials or on the field of fabrication process of nano-composite materials. Applicant should be within three years of receipt of Ph.D. degree on related field. For more information, please contact :

### ISRAEL (13 / 10 / 1999)
***** Postdoctoral Position Available for a Ph.D Physicist** (13th October 1999)

The High-Pressure (HP) group of the School of Physics & Astronomy at Tel Aviv University has a one year position available. This position could be extended to two years.
The HP group is known worldwide as one of the leading groups in experimental HP physics and is on the frontier of HP and diamond-anvil-cells based methodology. The main methods used are:
Mössbauer spectroscopy
X-ray diffraction with synchrotron radiation (ESRF, Grenoble) resistivity
Applicants must be between the final stage of dissertation and within three years of receipt of Ph.D diploma.
For more information, please contact:
Dr. Moshe P. Pasternak
School of Physics and Astronomy
Tel Aviv University - 69978 Tel Aviv, ISRAEL - email: hh136@ccsg.tau.ac.il

## FRANCE (2/07/99)

Joindre Pascal Viel , tél 01 69 08 41 47 CEA SRSIM Bt 461, 91191 Gif sur yvette
Proposition de post doc qui débuterait idéalement en septembre 99. Le financement du post doc est acquis (1 an) Le lieu de travail est le centre de Saclay (DSM/DRECAM/SRSIM) Le sujet concerne une étude très appliquée sur la dépollution des eaux de rejets industriels : Mise au point et étude d'un procédé d'élimination des métaux lourds basé sur la fabrication d'un filtre actif (complexation-décomplexation) utilisant la modification de surfaces métalliques par des films polymères électrogreffés en couches minces.
Conditions: avoir sa thèse depuis moins d'un an, ne pas avoir été salarié depuis sauf pour un an de postdoc à l'étranger et avoir moins de 30 ans

# Bibliographie Récente

## Livres ou "Special Issues"

**(05/05/2000)**
**EXTRACTIVE METALLURGY OF ACTIVATED MINERALS**
included in series Process Metallurgy, 10
by P. Balaz - Institute of Geotechnics, Slovak Academy of Sciences
ISBN : 0 - 444 - 50206 - 8 / Price USD 144, Euro 124.79)
http : // www.elsevier.nl/inca/publication
Description
Mechanical activation of solids is a part mechanochemistry, the science with a sound theoritical foundation exhibiting a wide range of potential application. Mechanical activation istself is an Innovative procedure where an improvement in technological processes can be attained via a combination of new surface area and defects formation in minerals.
Mechanical activation is o exceptional importance in extractive metallurgy and mineral processing and this area forms the topic of this book and is a result of more than twenty years of research and graduate teaching in the field.
In pyrometallurgy, the mechanical activation of minerals makes it possible to reduce their decomposition temperatures or causes such a degree of disordering that the thermal activation may be omitted entirely. The potential mitigation of environmental pollutants is becoming increasingly important in this context.
The lowering of reaction temperatures, the increase of the rate and amount of solubility, preparation of water soluble compounds, the necessity for simpler and less expensive reactors and shorter reaction times are some of the advantages of mechanical activation in hydrometallurgy. The environmental aspects of these processes are particularly attractive.
Several industrial processes are examined and the flowsheets are presented as successfull of activation. In these processes, the introduction of a mechanical activation step into the technological cycle significantly modifies the subsequent steps.
The book is designed for researchers, teachers, operators and students in the areas of extractive metallurgy, mineral processing, mineralogy, solid state chemistry and materials science. It will encourage newcomers to the mechanochemistry to do useful research and discover novel applications in this field.

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**(3/02/2000)**
Two new books on mechanical alloying are now available from Cambridge International Science Publishing (infos fournies par Anne Porter - Publishing Manager - Cambridge International Science Publishing http://www.demon.co.uk/cambsci/homepage.htm)
**1. MECHANICAL ALLOYING - FUNDAMENTALS AND APPLICATIONS**
http://www.demon.co.uk/cambsci/book52.htm Contents
Introduction (history, benefits of mechanical alloying); Mechanical alloying (alloying mills, mills in practice, improved mills, the process, parameters);
Variations of mechanical alloying (reaction milling, cryomilling, repeated rolling, double mechanical alloying, repeated forging); Process control agents in mechanical alloying; Mechanical alloying mechanisms (ductile-ductile system, ductile-brittle system, brittle-brittle system, metastable phase formation, amorphisation, nanocrystallization, extension of solid solubility, activation of solid state chemical interaction);
Energy transfer and energy maps;
Consolidation of mechanically alloyed powders (consolidation techniques, thermomechanical treatment); Mechanical properties of mechanically alloyed materials (tensile properties, fracture, creep, stress corrosion cracking susceptibility);
Modelling mechanical alloying (mechanistic models, deformation, coalescence and fragmentation, evolution of particle size, milling time, powder heating, powder cooling, atomistic model, thermodynamic and kinetic model)
Joining of mechanically alloyed materials; Rapid solidification and mechanical alloying; Applications (nickel-based superalloys, Al-based materials, supersaturated solutions, magnetic materials, mechanically alloyed powders for spray coatings, superplasticity, tribological materials, composites, amorphous solids, nanocrystalline materials, solid-state chemical reactions, etc). ISBN 1898326568, 160 pages 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 $Al-Al_4C_3$ material)

4. Static and dynamic mechanical properties (mechanical properties at elevated temperatures; mechanical properties at 20 °C; effect of interface on the mechanical properties; superplastic properties of the system; thermal stability of the system; creep characteristics; creep-fatigue characteristics)
References - ISBN 189832655X, 90 pages, 234 **156 mm, soft laminated cover,** £25.00, 1999

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## "Mechanical Alloying : Fundamentals and Applications"

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

web site : http://www.demon.co.uk/cambsi/book52.htm

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## "Non Equilibrium Processing of Materials"

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

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

web site : www.elsevier.nl/locate/isbn/0080426972

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## Bulk Amorphous Alloys : Preparation and Fundamental Characteristics

A. Inoue
Materials Science Foundation Vol. 4 - Trans Tech Publications : http ://www.ttp.net
Interest in bulk amorphous alloys has increased rapidly throughout the workd and these materials have now gained a position of great importance in basic science and engineering materials technology. Bulk amorphous alloys based upon the Zr - Al - Ni - Cu, Zr (Tin,Nb) - Al - Ni - Cu and Zr - Ti - Ni - Cu - Be systems have already achieved wide commercial success as components of various technical accessories ranging from sporting goods to optical instruments.
Here is a state of the art reviews on this new group of materials, covering all areas of interest, ranging from the synthesis of these special alloys and their fundamental properties, to their engineering characteristics and applications.
This work will therefore be of equal interest to those who wish to become fully acquainted with the subject, and to those who are already actively engaged in the field.

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## DISPERSION-STRENGTHENED ALUMINIUM PREPARED BY MECHANICAL ALLOYING

Michal Besterci, Institute of Materials Research, Slovak Academy of Sciences, Kosice
In the book, the author describes the theoretical and technological fundamentals of mechanical alloying the Al-C system. Special attention is given to material characteristics, the kinetics and mechanism of mechanical alloying, methods of mixture compaction and heat treatment of compacted parts. Models of dispersoid spatial arrangement, dispersoid evaluation and optimisation and experimental possibilities are discussed. The interpretation of the static and dynamic mechanical properties, especially strength and ductility properties at 20 °C, mechanical properties at elevated temperatures are discussed, with emphasis on the effect of interface, superplasticity, creep and creep-fatigue characteristics. Content
Introduction
1. Characteristics of dispersion-strengthened systems
2. Mechanical alloying (kinetics and mechanism of preparation of the Al-C system by mechanical alloying; compaction of powders and heat treatment of compacts;
3. Microstructure and quantitative evaluation of parameters of dispersion-strengthened materials (definition and properties of interparticle distance; experimental possibilities of determination of structural objects; models of heterogeneous structures and their evaluation; simulation of model structures; analysis of the spatial distribution of particles in the Al-$Al_4C_3$ material) 4. Static and dynamic mechanical properties (mechanical properties at elevated temperatures; mechanical properties at 20°C; effect of interface on the mechanical properties; superplastic properties of the system; thermal stability of the system; creep characteristics; creep-fatigue characteristics)
Index : ISBN 189832655X, 80 pages, 234 **156 mm, soft laminated cover,** £22.00, January 1999
Cambridge International Science Publishing 7 Meadow Walk, Great Abington, Cambridge CB1 6AZ, England Fax +44 1223 894539; Tel +44 1223 893295 Email: cisp@cisp.demon.co.uk
http://www.demon.co.uk/cambsci/homepage.htm

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## "Mechanical Alloying"

Auteurs : Li Lü & Man On Lai(National University of Singapore)
Kluwer Academic Publishers
**Contents :** Preface - Introduction to Mechanical Alloying - Experimental Set - Up - The Mechanical Alloying Process - Formation of New Materials - Characterization of Powders - Densification - Mechanical Properties - Mechanisms of Mechanical Alloying - Modeling of Mechanical Alloying - Index

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

Editors: Kenneth E. Gonsalves, Marie-Isabelle Baraton, Rajiv Singh, Heinrich Hofmann, Jerry X. Chen, and Joseph A. Akkara.
Materials Research Society, Symposium Proceedings Volume 501, 1998

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

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## "Nanomatériaux"

Auteurs : E. Gaffet, S. Begin - Colin, O. Tillement
Editeur : Innovation 128 - 24 Rue du Quatre Septembre - 75002 Paris - France - Fax : 33 1 42 65 47 76

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

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## CHEMISTRY FOR SUSTAINABLE DEVELOPMENT
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Proceedings of 2d International Conference on Mechanochemistry
(INCOME-2), which was held in Novosibirsk in 1997.

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

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## Mechanochemistry of Materials
## Cambridge International Science Publishing

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

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

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### BIBLIOGRAPHY ON MECHANICAL ALLOYING AND MILLING
**Suryanarayana (Inst for Materials and Advanced Processes, University of Idaho, USA )**

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

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**Proceeding du Congrès "Mechanically Alloyed, Metastable and Nancrystalline Materials"- Barcelone (1997)**
Editor : M.D. Baro, S. Surinach - Materials Science Forum 269 - 272 (1998)

## PERIODIQUES

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

**[65] USING EQUAL CHANNEL ANGULAR PRESSING FOR REFINING GRAIN SIZE**

Langdon TG. Furukawa M. Nemoto M. Horita Z. - JOM-Journal of the Minerals Metals & Materials Society. 52(4):30-33, 2000

Equal-channel angular pressing is an effective tool for attaining ultrafine grain sizes in bulk materials. An important advantage of this technique over conventional metal-working processes, such as extrusion and rolling, is that very high strains may be attained without any concomitant change in the cross-sectional dimensions of the sample. The microstructures introduced by equal-channel angular pressing critically depend on a number of experimental factors including the nature of the slip systems introduced during the pressing operation and the total strain imposed on the sample. These factors are illustrated by reference to experiments conducted on pure aluminum; results are also included to demonstrate the influence of alloying additions and especially the remarkably small grain sizes that may be achieved in materials having low rates of recovery.

