

RESEAU FRANÇAIS DE MECANOSYNTHESE

Lettre N°66

Septembre 2000

181 Groupes de Recherche
(dont 108 à l'étranger / 32 Pays)

Bureau : E. Gaffet (Président), G. Le Caër (Secrétaire Général), A.R. Yavari (Trésorier)

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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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Technical Announcements

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M.B.N.srl
via Roma, 4
I-31020 San Vendemiano (TV) - Italie

- **MBN srl**, based in Italy, announces the availability of laboratory (**kg range**) as well as industrial quantities (**ton range**) of materials produced by **mechanosynthesis**.
- More information are available at the Web site: <<http://www.mbn.it/>> .
- **Batches on-demand could be considered**

Contact : E-mail: info@mbn.it

Web site: <http://www.mbn.it>

Phone Number : +39 0422 718956

Fax Number : +39 0422 718964

<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: Fritsch GmbH (Andrea Köhler- Manufacturers of Laboratory Instruments 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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**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/icamp>

"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)

**International Conference on Trends in Mechanical Alloying
Science, Technology and Applications (TMA-2001)**

Jaipur, India Feb. 21-23, 2001.
Secretariat of Int. Conf. TMA-2001

Organizer : Dr. P. R. Soni
Department of Metallurgical Engineering,
Malaviya Regional Engineering College
Jaipur-302017, India
Tel : +91-0141-702042
Fax : +91-0141-702954
E-mail : psmt@arya.recjai.ernet.in

NOUVEAU

**Contact Persons : Dr. P. R. Soni
Prof. T. V. Rajan**

Technical Programmes

Paper Presentations

Technical sessions (oral and poster presentations) and special lectures are being scheduled.

Scope

All field of mechanical alloying, ranging from basic science to applied technology will be covered.

Papers will be called mainly on the following topics n The MA process

- Variations of the process
- Process control agents
- Mechanisms
- Energy considerations in mechanical alloying n Modelling mechanical alloying
- Consolidation of MA powder
- Joining the MA materials
- Mechanical alloying of rapidly solidified materials n Properties & applications of MA materials in areas such as - Dispersions strengthened materials, Hydrogen storage materials, Spray-coated powders, Superconducting materials, Composites, Superplasticity, Tribomaterials, Soft & hard magnetic materials, Amorpization, Nanocrystalline materials and Mechanically activated chemical reactions.

Call for the Papers

Detailed procedures for the submission of papers will be described in the second announcement to be issued around August 2000. Those who plan to submit papers should indicate so b marking the appropriate box in the attached response card to receive further information directly.

Exhibition

Exhibition will be held during the conference to provide all visitors with the latest technology & knowledge in the mechanical alloying worldwide. Display will include MA powders, alloying mills, MA products, and related publications.

Detailed procedures for applications for exhibition will be issued around August 2000. Those who plan to exhibit should indicate so by marking the appropriate box in the attached response card to receive further information directly.

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>

**COLLOQUE SUR LES INNOVATIONS
DANS LES MATERIAUX FRITES**

Poitiers-Futuroscope

3-4-5 juillet 2001

Ce colloque, organisé par une commission mixte SF2M/Groupe Français de la Céramique, vise à réunir la communauté industrielle et scientifique du domaine des poudres et matériaux frittés (métalliques et céramiques), afin de faire le point sur les innovations et avancées technologiques de la dernière décennie. Ces innovations portent sur toutes les étapes du procédé, de l'élaboration de la poudre jusqu'à la pièce frittée, ainsi que sur les applications. Elles peuvent être constituées aussi bien de nouvelles connaissances fondamentales que de nouvelles solutions techniques. Le colloque se déroulera sur 3 jours et comprendra une demi-journée consacrée à des visites de laboratoires ou de sites industriels.

Les contributions, sous forme de communications orales ou d'affiches, sont souhaitées dans les quatre thèmes suivants :

- Poudres et Mélanges
- De la poudre au matériau
- De la poudre à la pièce finalisée
- Propriétés et applications des matériaux frittés.

