

RESEAU FRANÇAIS DE MECANOSYNTHESE

Lettre N° 64

Juillet 2000

180 (+2) Groupes de Recherche
(dont 107 (+2) à l'étranger / 36 Pays)

Bureau :

International Editorial LRFM Committee

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J. Temuujon - Dpt Metallurgy and Ceramics Science - Tokyo - Japon
K. Uenishi - Dpt of Manufacturing Science - Osaka University - Japon

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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 à jour régulièrement
concernant les derniers résultats dans ce domaine.

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LAST CALL FOR PAPERS LAST CALL FOR PAPERS LAST CALL FOR PAPERS

**International Symposium on
Metastable Mechanically Alloyed and Nanocrystalline Materials**

St Catherines College, Oxford

9-14th July 2000

For the most up-to-date information please see

http://www.materials.ox.ac.uk/ocamac/ismanam/ismanam2000_news.htm

The International Symposium on Metastable, Mechanically Alloyed and Nanocrystalline Materials 2000 (ISMANAM-2000) will take place in Oxford (UK) during the week of 9-14th July 2000. The symposium will be based entirely within one of the Oxford colleges, St Catherines College. The symposium will cover amorphous, nanocrystalline, nanocomposite, mechanically alloyed, quasicrystalline and other metastable materials, providing a comprehensive overview of recent research into: synthesis and processing; structural and chemical characterisation; magnetic, electrical, mechanical and other properties; and applications in components and devices. Intended as a forum for novel ideas, the symposium should promote contact between basic research efforts and technological needs for industrial applications.

The sessions will include invited and keynote lectures from internationally distinguished scientists. Oral and poster research contributions are welcomed. Members of the Materials Science, Physics, Chemistry and Engineering communities are encouraged to participate and to contribute their work.

ISMANAM2000

International Symposium on Metastable Mechanically Alloyed and Nanocrystalline Materials

St Catherines College, Oxford

9-14th July 2000

Email: ismanam2000@materials.ox.ac.uk

Web: <http://www.materials.ox.ac.uk/Conferences/ISMANAM2000.html>

Technical Announcement

<p>Press release</p> <p>NEW – First presentation on the AICHEMA 2000</p> <p>VARIO-PLANETARY MILL "pulverisette 4"</p> <p>The "pulverisette 4" vario-planetary mill is capable of emulating ball mills of conventional design, precisely simulating the types of stress entailed and thus reproducing or optimising grinding processes. Due to the high flexibility available for selecting the grinding parameters, it is possible to achieve results unattainable with any other ball mills.</p> <p>This is the ideal mill for mechanical activation and alloying. The main applications are in the field of materials research and, of course, wherever a powerful, innovative planetary mill is required.</p> <p>When particles < 10 mm are fed in, a final fineness up to 0.1 µm can be achieved. The useful capacity is between 2 x 30 ml in the case of 80 ml grinding bowls and 2 x 125 ml when 250 ml grinding bowl are used.</p> <p>Method of operation:</p> <p>With standard planetary ball mills the grinding bowls are rotating and mounted eccentrically on a rotating support disc. The rotational speed of the supporting disc can be selected at will; the grinding bowl rotates at a fixed transmission ratio.</p> <p>Due to the overlapping of grinding bowls and supporting disc, the material to be ground and the grinding balls execute movements and trajectories in the grinding bowl, which are defined by the transmission ratio.</p> <p>With the "pulverisette 4" vario-planetary mill the rotational speeds of grinding bowls and supporting disc can be adjusted completely independently of each other. By varying the transmission ratio it is possible to control the movements and trajectories of the grinding balls at will so that the balls strike the inner wall of the bowl vertically (high impact energy), approach each other tangentially (high friction) or just roll down the inner wall of the bowl (centrifugal mills).</p>	<p>All intermediate levels and combinations of frictional and impact pressures can be set as required. By changing the transmission ratio it is therefore possible for the first time to carry out mechanical activation as well as mechanical alloying.</p> <p>Furthermore, it is also possible for the first time to optimally adjust a planetary ball mill to the material to be ground, the size of the grinding bowls and the grinding balls.</p> <p>Features of performance:</p> <ul style="list-style-type: none">• for the first time, all grinding parameters can be selected at will for optimal preparation of sample• Programming of the grinding parameters by PC software as desired• RS232 interface for programming and to transfer grinding parameters to the PC• Real-time display of the speeds to monitor the grinding process• Reversing option (direction of rotation reversed periodically) to improve the grinding results• Emulation of various ball mills• Variably adjustable pressure on sample (friction and/or impact)• Final fineness << 1 µm• Simultaneous grinding in up to 4 small or 2 large grinding bowls• Quick, secure fastening of the grinding bowls• Ease of cleaning <p>contact:</p> <p>Fritsch GmbH Manufacturers of Laboratory Instruments Andrea Köhler Industriestrasse 8 D-55743 Idar-Oberstein Phone: ++49/ 67 84/70-46 Fax: ++49/ 67 84/70-11 E-Mail: info@fritsch.de Internet: http://www.fritsch.de</p>
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**ANNONCE DE CONGRES ET / OU ECOLES
CONGRESS AND SCHOOL ANNOUNCEMENTS**

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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>

NCM8

**8th International Conference on the Structure of
Non - Crystalline Solid**

6 - 11 Aout 2000

Website : <http://www.sgt.org>

XIVth International Symposium on the Reactivity of Solids

Budapest, Hungary through 27-31 August 2000

<http://www.jate.u-szeged.hu/~isrs14>.

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>

Inorganic Materials Conference

University of California Santa Barbara, USA

13 - 16 September 2000 <http://www.elsevier.com/locate/im2000>

Congrès de la Société Française de Chimie

Univ. Rennes I

18 - 22 Septembre 2000

website : <http://www.sfc.fr>

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

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>

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

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

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

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>

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

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

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

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

PM2 TEC2001
2001 International Conference on Powder Metallurgy
& Particulate Materials
13 - 17 May 2001 - New Orleans - USA
Contact : MPIF

7th International Symposium on
Agglomeration
29, 30, 31 May 2001
Albi - France
Website : <http://www.univ-inpt.fr/~agglom>
or <http://www.enstimac.fr>

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.),

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

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)

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 Cherbourg M. A. ROUSSET Professeur, Université de Toulouse M BEAUGER Alain Ingénieur de Recherche et Développement TPC Saint Apollinaire M. 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 Apollinaire M. 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

H. SOUHA

Thèse de Doctorat d'Etat Marocaine

Faculté des Sciences Dahr El Mehras 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.)

O. Held

"Etude des réactions de combustions solide-solide ou solide liquide auto-entretenues pour différents

Elaboration de ces mêmes intermétalliques par broyage mécanique et étude de leur cinétique de cristallisation

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

D. Cracco

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

CNRS - Thiais - France - 25 Juin 1999

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

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

Cooperative Research on Related Areas

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.

**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

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. Placement is preferred prior to August with priority given to a qualified candidate with earlier availability.

Interested candidates should send correspondence 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

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

Bibliographie Récente

Livres ou "Special Issues"

(9/06/2000)

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

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

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

Preface

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

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

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

Contents

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Solution NiTiH by Reactive Ball Milling - References 157 - Mechanically Induced Solid-State Reduction -
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Mechanism of MSSR - Fabrication of Nanocrystalline WC and Nanocomposite WC-MgO Refractory Materials by
MSSR and Methods - References 189 - Mechanically Induced Solid-State Amorphi
Mechanical Solid State Amorphization of Fe₅₀W₅₀ Binary System - Special Systems and Applications - Amorphous
Austenitic Stainless Steel 254 - Fabrication of amorphous Fe₅₂Nb₄₈ Special Steel 257 - Fe-Zr-B 259 - Difference
between Mechanical Alloying and Mechanical Disordering in the Amorphization Reaction of Al₅₀Ta₅₀ in a Rod
Mill - Mechanically Induced Cyclic Crystalline-Amorphous Transformations During Mechanical Alloying -
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(05/05/2000)

EXTRACTIVE METALLURGY OF ACTIVATED MINERALS

included in series Process Metallurgy, 10

by P. Balaz - Institute of Geotechnics, Slovak Academy of Sciences

ISBN : 0 - 444 - 50206 - 8 / Price USD 144, Euro 124.79)

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

Description

Mechanical activation of solids is a part mechanochemistry, the science with a sound theoretical foundation exhibiting a wide range of potential application. Mechanical activation itself is an innovative procedure where an improvement in technological processes can be attained via a combination of new surface area and defects formation in minerals.

Mechanical activation is of exceptional importance in extractive metallurgy and mineral processing and this area forms the topic of this book and is a result of more than twenty years of research and graduate teaching in the field.

In pyrometallurgy, the mechanical activation of minerals makes it possible to reduce their decomposition temperatures or causes such a degree of disordering that the thermal activation may be omitted entirely. The potential mitigation of environmental pollutants is becoming increasingly important in this context.

The lowering of reaction temperatures, the increase of the rate and amount of solubility, preparation of water soluble compounds, the necessity for simpler and less expensive reactors and shorter reaction times are some of the advantages of mechanical activation in hydrometallurgy. The environmental aspects of these processes are particularly attractive.

Several industrial processes are examined and the flowsheets are presented as successful of activation. In these processes, the introduction of a mechanical activation step into the technological cycle significantly modifies the subsequent steps.

The book is designed for researchers, teachers, operators and students in the areas of extractive metallurgy, mineral processing, mineralogy, solid state chemistry and materials science. It will encourage newcomers to the mechanochemistry to do useful research and discover novel applications in this field.