**[64] EXTENDED SOLID SOLUBILITY FOR AL-W BINARY SYSTEM BY MECHANICAL ALLOYING**

Ouyang YF. Zhong XP. Wu WM. - Science in China, Series a (Mathematics, Physics, Astronomy). 43(2):180-184, 2000

$Al_{1-x}W_x$($x$ = 0.1, 0.2, 0.5, 0.9) powders have been prepared by mechanical alloying of the elements in a planetary ball mill. The structure and morphology of the milled powders were investigated using X-ray diffraction and electron microscopy. The solubility of Al in W is greatly extended, even more than 50%. The results are analyzed using embedded atom method (EAM) according to the scheme of mechanical alloying extending solid solubility. The theoretical results are in good agreement with the experimental.

**[63] EXPERIMENTAL STUDY OF THE SPIN DENSITY OF METASTABLE FCC FERROMAGNETIC FE-CU ALLOYS**

Bove LE. Petrillo C. Sacchetti F. Mazzone G. - Physical Review B. 61(14):9457-9466, 2000

Magnetization density measurements on metastable $Fe_xCu_{1-x}$ alloys at four compositions ($x$ = 20, 40, 50, and 60 at. %) and at 5 K temperature were carried out by means of polarized neutron diffraction. The samples were produced by high-energy ball milling and characterized by x-ray diffraction and fluorescence measurements. Additional bulk magnetization measurements were carried out on the two samples at high Fe concentration. Over the present concentration region, the Fe-Cu system is ferromagnetic and the four samples were found to be in the fcc phase. Fe-Cu is therefore a very suitable system to investigate the magnetic state of Fe in an fee environment. Other than confirming that the Fe-Cu system is not a simple dilution alloy, the present results were compatible with a two-state model for fee Fe-that is, two different coexisting electronic states associated with different magnetic moments and form factors.

**[62] THE SYNTHESIS OF TIN BY BALL-MILLING - A NEUTRON DIFFRACTION STUDY**

Campbell SJ. Hofmann M. Calka A. - Physica B. 276:899-900, 2000

Titanium nitride of FCC B1 structure forms readily on milling Ti in nitrogen and ammonia environments. Rietveld refinements of time-of-flight neutron diffraction patterns show that stoichiometric TIN is produced on milling Ti in nitrogen, whereas the off-stoichiometric nitride $TiN_{0.7}$, associated with the presence of hydrogen, is formed on milling in ammonia. The transformation of Ti to the nitride proceeds more rapidly in ammonia due to the catalytic effect of hydrogen.

**[61] THE COLD NEUTRON MULTILAYER PHASE-SPIN ECHO INTERFERENCE EXPERIMENT WITH MECHANICAL ALLOYING SAMPLES TO INVESTIGATE THE STATE TRANSITION WHICH DERIVES DECOHERENCE**

Otake Y. Fukunaga T. Tasaki T. Ebisawa T. Achiwa N. Hino M. - Physica B. 276:983-984, 2000

It has not been solved theoretically and experimentally how to explain the process which derives decoherence from coherent state though certain interactions between a quantum system such as a neutron and mesoscopic system, which consists of many particles. It is still an open question how to explicate decoherence and the quantum process through the quantum mechanical measurement process. In the last few decades this subject has become more important and discussed in various fields. Many quantum mechanical measurement theories derive decoherence from coherent state without experimental verification. Theoretically, it seems already clear that a kind of fluctuation of the mesoscopic system derives decoherence. We have done interference experiments to study this phenomena using spin interferometer for cold neutron inserting such a machanical alloying (MA) samples as $Ni_{25}T_{75}$ Of different alloying time. We chose such samples as Ni and Ti so that the total potential becomes zero but the fluctuation of weighted structure factor remains. We study this system to find the phenomena decreasing the coherence of wave function after once it improves in a diffuse scattering component.

**[60] CRYOGENIC MECHANICAL ALLOYING OF POLY(METHYL METHACRYLATE) WITH POLYISOPRENE AND POLY(ETHYLENE-ALT-PROPYLENE)**

Smith AP. Ade H. Balik CM. Koch CC. Smith SD. Spontak RJ. - Macromolecules. 33(7):2595-2604, 2000

Mechanical alloying is performed at cryogenic temperatures to incorporate polyisoprene (PI) or its hydrogenated analogue poly(ethylene-alt-propylene) (PEP) into poly(methyl methacrylate) (PMMA) as an example of high-energy solid-state blending. Morphological characterization of the blends by X-ray and electron microscopies confirms that the degree of dispersion of the constituent polymers improves with increasing milling time. Such dispersion in the PEP/PMMA blends is, however, ultimately compromised by phase coarsening when the materials are postprocessed above the PMMA glass transition temperature in the melt. Milling-induced PI cross-linking serves to suppress phase coarsening in PI/PMMA blends, which remain relatively well-dispersed even after postprocessing. These blends are generally less fracture-resistant than the as-received PMMA due mainly to the accompanying reduction in PMMA molecular weight. Their optical transparency is observed to decrease dramatically with increasing PEP or PI concentration until they appear opaque. An overall improvement in blend properties by mechanical alloying is, however, anticipated upon judicious selection of more degradation-resistant polymers.

**[59] EFFECTS OF ZR ADDITION ON DISCHARGE PROPERTIES OF MECHANICALLY ALLOYED MG2NI HYDROGEN-STORAGE ALLOY ELECTRODE**

Goo NH. Jeong WT. Lee KS. - Journal of Power Sources. 87(1-2):118-124, 2000

(Mg1-xZrx)(2)Ni (x = 0.0, 0.1, 0.2, 0.3 and 0.4) hydrogen-storage alloy electrodes are synthesized by means of a mechanical alloying process using a planetary ball mill. After milling for 160 h, (Mg1-xZrx)(2)Ni alloy with x = 0.0 remained the nanocrystalline phase. By contrast, Zr addition to this alloy enhances the structural disorder and amorphization. The discharge capacity of a (Mg1-xZrx)(2)Ni electrode increases with Zr content and reaches the highest capacity of 530 mAh g(-1) at x = 0.3, then decreases to 230 mAh g(-1) at x = 0.4, i.e., lower than that of pure Mg2Ni. Cyclic stability and rate capability vary with Zr addition.

**[58] MAGNETIC AND MOSSBAUER STUDY OF MULTIPHASE FE-ZR AMORPHOUS POWDERS OBTAINED BY HIGH ENERGY BALL MILLING**

Pizarro R. Garitaonandia JS. Plazaola F. Barandiaran JM. Greneche JM. - Journal of Physics-Condensed Matter. 12(13):3101-3112, 2000

The amorphous system Fe-Zr has been extensively studied due to its particular magnetic behaviour, especially in the concentration range around 90 at.% Fe, only accessible by rapid quenching. We have extended the study to lower Fe content alloys, by using mechanical alloying to synthesize FexZr100-x amorphous powders with x = 65, 70, 75. Mossbauer spectra and magnetization measurements have been performed as a function of temperature. Different magnetic measurements were performed to characterize the Curie temperatures of these multiphase samples and hyperfine field (B-hf) distributions, P(B-hf), were used to fit Mossbauer spectra below the Curie temperature (T-C) and quadrupole splitting distributions, P(Delta), above T-C, in agreement with the amorphous structures. The hyperfine field distributions of as-milled samples are characterized by two maxima and are fitted with two Gaussian curves that evolve with temperature in position, intensity and width. This bimodal behaviour has been also found in higher Fe content FeZr alloys as well as in ternary alloys with boron, and with nickel, and can be interpreted as arising from different neighbourhood Fe sites. The rather uncommon P(Delta) are also interpreted in the same terms.

**[57] NANO-STRUCTURED INTERMETALLIC COMPOUND TIAL OBTAINED BY CRYSTALLIZATION OF MECHANICALLY ALLOYED AMORPHOUS TIAL, AND ITS SUBSEQUENT GRAIN GROWTH**

Kambara M. Uenishi K. Kobayashi KF. - Journal of Materials Science. 35(11):2897-2905, 2000

The amorphization process during mechanical alloying (MA) was investigated for the Al-50at%Ti and Al-50at%Ti-10vol%TiB2 powder mixtures. Pure metallic powders of Al and Ti were finely mixed and transformed to the amorphous phase after being milled for about 2880 ks. In the case of Al-50at%Ti-10vol%TiB2 powder, the amorphous alloys with a fine dispersion of TiB2 particles could be obtained for a shorter milling times than that required for the powders without TiB2 ceramics. As a result of heat treatment for the mechanically alloyed amorphous powders, a nanocrystalline intermetallic compound of TiAl (gamma) could be produced. Subsequent grain growth of the gamma phase during heat treatment was investigated by estimating the grain-growth exponent and the activation energy for grain growth. It was found from this estimation that the grain growth was further suppressed as the powders were mechanically alloyed for longer times. Furthermore, the addition of the TiB2 particles that could be dispersed during MA finely and homogeneously in the amorphous matrix was found to be effective for suppression of the gamma grain growth especially at elevated temperatures as well as for a long annealing.

**[56] NANOPHASE FE ALLOYS CONSOLIDATED TO FULL DENSITY FROM MECHANICALLY MILLED POWDERS**

He L. Allard LF. Breder K. Ma E. - Journal of Materials Research. 15(4):904-912, 2000

Nanophase elemental Fe powders prepared by mechanical milling were sinter forged to full density with an average grain size in the nanophase range (below 100 nm). If Cu additions are introduced during milling to form supersaturated solid solutions (Fe85Cu15 and Fe60Cu40), grain sizes can be easily controlled to below 50 nm after consolidation. For Fe-Cu, it was observed that atomic level alloying between the two elements during milling was very helpful for obtaining a homogeneous microstructure and nanocrystalline grain/domain sizes in the consolidated product. The advantages of using sinter forging (upset die forging), as well as the role of the Cu addition, in the retention of nanocrystalline grain sizes are discussed. The consolidated Fe alloys exhibit very high strength under compression, further demonstrating that low populations of flaws and nanophase grain structures were attained in the consolidated products.