Pour toute information sur ce colloque et recevoir la circulaire de pré-inscription, s'adresser à SF2M. Les Fontanelles, 1 rue de Craïova. 92024 Nanterre Cedex, tél. : 01 41 02 03 90 ou consulter le site www.sf2m.asso.fr (rubriques « sommaires » puis « conférences »).

SOUTENANCES DE THESE

CH. GRAS

**Réactivité et Thermodynamique dans le procédé MASHS
(Mechanically Activated Self - Propagating High - Temperature Synthesis) :
Application aux systèmes Mo / Si et Fe / Si**

6 Septembre 2000 - Univ. de Bourgogne - Dijon

Jury : Y. Bienvenu (Rapp.), G. Le Caer (Rapp.), G. Bertrand, JP Bonnet, M. Gailhanou, JP Larpin, F. Bernard (Co - directeur) & E. Gaffet (Co - Directeur)

L'objectif principal de ce travail a été de traquer dans toutes les étapes du procédé MASHS (Mechanically Activated Self-propagating High-temperature Synthesis) les différentes modifications induites par l'adjonction d'un pré-broyage au processus de combustion autoentretenue SHS (Self-propagating High-temperature Synthesis).

Dans un premier temps, il a été nécessaire de définir la microstructure des réactifs après cobroyage. La poudre se présente, après traitement mécanique contrôlé, sous la forme d'agrégats microniques (0.2-200 µm) composés de nanocrystallites de métal et de silicium (Mo ou Fe : 50 nm et Si : 30 nm).

Lorsque celle-ci est mise en situation de réaction SHS, une diminution du temps nécessaire à l'ignition comme une accélération de l'onde de combustion liée à la dynamisation de la réaction chimique ont été mises en évidence. D'une manière générale, la multiplication des interfaces Métal/Si créées lors du cobroyage ainsi que l'énergie accumulée conduisent à la diminution de l'énergie seuil d'ignition. En effet, il a été possible d'instaurer une onde de combustion stable entre des nanocrystallites solides de fer et de silicium, et cela malgré les difficultés réactionnelles prédites par le critère semi-empirique d'autopropagation. En outre, la taille des cristallites de fer comme la présence de FeSi et a-FeSi2 mécano-induites sont capables de piloter le processus réactionnel tout en définissant la phase majoritairement produite.

En ce qui concerne la formation de MoSi2, la diffractions en temps réel (0.033 s) couplée à la thermographie infrarouge (0.0066s) met en évidence une étape de fusion du silicium sans pour autant être capable de déceler quelques intermédiaires de réaction que ce soient. Par contre, le produit final est pur, nanostructuré (60-90 nm) mais très poreux (45 à 50 %)

Par conséquent, le procédé MASHS pêche par son incapacité à produire un matériau dense. A contrario, une mise en température rapide effet joule et une mise sous charge simultanée à chaud, le pré-traitement mécanique s'est révélés comme l'unes des conditions nécessaires à la production de nano-disiliciure de molybdène (80 nm) dense (93%). Les premiers résultats tendent à prouver que ce nouveau matériau présente des modifications importantes de ses propriétés : une dureté à 298 K accrue (+55%) et une meilleure tenue à la corrosion haute température (1273 K)

Mots clés

Activation mécanique, Cobroyage à haute énergie, Combustion autoentretenue (SHS), Diffraction des rayons X en temps réel (TRXRD), Thermographie infrarouge, Nanomatériau, Synthèse/densification simultanée, Disiliciure de fer (FeSi2) et Disiliciure de molybdène (MoSi2)

M. ZOUGGAR

"Effets du broyage sur les propriétés structurales et mécaniques de poudres de fer pur et sur l'activation de la nitruration"

4 Septembre 2000 - LMP - Poitiers

Jury : A. Fnidiki(Rapp.), E. Gaffet (Rapp), P. Goudeau (Inv.), M. Grosbras (Inv.), A. Straboni , P. Chartier (Co - Dir) & J. Mimault (Co - Dir)