(3/02/2000)

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

1. MECHANICAL ALLOYING - FUNDAMENTALS AND APPLICATIONS

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

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

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

Energy transfer and energy maps;

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

Modelling mechanical alloying (mechanistic models, deformation, coalescence and fragmentation, evolution of particle size, milling time, powder heating, powder cooling, atomistic model, thermodynamic and kinetic model)

Joining of mechanically alloyed materials; Rapid solidification and mechanical alloying; Applications (nickel-based superalloys, Al-based materials, supersaturated solutions, magnetic materials, mechanically alloyed powders for spray coatings, superplasticity, tribological materials, composites, amorphous solids, nanocrystalline materials, solid-state chemical reactions, etc). ISBN 1898326568, 160 pages 234 **156 mm, cased**, £45.00, 1999

DISPERSION STRENGTHENED ALUMINIUM PREPARED BY MECHANICAL ALLOYING, by M Besterici

<http://www.demon.co.uk/cambsci/book51.htm> 1. Characteristics of dispersion-strengthened systems 2. Mechanical alloying (kinetics and mechanism of preparation of the Al-C system by mechanical alloying; compaction of powders and heat treatment of compacts;

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

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

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

"Mechanical Alloying : Fundamentals and Applications"

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

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

"Non Equilibrium Processing of Materials"

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

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

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

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 world and these materials have now gained a position of great importance in basic science and engineering materials technology. Bulk amorphous alloys based upon the Zr - Al - Ni - Cu, Zr (Ti,Nb) - Al - Ni - Cu and Zr - Ti - Ni - Cu - Be systems have already achieved wide commercial success as components of various technical accessories ranging from sporting goods to optical instruments.

Here is a state of the art reviews on this new group of materials, covering all areas of interest, ranging from the synthesis of these special alloys and their fundamental properties, to their engineering characteristics and applications.

This work will therefore be of equal interest to those who wish to become fully acquainted with the subject, and to those who are already actively engaged in the field.

DISPERSION-STRENGTHENED ALUMINIUM PREPARED BY MECHANICAL ALLOYING

Michal 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-Al₄C₃ material)
4. Static and dynamic mechanical properties (mechanical properties at elevated temperatures; mechanical properties at 20°C; effect of interface on the mechanical properties; superplastic properties of the system; thermal stability of the system; creep characteristics; creep-fatigue characteristics)

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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

"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/>)

"Nanomatériaux"

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

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

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

Au début des années 90, les japonais ont été les premiers à 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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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.

Mechanochemistry of Materials
Cambridge International Science Publishing

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

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

BIBLIOGRAPHY ON MECHANICAL ALLOYING AND MILLING

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

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

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

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

PERIODIQUES

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

[62] TECHNICAL NOTE - SYNTHESIS OF HIGH DENSITY ULTRAFINE W/CU COMPOSITE ALLOY BY MECHANO-THERMOCHEMICAL PROCESS

Lee GG. Ha GH. Kim BK. - Powder Metallurgy. 43(1):79-82, 2000

It is difficult to obtain a high density W/Cu composite without the addition of a sintering activator as there is no mutual solubility but a great difference in melting temperature and specific gravity. Ultrafine starting powders are needed in order to achieve the desired full density W/Cu composite. Recently, the authors have developed a new manufacturing process to achieve the full density ultrafine W/Cu composite alloy by combining a thermochemical and a mechanical process (mechano-thermochemical process). W/Cu oxide and W/Cu metal composite powders, which are loosely agglomerated homogeneous clusters of nanoscale sized particles, can be synthesised by a thermochemical process and then these powders are treated again by a mechanical process to obtain ultrafine powders. In the case of the mechano-thermochemical process, a sintered W/Cu composite alloy, obtained from the W/Cu oxide composite powder passing through a multistep reduction process, shows a higher density than that produced from the W/Cu metal composite powder. A full density W+20wt-%Cu composite alloy with an average W particle size of 1 μ m can be obtained without the addition of a sintering activator through a combination of the mechano-thermochemical process and liquid phase sintering.

[61] FABRICATION OF Mg₂NiHx FROM MG AND NI CHIPS BY HYDROGEN INDUCED PLANETARY BALL MILLING

Hong TW. Kim SK. Park GS. Kim YJ. - Materials Transactions Jim. 41(3):393-398, 2000

For manufacturing Mg₂NiHx hydrogen absorbing materials economically, Mg and Ni chips were mechanically alloyed under 2 MPa hydrogen atmosphere by planetary ball milling. The homogeneous Mg₂NiHx was formed after 72 h mechanical alloying (MA) in both 30:1 and 66:1 BCR (ball to chips mass ratio). The maximum absorbed hydrogen content of the Mg₂NiHx reached 3.9 mass% after 48 h MA in 30:1 BCR and the dehydrogenation reaction of them started around 473 similar to 503 K from the result of TG analysis. On the other hand, in 66:1 BCR, the fully hydrogenated alloys were obtained after 96 h MA, the total hydrogen content was 2.25 mass% and the dehydrogenation temperature was around 453 similar to 493 K. The composite phase obtained after 96 h MA in 66:1 BCR is composed of the nanocrystalline and amorphous phases, the structure of which seems to be the reason of the improved hydriding and dehydriding kinetics. It was deduced that the conditions of BCR affected strongly the synthesis of the Mg₂NiHx.

[60] MICROSTRUCTURES AND MECHANICAL PROPERTIES OF Nb/Nb-SILICIDE IN-SITU COMPOSITES SYNTHESIZED BY REACTIVE HOT PRESSING OF BALL MILLED POWDERS

Ma CL. Kasama A. Tanaka H. Tan Y. Tanaka R. Mashima Y. Hanada S. - Materials Transactions Jim. 41(3):444-451, 2000

To develop advanced structural materials at very high temperatures, ball milling followed by reactive hot pressing was employed to fabricate binary Nb/Nb-silicide in-situ composites with 3.5 to 16 mol% silicon content. Changes in morphology during milling of niobium and silicon elemental powders were studied, and microstructures and mechanical properties of reactively hot pressed composites were evaluated by OM, SEM, XRD, DTA and EPMA. No detectable niobium silicide was formed during milling. Reactive hot pressing of the milled powders yields two kinds of silicides, Nb₃Si and Nb₅Si₃, through in-situ reaction between heavily deformed niobium particles and fragmented silicon particles. DTA revealed that silicide formation occurs at 940 K for Nb₃Si and 1050 K for Nb₅Si₃. Fully dense Nb/Nb-silicide in-situ composites with equiaxed grain microstructures are produced by the reactive hot pressing of milled powders. All of the composites exhibit considerable compressive ductility without cracking even at room temperature. Annealing at high temperature results in growth of equiaxed grains of niobium and silicide, thereby increasing high temperature strength.

[59] PREPARATION OF FUNCTIONALLY GRADED MATERIALS BY PULSE CURRENT PRESSURE SINTERING OF BALL MILLED AL-50 AT% TI POWDER

Nagae T. Saji S. Yanagimoto T. Nose M. Yokota M. - Materials Transactions Jim. 41(3):457-460, 2000

A functionally graded material (FGM) laminated with five layers composing of Al, Ti and Al₃Ti phases was prepared from an equimolar mixture of Al and Ti powders by combination of ball milling and pulse current pressure sintering (PCPS) processes. The characteristics of the FGM are as follows. (1) The volume fraction of Al₃Ti phase in the laminated layers increases with increasing milling period of Al-50 at%Ti mixed powder. This results from the accelerated formation of Al₃Ti in the PCPS process due to refinement of Al and Ti composite particles during milling. (2) The average value of Vickers hardness for each laminated layer of the FGM increases with increasing content of Al₃Ti phase in the layer. The average hardness values of the five layers are 28, 165, 258, 425 and 520 HV, respectively.

[58] MECHANO-CHEMICAL MODIFICATION OF POLYSTYRENE AND POLY(METHYL METHACRYLATE)

Frendel A. Drache M. Janke G. Schmidt-Naake G. - Chemie Ingenieur Technik. 72(4):391-396, 2000

[57] THERMAL AND MORPHOLOGICAL STUDIES OF BINARY AND TERNARY COMPOSITES OF POLY(VINYLALCOHOL) WITH ALUMINA AND ZIRCONIA

Mishra R. Rao KJ. - Ceramics International. 26(4):371-378, 2000

Composites of PVA with alumina (PVA-Al₂O₃), zirconia (PVA-ZrO₂), and both alumina-zirconia (PVA-Al₂O₃-ZrO₂) were prepared by ball milling the dispersed solid powder of Al₂O₃, ZrO₂ and Al₂O₃-ZrO₂ in a PVA matrix at room temperature. The composites were cast into films. The films were later decomposed and the residual ceramics were sintered at 1400 degrees C. The surfaces of the composites have been examined using scanning electron microscopy at various stages. The nature of decomposition of PVA as influenced by ceramic particle surfaces has also been studied. There is clear evidence that controllable morphologies and microscopic porosities can be achieved in

these composites.

[56] THERMAL STABILITY OF NANOCRYSTALLINE NI SILICIDES SYNTHESIZED BY MECHANICAL ALLOYING

Datta MK. Pabi SK. Murty BS. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 284(1-2):219-225, 2000

Solid state reactions induced by mechanical alloying followed by isothermal annealing in elemental blends of Ni and Si have been studied over the entire composition range of the Ni-Si system. Above a critical crystallite size, the congruent melting phases, γ -Ni₃₁Si₁₂ and NiSi, become unstable and react with second phase available to form equilibrium non-congruent, β (1)-Ni₃Si and α -NiSi₂, after isothermal annealing at 723 and 1023 K, respectively. Formation of equilibrium ϵ -Ni₃Si₂ has not been observed in the composition range 33-50at.% Si even after annealing at 1073 K. The results also indicate that the crystallite size of non-congruent melting phases is well above 100 nm even at the stage of their evolution, suggesting that they are probably stable only in the bulk state. The grain growth is faster for the non-congruent phases when compared to that of congruent melting compounds. The interfacial energy of the compounds plays an important role in controlling their stability and grain growth in the nanocrystalline state.