**[55] EFFECT OF SURFACE MODIFICATION OF TIV2.1NI0.3 BY BALL-MILLING WITH MGNI ON CHARGE-DISCHARGE CYCLE PERFORMANCE**

Choi WK. Tanaka T. Morikawa T. Inoue H. Iwakura C. - Journal of Alloys & Compounds. 302(1-2):82-86, 2000

Charge-discharge cycle performance of TiV2.1Ni0.3 alloy was markedly improved by surface modification with MgNi alloy using a ball-milling method. For the purpose of clarifying the effect of the surface modification, the change in the surface structure before and after the charge-discharge measurement was investigated by X-ray photoelectron spectroscopy (XPS). As a result, it was found that the mutual diffusion layer was formed after ball-milling for 3 h and it could work as a barrier against the deep corrosion of the components. This is thought to be responsible for the improvement of the charge-discharge cycle performance. However, the extension of the mutual diffusion layer by ball-milling for longer duration led to the acceleration of the corrosion, resulting in the formation of the thick oxide layers, probably due to the decrease in surface concentration of the Mg and Ni components and to the increase in those of the Ti and V components, causing the deterioration of the charge-discharge cycle performance.

**[54] DECOMPOSITION OF NICKEL-DOPED MAGNESIUM HYDRIDE PREPARED BY REACTIVE MECHANICAL ALLOYING**

Tessier P. Akiba E. - Journal of Alloys & Compounds. 302(1-2):215-217, 2000

Magnesium hydride is prepared by reactive mechanical alloying. The catalytic effect of a small amount of nickel, added to the starting mixture, is examined. The influence of nickel on the decomposition temperature of the hydride is subsequently studied.

**[53] TENSILE PROPERTIES OF NITRIDE DISPERSED AL-TI ALLOY SYNTHESIZED BY REACTIVE BALL MILLING IN N-2 GAS**

Moon KO. Oh MS. Lee KS. - Journal of Alloys & Compounds. 302(1-2):227-234, 2000

Nitride dispersed Al-Ti alloys have been prepared by reactive ball milling (RBM) of elemental powders of Al and Ti in nitrogen gas. The particle size and the grain size were effectively reduced by the hard and brittle TiN and the dissolved nitrogen atoms. The as-milled powder was consolidated by hot extrusion at 500 degrees C. The grain size of the as-extruded specimen was about 50-100 nm, and TiN with a grain size of 10 nm existed all over the specimen. UTS of Al-10 wt.%(Ti+TiN) alloy produced by two-step RBM in N-2 and hot extrusion was lower than that of Al-5 at.%Ti alloy prepared by RBM in H-2 and hot extrusion, although the hardness of the former was higher than that of the latter. The ductilities of Al-10 wt.%(Ti+TiN) were very poor and even less than those of specimens prepared by RBM in H-2. The dispersoids existing at the grain boundary appeared to restrict deformation through the grain boundary and promote the formation of micro-voids at the grain boundaries. Thus, fracture easily occurred intergranularly in this ahoy and the ductility was poor at all tested temperatures.

**[52] EFFECTS OF A PARTIAL SUBSTITUTION OF FE BY MO AND MO PLUS CO ON THE STRUCTURE AND MAGNETIC PROPERTIES OF ND8.4FE87.1B4.5 ALLOYS PREPARED BY MECHANICAL ALLOYING**

Cui BZ. Sun XK. Liu W. Geng DY. Yang ZQ. Zhang ZD. - Journal of Alloys & Compounds. 302(1-2):281-286, 2000

The effects of the addition of Mo alone and the addition of Mo pins Co an the structure and magnetic properties of mechanically alloyed Nd8.4Fe87.1B4.5 alloy were studied. Both the addition of Mo alone and the addition of Mo plus Co result in an increase in the Curie temperatures of the hard phases. The addition of Mo alone, especially for the 1.6 at% Mo-added case, results in the improvement of the intrinsic coercivity H-0(c) and the maximum magnetic energy product (BH)(max). The addition of Mo alone results in refinement of alpha-Fe grains but in coarsening of the hard grains. The addition of both Co and Mo can both refine the soft grains and inhibit the trend of coarsening of the hard grains due to the addition of Mo alone. The best magnetic properties: mu(0)H(c) = 0.58 T (5.8 kOe), J(r) = 1.04 T (10.4 kG), (BH)(max) = 113.8 kJ m(-3) (14.3 MGOe), J(r)/J(s) = 0.68, are achieved for the Nd8.4Fe80.4Co5.6Mo1.1B4.5 alloy.

**[51] THE ELECTRONIC AND ELECTROCHEMICAL PROPERTIES OF THE ZRV2 AND ZR(V0.75NI0.25)(2) SYSTEMS**

Szajek A. Jurczyk M. Rajewski W. - Journal of Alloys & Compounds. 302(1-2):299-303, 2000

The ZrV2 and Zr(V0.75Ni0.25)(2) systems crystallize in the Laves-phase MgCu2 (C15) crystal structure. These materials have been prepared using a simple mechanical alloying (MA) followed by annealing. The amorphous phase forms directly from the starting mixture of the elements, without other phase formation. Heating the MA samples at 1070 K for 1 h resulted in the creation of ordered ZrV2-type alloys. In mechanically alloyed and annealed ZrV2 and Zr(V0.75Ni0.25)(2) materials discharge capacities of 0 and 100 mAhg(-1) were obtained, respectively. The electronic structure has been studied by the tight binding version of the linear muffin-tin orbital method in the atomic sphere approximation (TB-LMTO ASA). The Ni impurities cause a charge transfer from the Zr and V atoms to the Ni atom, the valance band is wider and the density of electronic states at the Fermi level decreases by about 30%.

**[50] EFFECT OF RESPECTIVE ADDITIONS OF GA, ZR AND COMBINED ADDITIONS OF CO, GA, ZR ON MAGNETIC PROPERTIES OF REMANENCE-ENHANCED ND8.4FE87.1B4.5 ALLOY**

Cui BZ. Sun XK. Liu W. Zhang ZD. Geng DY. Zhao XG. - Journal of Materials Science & Technology. 16(2):118-120, 2000

The effect of a partial substitution of Zr; Ga; Co and Ga; Co, Ga and Zr; respectively for Fe on the structure and magnetic properties of Nd8.4Fe87.1B4.5 alloy prepared by mechanical alloying has been studied in detail. It has been shown that, intrinsic coercivity mu(o)H(c), and maximum magnetic energy product (BH)(max) increase for only Zr or Ga-containing samples, which is mainly due to the refinement of soft grains. The combined addition of Co and Ga is less effective for improving mu(o)H(c) and (BH)(max) than the respective addition of Co or Ga, which may be due to the formation of CsCl-type CoGa. mu(o)H(c) and (BH)(max) for combined Co, Ga, Zr-added alloy are higher than those for the combined Co, Ga-added alloy but lower than those for only Zr-containing alloy.

**[49] HARD MAGNETIC PROPERTIES OF SM-FE-C BASED ALLOYS PREPARED BY MECHANICAL ALLOYING**

Geng DY. Zhang ZD. Cui BZ. Guo ZJ. Liu W. Zhao XG. - Journal of Materials Science & Technology. 16(2):125-126, 2000

The structure and magnetic properties of SmyFe(100-1.5)yC(0.5y)(y=8 similar to 20) alloys prepared by mechanical alloying (MA) from Sm, Fe and graphite have been investigated systematically. In order to improve hard magnetic properties of the alloys prepared by mechanical alloying, a new method consisting of re-milling and re-annealing was developed. After being re-milled and re-annealed, the Curie temperature T-C of the Sm-Fe-C alloys changes. The T-C of 2:17 phase increases, whereas the T-C of 2:14:1 phase decreases. After being re-annealed at low temperatures, the grain sizes of hard phases are smaller than those in the alloys annealed at high temperatures. The effects of Co or Ti substitution for Fe are studied.

**[48] HIGH ENERGY AND HIGH COERCIVITY SINTERED NDFEB MAGNETS BY LOW OXYGEN PROCESS**

Ding KH. Liu GZ. Li ZJ. Yan JM. Tao YJ. Wu B. - Journal of Materials Science & Technology. 16(2):127-128, 2000

Using ball milling and single direction pressing, we can produce high performance NdFeB sintered' magnets. The oxygen content of sintered magnets can be controlled under 1500x10(-6) and the magnetic performance can be improved by using low oxygen processing. The high performance NdFeB sintered magnets with B-r=(1.4+/-0.2)T, (j)H(c)greater than or equal to 796kA/m and (BH)(max)=(390+/-16) kJ/m(3), have been batch produced.

**[47] NANOCOMPOSITES OF SN AND LI2O FORMED FROM THE CHEMICAL REDUCTION OF SNO AS NEGATIVE ELECTRODE MATERIAL FOR LITHIUM-ION BATTERIES**

Foster DL. Wolfenstine J. Read JR. Behl WK. - Electrochemical & Solid-State Letters. 3(5):203-204, 2000

A nanocomposite of lithium oxide and tin was prepared by reducing SnO with Li3N through a powder milling process. The tin particles were found to be uniformly distributed within a lithium oxide matrix with the majority of tin particles on the order of 100 nm or less. The nanocomposite was evaluated as an electrode material. Most of the irreversible capacity on the first cycle associated with SnO electrodes was eliminated.

**[46] BINARY MIXTURES OF SEMICRYSTALLINE/NONCRYSTALLINE POLYMERS FORMED BY BALL MILLING**

Font J. Muntasell J. Cesari E. -Materials Research Bulletin. 34(14-15):2221-2230, 1999

Ball milling technique was applied to prepare binary mixtures of poly(ether imide) (PEI) with poly(ethylene terephthalate) (PET), poly(butylene terephthalate) (PBT), and poly(ether ether ketone) (PEEK). From differential scanning calorimetry (DSC) and X-ray diffraction (XRD) measurements, we have analyzed the influence that PEI has on the thermal behavior of these mixtures. A comparison with the blends formed by conventional methods has been performed.