Résumé : Ce travail porte sur l'évolution microsturale et mécanique de la poudre de fer élaborée par broyage mécanique, et sur celle subissant par la suite de traitements de recuits sous vide, de nitruration dans l'enceinte d'une presse isostatique à chaude (HIP) ou bien des traitements thermiques en plasma d'ammoniac ••

C. GOUJON

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

25 Mai 2000 - ENSMSE

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

J. JOARDAR

"Synthesis of nanocrystalline aluminides in Al - Ni - Fe system"

by Mechanical Alloying"

Avril 2000

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

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

Christine BARBEAU

(Laboratoire de Métallurgie Physique - Futuroscope)

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

13 mars 2000

Thèse de Doctorat de l'Université de Poitiers

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

Hugues GUÉRAULT

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

28 Janvier 2000

THÈSE DE DOCTORAT - Université du Maine - Physique des Matériaux et des Surfaces

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

Cyril LENAIN

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

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

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

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 :

Bibliographie Récente

Livres ou "Special Issues"

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

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

MACROMOLECULAR MECHANOCHEMISTRY

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

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

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

Contents

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

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

Send your preliminary order to orders@cisp.demon.co.uk

(9/06/2000)

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

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

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

Preface

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

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

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

Contents

Introduction - Background - History of Mechanical Alloying - Milling - Factors Affecting the Mechanical Alloying 8
- Types of Mills 8 - High Energy Ball mill 9 - Attritor Ball Mill 9 - Planetary Ball Mill 11 - Vibratory Ball Mill 12 - Low Energy Ball Mill 15 - Tumbler Ball Mill 15 - Tumbler Rod Mill 16 - Effect of Ball-to-Powder Weight Ratio 19 - Effect of Milling Atmosphere 22 - Mechanism of Mechanical Alloying 23 - Ball-Powder-Ball Collision 24 - Necessity of Mechanical Alloying 25 - References 27

PART I GRAIN REFINING, SIZE CONTROLLING AND HOMOGENIZATION

Fabrication of ODS Alloys - Introduction and Background - Applications and Examples - ODS Ni-Base Superalloys and Fe-Base High Temperature Alloys 34 - INCONEL MA 754 35 - INCONEL MA 6000 37 - INCOLOY MA 956 38 - ODS Al Base Alloys 38 - References 45 - Fabrication of Nanophase Materials - Introduction - Influence of Nanocrystalline on the Mechanical Properties: Strengthening by the Grain size Reduction - Formation of Nanocrystalline Materials by Ball Milling Technique - Mechanism(s) 52 - Selected Examples 53 - Formation of Nanocrystalline NixMo100-x 53 - Formation of Nanocrystalline FCC Metals 54 - Consolidation of the Nanocrystalline Milled Powders - References 59 - Fabrication of Nanocomposite Materials - Introduction and Background - Fabrication of SiCp/Al Composites by Mechanical Alloying - Properties of Mechanically Solid State Fabricated SiCp/Al Composites - Mechanism of Fabrication - References 82

PART II ROOM TEMPERATURE REACTIVE MILLING

Mechanically Induced Solid-State Carbonization - Introduction - Difficulties of Preparations - Fabrication of

Nanocrystalline TiC by Mechanical Alloying Method - Properties of Mechanically Solid State Reacted TiC Powders - Other Carbides Produced by Mechanical Alloying - References 124 - Mechanically Induced Solid-Gas Reaction - Introduction - Fabrication of Nanocrystalline TiN by Reactive Ball milling - Properties of Reacted Ball Milled TiN Powders - Mechanism of Fabrication - Other Nitrides Produced by RBM - Fabrication of Nanocrystalline Solid Solution NiTiH by Reactive Ball Milling - References 157 - Mechanically Induced Solid-State Reduction - Introduction - Reduction of Cu₂O with Ti by Room Temperature Rod Milling - Properties of Rod-Milled Powders - Mechanism of MSSR - Fabrication of Nanocrystalline WC and Nanocomposite WC-MgO Refractory Materials by MSSR and Methods - References 189 - Mechanically Induced Solid-State Amorphization - 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 - References 295 -