[55] ENHANCEMENT OF SELF-SUSTAINING REACTION CU₃SI PHASE FORMATION STARTING FROM MECHANICALLY ACTIVATED POWDERS

Bernard F. Souha H. Gaffet E. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 284(1-2):301-306, 2000

Mechanical high-energy ball milling of an 3Cu + Si elemental powders mixture was used to activate a self-sustaining combustion reaction or so-called self-sustaining high-temperature synthesis (SHS) to form the copper silicide phase, a reaction for which the thermodynamic criterion proposed by Munir for self-propagation reaction is not favorable. A complete characterization of the end-products was performed with X-ray diffraction analysis and scanning electron microscopy. Thermal and structural information describing the combustion front initiated by heating up a sample to 180 degrees C in a Cu/Si system is communicated. This paper clearly shows that the mechanically activated self-sustaining high-temperature synthesis process produces a pure Cu₃Si compound in spite of the limitation imposed by the thermodynamic criterion. In addition, it stems that the reactivity of Cu₃Si elaborated from the mechanically activated SHS process towards CuCl is greater than the reactivity of Cu₃Si reference powder. This difference is mainly due to the crystallite size.

[54] TEMPERATURE-INDUCED MORPHOLOGICAL EVOLUTION IN POLYMER BLENDS PRODUCED BY CRYOGENIC MECHANICAL ALLOYING

Smith AP. Spontak RJ. Koch CC. Smith SD. Ade H. - Macromolecular Materials & Engineering. 274(1):1-12, 2000

Novel nanoscale morphologies are achieved in blends of poly(methyl methacrylate) (PRIMA) and either polyisoprene (PI) or poly(ethylene-alt-propylene) (PEP) produced by high-energy mechanical alloying at cryogenic temperatures. Since the blends are prepared as solid powders, they must be post-processed in the melt to be of practical relevance. In this study, we employ scanning transmission X-ray microscopy (STXM), transmission electron microscopy (TEM) and light microscopy (LM) to investigate the stability of nanostructural features and the dynamics of phase coarsening at elevated temperatures (above the glass transition temperature of milled PMMA). Substantial coarsening is observed in PEP/PMMA blends at relatively short times, indicating that the two polymers are highly immiscible and mobile at the conditions examined. The nanostructure evident in PV PMMA blends is, however, more robust and undergoes less evolution due to milling-induced PI crosslinking. Instead of forming discrete dispersions in a PMMA matrix, the PI molecules organize into a fine network morphology upon annealing in the melt.

[53] SELECTIVE NO REDUCTION BY PROPANE OVER COMMERCIAL OXIDE CATALYSTS AND THEIR MECHANICAL MIXTURE: I. SYNERGISTIC EFFECT

Tret'yakov VF. Burdeinaya TN. Matyshak VA. Ukharskii AA. Mokrushin OS. Glebov LS. - Kinetics & Catalysis. 41(2):236-241, 2000

In the reaction of NO reduction by propane in excess oxygen, the activity of a binary mixture of commercial oxide catalysts is higher than should be expected if it were proportional to the activities of separate catalyst components. This synergism reveals itself in the fact that the conversion of NO and C₃H₈ is higher when the catalyst is a mechanical mixture than the sum of conversions over separate catalysts, all other conditions being the same. Based on the kinetic and thermal desorption measurements, the experimental conditions are found under which the synergism is observed. A hypothetical mechanism is proposed.

[52] MECHANICALLY DRIVEN PHASE TRANSITION OF FULLERENE

Liu ZG. Ohi H. Masuyama K. Tsuchiya K. Umemoto M. -Journal of Physics & Chemistry of Solids. 61(7):1119-1122, 2000

Structural evolution that takes place during mechanical milling of fullerene (C-60(C-70)) was investigated. Mechanical milling did not destroy the molecular structure of C-60(C-70), but the fee crystalline structure with long-range periodicity. Longer milling time results in the formation of C-60(C-70) polymer, including C₆₀ dimer. Thermal analysis revealed a depolymerization enthalpy close to the theoretical calculation of the (2 + 2) cycloaddition bonding energy of polymerization. Mechanical milling may be one effective means to produce C-60(C-70) polymer.

[51] RAMAN SPECTRUM OF BALL-MILLED ALPHA-TiCl₃

Miyaoka H. Kuze T. Sano H. Mori H. Mizutani G. Ushioda S. Otsuka N. Terano M. -Journal of Luminescence. 87-9:709-711, 2000

We have obtained the Raman spectrum of ball-milled alpha-TiCl₃. We found six bands in the Raman spectrum. Five of them were assigned to the same bands as those observed in crystalline alpha-TiCl₃. A new band appeared at 224 cm⁻¹ for ball-milled alpha-TiCl₃, probably due to the breakdown of the crystal symmetry.

[50] MECHANOCHEMICAL SYNTHESIS OF UREA ADDUCTS WITH LONG CHAIN ALKYL DERIVATIVES

Fernandez-Bertran J. Alfonso LM. Alvarez JC. Reguera E. - Journal of Inclusion Phenomena & Molecular

Recognition in Chemistry. 37(1-4):131-136, 2000

The adducts of urea and solid alkyl derivatives can be obtained mechanochemically in good yields. The products synthesized by grinding the solid reagents have identical IR spectra and XRD powder patterns to those obtained by crystallization from ethanolic solutions.

[49] EFFECT OF OXYGEN AND TITANIUM CONTENTS ON THE STABILITY OF NANOCRYSTALLINE TI-RU-Fe-O CATHODE MATERIALS FOR CHLORATE ELECTROLYSIS

Roue L. Bonneau ME. Guay D. Blouin M. Schulz R. - Journal of Applied Electrochemistry. 30(4):491-498, 2000
Electrodes made from nanocrystalline Ti:Ru:Fe (2 - y:1 + y/2:1 + y/2), with y varying from 0 to 1 by step of 0.25, and Ti:Ru:Fe:O (2:1:1:w), with w varying from 0 to 2 by step of 0.5, were prepared and tested as activated cathodes for the hydrogen evolution reaction in typical chlorate electrolysis conditions. These electrodes were subjected to an accelerated aging test, consisting of a succession of cycles of hydrogen discharge (HER) and open-circuit (OCP) conditions. In addition to monitoring the cathodic overpotential value during the aging test, visual inspection and mass loss measurements were performed on the electrodes at the end of the test to assess their stability. In the case of Ti:Ru:Fe (2:1:1), a large increase of the cathodic overpotential value is observed after 20 cycles. Adding O to the formulation causes a remarkable improvement of the long-term stability of the electrodes. As little as [O] = 10 at.% in nanocrystalline Ti:Ru:Fe:O (2:1:1:w) materials is sufficient for the electrode to show absolutely no sign of degradation after 50 cycles of HER/OCP, the longest accelerated test conducted. Adding more O to the formulation of the material does not lead to further stability improvement. A better stability under the conditions of the accelerated aging test can also be observed for nanocrystalline Ti:Ru:Fe (2 - y:1 + y/2:1 + y/2) materials with y > 0. In that case however, the level of improvement is dependent on the value of y. The best results are obtained for y = 0.75. A hypothesis is proposed to explain the improved stability obtained by lowering the Ti content and/or by adding O. The similarity and difference between both ways of improving the stability of the nanocrystalline Ti:Ru:Fe materials are also discussed.

[48] A MORE CONSISTENT EXPLANATION OF THE STRENGTH OF AL2O3/SiC NANOCOMPOSITE AFTER GRINDING AND ANNEALING

C. Anya - Ceramics International. 26(4):427-434, 2000

In a recent paper it was proposed that the strengthening of 5 vol% SiC Al₂O₃-SiC nanocomposite after grinding and annealing is attributable to flaw healing, in parallel with an oxidation reaction, which lead to a reduction in the density and size of flaws on annealing. Other authors have also favoured the flaw healing mechanism. However, the possibility that a mechanism based on dislocation activities might be mostly responsible has never been fully investigated. In this report the two mechanisms are investigated and it is demonstrated that the latter is more consistent with experimental observations. Possible sources of the ambiguity met in some of the explanations of the mechanisms behind the strengthening phenomenon are also identified.

[47] LOW-ENERGY BALL-MILLING: TRANSFORMATIONS OF BORON NITRIDE POWDERS. CRYSTALLOGRAPHIC AND CHEMICAL CHARACTERIZATIONS.

Gasgnier M. Szwarc H. Ronez A. - Journal of Materials Science. 35(12): 3003-3009, 2000.

BN powders (with and without water) which have undergone low-energy ball-milling have been studied through X-ray diffraction, transmission electron microscopy, granulometry, electron energy loss (EELS) and IR spectroscopies. The main result is the formation of the orthorhombic and cubic high pressure phases of BN. A "turbostratic" behaviour of hexagonal BN has also been observed. As expected, the grain sizes decrease markedly. It is to be noticed that the presence of water induces some chemical reactions which are revealed through ammoniac and nitride compounds scents. However, according to EELS measurements, the main transformations are structural ones.