**[45] RAPID HARDENING OF CEMENT BY THE ADDITION OF A MECHANICALLY ACTIVATED AL(OH)(3)-CA(OH)(2) MIXTURE**

Kitamura M. Kamitani M. Senna M. - Journal of the American Ceramic Society. 83(3):523-527, 2000

Rapid hardening of cement was achieved in the present study by adding a mechanically activated AI(OH)(3)-Ca(OH)(2) mixture to the starting cement paste. Among the dominant parameters for hardening were the mechanical treatment time for the Al(OH)(3) powder and the Al(OH)(3)/Ca(OH)(2) ratio. The hardening mechanisms are discussed here in terms of the ionic concentration of the solution and the hydration products created when the Al(OH)(3)-Ca(OH)(2) mixture was added to water. Mechanical activation of the Al(OH)(3) powder accelerated dissolution into an aqueous alkaline solution and induced the formation of calcium aluminate hydration products. Those hydration products increased the compressive strength of the cement paste at a very early stage of hardening.

**[44] INTERCALATION COMPOUNDS OF ALUMINUM HYDROXIDE**

Isupov VP. - Journal of Structural Chemistry. 40(5):672-685, 1999

Crystalline aluminum trihydroxides Al(OH)(3) (gibbsite, bayerite, and nordstrandite) can serve as layered intercalation matrices in which metal salts are arranged in a specific way. Small cations (lithium, magnesium, and transition metals) lie in the octahedral voids of aluminum hydroxide layers, and water molecules are located between the layers. This localization of small cations gives rise to the molecular sieve effect, where alkaline and alkaline earth cations (Na+, K+, Ca2+, etc.), which are large relative to the octahedral voids, are not intercalated into aluminum trihydroxides. In the first step of lithium salt intercalation, the cations, the anions, and the water molecules are incorporated into the interlayer space of aluminum hydroxide with subsequent transition of lithium into the voids of the layer.

**[43] UNDERSTANDING THE PHASE TRANSITIONS AND TEXTURE IN SUPERIONIC PBSNF4 - THE KEY TO REPRODUCIBLE PROPERTIES**

Collin A. Denes G. Le Roux D. Madamba MC. Parris JM. Salaun A.- International Journal of Inorganic Materials. 1(5-6):289-301, 1999

The recent use of high-performance fluoride ion conductor PbSnF4 for the fabrication of a solid state room temperature amperometric oxygen sensor implies that the materials used for its fabrication is well defined and undergoes no transformation over the lifetime of the sensor in the conditions of use. There are many ways to prepare PbSnF4, and subtle differences can lead to different PbSnF4 phases. Furthermore, moderate applications of heat and mechanical energy (ball milling), or minor changes in the composition of the reaction medium, can result in phase transitions, including order/disorder phenomena. In addition, it was also found that several phases, that appear stable, are in fact metastable and undergo transformations over prolonged periods of time. Furthermore, most phases of PbSnF4 show a considerable amount of preferred orientation due to the layered structure, causing highly anisotropic properties of polycrystalline samples. We have discovered methods for eliminating the preferential orientation of the crystallites and for enhancing it, close to the situation of a single crystal, in one direction. The adequate choice of the method of preparation, proper control of the preferred orientation, and knowledge of the phase transitions, should make possible the production of a material with stable and reliable properties

**[42] NEW EQUILIBRIUM PHASE IN THE FE-GE SYSTEM OBTAINED BY MECHANICAL ALLOYING**

Gerasimov KB. Pavlov SV. - Intermetallics. 8(4):451-452, 2000

A new equilibrium phase has been found in the Fe-Ge system. It has the composition Fe2Ge3, and decomposes at about 580 degrees C due to a peritectoid reaction, giving FeGe (cubic modification) and FeGe2. The phase forms by a eutectoid reaction from FeGe2 at about 530 degrees C; however, this transformation is kinetically inhibited. The Fe2Ge3 compound is likely to have a tetragonal lattice; structure type of Ru2Sn3, a = 5.59 Angstrom, c = 8.92 Angstrom.

**[41] MANUFACTURING OF ALUMINUM FLAKE POWDER FROM FOIL SCRAP BY DRY BALL MILLING PROCESS**

Hong SH. Lee DW. Kim BK. - Journal of Materials Processing Technology. 100(1-3):105-109, 2000

A feasibility study for producing aluminum flake powder from aluminum foil scrap by dry ball milling under an inert argon with a few % of oxygen was carried out. It was found that the pieces of aluminum foil scrap were laminated to each other, elongated by micro-forging of the falling balls, fragmented into small pieces of foil and finally formed into flake powder, during the ball milling. A larger ball is more beneficial to the milling of aluminum foil into the flake powder due to the large impact energy during the falling of the ball. Intermediate stops during the ball milling produced a finer aluminum flake powder than non-stop milling for up to 25 h due to cooling of the vial. A larger amount of stearic acid reduces friction between the foil or the balls and vial wall, thus give less milling efficiency, 3 wt.% of stearic acid as additive being verified as the optimum content. The aluminum flake powder produced by the dry ball milling of aluminum foil scrap can be applied to fingerprint detection or to aerate light-weight concrete.

**[40] THERMAL PROPERTIES OF MECHANOCHEMICALLY PRETREATED PRECURSORS OF BATIO3 SYNTHESIS**

Balaz P. Plesingerova B. - Journal of Thermal Analysis. 59(3):1017-1021, 2000
The changes of physico-chemical properties of mechanochemically pretreated ($BaCO_3+TiO_2+PbO$) powders were investigated. The values of apparent activation energy of $BaTiO_3$ formation calculated by the Freeman and Carroll method decrease with milling time. The changes of precursors density may be interpreted as a consequence of mechanochemical reactions during milling.

**[39] EFFECT OF MECHANOCHEMICAL TREATMENT ON THE SYNTHESIS OF CALCIUM DIALUMINATE**

Temuujin J. MacKenzie KJD. Jadambaa T. Namjildorj B. Olziiburen B. Smith ME. Angerer P. - Journal of Materials Chemistry. 10(4):1019-1023, 2000
Calcium dialuminate ($CaAl_4O_7$) powders were synthesised from mechanochemically treated mixtures of aluminium hydroxide + calcium hydroxide and aluminium hydroxide + calcium carbonate. On grinding, both mixtures produce X-ray amorphous precursor phases which show Al-27 MAS NMR resonances characteristic of Al in octahedral and tetrahedral sites, and a site identified by a resonance at 37-39 ppm (possibly pentahedral Al). Although grinding does not completely destroy the carbonate XRD reflections in the carbonate mixture, both precursors show a high degree of homogeneity and behave similarly on heating, forming monophase $CaAl_4O_7$ at < 1050 degrees C. By contrast, the same unground compositions form mixtures of alpha-alumina and various calcium aluminates (but not $CaAl_4O_7$) on heating as high as 1250 degrees C. Calcium dialuminate synthesised from the carbonate-containing mechanochemical precursor had a smaller particle size which may be advantageous for subsequent fabrication and sintering.

**[38] MAGNETIC COLLECTION OF GRINDING BALL FRAGMENTS FROM SAG AND BALL MILL CIRCUITS**

DA Norrgran, MJ Mankosa - MINERAL PROCESSING/ENVIRONMENT, HEALTH AND SAFETY, 1999, pp 177-189 - 4TH INTERNATIONAL CONFERENCE OF THE COPPER 99/COBRE 99; PHOENIX, ARIZONA. OCTOBER 10-13, 1999
Grinding ball fragments discharging from SAG mills and ball mills causes extreme wear to downstream processing equipment. These bail fragments, recirculating in a milling circuit, will cause excessive wear to sumps, pumps, hydrocyclones, and interconnecting piping. A magnetic separation system to remove grinding ball fragments from the mill discharge has been developed and successfully applied in the milling circuit. This magnetic separation system - termed Trommel Magnet - was first applied at the Escondida copper concentrator in Chile. The Trommel Magnet consists of an are of permanent magnets mounted at the discharge end of the trommel screen. The magnetic are attracts the ball fragments and removes them from the process stream. This retrofitted magnetic separation system removed 126 metric tons of ball fragments from the circulating load of a single ball mill in the initial 24 hour period. The mill has since stabilized and the magnetic separator is currently removing 7 metric tons per day of ball fragments. The retrofit of the Trommel Magnet has: 1. Extended the pump life and the hydrocyclone life approximately 300 percent. 2. Provided a 5 percent increase in the throughput of the mills by removing grinding ball fragments that contribute very little to the grinding process. Variations of the Trommel Magnet have also been developed for removing grinding ball fragments directly from the mill discharge without the use of a trommel screen. In each case, permanent magnets are used to collect the ball fragments from the mill. Discharge stream prior to reporting to the sump. These other systems have been designed and fabricated for in-plant test work.

**[37] MILLING FOR THE MILLENNIUM**

SM Jones, RF Pena - MINERAL PROCESSING/ENVIRONMENT, HEALTH AND SAFETY, 1999, pp 191-204 - 4TH INTERNATIONAL CONFERENCE OF THE COPPER 99/COBRE 99; PHOENIX, ARIZONA. OCTOBER 10-13, 1999
This paper describes the state of the art of grinding in the Copper Industry today including descriptions of the largest equipment and design features which allow processing of as much as 100,000 mtpd in a single mill line. We include a typical flowsheet, control philosophies, and maintenance techniques which result in low cost production which is essential in today's market.

**[36] APPLICATION OF MECHANICAL ALLOYING PROCESSING TO THE FORMATION OF COPPER-CARBIDE ALLOYS**

V Vergara, M Lopez, R Benavente, C Camurri, B Cartes, J Jimenez - PLENARY LECTURES/MOVEMENT OF COPPER AND INDUSTRY OUTLOOK/COPPER APPLICATIONS AND FABRICATION, 1999, pp 303-310 - 4TH INTERNATIONAL CONFERENCE OF THE COPPER 99/COBRE 99; PHOENIX, ARIZONA. OCTOBER 10-13, 1999
Four copper composites were obtained by mechanical alloying, using as dispersed phases of four carbide powders; they were: boron carbide, chromium carbide, silicon carbide and zirconium carbide. The purpose of this work was to study some characteristics of these compounds and their feasibility for use as materials for electrical equipment. Raw materials used were copper powder finer than 150 mesh, and 2-4 vol % of powder carbides finer than 325 mesh. The alloying process was carried out using both a Spex 800-D mill with tungsten jars and balls and a hardened stainless steel jar and balls, the purpose of which was to measure the degree of contamination induced by the milling media. The changes in particle size, morphology and microstructure of copper alloys were studied with SEM and TEM for each alloyed powder. The degree of saturation of copper by the carbides was measured by X-ray diffraction. Sound samples were obtained by hot isostatic pressing at 1073 degrees K. The best characteristics were obtained for the compound copper-chromium carbide, based on their hardness, tensile strength and electrical conductivity values. The other alloys failed due to their excessive brittleness after hot pressing.