(05/05/2000)

EXTRACTIVE METALLURGY OF ACTIVATED MINERALS

included in series Process Metallurgy, 10

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

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

http : // www.elsevier.nl/inca/publication

Description

Mechanical activation of solids is a part mechanochemistry, the science with a sound 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 Besterçi

<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 Besterçi, 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)

Index : ISBN 189832655X, 80 pages, 234 **156 mm**, **soft laminated cover**, £22.00, January 1999

Cambridge International Science Publishing 7 Meadow Walk, Great Abington, Cambridge CB1 6AZ, England Fax

+44 1223 894539; Tel +44 1223 893295 Email: cisp@cisp.demon.co.uk

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

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

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

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

CHEMISTRY FOR SUSTAINABLE DEVELOPMENT

Vol. 6, No. 2-3, MARCH-JUNE 1998

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)

MEETING REPORTS

Contribution from Peter Balaz

I have participated in July at the **XXI. International Mineral Processing Congress** in Rome (23.7.-27.7.2000). The Proceedings were published by Elsevier under edition of P. Massacci.

Few papers on the application of mechanochemistry were published :

BENEFICATION AND MECHANO-CHEMICAL ALTERATION OF NATURAL ZEOLITES, PAPER

T.S. Yusupov, H. Heegn, L.G. Shumskaya - A1:44-51

MECHANICALLY INDUCED CHANGES IN STRUCTURE AND PROPERTIES OF SOLIDS, PAPER

#H. Heegn - A4:52-59

THIOSULFATE LEACHING OF GOLD FROM A MECHANOCHEMICALLY PRETREATED COMPLEX SULFIDE CONCENTRATE, PAPER

P. Balaz, J. Ficeriova, E. Boldizarova, M. Haber, S. Jelen, R. Kammel - A6:74-81

CONGRESS PROCEEDINGS

[84] STRUCTURE AND MAGNETIC PROPERTY CORRELATION IN NANOCRYSTALLINE SMFe9

C Djega Mariadassou, L Bessais - NANOPHASE AND NANOCOMPOSITE MATERIALS III (Series: MATERIALS RESEARCH SOCIETY SYMPOSIUM PROCEEDINGS), 2000, Vol 581, pp 95-100 - SYMPOSIUM ON NANOPHASE AND NANOCOMPOSITE MATERIALS III HELD AT THE 1999 MRS FALL MEETING; BOSTON, MASSACHUSETTS. NOVEMBER 29-DECEMBER 2, 1999

Sm_xFe_{100-x} samples with x = 7.6, 10.5 and 12.5 were prepared by high energy ball-milling and subsequent annealing at various temperature T-a, 600 < T-a < 1200 degrees C. Rietveld analysis coupled to Curie temperature measurements and Mossbauer spectroscopy revealed for 600 < T-a < 900 degrees C an hexagonal phase P6/mmm derived from TbCu₇ with stoichiometry SmFe₉. At T-a > 900 degrees C, the ordered R $\overline{3}m$ Sm₂Fe₁₇ structure is obtained so that SmFe₉ appears as the out-of-equilibrium precursor of Sm₂Fe₁₇. The Curie temperature and hyperfine field augmentation found for SmFe₉ results from an increase of the interatomic distance in the Fe-Fe dumbbell, responsible for a reduction of the negative exchange interaction.

[83] NANOCOMPOSITES IN THE SYSTEMS Fe_{1-x}O-Fe₃O₄ AND MgO-MgFe₂O₄ PRODUCED BY MECHANICAL ALLOYING

A Huerta, HA Calderon, H Yee Madeira, M Umamoto, K Tsuchiya - NANOPHASE AND NANOCOMPOSITE MATERIALS III (Series: MATERIALS RESEARCH SOCIETY SYMPOSIUM PROCEEDINGS), 2000, Vol 581, pp 253-258 - SYMPOSIUM ON NANOPHASE AND NANOCOMPOSITE MATERIALS III HELD AT THE 1999 MRS FALL MEETING; BOSTON, MASSACHUSETTS. NOVEMBER 29-DECEMBER 2, 1999