[46] THERMOELECTRIC PROPERTIES OF THE HOT-PRESSED (BI,SB)₂(TE,SE)₃ ALLOYS

Oh TS. Hyun DB. Kolomoets NV. - Scripta Materialia. 42(9):849-854, 2000

[45] FORMATION/CONSOLIDATION OF WC-CO CERMETS BY SIMPLE SHEAR

Parasiris A. Hartwig KT. Srinivasan MN. - Scripta Materialia. 42(9):875-880, 2000

[44] INDENTATION CREEP OF NANOCRYSTALLINE CU-TiC ALLOYS PREPARED BY MECHANICAL ALLOYING

Shen BL. Itoi T. Yamasaki T. Ogino Y. - Scripta Materialia. 42(9):893-898, 2000

[43] INFLUENCE OF MECHANICAL TREATMENT OF ASPIRIN ON THE KINETICS OF ITS DISSOLUTION IN WATER

Poluboyarov VA. Korotaeva ZA. Kiselevich SN. Pankrat'ev YD. Sysoev VF. Andryushkova OV. - Russian Journal of Physical Chemistry. 73(7):1094-1099, 1999

The characteristic constant for the dependence of the excess saturation concentration of aspirin in aqueous solution (saturated for 20 min) on the time of mechanical treatment of aspirin is consistent with the rate of accumulation of energy by aspirin, which is determined by the duration of mechanical treatment. Measurements of pH value show that the constants of dissociation for mechanically treated aspirin with the formation of monomers in an aqueous solution are three to ten times higher than those for the initial aspirin. This suggests that a different conformation of the aspirin monomer is produced by mechanical treatment. C-13 NMR measurements show that aspirin molecules form self-associates. Saturated solutions of mechanically activated aspirin contain self-associates that differ from those typical of saturated solutions of initial aspirin. Mechanical energy is accumulated in solid aspirin at a time-dependent rate. The process of energy relaxation begins after a certain number of intermolecular bonds are broken with the formation of new intermolecular bonds.

[42] THE ALPHA ->BETA POLYTYPIC TRANSFORMATION IN SiC INDUCED BY BALL MILLING

Yang XY. Wu YK. Ye HQ. - Philosophical Magazine Letters. 80(5):333-339, 2000

The alpha --> beta polytypic transformation in SiC was observed during ball milling at room temperature. Microstructural evolution was characterized on an atomic scale by high-resolution electron microscopy (HREM). HREM observations showed that partial dislocations play an important role in the transformation under the complicated conditions during ball milling.

[41] HYDROGEN STORAGE PROPERTIES OF NANO-COMPOSITES OF Mg AND Zr-Ni-Cr ALLOYS

Yang J. Ciureanu M. Roberge R. - *Materials Letters*. 43(5-6):234-239, 2000

Mg and Zr-Ni-Cr alloy nano-composite hydrogen storage materials have been prepared by high energy mechanical milling (MM) of Mg powders with either crystalline ZrNiCr and ZrNi_{1.6}Cr_{0.4} powders or mechanically milled amorphous ZrNiCr and ZrNi_{1.6}Cr_{0.4} powders. Nano-composites of amorphous Zr-Ni-Cr alloy and Mg have better desorption kinetics compared to crystalline Zr-Ni-Cr alloy and Mg nano-composites. Amorphous ZrNi_{1.6}Cr_{0.4} and Mg nano-composites desorb larger amount of H₂ much faster than amorphous ZrNiCr and Mg nano-composites, The nano-composite of 35 wt.% amorphous ZrNi_{1.6}Cr_{0.4} and Mg releases 4.3 wt.% H₂ at 300 degrees C in 30 min. X-ray diffraction revealed that there are no reactions between Mg and Zr-Ni-Cr alloys in the milling, activation, and subsequent cycling processes, proving that amorphous ZrNi_{1.6}Cr_{0.4} is an effective hydrogen absorption and desorption catalyst.

[40] PREPARATION OF IRON-NICKEL CATALYSTS BY MECHANICAL ALLOYING

Zeifert BH. Salmones J. Hernandez JA. Reynoso R. Nava N. Cabanas-Moreno JG. Aguilar-Rios G. - *Materials Letters*. 43(5-6):244-248, 2000

Raney type catalysts were prepared by means of a two-step procedure: (i) mechanical alloying (MA) of the metals and (ii) alkaline aluminum leaching. MA is a novel alternative related to the synthesis of skeletal Ni catalysts. Catalyst characterization was performed by atomic absorption (AA), X-ray diffraction (XRD), and Mossbauer spectroscopy (MS). Textural studies were also carried out. Binary Al-Ni and ternary Al-Ni-Fe alloys were produced by MA from pure metallic powders; in particular, the intermetallic beta-(AlNi) phase was formed with a fine microstructure as a non-equilibrium phase; then, aluminum was selectively removed. After aluminum leaching, the beta-(AlNi) phase was transformed into the more stable nickel fee structure.

[39] HYSTERESIS AND RELAXATION OF HARD-SOFT NANOCOMPOSITE SAMPLES

Gonzalez JM. Montero MI. Crespo P. Marin P. Hernando A. - *Journal of Applied Physics*. 87(9 Part 2):4759-4761, 2000

We present experimental results on the hysteresis and relaxation of Fe/Ba hexaferrite composite samples prepared by ball milling starting from precursor Fe and Ba hexaferrite particles. Our results show that, for maximum applied fields of up to 8 kOe, the measured loops show a shift of up to 85 Oe. The compositional dependence of that loop displacement presents two maxima (at approximately $x=0.3$ and $x=0.7$) and a minimum at $x=0.5$. These results are discussed in terms of the dipolar coupling between both phases.

[38] EFFECTS OF THE SUBSTITUTION OF CU OR NB FOR FE ON THE STRUCTURE AND MAGNETIC PROPERTIES OF Nd8Fe84Ti8 ALLOYS PREPARED BY MECHANICAL ALLOYING

Cui BZ. Sun XK. Liu W. Geng DY. Zhao XG. Zhang ZD. - *Journal of Applied Physics*. 87(9 Part 2):5287-5289, 2000

The effects of a partial substitution of Cu or Nb for Fe on the structure and magnetic properties of mechanically alloyed Nd₈Fe₈₄Ti₈ alloys were studied. Compared with Nd₈Fe₈₄Ti₈ alloys prepared under the same conditions, the temperature necessary for the formation of the Nd(Fe, Ti)(12) phase decreases from 900 to 750 degrees C with the addition of 3 at. % Cu. The addition of Cu hinders the nitrogenation of the Nd(Fe, Ti)(12) phase. The pretreatment of hydrogenation on the Cu-added alloys at 300 degrees C facilitates the formation of nitrides. With increasing annealing temperature, the intrinsic coercivity of the nitrides increases until it attains a maximum at 850 degrees C. The addition of Nb is harmful to the formation of the perfect Nd(Fe, Ti)(12) phase. For the 3 at. % Nb-containing alloys, annealing at 960 degrees C makes a metastable phase Nd(Fe, Ti)(7) be formed. The addition of Nb is unfavorable for the intrinsic coercivity and the maximum magnetic energy product of the nitrides.

[37] SM-FE-C NANOCOMPOSITE MAGNETS PREPARED FROM POWDERS OF SM, FE, AND GRAPHITE BY MECHANICAL ALLOYING

Geng DY. Zhang ZD. Cui BZ. Zhao XG. Liu W. Guo ZJ. - *Journal of Applied Physics*. 87(9 Part 2):5296-5298, 2000

The effects of further addition, of carbon on structure and magnetic properties of the Sm₂₀Fe₇₀C₁₀ alloy synthesized by mechanical alloying have been investigated. The main phase is Sm₂Fe₁₇C_x or Sm₂Fe₁₄C, depending on the composition of starting materials. The Sm₂Fe₁₄C phase tends to form when more carbon is added. Remilling and reannealing at a low temperature (600 degrees C) improve the magnetic properties of the alloys. The ratio of remanence to saturation magnetization as high as 0.79 indicates the existence of enhanced exchange coupling between the hard Sm₂Fe₁₇C_x and soft Sm₂Fe₁₄C/alpha-Fe magnetic phases in the Sm₂₀Fe₇₀C₁₅ alloy.

[36] COERCIVITY IN MECHANICALLY MILLED PR-CO-ZR POWDERS WITH TBCU7 STRUCTURE

Chen ZM. Meng-Burany X. Hadjipanayis GC. - *Journal of Applied Physics*. 87(9 Part 2):5302-5304, 2000

The structural, microstructural, and magnetic properties of the PrCo_{7-x}Zr_x ($x=0, 0.2, 0.3, \text{ and } 0.4$) powders produced by mechanical milling have been studied with the aim of developing fine microstructure and high coercivity in these powders. X-ray diffraction, transmission electron microscope, and magnetic measurements show that a single phase with TbCu₇-type structure and with high anisotropy of up to 100 kOe is obtained in as-cast PrCo_{6.7}Zr_{0.3} alloy. Magnetic hardening is developed in the mechanically milled powders. An optimum coercivity of 5.3 kOe has been obtained in PrCo_{6.7}Zr_{0.3} powders milled for 2 h and annealed at 800 degrees C for 1 min, which shows a uniform microstructure of the 1:7 phase with an average grain size of about 10-15 nm. The observed magnetic hardening is believed to arise from the high anisotropy field of the Pr(Co, Zr)(7) phase and the uniform nanoscale microstructure developed by mechanical milling and subsequent annealing.