**[35] STUDY-ON MECHANICAL ALLOYING AND SUBSEQUENT HEAT TREATMENT OF THE TI-SI SYSTEM**

Yang JY. Wu JS. Hua W. - Physica B. 279(4):241-245, 2000
Elemental powder mixtures of $Ti_xSi_{100-x}$ were mechanically alloyed by using a planetary ball mill. XRD, SEM, TEM and DTA were used to characterize their mechanical alloying process. After milling for 45 h, $Ti_{75}Si_{25}$, $Ti_{62.5}Si_{37.5}$ and $Ti_{50}Si_{50}$ powders were amorphized completely; however, no obvious amorphization was observed for $Ti_{33.3}Si_{66.7}$ and $Ti_{25}Si_{75}$ powders. By using Miedema model, a thermodynamics analysis was performed. Annealing the as-milled powders in vacuum at 860 degrees C for 1 h, the as-milled $Ti_{62.5}Si_{37.5}$ powders transformed into $Ti_5Si_3$ intermetallics, and $Ti_{33.3}Si_{66.7}$ transformed into $TiSi_2$ intermetallics completely.

**[34] THERMOELECTRIC PROPERTIES OF THE P-TYPE BI2TE3-SB2TE3-SB2SE3 ALLOYS FABRICATED BY MECHANICAL ALLOYING AND HOT PRESSING**
Kim HC. Oh TS. Hyun DB. - Journal of Physics & Chemistry of Solids. 61(5):743-749, 2000
The p-type (100 - x)% Bi2Te3-x% Sb2Te3 (75 less than or equal to x less than or equal to 85) and 20% Bi2Te3-(80 - y)% Sb2Te3-gamma% Sb2Se3 (0 less than or equal to gamma less than or equal to 7) alloys were fabricated by mechanical alloying and hot pressing, and their thermoelectric properties were characterized. Among the hot-pressed (100 - x)% Bi2Te3-x% Sb2Te3 alloys, the 20% Bi2Te3-80% Sb2Te3 alloy exhibited a maximum figure-of-merit of 3.05 x 10(-3)/K. The Seebeck coefficient and the electrical resistivity of the hot-pressed 20% Bi2Te3-(80 - gamma) Sb2Te3-gamma% Sb2Se3 alloys increased with increasing the Sb2Se3 content due to the reduction of the hole concentration. The figure-of-merit of the hot-pressed 20% Bi2Te3-(80 - gamma)% Sb2Te3-gamma% Sb2Se3 alloys decreased from 3.05 x 10(-3)/K to 2.5 x 10(-3)/K with increasing the Sb2Se3 content up to 7 mol%, because the increment of the electrical resistivity was much larger than the decrement of the thermal conductivity and the increment of the Seebeck coefficient.

**[33] EVIDENCE OF FERRIMAGNETIC ORDERING IN FEMNO3 PRODUCED BY MECHANICAL ALLOYING**
Seifu D. Kebede A. Oliver FW. Hoffman E. Hammond E. Wynter C. Aning A. Takacs L. Siu IL. Walker JC. Tessema G. Seehra MS. - Journal of Magnetism & Magnetic Materials. 212(1-2):178-182, 2000
Mechanical alloying of alpha-Fe2O3 with Mn2O3 is shown to produce FeMnO3, whose X-ray diffraction pattern fits the cubic structure (space group Ia3. lattice constant, approximate to 9.40 Angstrom) identical to that of Mn2O3. Tempcrilture variation of its magnetic susceptibility chi shows a phase transition near T-c approximate to 40 K and the chi(-1) versus T data for T > T-c fit the variation expected for a ferrimagnet with a magnetic moment of 2.8 mu(B) per formula unit. The Mossbauer spectrum for T > T, is a doublet with quadrupole splitting approximate to 1 mm/s and isomer shift approximate to 0.4 mm/s, changing to a sextet for T < T-c with a hyperfine field of 445 kOe at 16 K.

**[32] MAGNETIC PROPERTIES AND MICROSTRUCTURE OF MECHANICALLY MILLED SM-2(CO,M)(17)-BASED POWDERS WITH M=ZR, HF, NB, V,** Ti, Cr, Cu and Fe
Chen ZM. Meng-Burany X. Okumura H. Hadjipanayis GC. - Journal of Applied Physics. 87(7):3409-3414, 2000
The structure, microstructure, and magnetic properties of nanostructured Sm2Co17-based powders synthesized by mechanically milling and subsequent annealing have been systematically studied. It has been found that a nanoscale 2:17 phase with an average grain size of about 30 nm is developed within the powders, which have an average particle size of about 5 mu m. Optimum magnetic properties of M-s = 110.5 emu/g, M-r = 66.2 emu/g, M-r/M-s = 0.60, H-c = 9.6 kOe, and (BH)(m) = 10.8 MGOe have been obtained in stoichiometric Sm2Co17 powders milled for 6 h and annealed at 800 degrees C for 30 min. The observed magnetic hardening is believed to arise from the high anisotropy of the Sm2Co17 phase and its nanoscale grain size. A small amount of Zr substitution for Co significantly increases the coercivity by increasing the anisotropy field of the Sm2Co17 phase. Cu substitution in Zr-contained samples further increases the coercivity by introducing a nanoscale 1:5 phase which forms a uniform mixture with the 2:17 nanograins. The highest coercivity of 20.6 kOe has been obtained in the Sm-12(Co0.92Cu0.06Zr0.02)(88) powders. Fe substitution enters the Co lattice sites of the 2:17 structure, leading to an increase of the magnetization but a decrease of the coercivity. An optimum maximum energy product of 14.0 MGOe is obtained in the Sm-12(Co0.7Fe0.3)(88) powders.

**[30] ENHANCEMENT OF CRITICAL TEMPERATURE IN FINE LA0.7CA0.3MNO3 PARTICLES PREPARED BY MECHANOCHEMICAL PROCESSING**
Muroi M. Street R. McCormick PG. - Journal of Applied Physics. 87(7):3424-3431, 2000
Fine La0.7Ca0.3MnO3 powders have been synthesized by mechanochemical processing, involving high-energy ball milling of LaCl3, CaCl2, MnCl2, and Na2CO3 and subsequent heat treatment. Single-phase La0.7Ca0.3MnO3 particles, dispersed in a NaCl matrix, were obtained for heat-treatment temperatures (T-ht) of 750 degrees C or above, with the crystallite size increasing with T-ht, from about 20 nm at 750 degrees C to about 1 mu m at 1200 degrees C. Magnetic measurements showed that as the crystallite size decreases, the saturation magnetization at low temperature decreases from the theoretical value for full polarization of Mn spins, 3.7 mu(B) per Mn ion, while T-c increases from 250 up to 278 K for the crystallite size of similar to 20 nm; and that there exists a narrow temperature range near T-c in which the magnetization for a larger crystallite size is smaller in low fields but becomes larger in high fields than for a smaller crystallite size. These observations are discussed in terms of the ferromagnetic transition unique to perovskite manganites, involving the formation and growth of magnetic polarons above T-c.

**[29] THE INFLUENCE OF BALL-MILLING ON THE STRUCTURE OF GRAPHITE [CHINESE]**
Yang HS. Wu GT. Zhang XB. Chen XH. Lu XN. Wang M. Wang CS. He PM. Xu ZD. Li WZ. - Acta Physica Sinica. 49(3):522-526, 2000
After 150 h of ball milling, the structure of the pristine well-crystalline graphite is damaged and a great many of defects are induced. The ball milling produces a mixture consisting of high curved carbon sheets, carbon nanoarches, onions and tripods. The characters of above graphite structures and their possible mechanism of structural transformation are also discussed.

**[28] MILLING EFFECTS UPON QUANTITATIVE DETERMINATIONS OF CHRYSOTILE ASBESTOS BY THE REFERENCE INTENSITY RATIO METHOD**
De Stefano L. De Luca F. Buccolieri G. Plescia P. - Powder Diffraction. 15(1):26-29, 2000
As is well known from literature, the grinding process, which is an unavoidable step in sample preparation, may strongly modify the physical properties of chrysotile through amorphisation. The aim of this work is to establish the proper milling time to apply to the samples before all accurate X-ray powder diffraction quantitative analysis. We have used the RIR (reference intensity ratio) analytical method, based on the measurement of the ratio I/I-s between the intensity of the strongest line of an analyte and the intensity of the analytical peak of a standard material, when they are thoroughly mixed 50:50 by weight. We have studied how the RIR value changes as a function of the milling time of the sample and how the accuracy of this: quantitative method is affected.