Wustite-magnetite and magnesia-magnesioferrite nanocrystalline ceramics have been prepared by mechanical milling and spark plasma sintering. As-milled powders have a nanocrystalline structure in both systems. Low energy milling gives rise to an increasingly higher volume fraction of wustite as a function of milling time in the Fe_{1-x}O-Fe₃O₄ system. Similar results are obtained in the MgO-MgFe₂O₄ system with increasingly larger amounts of MgFe₂O₄ produced by milling. Composites of magnetic particles (Fe₃O₄ or MgFe₂O₄) in a nonconductive matrix (FeO or MgO, respectively) are found in the sintered samples. Measurement of magnetic properties can be used to determine conclusively the nature of the developed phases and the effect of grain size.

[82] MAGNETIC HARDENING OF MECHANICALLY ALLOYED SMFe_{11-x}COxTi

L Bessais, C Djega Mariadassou - NANOPHASE AND NANOCOMPOSITE MATERIALS III (Series: MATERIALS RESEARCH SOCIETY SYMPOSIUM PROCEEDINGS), 2000, Vol 581, pp 259-264 - SYMPOSIUM ON NANOPHASE AND NANOCOMPOSITE MATERIALS III HELD AT THE 1999 MRS FALL MEETING; BOSTON, MASSACHUSETTS. NOVEMBER 29-DECEMBER 2, 1999

Mechanical alloying of ternary SmFe_{11-x}CoxTi (x = 0, 0.5, 1, 1.5, 2) alloys was carried out under an Ar atmosphere. Milled samples were annealed for 30 min in a vacuum at different temperatures T-a, from 650 degrees C to 1150 degrees C. The effects of heat treatment, on structure and magnetic property changes, have been investigated by means of x-ray diffraction using the Rietveld method, Mossbauer spectroscopy and differential sample magnetometer. Tetragonal ThMn₁₂-type structure is observed for samples annealed at T-a > 900 degrees C. For 650 < T-a < 800 degrees C the TbCu₇ type phase was identified as the major phase. Between these two regions a mixture of TbCu₇ and ThMn₁₂-type nanocrystalline phases is obtained with a maximum of the coercive field H_c (H_c > 5kOe). The Mossbauer spectra relative to the hexagonal phase show sextuplets broadened by the statistical occupancies of the iron sites. An enhancement of the magnetic properties results from the Co substitution.

[81] THE ROLE OF BORON IN THE MECHANICAL MILLING OF TITANIUM-6% ALUMINIUM-4% VANADIUM POWDERS

AP Brown, R Brydson, C Hammond, TMT Godfrey, A Wisbey - NANOPHASE AND NANOCOMPOSITE MATERIALS III (Series: MATERIALS RESEARCH SOCIETY SYMPOSIUM PROCEEDINGS), 2000, Vol 581, pp 265-270 - SYMPOSIUM ON NANOPHASE AND NANOCOMPOSITE MATERIALS III HELD AT THE 1999 MRS FALL MEETING; BOSTON, MASSACHUSETTS. NOVEMBER 29-DECEMBER 2, 1999

The reduction in grain size of a metal can lead to significant improvement in mechanical properties. Mechanical alloying (MA) with a second phase is a possible route to producing fine-grained, particulate reinforced material. This study describes the microstructural development of Ti-6%Al-4%V milled with increasing concentrations of boron. Mechanical milling of Ti-6%Al-4%V powder produces a nanocrystalline material. MA of Ti-6%Al-4%V with boron results in the alloying of the two to form either a boride or an amorphous phase when the local concentration of boron is similar to 50 at.%. During milling, the boron tends to remain near to its original particle form and in these boron-

rich regions TIE is formed. Beyond these regions small amounts of boron (a few at.%) mix with the titanium matrix and reduce further the grain size of the titanium. An increase in the global concentration of boron increases the volume fraction of boride produced.