[35] STRUCTURE AND MAGNETIC PROPERTIES OF N-CONTAINING ND-FE-B ALLOYS PREPARED BY MECHANICAL ALLOYING

Liu W. Zhang ZD. Liu JP. Sun XK. Sellmyer DJ. - *Journal of Applied Physics*. 87(9 Part 2):5332-5334, 2000

The structure and magnetic properties of Nd₂Fe₁₄B_Ndelta-based alloys prepared by mechanical alloying have been investigated. For the Nd₁₄Fe₇₈B₈N_y series, a large amount of NdN, alpha-Fe, and a small Nd₂Fe₁₄B_Ndelta phase are observed in x-ray diffraction patterns. With increasing Nd content, the amount of the Nd₂Fe₁₄B_Ndelta phase increases and alpha-Fe decreases gradually. When x greater than or equal to 25 in the Nd_xFe_{92-x}B₈N_y systems, some

amount of the N-containing, Nd-rich phase appears. Meanwhile, the content of the nitrogen in the Nd₂Fe₁₄N_{delta} phase decreases and, correspondingly, the Curie temperature decreases. The coercivity and the maximum magnetic energy product of this series attain maxima at x=25. A coercivity as high as 20 kOe has been achieved. The effect of nitrogen on the formation, composition, and magnetic properties of the Nd₂Fe₁₄N_{delta} compound is discussed.

[34] EFFECTS OF GA SUBSTITUTION FOR FE ON THE STRUCTURE AND MAGNETIC PROPERTIES OF Nd_{8.4}Fe_{87.1}XGa_xB_{4.5} (X=0-2.2) ALLOYS PREPARED BY MECHANICAL ALLOYING

Cui BZ. Sun XK. Liu W. Zhang ZD. Geng DY. Zhao XG. Liu JP. Sellmyer DJ. - Journal of Applied Physics. 87(9 Part 2):5335-5337, 2000

The effects of a partial substitution of Ga for Fe on the structure and magnetic properties of mechanically alloyed Nd_{8.4}Fe_{87.1}B_{4.5} were studied. With increasing Ga content, the magnetic properties of the nanocomposite magnets first improve, reach maxima at 1.1 at. % Ga, and then degrade. The addition of Ga helps to control the morphology, especially the grain sizes of both alpha-Fe and Nd₂Fe₁₄B.

[33] SPIN-GLASS BEHAVIOR IN ULTRAFINE La_{0.7}Ca_{0.3}MnO_z POWDERS PREPARED BY MECHANICAL ALLOYING

Muroi M. Street R. McCormick PG. - Journal of Applied Physics. 87(9 Part 2):5579-5581, 2000

Ultrafine La_{0.7}Ca_{0.3}MnO_z powders with controlled oxygen stoichiometry have been synthesized by mechanical alloying at ambient temperature. It is found that high-energy ball milling of La₂O₃, CaO, MnO₂, and Mn₃O₄, mixed in the stoichiometric cation ratio, yields single-phase La_{0.7}Ca_{0.3}MnO_z powders having crystallite sizes of about 10 nm and various oxygen stoichiometries (2.68 less than or equal to z less than or equal to 3.35), adjustable by changing the MnO₂/Mn₃O₄ ratio. The spontaneous magnetization (M_s) of the as-milled powder depends less on the nominal Mn valence (v(Mn)) than in bulk (La, Ca)MnO₃ and the maximum M_s, observed for epsilon(Mn) = 3.3, is much smaller. Annealing in air at temperatures above 500 degrees C increases M_s, but the way M_s approaches the bulk value (90 emu/g) depends on epsilon(Mn). These observations are discussed in terms of magnetic disorder resulting from defects induced by high-energy milling and surface effects dominant in small crystals.

[32] PRODUCTION OF IRON NITRIDES BY MECHANICAL ALLOYING

Saito T. - Journal of Applied Physics. 87(9 Part 3):6514-6516, 2000

The iron nitrides were prepared by mechanical alloying followed by solid state reaction. Mechanical alloying of alpha-Fe and epsilon-Fe₃N powders resulted in the formation of alpha-Fe solid solution with the composition of Fe₁₆N₂. The mechanically alloyed powders, when annealed at low temperatures (100-200 degrees C), consisted of alpha'-Fe₁₆N₂, alpha-Fe, and epsilon-Fe₃N phases and consisted of alpha'-Fe₁₆N₂, alpha-Fe, and gamma'-Fe₄N phases when annealed at intermediate temperatures (300-500 degrees C). On the other hand, the mechanically alloyed powders consisted mostly of alpha'-martensite phase and showed a higher saturation magnetization than iron powders when annealed at high temperatures (700-800 degrees C).

[31] SPIN-GLASS-LIKE STATIC AND DYNAMIC PROPERTIES OF MECHANICALLY ALLOYED FE-RE-CR

De Toro JA. de la Torre MAL. Arranz MA. Riveiro JM. Martinez JL. - Journal of Applied Physics. 87(9 Part 3):6534-6536, 2000

We report the observation of spin-glass-like features in a Fe₆₁Re₃₀Cr₉ sample obtained after high energy milling for 460 h. X-ray diffraction showed a broad nanocrystalline peak. The zero-field-cooled and field-cooled magnetization curves exhibited a clear irreversibility, the former showing a relatively sharp maximum for low fields at T(max) approximate to 60 K. In addition, the temperature dependence of the magnetic AC susceptibility (chi(ac)) also showed a peak at approximately the same temperature. The frequency sensitivity of such maximum (p=Delta T-max/T(max)Delta log w approximate to 0.02) is small compared to that of typical superparamagnets. However, we discuss the insufficiency of this set of phenomena to assert the existence of a true spin-glass transition and propose the blocking of interacting ferromagnetic nanoparticles as the origin of the observed behavior. The effect of annealing, rendered into nanocrystallite growth, on the chi(ac) maximum is studied in an attempt to test this explanation.

[30] MICROMAGNETIC ANALYSIS OF REMANENCE AND COERCIVITY OF NANOCRYSTALLINE PR-FE-B MAGNETS

Suess D. Dahlgren M. Schrefl T. Grossinger R. Fidler J. - Journal of Applied Physics. 87(9 Part 3):6573-6575, 2000

Nanocrystalline exchange coupled Pr_{11.76}Fe_{82.36}B_{5.88} single-phase and Pr₉Fe₈₅B₆ two-phase magnets containing 20 vol % alpha-Fe were produced using mechanical alloying. Micromagnetic finite element simulations were used to analyze the temperature dependence of the magnetic properties. In the single-phase exchange-coupled magnet a significant enhancement of the remanence J(r)=1.1 T was achieved at room temperature. A further enhancement of the remanence was observed in two-phase alpha-Fe containing magnets reaching a remanence of J(r)=1.23 T. The corresponding values of the coercive field are mu(0)H(c)=0.81 T and mu(0)H(c)=0.57 T for the single-phase magnet and the two-phase magnet, respectively. Remanence enhancement becomes more effective with increasing temperature, compensating the decrease of the saturation polarization. In the two-phase magnet the remanence increases from J(r)=1.12 to J(r)=1.23 T as the temperature is increased from 200 to 300 K. Micromagnetic calculations clearly show that the increase of the exchange length with increasing temperature improves the effective coupling between the Pr₂Fe₁₄B and the alpha-Fe phase. The decrease of the coercive field with increasing temperature has to be attributed to the temperature dependence of the anisotropy field.

[29] STRUCTURAL AND MAGNETIC PROPERTIES OF MECHANICALLY MILLED SMC05 : C

Kirkpatrick EM. Leslie-Pelecky DL. - Journal of Applied Physics. 87(9 Part 3):6734-6736, 2000

Mechanically milling SmCo₅ powder significantly increases coercivity and remanence ratio by introducing defects; however, these defects can be removed by room-temperature aging, with a resultant decrease in coercivity. A series of (SmCo₅)_x:C_{1-x} (0.15 less than or equal to x less than or equal to 1) samples has been fabricated to investigate the effect of C on oxidation protection and magnetic properties. SmCo₅ was premilled for 1 h, then added to C powder and milled for times ranging from 15 min to 7 h. X-ray diffraction indicates the presence of crystalline graphite and SmCo₅ for milling times less than or equal to 6 h and also shows the presence of fcc Co for milling times > 7 h. The

magnetic properties are very weakly dependent on milling time after the C addition, which is attributed to the lack of further grain refinement. The saturation magnetization scales linearly with the wt % of SmCo₅. Remanence ratios are approximately 0.7 and independent of volume fraction. The maximum coercivity of 16.5 kOe is comparable to the maximum obtained by milling SmCo₅ without C. Samples exposed to air for times up to two months show no decrease in coercivity or remanence ratio for x less than or equal to 0.70. The addition of C has no detrimental effect on the magnetic properties obtained by milling, except the expected reduction of M_s.

[28] FORMATION OF YTTRIUM ALUMINUM PEROVSKITE AND YTTRIUM ALUMINUM GARNET BY MECHANICAL SOLID-STATE REACTION

Guo XM. Sakurai K. - Japanese Journal of Applied Physics Part 1-Regular Papers Short Notes & Review Papers. 39(3A):1230-1234, 2000

Polycrystalline fine powder of yttrium aluminum perovskite (YAP) and yttrium aluminum garnet (YAG) have been successfully synthesized for the first time by mechanical solid-state reaction. The reaction scheme has been studied in detail. In short, the technique uses two reaction steps. First, a high-energy ball mill is used for the formation of a non-equilibrium disordered phase of Y-Al-O from a stoichiometric mixture of alumina and yttria at room temperature. Then, subsequent heating crystallizes the powder and obtains either YAP or YAG, depending on the initial chemical composition. Preparing the Al₂O₃-YAG system is also possible. The crystallization is completed at a maximum temperature of 1150 degrees C, and therefore special high-temperature equipment is not required, unlike in conventional solid-state-reaction procedures.