**[27] EFFECTS OF ADDITIONAL ELEMENTS ON THE STRUCTURE AND MAGNETIC PROPERTIES OF ND2FE14B/ALPHA-FE-TYPE NANOCOMPOSITE MAGNETS**
Cui BZ. Sun XK. Liu W. Zhang ZD. Geng DY. Zhao XG. - Journal of Physics D-Applied Physics. 33(4):338-344, 2000
The effects of a partial substitution of Mo, Ga, Si or Al, respectively, for Fe on the structure and magnetic properties of Nd-8.4 Fe-87.1 BJ(4.5) alloy prepared by mechanical alloying have been studied in detail. The addition of 1.1 at% Mo, Si or Ga results in the improvement of the intrinsic coercivity of the nanocomposite magnets. In contrast, the substitution of 1.1 at% Al is unfavourable for improving the magnetic properties. The optimum average grain size d(op) of the alpha-Fe is different for various element substitutions. It is found that in addition to the grain size of alpha-Fe. the grain size of Nd2Fe14B is another important factor in determining the magnetic properties of nanocomposite magnets. The main effects of these substitutions on the magnetic properties of the nanocomposite magnets originate Rom modifications of the grain sizes of the soft magnetic phase and the hard magnetic phase. The best magnetic properties: mu(0)H(c) = 0.4 T (4 kOe), J(r) = 1.07 T (10.7 kGs), (BH)(max) = 93.2 kJ m(-3) (11.7 MGOe), J(r)/J(s) = 0.68, are achieved for the Nd8.4Fe86Ga1.1B4.5 alloy.
**[26] A NEW CLASS OF ULTRA-HARD MATERIALS BASED ON ALMGB14**
Cook BA. Harringa JL. Lewis TL. Russell AM. - Scripta Materialia. 42(6):597-602, 2000
**[25] KINETIC, CHEMICAL AND MECHANICAL FACTORS AFFECTING MECHANICAL ALLOYING OF NI-BCC TRANSITION METAL MIXTURES**
Streletskii AN. Courtney TH. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 282(1-2):213-222, 2000
The influence of enthalpy of mixing and elemental mechanical characteristics on the kinetics of mechanical alloying (MA) of 80 at.% Ni-20 at.% bcc transition metals (Fe, Cr, W, Nb, and Ta) has been studied. The features and structures of milled powders were characterized by X-ray diffraction, optical microscopy, and particle size analysis. Powder caking of the grinding media in varying amounts depending On the materials alloyed and the alloying time - occurs when these elemental combinations are mechanically alloyed. Differences, if any, between the structures of 'free' and 'caked' powders were also determined. As expected, for systems with low enthalpies of mixing (Ni/Fe, Ni/Cr, Ni/W) crystalline solid solutions form during MA. Moreover, the compositions of these solid solutions are the same in both free and caked powders. Also as anticipated, for systems with high enthalpies of mixing (Ni/Nb, Ni/Ta), extended MA produces amorphous phases that form from precursor solid solutions. In distinction to systems with low enthalpies of mixing, the structures of the free and caked powders differ for systems with high mixing enthalpies. Caked powders were inhomogeneous, consisting of powder in varying degrees of solid solution and, if the milling time was sufficiently long, also some amorphous powder. However, the free powder was almost entirely noncrystalline With extended milling, the fraction of free powder increases suggesting that the formation of the amorphous phase takes place on the surface layer of the coated grinding media. Subsequent to its formation, the amorphous phase is abraded from the coated surfaces. The kinetics of solid solution and/or amorphous phase formation is discussed in terms of the differing mechanical characteristics of the bcc transition elements, as well as the system thermodynamics.
**[24] MICROSTRUCTURE AND TENSILE PROPERTIES OF MECHANICALLY ALLOYED TI-6AL-4V WITH BORON ADDITIONS**
Godfrey TMT. Wisbey A. Goodwin PS. Bagnall K. Ward-Close CM. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 282(1-2):240-250, 2000
Particulate titanium metal matrix composites (MMCs) offer advantages for a wide range of structural applications with increases in stiffness and possibly wear resistance. A powder metallurgy/mechanical alloy route was used successfully to give a fine, uniform distribution of boron in titanium alloy powder. This powder was hot isostatically pressed and during the process TiB reinforcement was formed by an in-situ reaction. Significant tensile ductility in MA titanium ahoy (without boron), equivalent to wrought material was achieved, along with good tensile strength. In materials with high TiB volume fractions it is thought that the fine boride size and high strength matrix caused embrittlement. This suggests that the use of a lower strength matrix, such as commercial purity titanium, and shorter milling times would provide a better balance of strength, stiffness and ductility for this type of composite.
**[23] SYNTHESIS OF SIC-AIN POWDER AND CHARACTERIZATION OF ITS HIP-SINTERED COMPACTS**
Li JF. Sugimori M. Kobayashi Y. Watanabe R. - Nippon Seramikkusu Kyokai Gakujutsu Ronbunshi-Journal of the Ceramic Society of Japan. 108(3):265-270, 2000
SiC-AlN powder was synthesized from a powder mixture of Si, Al and C through direct reaction between the constituent elements, i.e., (l-x)Si+(l-x)C+xAl+(x/2)N-2 = (Sic)(1-x)(AlN)(x). A green compact of the mixed elemental powders was fired in nitrogen, and a porous compact composed of SiC and AlN and their solid solutions was obtained. This porous compact could be easily pulverized to fine SiC-AlN powder by ball-milling. Ultrafine microstructure with fine grains smaller than 100 nm was obtained by hot-isostatic pressing (HIP) of the synthesized powders without external additives. Most grains therein were SiC-AlN solid solutions with compositions close to the nominal mixed ratio. The obtained SiC-AlN ceramic alloys, here referred to as homogeneous solid solutions and their mixture, showed better mechanical properties than SiC and AlN. In particular, high strength, more than twice that of the conventional monolithic SiC, was obtained in the SiC-50 mol%AlN composition mainly because of its ultrafine-grained microstructure. This was also the main reason for the present SiC-AlN ceramic alloys to exhibit ductile deformation at relatively low temperatures.
**[22] MECHANOCHEMICALLY INDUCED FORMATION OF LA2SIO5**
Tzvetkov G. Minkova N. - Journal of Materials Science. 35(10):2435-2441, 2000
Mechanochemical technique was applied to prepare La2SiO5 under conditions where the conventional solid-state synthesis shows unsatisfactory results. The effects of the mechanochemical treatment of a mixture of lanthana and silica gel (in molar ratio La2O3/SiO2 = 4:3) has been studied by X-ray diffractometry (XRD), infrared spectroscopy (IR) and using a scanning electron microscope (SEM). Differential thermal analysis (DTA) and thermogravimetry (TG) have been used to follow the thermal behaviour of initial and milled samples. It was found that the amorphous

silicate precursor of La-4.67(SiO4)(3)O is formed as a result of a mechanochemical solid-state reaction. The crystallization of the latter silicate occurs at 880 degrees C during the subsequent heat treatment of the milled samples. The formation of La2SiO5 without any XRD-detectable traces of La-4.67(SiO4)(3)O takes place after heating at 1100 degrees C for 2 h. The rate of conversion increases with increasing the milling time, reaching 96% after mechanochemical treatment for 3 h and subsequent heating at 1100 degrees C.

**[21] ROOM TEMPERATURE STABILISATION OF GAMMA-BI2VO5.5 AND SYNTHESIS OF THE NEW FLUORITE PHASE F-BI2VO5 BY A MECHANOCHEMICAL ACTIVATION METHOD**

Castro A.  Millan P.  Ricote J.  Pardo L. - Journal of Materials Chemistry. 10(3):767-771, 2000

Mechanochemical activation followed by annealing at moderate temperatures results in the stabilisation at room temperature of the high ionic conductor oxide gamma-Bi2VO5.5 belonging to the Bi-V-V-O-2 system, while in the Bi-V-IV-O-2 system a non-previously reported fluorite-type f-Bi2VO5 phase is isolated. All alpha-, beta- and gamma-Bi2VO5.5 and f-, alpha- and beta-Bi2VO5, as well as amorphous powders with 2Bi(2)O(3):V2O5 and Bi2O3:VO2 compositions, are studied by X-ray powder diffraction at ambient and high temperatures, thermal analysis and scanning electron microscopy.

**[20] MODELLING AND CONTROL OF A JET MILL PLANT**

Gommeren HJC.  Heitzmann DA.  Moolenaar JAC.  Scarlett B. - Powder Technology. 108(2-3):147-154, 2000

Although jet milling is a very energy consuming grinding process it is increasingly used in industry because very fine grinding product with a narrow size distribution is attained without contamination as the milling occurs by inter particle collisions. At Delft University of Technology a project has been started to achieve a considerable energy reduction in jet milling processes. The grinding plant consists of a spiral jet mill in closed loop with an external classifier. Main feature of the system will be an operation control based on in-line particle size measurements using laser diffraction. In industrial practice the operating conditions are often determined by trial and error. To avoid off-spec material the mill is often tuned to lower risk. This results in a relatively large amount of overground material. With respect to the control strategy, the first step was real time particle size monitoring to explore the operating ranges of the jet mill. The controlability is studied in relation to several process inputs and process configurations. A dynamic model of the closed loop grinding plant is developed. particle transport and size reduction inside the mill show a stochastic behaviour and are described by size and state dependent probability functions. Separate experiments are carried out to derive equations for the breakage kinetics of different materials. Numerical flaw simulations are carried out to provide statistic data about the frequency and intensity of collisions between particles in relation to state conditions in the mill. A glass bottom plate will be placed on the mill to observe flow patterns. The influence of several process input variables on the dynamics of the grinding plant and the final product are simulated. Pilot plant experiments are carried out to verify and optimize the dynamic model by direct measurement of the PSD under actual system conditions. The ultimate objective of the dynamic model will be the implementation in a control system. The required setpoints of the mill/classifier system are predicted to obtain the desired product quality at minimum energy use. Furthermore the model can be used for scale up and plant design.

**[19] THE ROLE OF DISPERSED PARTICLES IN STRENGTHENING AND FRACTURE MECHANISMS IN A MO-ZRC ALLOY PROCESSED BY MECHANICAL ALLOYING**

Takida T.  Mabuchi M.  Nakamura M.  Igarashi T.  Doi Y.  Nagae T. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 31(3):715-721, 2000

The tensile properties of a ZrC particle-dispersed Mo, which was processed by spark plasma sintering with mechanically alloyed powder, were investigated at room temperature and at elevated temperatures of 1170 to 1970 K. The Mo-ZrC alloy showed much higher strength at room temperature than a fully recrystallized pure Mo. The high strength of Mo-ZrC is mainly attributed to a very small grain size (about 3 mu m). The main role of the ZrC particle is not to increase strength due to the particle-dislocation interaction, but to limit grain growth during sintering and to attain the very small grain size. The elongation at room temperature of Mo-ZrC was much lower than that of pure Mo. This is probably related to the higher interstitial contents. However, Mo-ZrC showed a large elongation of 180 pet at 1970 K and 6.7 x 10(-4) s(-1). It was suggested that the ZrC particles stabilized the fine-grained microstructure yet provided no cavitation sites at 1970 K; as a result, the large elongation was attained.

**[18] PHASE TRANSITIONS IN REACTIVE FORMATION OF TI5SI3/TIAL IN SITU COMPOSITES**

Du YJ.  Rao KP.  Chung JCY.  Han XD. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 31(3):763-771, 2000

A new method is developed for preparing Ti5Si3/TiAl in situ composites by incorporating metastable phases (called metastable precursors) into TiAl (a mixture of elemental Ti and Al) matrix powders. Metastable precursors with a starting composition of Ti-14Al-21Si are prepared by mechanical alloying (MA). They have been proven through X-ray diffraction (XRD) analysis and transmission electron microscope (TEM) observations to be mainly consisting of mixtures of nanostructured solid solutions and milling-formed TiAl compound. Particularly, phase reactions and transitions in the precursors and the composites during heating have been investigated in detail by using diffraction thermal analysis (DTA) in conjunction with XRD. It has been found that Ti5Si3 is in situ formed through a phase transition chain, TiSi2 --> Ti5Si4 --> Ti5Si3. When the composite powder (precursor, Ti and Al) is heated, a combustion reaction first occurs in the matrix, which results in the formation of TiAl3 and/or TiAl followed by the completion of the previously mentioned silicide transitions in a very short time. Scanning electron microscope (SEM) observations indicated the locations of reinforcements in the reaction-formed composite, and TEM observation provided some details of the structures for the reinforcements and their neighborhood. This method is intriguing because a designed phase hierarchy is possible.