[80] STEADY STATE PHASE DIAGRAM OF CU-AG UNDER BALL MILLING: AN XRD AND APFIM STUDY
F Wu, P Bellon, AJ Melmed, TA Lusby - NANOPHASE AND NANOCOMPOSITE MATERIALS III (Series: MATERIALS RESEARCH SOCIETY SYMPOSIUM PROCEEDINGS), 2000, Vol 581, pp 271-276 - SYMPOSIUM ON NANOPHASE AND NANOCOMPOSITE MATERIALS III HELD AT THE 1999 MRS FALL MEETING; BOSTON, MASSACHUSETTS. NOVEMBER 29-DECEMBER 2, 1999

The nature of the steady state reached during ball milling of Cu_xAg_{1-x} powders ($x = 35$ to 75) is studied as a function of the milling temperature (85K less than or equal to T less than or equal to 503K). The characterization of the powders is performed by using x-ray diffraction. Differential calorimetry and atom probe field ion microscopy. A steady-state phase diagram is built. Three-phase coexistence is shown to generally take place at intermediate milling temperatures. Atom probe data reveals that the solid solution stabilized by low milling temperature is nearly random, where as milling at elevated temperatures results in the decomposition of the elements at a lengthscale of 20 similar to 30 nm.

[79] CRACK HEALING IN AN ALUMINA/SILICON CARBIDE NANOCOMPOSITE AFTER GRINDING AND ANNEALING

HZ Wu, JM Titchmarsh, SG Roberts, B Derby - NANOPHASE AND NANOCOMPOSITE MATERIALS III (Series: MATERIALS RESEARCH SOCIETY SYMPOSIUM PROCEEDINGS), 2000, Vol 581, pp 327-332 - SYMPOSIUM ON NANOPHASE AND NANOCOMPOSITE MATERIALS III HELD AT THE 1999 MRS FALL MEETING; BOSTON, MASSACHUSETTS. NOVEMBER 29-DECEMBER 2, 1999

Alumina/silicon carbide nanocomposites are known to show their highest strength levels after surface grinding followed by annealing. After annealing in flowing argon, nanocomposites with very coarsely ground surfaces have strengths exceeding those with a finely polished surface. Specimens with lapped surfaces also show a small improvement in strength on annealing. TEM investigations of annealed cross-sections show that the annealing process leads to surface crack healing. The chemical composition of the subsurface region has been studied, and reactive products on and close to the nanocomposite surface after annealing have been investigated by energy dispersive X-ray analysis in the STEM.

[78] THERMOELECTRIC PROPERTIES OF Bi_2Te_3 - Sb_2Te_3 COMPOUNDS PREPARED BY MA-PULSE DISCHARGE SINTERING PROCESS

RE Park, YH Park, T Abe - NANOPHASE AND NANOCOMPOSITE MATERIALS III (Series: MATERIALS RESEARCH SOCIETY SYMPOSIUM PROCEEDINGS), 2000, Vol 581, pp 591-596 - SYMPOSIUM ON NANOPHASE AND NANOCOMPOSITE MATERIALS III HELD AT THE 1999 MRS FALL MEETING; BOSTON, MASSACHUSETTS. NOVEMBER 29-DECEMBER 2, 1999

The Bi_2Te_3 - Sb_2Te_3 compounds with the composition of useful thermoelectric cooling materials were prepared by mechanical alloying-pulse discharge sintering process. Effects of the process on the Seebeck coefficient, electrical resistivity and thermal conductivity were investigated. Temperature dependence of the Hall coefficient was also observed in the temperature range 80 - 325 K. The figure of merit, Z , was found to be about $4.0 \times 10^{-3} K^{-1}$ at room temperature in the 25% Bi_2Te_3 -75% Sb_2Te_3 composition sintered at 618K using grain refined mechanically alloyed powders which had the size of under 32 μm . The value of Z was remarkably improved with a decrease of the thermal conductivity shown in the fine grain compacts fabricated by mechanical alloying-pulse discharge sintering process.