[27] LARGE ZINC CATION OCCUPANCY OF OCTAHEDRAL SITES IN MECHANICALLY ACTIVATED ZINC FERRITE POWDERS

Oliver SA. Harris VG. Hamdeh HH. Ho JC. - Applied Physics Letters. 76(19):2761-2763, 2000

The cation site occupancy of a mechanically activated nanocrystalline zinc ferrite powder was determined as (Zn_{0.552}+Fe_{0.183})(tet)[Zr_{0.452}+Fe_{1.823}](oct)O₄ through analysis of extended x-ray absorption fine structure measurements, showing a large redistribution of cations between sites compared to normal zinc ferrite samples. The overpopulation of cations in the octahedral sites was attributed to the ascendance in importance of the ionic radii over the crystal energy and bonding coordination in determining which interstitial sites are occupied in this structurally disordered powder. Slight changes are observed in the local atomic environment about the zinc cations, but not the iron cations, with respect to the spinel structure. The presence of Fe³⁺ on both sites is consistent with the measured room temperature magnetic properties.

[26] PIGMENTATION OF VAT BLUE RS BY BALL MILLING IN SOLVENTS

Zhang TY. Fei XN. Wang SR. Zhou CL. - Dyes & Pigments. 45(1):15-21, 2000

Pigmentation of Vat Blue RS by ball milling in the presence of organic solvents or water was carried out and the effects of ball milling time, characteristics of solvent used and two-step ball milling on the transformation of crystal phase and the resulting pigment properties were studied. Experimental results showed that the alpha-phase of C.I. Pigment Blue 60 can be obtained by ball milling 2 h in the presence of xylene and NaCl. The rate of transformation to the alpha-phase was improved with an increase in the proportion of aromatic solvents in the medium employed. By milling in an aqueous medium without organic solvents, the original pigment crystal phase was destroyed.

[25] COMPARATIVE STUDY OF MECHANOCHEMICAL PREPARATION OF ALUMINOSILICATE PRECURSORS FROM VARIOUS ALUMINIUM HYDROXIDES AND AMORPHOUS SILICA

Temuujin J. Okada K. MacKenzie KJD. Amgalan J. - British Ceramic Transactions. 99(1):23-25, 2000.

The mechanochemical formation and homogeneity of aluminosilicate precursors was investigated with respect to the influence of particle size and chemical form of the aluminium hydroxide starting material. Aluminium hydroxide in the form of bayerite with a large particle size was found to be more reactive than gibbsite of small particle size. Si-29 and Al-27 MAS-NMR, DTA-TG, and XRD measurements showed that ground mixtures of aluminium hydroxide and silica gel are more homogeneous than mixtures containing silicic acid.

[24] EFFECT OF HETEROGENEOUS PRECIPITATION ON AGE-HARDENING OF AL₂O₃ PARTICLE DISPERSION AL-4MASS%CU COMPOSITE PRODUCED BY MECHANICAL ALLOYING

Arakawa S. Hatayama T. Matsugi K. Yanagisawa O. - Scripta Materialia. 42(8):755-760, 2000

[23] THE USE OF THERMOGRAVIMETRY TO ASSESS THE EFFECT OF MECHANICAL ACTIVATION OF SELECTED INORGANIC SALTS

Wieczorek-Ciurowa K. Shirokov JG. Parylo M. - Journal of Thermal Analysis. 60(1):59-65, 2000.

The thermal decomposition of malachite and calcite and their physical mixture was studied by thermogravimetry employed as a tool for the estimation of physicochemical changes in solids, caused by dry grinding. For the identification of solids X-ray powder diffraction and IR spectroscopy were used.

[22] EFFECT OF POWDER TREATMENT ON INJECTION MOULDED ZIRCONIA CERAMICS

Trunec M. Dobsak P. Cihlar J. - Journal of the European Ceramic Society. 20(7):859-866, 2000

Agglomerated fine zirconia powder was exposed to dry and wet ball milling and to wet mixing. The subject of study was the effect of powder treatment on the disintegration of agglomerated particles, on the rheological properties of thermoplastic ceramic mixtures, and on the properties of sintered yttria-stabilized tetragonal zirconia polycrystalline ceramics (Y-TZP). Test specimens in the shape of bars and discs were produced by injection moulding of ceramic mixtures containing 52.5 and 49 vol% of powder. The powder treatment was found to yield improved rheological properties of ceramic mixtures and improved mechanical properties of sintered specimens in the case of 52.5 vol% of powder in the ceramic mixture. However, the same or even better mechanical properties of sintered components were found when the loading of ceramic mixture was reduced to 49 vol% of powder and non-treated powder was used.

[21] SURFACE MODIFICATION OF DY₂O₃-NB₂O₅ DOPE MIX FOR DIELECTRIC MATERIALS IN AQUEOUS DISPERSION

Lee WH. Tseng TY. Hennings D. - Journal of the European Ceramic Society. 20(7):977-982, 2000

The electrokinetic mismatch between the basic dielectric compound (Ba,Ca)(Ti,Zr)O₃ (BCTZ) and Dy₂O₃-Nb₂O₅ dope mix in aqueous suspensions has been improved by a pre-firing and milling treatment of the dope mix. Pre-fired

dope mix showed a much higher zeta potential which resulted in a drastic improvement of the particle dispersion in aqueous slurries. By use of surface-modified dope mix the homogeneity of the micro structure of multilayer ceramic capacitors is improved and the break-down voltage of thin (7 μ m) dielectric layers increased. The results observed indicate the critical influence of the micro structural homogeneity on the breakdown voltage of MLCCs.

[20] CRYSTALLIZATION DYNAMICS AND MAGNETORESISTANCE OF PEROVSKITE-LIKE MANGANATE SYNTHESIZED BY MECHANICAL ALLOYING

Jin ZQ. Qin HX. Zhang JR. Du YW. - Journal of Physics-Condensed Matter. 12(14):3497-3503, 2000
Nanocrystalline ferromagnetic La_{0.7}Ca_{0.3}MnO₃ perovskites with magnetoresistance effect have been successfully prepared by mechanical alloying. Thermal transformation of the amorphous phase, resulting from ball milling, to perovskite structure was studied by differential scanning calorimetry. Following the law of mass action, we discuss the crystallization dynamics of the amorphous phase. The activation energy for crystallization transformation is calculated to be about +1.25 kJ g⁻¹. The characterization of resistivity $\rho(T)$ for La_{0.7}Ca_{0.3}MnO₃ perovskites has also been investigated. At low temperature T, $\rho(T)$ has a direct-proportion dependence on T⁻². With increasing annealing temperature, the slope of the ρ -T⁻² curve decreases. The temperature dependent magnetoresistance effect at temperature far below the Curie temperature can be well expressed as the equation $\Delta \rho / \rho(0) = p(1) - p(2) T^{-3/2} - p(3) T^{5/2}$.

[19] MECHANOCHEMICAL SYNTHESIS OF METASTABLE SOLID SOLUTIONS: PHASE COMPOSITION AND MICROSTRUCTURE EVOLUTION

Grigor'eva TF. Tsybulya SV. Cherepanova SV. Kryukova GN. Barinova AP. Belykh VD. Boldyrev VV. - Inorganic Materials. 36(2):143-149, 2000

The formation of Cu-Sn, Cu-In, Ni-Sn, and Ni-In supersaturated solid solutions during mechanochemical synthesis was studied. It was found that, in the process of synthesis, intermetallic compounds were formed first. Electron-microscopic examination revealed the presence of stacking faults and microstrains nonuniformly distributed in the initial stages of mechanochemical synthesis. Further mechanical activation makes the microstrain distribution more uniform. The microstructure of the metastable solid solutions is well described by models taking into account the major types of structural imperfections-second-order microstrains and deformation stacking faults.

[18] INFLUENCE OF THE MECHANOCHEMICAL ACTIVATION ON CONCENTRATION LIMITS OF SELF-PROPAGATING HIGH-TEMPERATURE SYNTHESIS [RUSSIAN]

Grigor'eva TF. Korchagin MA. Barinova AP. Lyakhov NZ. - Doklady Akademii Nauk. 369(3):345-347, 1999

[17] VIDEO SAMPLING FOR MINE-TO-MILL PERFORMANCE EVALUATION: MODEL CALIBRATION AND SIMULATION

JA Herbst, SL Blust - CONTROL 2000: MINERAL AND METALLURGICAL PROCESSING, 2000, pp 157-166 - 3RD SYMPOSIUM ON CONTROL 2000 - MINERAL AND METALLURGICAL PROCESSING; SALT LAKE CITY, UTAH. FEBRUARY, 2000

Optimization of blasting, crushing and grinding operations is filled with challenges. One of the more difficult tasks is accurately sampling and determining the size distributions of blasted and crushed materials at a reasonable cost. The task is difficult because of the large size of fragments and the tonnage involved. However, the size distribution measurements are necessary for models that predict the performance of mine-through-mill operations. This paper is concerned with the use of video sampling for this task at National Steel Pellet Company's operation in Keewatin, Minnesota. Data gathering data analysis, model building, and mine-to-mill simulation are all described.

[16] USING MULTIVARIABLE PREDICTIVE CONTROL TO OPTIMIZE THE ASARCO MISSION MILL

A Nix, A Morrow, L Gordon - CONTROL 2000: MINERAL AND METALLURGICAL PROCESSING, 2000, pp 201-207 - 3RD SYMPOSIUM ON CONTROL 2000 - MINERAL AND METALLURGICAL PROCESSING; SALT LAKE CITY, UTAH. FEBRUARY, 2000

At the ASARCO Mission Mill, a multivariable, predictive controller was used to optimize parallel grinding circuits, improving throughput and product size. Using a dynamic model of the grinding circuit developed from on-line testing, the software manipulates 4 variables to control 14 quality and constraint variables in each circuit. Traditionally, model-based control in grinding is difficult due to ore changes. This is addressed via a novel method to predict mill overload and automatic model switching.