**[17] THE EFFECT OF TUNGSTEN ADDITION ON THE MAGNETIC PROPERTIES AND MICROSTRUCTURE OF SMFEN-ALPHA-FE NANOCOMPOSITES**

Kaszuwara W.  Leonowicz M.  Kozubowski JA. - Materials Letters. 42(6):383-386, 2000

The effect of tungsten addition, in the range of 0-17 at.%, on the magnetic properties and microstructure of SmFeN-alpha-Fe permanent magnet nanocomposites is presented. The magnets were prepared by mechanical alloying of elementary powders of Sm, Fe and W, followed by annealing and nitriding. The addition of tungsten leads to initial increase and further decrease of the coercivity with a maximum value of 250 kA/m for 7 at.% W. The remanence, however, continuously decreases from 1.2 T for 0% W down to 0.7 T for 17 at.% W. The maximum value of (BH)(max) (120 J/m(3)), was obtained for 2 at.% W. The measurements of Curie temperature vs. W content showed a constant and decreasing values for alpha-Fe and the Sm2Fe17 phase, respectively. This behaviour can be explained by the dissolution of some W atoms in the Sm2Fe17 phase. X-ray diffraction analysis indicated, beside the alpha-Fe and Sm2Fe17 phases, the existence of pure W in the microstructure. Addition of W leads to reduction of the a-Fe crystallite size (20 nm for 0% W, 18 nm for 7% W and 8 nm for 17% W). The high resolution electron microscopy investigations show randomly distributed small (below 20 nm) crystallites of tungsten. Thus for a small amount of W the remanence is still substantially enhanced. The increase of the coercivity, for W contents up to about 7 at.%, can possibly be attributed to increased magnetic anisotropy of the Sm-2(Fe,W)(17)N-3 phase.

**[16] WET MILLING OF FE/AL/AL2O3 AND FE2O3/AL/AL2O3 POWDER MIXTURES**

Schicker S. Garcia DE. Gorlov I. Janssen R. Claussen N. - Journal of the American Ceramic Society. 82(10):2607-2612, 1999

Wet milling of Al2O3-aluminide alloy (3A) precursor powders in acetone has been investigated by milling Fe/Al/Al2O3 and Fe2O3/Al/Al2O3 powder mixtures. The influence of the milling process on the physical and chemical properties of the milled powders has been studied. Particle refinement and homogenization were found not to play a dominant role, whereas plastic deformation of the metal particles leads to the formation of dislocations and a highly disarranged polycrystalline structure. Although no chemical reactions among the powder components in Fe2O3/Al/Al2O3 powder mixtures were observed, the formation of a nanocrystalline, ordered intermetallic FeAl phase in Fe/Al/Al2O3 powder mixtures caused by mechanical alloying was detected. Chemical reactions of Fe and Al particle surfaces with the atmosphere and the milling media lead to the formation of highly porous hydroxides on the particle surfaces. Hence the specific surface area of the powders increases, while the powder density decreases during milling. The fraction of Fe oxidized during milling was determined to be 0.13. The fraction of Al oxidized during milling strongly depends on the metal content of the powder mixture. It ranges between 0.4 and 0.8.

**[15] LOW-TEMPERATURE SINTERING AND MICROWAVE DIELECTRIC PROPERTIES OF ZINC METATITANATE-RUTILE MIXTURES USING BORON**

Kim HT. Kim SH. Nahm S. Byun JD. Kim Y. - Journal of the American Ceramic Society. 82(11):3043-3048, 1999

Mixtures of zinc metatitanate and rutile (ZnTiO3 + xTiO(2), where x = 0-0.5) have been prepared via the conventional mixed-oxide method. Centrifugal planetary milling with zirconia beads 1 mm in diameter produced very fine powders (mean particle size of 0.2 mu m), which allowed the synthesis of ZnTiO3 and sintering at temperatures <945 degrees C, which is the decomposition temperature of ZnTiO3. Sintering of the mixtures was enhanced further by the addition of B2O3. Densities of >94% of the theoretical density have been attained for the specimens that were sintered at 875 degrees C for 4 h with B2O3 additions of <1 wt%. Microwave dielectric properties of the aforementioned compositions were as follows: dielectric constant of 29-31, normalized quality factor of 56000-69000 GHz, and a temperature coefficient of resonance frequency between -10 and +10 ppm/degrees C, Sintering was enhanced by the formation of a ZnO-B2O3 liquid phase, which affected the microwave properties, because of variation in the phase composition.

**[14] EFFECT OF CARBIDES ON THE MICROSTRUCTURE AND PROPERTIES OF TI(C, N)-BASED CERAMICS**

Authors Park DS. Lee YD. Kang SH. - Journal of the American Ceramic Society. 82(11):3150-3154, 199

Ti(C,N)-based ceramics that contained various carbides were prepared via pressureless sintering, to study the effects of the carbides on the microstructure and properties of the ceramics. All the systems showed good densification behavior and formed solid solutions. Impurities introduced from the milling media were sufficient to promote the densification of the ceramics. The grain structures of the systems were a function of carbide crystal structure and their affinity with nitrogen. Microhardness, three-point flexural strength, and fracture toughness of the Ti(C,N)-based ceramics were measured and determined to be 16-18 GPa, 0.7-1.1 GPa, and 3.5-5.5 MPa.m(1/2), respectively.

**[13] INFLUENCE OF MAGNESIA ADDITION ON THE RHEOLOGICAL PROPERTIES OF MULLITE SUSPENSIONS**

Galassi C. Roncari E. Bassarello C. Lapasin R. - Journal of the American Ceramic Society. 82(12):3453-3458, 1999

Water-based suspensions of mullite that are used to form porous bodies through the impregnation of sponges were investigated. The rheological properties of the suspensions are strongly influenced by the amount of magnesia added, as well as the solid loading and the preparation procedure. Amounts of magnesia higher than 0.15 wt% change both the steady shear behavior from nearly Newtonian to plastic (with an increasing apparent yield stress) and time dependent, as well as the viscoelastic properties (the storage modulus increases more than 5 orders of magnitude). Similar effects are shown for long milling procedures, or solids loadings higher than 25 vol%, while the elastic properties are not strictly related to the total volume fraction. The results of the rheological characterization, under either continuous or dynamic conditions, are discussed in relation to the properties of the structure built up within the suspension. The density of the cast bodies increases with decreasing magnesia content.

**[12] LOW-FIRED (ZN,MG)TIO3 MICROWAVE DIELECTRICS**

Kim HT. Nahm S. Byun JD. Kim Y. - Journal of the American Ceramic Society. 82(12):3476-3480, 1999

A dielectric ceramic comprised of (Zn1-xMgx)TiO3 (x = 0 to x = 0.5) with low sintering temperature and promising microwave properties was prepared by applying a semichemical synthesis route and a microbeads milling technique. X-ray diffractometry and thermal analyses results indicated that the phase stability region of the hexagonal (Zn,Mg)TiO3 extended to higher temperatures as the amount of magnesium increased. The dielectric properties in this system exhibited a significant dependence on the sintering conditions, especially near the phase decomposition temperature. From 950 degrees C, the temperature compensation characteristics occurred as the phase composition changed from hexagonal (Zn,Mg)TiO3 to two phases: (Zn,Mg)(2)TiO4 and rutile. The magnesium content for zero temperature coefficient (tau(f)) was similar to 3 mol% at 950 degrees C; however, tau(f) increased with the sintering temperatures because of the shift of the decomposition temperature.

**[11] ACTIVATION-INDUCED PYROCHLORE-TO-PEROVSKITE CONVERSION FOR A LEAD MAGNESIUM NIOBATE PRECURSOR**

Xue JM. Wang J. Ng WB. Wan DM. - Journal of the American Ceramic Society. 82(8):2282-2284, 1999

One or more pyrochlore phases are often involved as transitional phases prior to the formation of the perovskite phase in lead magnesium niobate (PMN) precursors. The elimination of these pyrochlore phases requires careful control of the calcination temperature and therefore is often difficult to complete. We report on the pyrochlore-to-perovskite conversion triggered by mechanical activation in a freeze-dried PMN precursor. The resulting PMN powder exhibits a well-established crystalline perovskite structure which is stable for calcination temperatures ranging from 400 degrees to 800 degrees C. A refined particle size in the range of 30-50 nm was obtained.

**[10] AMBIENT-TEMPERATURE MECHANOCHEMICAL FORMATION OF TITANIUM NITRIDE-ALUMINA COMPOSITES FROM TIO2 AND FETIO3**

Welham NJ. Kerr T. Willis PE. - Journal of the American Ceramic Society. 82(9):2332-2336, 1999

The fabrication of a homogeneous submicrometer-sized powder composed of nanocrystalline (<10 nm) alumina and titanium nitride during high-energy ball-milling is reported in this paper. The starting materials were rutile (TiO2) and aluminum powder. A similar composite with iron was also produced using the mineral ilmenite (FeTiO3) as the starting material. The powders were ball-milled together under a nitrogen atmosphere for 100 h in a laboratory-scale mill and subjected to thermal analysis and isothermal annealing at up to 1200 degrees C. X-ray diffraction shouted that all of the phases formed within the milling step and underwent grain growth on annealing. Differential thermal analysis indicated no residual elemental aluminum, confirming that the reaction was completed during the milling operation.

**[9] MECHANOCHEMICAL FORMATION OF METAL-CERAMIC COMPOSITES**

Welham NJ. Willis PE. Kerr T. - Journal of the American Ceramic Society. 83(1):33-40, 2000

A mechanical activation technique has been used to form composites of alumina with titanium carbide, nitride, or carbonitride, both with and without elemental iron. The composites were formed by reacting elemental aluminum with either ilmenite (FeTiO3) or rutile (TiO2) concentrates in the presence of carbon and/or nitrogen in a ball-mill at ambient temperature. The reaction was complete for the ilmenite samples after milling but was completed only for rutile under hot pressing conditions. Microhardness measurements indicated that the composites had hardnesses in the range 19-30 GPa (1740-2750 VHN), with only a small variation within each sample. Elemental mapping of the pressed pellets indicated that titanium and aluminum were evenly distributed on a submicrometer level whereas iron tended to coalesce into <20 mu m particles in the presence of TiC. The coalescence decreased with the carbon content of the hard material until iron was evenly distributed with TiN. A superstoichiometric amount of aluminum led to the formation of iron-aluminum phases which decreased the iron coalescence. The XRD crystallite size of the alumina was 30-50 nm and was 25-50 mm for the titanium phases, confirming the extremely fine microstructure.