[77] MAGNETIC HARDENING INDUCED BY EXCHANGE COUPLING IN MECHANICALLY MILLED ANTIFERROMAGNETIC - FERROMAGNETIC COMPOSITES

J Sort, J Nogués, X Amils, S Surinach, JS Muñoz, MD Baro - NANOPHASE AND NANOCOMPOSITE MATERIALS III (Series: MATERIALS RESEARCH SOCIETY SYMPOSIUM PROCEEDINGS), 2000, Vol 581, pp 641-646 - SYMPOSIUM ON NANOPHASE AND NANOCOMPOSITE MATERIALS III HELD AT THE 1999 MRS FALL MEETING; BOSTON, MASSACHUSETTS. NOVEMBER 29-DECEMBER 2, 1999

Mechanical milling has been used to synthesize ferromagnetic (FM, Co) antiferromagnetic (AFM, NiO) composites. The coercivity, H_C , and energy product, BH_{Max} , of these composites can be enhanced at room temperature after appropriate heat treatments above the Neel temperature of the AFM, T_N . Although the maximum H_C is achieved for the (NiO) $_1$:1(Co) weight ratio, BH_{Max} is further enhanced for the (NiO) $_2$:3(Co) ratio, where higher saturation magnetization is obtained due to the larger amount of FM. Exchange coupling, responsible for these effects, decreases as the temperature is increased and vanishes close to T_N . The thermal stability of the coercivity enhancement remains rather insensitive to the somewhat broad distribution of blocking temperatures of this system.

[76] CRYOGENIC MECHANICAL ALLOYING OF POLY (ETHER ETHER KETONE) - POLYCARBONATE COMPOSITE POWDERS FOR SELECTIVE LASER SINTERING

J Schultz, R Kander, C Suchicital - SOLID FREEFORM FABRICATION PROCEEDINGS, AUGUST 1999 (Series: SOLID FREEFORM FABRICATION PROCEEDINGS (SERIES)), 1999, pp 311-318 - 10TH SOLID FREEFORM FABRICATION SYMPOSIUM (SFF); AUSTIN, TEXAS. AUGUST 9-11, 1999

Mechanical alloying is a solid state processing technique traditionally used in the metallurgical industry to extend solubility limits in alloy systems. Mechanical alloying can also be used to blend polymer systems at ambient or cryogenic temperatures. In this work, cryogenic mechanical alloying was employed to create composite powders of Poly (ether ether ketone) (PEEK) - Polycarbonate (PC) for use in selective laser sintering applications. The microstructural development of the PEEK-PC system that occurs during laser sintering and the effects of this microstructure on mechanical properties of the laser sintered parts was investigated.

[75] SYNTHESIS OF NANOCRYSTALLINE INCONEL 625 POWDERS BY CRYOMILLING

DG Cheng, JH He, R Rodriguez, EJ Lavernia - SURFACE ENGINEERING: IN MATERIALS SCIENCE I, 2000, pp 13-21 - SYMPOSIUM ON SURFACE ENGINEERING IN MATERIALS SCIENCE I HELD AT THE 2000 TMS

ANNUAL MEETING; NASHVILLE, TENNESSEE. MARCH 12-16, 2000

The present paper reports on the successful synthesis of nanocrystalline Inconel 625 powders by cryogenic high energy ball milling (cryomilling). Commercially available Inconel 625 powders (Diamalloy 1005 AMDRY 625) were milled in liquid nitrogen for 20 hours. The characteristics of the milled powders, i.e., morphology, powder size, grain size, chemistry and structure evolution during milling, were analyzed using X-ray diffraction (XRD), chemical analysis and scanning electron microscopy (SEM).