[15] SEMI-AUTOGENOUS MILL OPTIMIZATION WITH DEM SIMULATION SOFTWARE

RK Rajamani - CONTROL 2000: MINERAL AND METALLURGICAL PROCESSING, 2000, pp 209-214 - 3RD SYMPOSIUM ON CONTROL 2000 - MINERAL AND METALLURGICAL PROCESSING; SALT LAKE CITY, UTAH. FEBRUARY, 2000

The processing capability of a semi-autogenous (SAG) mill is greatly affected by ore geology and operating variables within the mill. The SAG mill is problematic because it cannot be conveniently studied on a smaller, laboratory-scale mill. The interior of the mill, where grinding takes place, cannot be observed using instrumentation available today. Yet the motion of the rock and ball charge determines the power draft and hence capacity of the mill. Therefore, mill optimization is pursued with a charge motion simulation software. This software uses the discrete element method to describe the individual motion of numerous ball and rock particles in the mill. It is shown that by simply increasing the chord length between the lifters and increasing the face angle of the lifters the capacity of the mill can be increased. Such a configuration of lifters allows the operator to increase mill speed, hence drawing higher power while considerably minimizing direct ball strikes against shell plates and lifters. A case study of a 36 ft. Diameter x 15 ft. Long mill is presented. Reducing the direct ball strikes leads to lesser steel consumption both in terms of balls and lifters.

[14] IN-SITU OBSERVATION OF THE COMBUSTION PHASE TRANSFORMATION IN A MECHANICALLY ALLOYED Ti-AL POWDER MIXTURES

CE Wen, K Yasue, JG Lin, YG Zhang - JAPAN INSTITUTE OF METALS, PROCEEDINGS, VOL 12, (JIMIC - 3), PTS 1 AND 2, 1999, pp 281-284 - INTERNATIONAL CONFERENCE ON SOLID-SOLID PHASE TRANSFORMATIONS (PTM 99); KYOTO, JAPAN. MAY 24-28, 1999

TEM in-situ observations are performed on the combustion phase transformations of Ti-Al mechanically alloyed powder mixtures. Results indicate that different kinds of combustion phase transformations occur under the irradiation of electron beam depending on the compositions and the mechanically alloying time of the powder mixtures. After mechanically alloyed for 360ks, the combustion phase transformation starts immediately as being irradiated and progresses gradually from an amorphous state in the mixture of the stoichiometrical TiAl; and two steps of combustion phase transformations react from a supersaturated solid solution of Al(Ti)_{ss} after being irradiated for different times in the mixture of the stoichiometrical TiAl₃. After mechanically alloyed for 180ks, the same avalanche-like reaction occurs after a period of irradiating from a mixture of supersaturated solid solutions of Ti(Al)_{ss} and Al(Ti)_{ss} with nanocrystalline size in both kind of composition powders.

[13] SHOCK INDUCED REGULARIZATION TO REFRACTORY METAL DI-SILICIDES FROM MA PRECURSOR

T Aizawa - JAPAN INSTITUTE OF METALS, PROCEEDINGS, VOL 12, (JIMIC - 3), PTS 1 AND 2, 1999, pp 353-356 - INTERNATIONAL CONFERENCE ON SOLID-SOLID PHASE TRANSFORMATIONS (PTM 99); KYOTO, JAPAN. MAY 24-28, 1999

In the shock induced synthesis from the mechanically premixed constituents or the MA precursors into transition-metal silicides, the regularization process is investigated in order to discuss the shock induced reaction process. Mo-Si system was employed to describe experimentally the shock induced regularization process into MoSi₂.

[12] MARTENSITIC NUCLEATION BY THERMAL AND MECHANICAL ACTIVATION IN IRON BASED ALLOYS

XQ Zhao - JAPAN INSTITUTE OF METALS, PROCEEDINGS, VOL 12, (JIMIC - 3), PTS 1 AND 2, 1999, pp 943-946 - INTERNATIONAL CONFERENCE ON SOLID-SOLID PHASE TRANSFORMATIONS (PTM 99); KYOTO, JAPAN. MAY 24-28, 1999

The thermodynamic and kinetic aspects of martensitic nucleation in iron based alloys were considered based on the classical nucleation theory. It is indicated that the activation energy for homogeneous nucleation is far below the energy involved in thermal vibration at appropriate temperature, and thus martensitic nucleation may occur by thermal activation. The kinetic characteristics of homogeneous nucleation is in good compatibility with various experimental studies concerning the kinetics of martensitic transformations. It is also found that the mechanical effect may play a key role in activation of nucleation at very low temperature when martensitic transformation occurs under rapid quenching.

[11] ATOMIC AND MAGNETIC STRUCTURES OF FE-CU ALLOYS PREPARED BY MECHANICAL ALLOYING TECHNIQUE

H Ino, K Tokumitsu - JAPAN INSTITUTE OF METALS, PROCEEDINGS, VOL 12, (JIMIC - 3), PTS 1 AND 2, 1999, pp 1255-1258 - INTERNATIONAL CONFERENCE ON SOLID-SOLID PHASE TRANSFORMATIONS (PTM 99); KYOTO, JAPAN. MAY 24-28, 1999

We studied atomic and magnetic structures of supersaturated solid solutions of iron and copper prepared by mechanical alloying technique. The binary bcc solid solution was formed up to 35 at % Cu and fee solution over the composition. The lattice constant of each phase is large by 0.5% compared with the original bcc iron or fee copper. Measurements of magnetization and Mossbauer effect showed that the magnetic state of the fee solution is strongly dependent on the alloy composition and it changes from ferromagnetic to paramagnetic with increasing copper content. The origin of the appearance of ferromagnetism of iron atoms in the fee solid solution is discussed in terms of the average interatomic distance and the sort of neighboring atoms.

[10] PHASE TRANSFORMATIONS INDUCED IN NANOCRYSTALLINE MATERIALS DURING MECHANICAL ALLOYING

BS Murty, J Joardar, MK Datta, SK Pabi - JAPAN INSTITUTE OF METALS, PROCEEDINGS, VOL 12, (JIMIC - 3), PTS 1 AND 2, 1999, pp 1259-1262 - INTERNATIONAL CONFERENCE ON SOLID-SOLID PHASE TRANSFORMATIONS (PTM 99); KYOTO, JAPAN. MAY 24-28, 1999

The paper reports allotropic transition from fee to hcp Ni(Si) and bcc to omega Fe(Si) on nanocrystallization (<10nm) during mechanical alloying (MA). MA of elemental blends of Ni and Si has resulted in the formation of only congruent melting silicides and the formation of non-congruent melting phases is bypassed in the nanocrystalline state. Nanocrystallization has been found to be the prerequisite for alloying during MA based on studies on a number of systems such as Cu-Ni, Cu-Zn, Cu-Al, Ni-Al and Nb-Al. The mechanism of alloying is sensitive to the enthalpy of formation of the phases and their ordered nature. A new mechanism christened as "discontinuous additive mixing" has been observed in case of ordered compounds with large enthalpy of mixing below a critical crystallite size (CS). The phase transformations have been discussed based on thermodynamic and mechanical instability criteria.

[9] TRANSFORMATIONS IN MECHANICALLY ALLOYED CO-Fe-TI POWDERS

HA Calderon, JG CabanasMoreno, R MartinezSanchez, L Rendon, M Umamoto, E VanCappellen - JAPAN INSTITUTE OF METALS, PROCEEDINGS, VOL 12, (JIMIC - 3), PTS 1 AND 2, 1999, pp 1263-1266 - INTERNATIONAL CONFERENCE ON SOLID-SOLID PHASE TRANSFORMATIONS (PTM 99); KYOTO, JAPAN. MAY 24-28, 1999

By applying a fast, hot consolidation process, mechanically alloyed Co-Ti powders, consisting of nanocrystallites dispersed in an amorphous matrix, transform into two-phase, micro- and nanocrystalline intermetallic compounds. Examination by TEM and EDAX, down into the nanometer scale, reveal the existence of Fe and Cr contaminants finely dispersed in the alloyed materials. Dissolution of the nanocrystals in the amorphous matrix, followed by the crystallization of the later, is suggested as the transformation sequence in these materials.

[8] MECHANICAL MILLING OF INTERMETALLIC COMPOUNDS R₂TM₁₇ (R=Y, Gd AND TM=FE, NI)

H Takano, K Honda, Y Mitsuboshi, S Murayama, K Hoshi, KHJ Buschow, H Bakker - APAN INSTITUTE OF METALS, PROCEEDINGS, VOL 12, (JIMIC - 3), PTS 1 AND 2, 1999, pp 1267-1270 - INTERNATIONAL CONFERENCE ON SOLID-SOLID PHASE TRANSFORMATIONS (PTM 99); KYOTO, JAPAN. MAY 24-28, 1999

Mechanical milling of R₂TM₁₇ compounds (R=Y, Gd and TM=Fe, Ni) was performed, and the structural stability and the magnetic changes of these compounds were studied. X-ray diffraction patterns, magnetization and Mossbauer

spectra for several periods of milling are presented. After long periods of milling, the structure of Y₂Fe₁₇ (Gd₂Fe₁₇) changed from hexagonal (rhombohedral) to BCC. This BCC material has the same lattice parameter as BCC Fe. But the magnetic properties of the BCC material are different from those of BCC Fe. Mechanical milling of Y₂Ni₁₇ induces a phase decomposition into FCC Ni and nano-crystalline original material. Concluding, the structure of the R₂TM₁₇ compounds is unstable with respect to mechanical impact such as mechanical milling. The structure and the magnetic properties of the milled products depend on the transition metal component TM.