**[8] MECHANOCHEMICAL SYNTHESIS OF 0.9[9.6PB(ZN1/3NB2/3)O-3 CENTER DOT O.4PB(MG1/3NB2/3)O-3]CENTER DOT 0.1PBTIO(3)**

Wan DM. Xue JM. Wang J. - Journal of the American Ceramic Society. 83(1):53-59, 2000

Lead zinc niobate-lead magnesium niobate-lead titanate (PZN-PMN-PT) ceramic powders of perovskite structure have been prepared via a mechanochemical processing route. A single-phase perovskite powder of ultrafine particles in the nanometer range,vas successfully synthesized when a MZN powder (columbite precursor) was mechanically activated for 10 h together with mixed lead and titanium oxides. The following steps are involved when the ternary oxide mixture is subjected to an increasing degree of mechanical activation. First, the starting materials are significantly refined in particle size as a result of the continuous deformation, fragmentation and then partially amorphized at the initial stage of mechanical activation. This is followed by the formation of perovskite nuclei and subsequent growth of these nuclei in the activated oxide matrix with increasing activation time. When calcined at various temperatures in the range of 500-800 degrees C, pyrochlore phase was not detected by XRD phase analysis in the mechanochemically synthesized powder. Only a minor amount (similar to 2%) of pyrochlore phase was observed when the calcination temperature was raised to 850 degrees C. The PZN-PMN-PT derived from the mechanochemically synthesized powder can be sintered to similar to 98% relative density at a sintering temperature of 950 degrees C, The PZN-PMN-PT sintered at 1100 degrees C for 1 h exhibits a dielectric constant of similar to 18 600 and a dielectric loss of 0.015 at the Curie temperature of 112 degrees C when measured at a frequency of 0.1 kHz, together with a d(33) value of 323 x 10(-12) pC/N.

**[7] NANOSIZED BARIUM TITANATE POWDER BY MECHANICAL ACTIVATION**

Xue JM. Wang J. Wan DM. - Journal of the American Ceramic Society. 83(1):232-234, 2000

Mechanical activation, without any additional heat treatment, is used to trigger the formation of a perovskite BaTiO3 phase in an oxide matrix that consists of BaO and TiO2 in a nitrogen atmosphere. The resulting BaTiO3 powder exhibits a well-established nanocrystalline structure, as indicated by phase analysis using X-ray diffractometry. A crystallite size of similar to 14 nm is calculated, based on the half-width of the BaTiO3 (110) peak, using the Scherrer equation, and an average particle size of 20-30 mm is observed using transmission electron microscopy for the activation derived BaTiO3 powder.

**[6] PREPARATION OF NANOSIZED HEMATITE PARTICLES BY MECHANICAL ACTIVATION OF GOETHITE SAMPLES**

Subrt J. Perez-Maqueda LA. Criado JM. Real C. Bohacek J. Vecernikova E. - Journal of the American Ceramic Society. 83(2):294-298, 2000

Nanosized single crystals of hematite with a very narrow particle size distribution were prepared by mechanical activation of two different goethite samples. Both goethite samples transformed completely into hematite after 70 h grinding time. Transmission electron microscopy showed that the final particles were spherical in shape and of similar to 17 nm average particle size. This particle size was coincident with that estimated from specific surface measurements, indicating that the hematite samples consisted of nonporous and nonaggregated particles. The crystallite size, calculated from the broadening of the XRD peaks, in the hematite samples indicated that particles consisted of single crystals, No influence of the precursor was observed in the products, so both goethite samples yielded identical rounded single crystals with a narrow particle size distribution.

**[5] THE REACTION-BONDED ALUMINUM OXIDE PROCESS: I, THE EFFECT OF ATTRITION MILLING ON THE SOLID-STATE OXIDATION OF ALUMINUM POWDER**

Suvaci E. Simkovich G. Messing GL. - Journal of the American Ceramic Society. 83(2):299-305, 2000

The effect of attrition milling on the solid-state oxidation of aluminum powder is important for the reaction-bonded aluminum oxide process. Attrition milling increased the surface area to 14.4 and 20.2 m(2)/g versus 1.2 m(2)/g for unmilled powder and smeared the Al particles, and the surface was hydrolyzed to form bayerite and boehmite, Upon heating the hydroxides decompose to form an 11-13 nm thick amorphous plus gamma-Al2O3 layer which subsequently retards oxidation kinetics. The oxidation per unit area decreases for the higher surface area powders at temperatures below the critical temperature but the total oxidation of the milled powder is similar to 70% versus similar to 9% for the as-received powder because of the higher surface: area. The critical temperature depends on Al particle surface characteristics and is defined as the transition temperature above which the initial rate of oxidation is linear, not parabolic. Above the critical temperature the oxidation per unit area decreases significantly. In addition, linear oxidation occurs faster than parabolic oxidation and thus the initial fast oxidation kinetics (i.e., linear) can cause thermal runaway during oxidation, Therefore, oxidation below the critical temperature is essential to maximize solid-state oxidation and to prevent thermal runaway. The critical temperatures for the as-received (1.24 m(2)/g), the 6 h (14.4 m(2)/g), and 8 h (20.2 m(2)/g) attrition-milled Al powders were 500 degrees, 475 degrees, and 500 degrees C, respectively. A model for oxidation during the parabolic and linear oxidation stages is presented.

**[4] HRTEM AND EELS STUDIES ON THE AMORPHIZATION OF HEXAGONAL BORON NITRIDE INDUCED BY BALL MILLING**

Huang JY. Yasuda H. Mori H. - Journal of the American Ceramic Society. 83(2):403-409, 2000

We present a new approach, namely ball milling, to synthesize amorphous boron nitride (a-BN), The amorphization kinetics are revealed by X-ray diffractometry (XRD), high-resolution transmission electron microscopy (HRTEM), and electron energy loss spectroscopy (EELS), HRTEM investigations indicate that, in the early stage of milling, the thick sp(2) layers are sliced into many thinner sheets because of cleavage;along the basal planes, In the intermediate stage of milling, deformation is accommodated primarily by simultaneous shearing along the basal planes. As a result of sustained shearing, a number of defects, such as stacking faults, (0002) [11 (2) over bar 0] twinning, simultaneous shearing of lattice planes, and half Frank loops with Burgers vectors of 1/2[0001], are introduced in the hexagonal BN (h-BN) grains. Simultaneous shearing also causes significant change in the lattice symmetry of most grains. In the final stage of milling, the fiberlike tightly bonded sp2 sheets are broken and refined further, until a nanocrystalline and amorphous mixture is formed. XRD of the sample mined for 180 b exhibits an amorphous halo pattern; nevertheless, HRTEM demonstrates that the end product is essentially a nanocrystalline and amorphous mixture. The grain sizes of the nanocrystals are <3 nm, and their stacking is turbostratic. EELS investigations of the a-BN indicate that bonding is :primarily sp2, but a small fraction of sp(3) a-BN is also found, which is assumed to be the nuclei of the cubic phase (c-BN) in the high-pressure and high-temperature experiments and thus facilitates the hexagonal to cubic transition, The present a-BN fabrication method can provide an effective way to facilitate the synthesis of sintered bulk c-BN materials.

**[3] MECHANOCHEMICAL SYNTHESIS OF LANTHANUM ALUMINATE BY GRINDING LANTHANUM OXIDE WITH TRANSITION ALUMINA**

Zhang Q. Saito F. - Journal of the American Ceramic Society. 83(2):439-441, 2000

Grinding lanthanum oxide (La2O3) with Al2O3 was conducted to investigate their mechanochemical reactions to form lanthanum aluminate (LaAlO3) powder using a planetary ball mill. Grinding for 120 min allowed us to obtain single-phase LaAlO3, with a large surface area when transition alumina was used, whereas no formation of LaAlO3 was achieved when alpha-Al2O3 was used. The mechanochemical process can be applied to synthesize other rare-earth (RE) aluminates (REAlO3) from mixtures of a rare-earth oxide and transition alumina.

**[2] INFLUENCE OF MECHANICAL AND THERMAL TREATMENTS ON RAW MATERIALS CONTAINING PYROPHYLLITE**

Soto PJS. De Haro MCJ. Cosp JP. Pichardo MR. Rodriguez JLP. - Boletin de la Sociedad Espanola de Ceramica y Vidrio. 39(1):119-134, 2000

In the present work results obtained in a study on the influence of thermal, mechanical by dry grinding, and their combination, in raw materials containing pyrophyllite, are discussed. First of all,it is studied the influence of thermal treatment concerning the development and evolution of crystalline phases (mullite and cristobalite) from dehydroxylated pyrophyllite. On the basis of these results, it is analyzed what happens in a natural raw mixture of pyrophyllite with kaolinite and mica (sericite) submitted to thermal treatments. The raw pyrophyllite materials are altered under laboratory conditions using mechanical treatments by dry grinding. It is noted that the increase of surface area and particle size reduction is produced by grinding, but other effects are produced on the structure and properties of the solid submitted to grinding. In general, grinding leads to a progressive destruction of the original crystal structure of the present layered silicates, but it is,preferentially produced along the "c" axis. In other words, mechanochemical reactions are induced by dry grinding due to the increase of reactivity of the system. Between these reactions, it is enhanced the reagglomeration process that occurs above a determinate limit of grinding time. The grinding treatment can be combined with a subsequent thermal treatment that enhances the increase of reactivity, producing the formation of crystalline phases (mullite and cristobalite) at lower temperatures that in unground samples with energy saving. The results are compared taking into account the crystal structures of both kaolinite and pyrophyllite, the thermal transformation of kaolinite to mullite, and the process of grinding kaolinite because this layer silicate is present in the raw materials containing pyrophyllite.

**[1] ENHANCED DISSOLUTION FOLLOWING EXTENDED MILLING**

Fletcher NH. Welham NJ. - AICHE Journal. 46(3):666-669, 2000

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