[7] SOLID STATE AMORPHIZATION OF STRUCTURAL CERAMICS BY MECHANICAL ALLOYING

H Kimura, K Hongo - JAPAN INSTITUTE OF METALS, PROCEEDINGS, VOL 12, (JIMIC - 3), PTS 1 AND 2, 1999, pp 1271-1274 - INTERNATIONAL CONFERENCE ON SOLID-SOLID PHASE TRANSFORMATIONS (PTM 99); KYOTO, JAPAN. MAY 24-28, 1999

This article reports high-quality and efficient powder production of amorphous oxide ceramics using the rotating-arm reaction ball mill with a control system of water cooling. The reaction milling technique makes it possible to characterize pulverizing, the formation of multilayer, an amorphization reaction and homogenizing during mechanical alloying of the powder mixture, ZrO₂- 20mass% Al₂O₃ through the temperature inside the tank and the torque applied to agitating balls. The amorphous ZrO₂-20mol% Al₂O₃ powder has an endothermic peak at glass transition prior to a sharp crystallization peak in differential scanning calorimetry. The ball-milled ZrO₂-20mol% Al₂O₃ is confirmed to be a uniform amorphous structure without crystallite via high-resolution electron microscopy. The solid state amorphization of ZrO₂-20mol% Al₂O₃, occurs below approximately 350 K, and the milling time necessary to complete the synthesis decreases with increasing temperature.

[6] SYNTHESIS OF LITHIUM STORAGE INTERMETALLIC COMPOUNDS AND THEIR ANODE BEHAVIORS IN SECONDARY BATTERIES

H Sakaguchi, H Honda, H Maeta, T Esaka - JAPAN INSTITUTE OF METALS, PROCEEDINGS, VOL 12, (JIMIC - 3), PTS 1 AND 2, 1999, pp 1305-1308 - INTERNATIONAL CONFERENCE ON SOLID-SOLID PHASE TRANSFORMATIONS (PTM 99); KYOTO, JAPAN. MAY 24-28, 1999

Mg₂Ge and Mg₂Si were synthesized as anode active materials for lithium secondary batteries using mechanical alloying (MA). Samples with various internal energies were obtained by varying MA time from 25 h to 100 h. The first discharge (release of Li) capacity of the Mg₂Ge electrode decreased with increasing MA time. The decrement of discharge capacity with repeating charge-discharge cycle, however, did not depend on the order. The sample prepared by mechanical alloying for 40 h exhibited the longest charge-discharge cycle life, suggesting that there is the optimum degree of internal energy. Thus, it was found that the electrode performance was strongly influenced by degree of internal energy of the compounds. For the Mg₂Si electrode, the cycle life was short in comparison with the Mg₂Ge electrode, though the first discharge capacity was slight larger than that of the Mg₂Ge electrode.

[5] FORMATION OF A SUBNANOSTRUCTURED PHASE NEAR A CRACK IN A STEEL SUBJECTED TO ROLLING CONTACT FATIGUE UNDER SEVERE CONDITIONS

A Muroga, T Yasui, H Saka - JAPAN INSTITUTE OF METALS, PROCEEDINGS, VOL 12, (JIMIC - 3), PTS 1 AND 2, 1999, pp 1577-1580 - INTERNATIONAL CONFERENCE ON SOLID-SOLID PHASE TRANSFORMATIONS (PTM 99); KYOTO, JAPAN. MAY 24-28, 1999

Microstructure of a steel subjected to radial-type rolling contact fatigue has been examined by transmission electron microscopy (TEM). The microstructure near a crack shows a peculiar feature in the sense that it appears bright when etched with Nital. Such a region is named a bright etched region (BER). Most of the area in a BER consists of fine crystals, the diameter of which is about a few tens nanometers. However, in the very vicinity of a crack a region consisting of much finer grains, the diameter of which is less than 1nm, is observed. The grain size of a nanostructure in a BER was controlled by annealing and the Vickers hardness was measured as a function of the grain size. It is concluded that Hall-Petch law does not hold below 50nm.

[4] NANO-CRYSTALLINE STRUCTURE FORMATION IN MECHANICALLY MILLED 304L STEEL BY REVERSION OF DEFORMATION-INDUCED MARTENSITE

R Ishibashi, T Kamino, T Abe, Y Aono - JAPAN INSTITUTE OF METALS, PROCEEDINGS, VOL 12, (JIMIC - 3), PTS 1 AND 2, 1999, pp 1649-1652 - INTERNATIONAL CONFERENCE ON SOLID-SOLID PHASE TRANSFORMATIONS (PTM 99); KYOTO, JAPAN. MAY 24-28, 1999

Grain refinement by heavy cold working of mechanical milling was examined for gas atomized powder with the composition of 304L steel. Furthermore, microstructural changes of the milled powder were investigated during heating which simulated the sintering process. Also RT tensile properties of the sintered materials by HIP were investigated. About 90 vol.% of gamma-phase transformed to alpha'-phase during milling by the deformation-induced-martensite process. The microstructure contained grains with sizes from 50 to 200 nm, in which subgrains or dislocation cells with sizes from 10 to 20 nm were present. According to the XRD results, reversion to gamma-phase began under 773 K and was completed between 923 and 973 K. Microstructural changes were investigated, in-situ, by TEM with temperature control. Sintered materials had 0.2% proof strength of 757 MPa and tensile strength of 865 MPa.

[3] ALCOHOL SENSOR BASED ON A NON-EQUILIBRIUM NANOSTRUCTURED xZrO(2)-(1-x)ALPHA-FE2O3 SOLID SOLUTION SYSTEM

Tan OK. Cao W. Zhu W. - Sensors & Actuators B-Chemical. 63(1-2):129-134, 2000

Non-equilibrium nanostructured solid solution xZrO(2)-(1-x)alpha-Fe₂O₃ powders have been prepared using the high-energy ball milling technique, and their particle size, structural properties and alcohol gas properties have been systematically characterized using X-ray diffraction (XRD) and gas sensing measurements. Experimental results show that particle size of the powder is drastically milled down to less than 10 nm after 20 h of high-energy ball milling. Our modified structural model, square(1/3) + ZrO₂ + yO(2) --> Zr-1/3(4+) + 2(1-y)O-s(2) + 4yO(s)(-); with square(1/3) denoting the 1/3 available octahedral sites of the corundum structure; for these non-equilibrium nanostructured solid solution xZrO(2) - (2-x)alpha-Fe₂O₃ materials can explain both the lattice expansion of these high energy milled sample as well as the charge neutrality in terms of additional oxygen dangling bonds at the

particle surfaces. The screen-printed thick film gas sensors made from such mechanically alloyed materials demonstrate a very high gas sensitivity value of 1097 at 1000 ppm of alcohol gas in air. It is believed that the high gas sensitivity value is related to the enormous oxygen-dangling bonds at the particle surfaces.

[2] PREPARATION METHOD OF SUBMICROMETER-SIZED ALPHA-ALUMINA BY SURFACE MODIFICATION OF GAMMA-ALUMINA WITH ALUMINA SOL

Han KR. Lim CS. Hong MJ. Jang JW. Hong KS. - Journal of the American Ceramic Society. 83(4):750-754, 2000
A method is introduced to prepare almost-spherical submicrometer-sized α -alumina via surface modification of gamma-alumina with an alumina sol. Milled gamma-alumina, in the presence of 3 wt% of alpha-alumina with a median particle size ($d(50)$) of 0.32 μm (AKP-30), produced irregularly shaped alpha-alumina with $d(50)$ approximate to 0.3 μm after heat treatment at 1100 degrees C for 1 h. gamma-alumina that had been surface-modified by milling in the presence of 3 wt% of the alumina sol resulted in almost-monosized, spherical α -alumina similar to 0.3 μm in size after heat treatment at 1100 degrees C for 1 h. Furthermore, almost-spherical α -alumina 0.1-0.2 μm in size was obtained by milling gamma-alumina with 3 wt% of AKP-30 alumina in the presence of 3 wt% of the alumina sol, followed by heat treatment at 1100 degrees C for 1 h. The alumina sol that has been introduced in this work seems to act as a dispersant, in addition to helping to form a spherical shape.

[1] DEFORMATION-INDUCED MICROSTRUCTURAL CHANGES IN FE40NI40P14B6 METALLIC GLASS

Fan GJ. Quan MX. Hu ZQ. Loser W. Eckert J. - Journal of Materials Research. 14(9):3765-3774, 1999
The effect of mechanical deformation via high-energy ball milling on the structure of the Fe40Ni40P14B6 metallic glass was studied by means of x-ray diffraction, transmission electron microscopy (TEM), and differential scanning calorimetry (DSC). After 5 h of milling, TEM observations indicated that some nanocrystallites with a diameter of about 6 nm precipitated from surface layers of the amorphous ribbons, whereas the bulk remained amorphous. When milling time was increased to 11 h, bulk crystallization occurred. The amorphous Fe40Ni40P14B6 alloy crystallized into a mixture of gamma-(Fe,Ni) and (Fe,Ni)₃(P,B). To understand the microstructural changes occurring in the amorphous ribbons before the onset of bulk crystallization, the isothermal crystallization behavior of as-deformed amorphous ribbons was studied. Compared with as-quenched amorphous ribbons, the local value of the Avrami exponent, derived from isothermal DSC data, increased from 3.5 to 4.1 for bulk crystallization. The thermal crystallization mechanism of deformed amorphous Fe40Ni40P14B6 ribbons changed from an eutectic-type reaction with simultaneous precipitation of gamma-(Fe,Ni) and (Fe,Ni)₃(P,B) from the amorphous matrix to a primary-type reaction with precipitation of alpha-Fe(P,B) preceding the formation of gamma-(Fe,Ni) and (Fe,Ni)₃(P,B). Our results suggest that several hours of mechanical milling cause surface crystallization and some atomic rearrangements in the amorphous alloy. The latter effect may be responsible for the observed primary-type reaction for crystallization of the deformed amorphous alloy.

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