[74] MECHANICAL ACTIVATION OF BaCO₃-Al₂O₃-SiO₂

S Boskovic, D Kosanovic, D BahloulHourlier, P Thomas, S Kis - ADVANCED SCIENCE AND TECHNOLOGY OF SINTERING, 1999, pp 167-173 - 9TH WORLD ROUND TABLE ON SINTERING (IX WRTCS); BELGRADE, YUGOSLAVIA. SEPTEMBER 1-4, 1998

A BaCO₃-Al₂O₃-SiO₂ mixture was mechanically activated for 2 and 4 hours. Chemical composition of the mixture corresponded to BaAl₂Si₂O₈. Reaction course was followed in the temperature range 750 degrees-1200 degrees C as a function of activation time. Reaction of celsian formation was followed using thermogravimetry as well as conventional and high-temperature X-ray diffractions. The obtained data show that reaction rate increases with prolonged activation time, under the same conditions of thermal treatment. Formation of hexacelsian was favoured with increasing activation time. Formation of monoclinic celsian was retarded, with longer activation.

[73] MECHANOCHEMICAL SYNTHESIS OF NANOPHASED CARBIDE TiC

I Uvarova, M Savyak, I Timofeeva, L Isayeva - ADVANCED SCIENCE AND TECHNOLOGY OF SINTERING, 1999, pp 175-179 - 9TH WORLD ROUND TABLE ON SINTERING (IX WRTCS); BELGRADE, YUGOSLAVIA. SEPTEMBER 1-4, 1998

[72] MATHEMATICAL MODELING OF COMBUSTION-TYPE INTERACTION DURING MECHANICAL ALLOYING

BB Khina, YS Sholpan - ADVANCED SCIENCE AND TECHNOLOGY OF SINTERING, 1999, pp 181-186 -9TH WORLD ROUND TABLE ON SINTERING (IX WRTCS); BELGRADE, YUGOSLAVIA. SEPTEMBER 1-4, 1998

[71] EVOLUTION OF THE MICROSTRUCTURE OF DISPERSE ZNO DURING MECHANICAL ACTIVATION AND ITS INFLUENCE ON SINTERABILITY

TV Sreckovic, MG Kakazey, VA Melnikova, TV Tomila, MM Ristic - ADVANCED SCIENCE AND TECHNOLOGY OF SINTERING, 1999, pp 187-192 - 9TH WORLD ROUND TABLE ON SINTERING (IX WRTCS); BELGRADE, YUGOSLAVIA. SEPTEMBER 1-4, 1998

[70] CHANGE OF THE PHYSICAL CHEMICAL CHARACTERISTICS OF THE MATERIALS BY MECHANICAL ACTIVATION

L Andric, D Zivanovic, S Milosevic - ADVANCED SCIENCE AND TECHNOLOGY OF SINTERING, 1999, pp 199-204 - 9TH WORLD ROUND TABLE ON SINTERING (IX WRTCS); BELGRADE, YUGOSLAVIA. SEPTEMBER 1-4, 1998

Mechanical activation-milling of a material changes the size and structure of its particles, leading to an increase of the system potential energy, and also material chemical activity as a consequence of increased free surface and concentration of the lattice defects. These changes show that new change the properties of treated materials. Control of the mechanical activation process and properties of activated materials are utilized in several technologies (sintering, chemical synthesis, hide metallurgy etc.). In this paper experimentally obtained results on changes of physico-chemical properties of alumina during its mechanical activation in high energy vibro mill are presented.

[69] MECHANICAL ACTIVATION OF MICA

D Zivanovic, L Andric, Z Sekulic, S Milosevic - ADVANCED SCIENCE AND TECHNOLOGY OF SINTERING, 1999, pp 211-217 - 9TH WORLD ROUND TABLE ON SINTERING (IX WRTCS); BELGRADE, YUGOSLAVIA. SEPTEMBER 1-4, 1998

In this paper, the investigation results on the changes occurring during mechanochemical activation of mica performed in planetary and vibrational mills, are presented. The model of the particle size distribution and specific surface change developed by Bet method is propose. Mechanochemically activated mica can be used as filler in industry of pigments.

[68] THE INFLUENCE OF MECHANICAL TREATMENT ON ACTIVATION OF THE ZNO-SNO₂ SYSTEM

A Tucic, N Nikolic, B Marinkovic - ADVANCED SCIENCE AND TECHNOLOGY OF SINTERING, 1999, pp 219-222 - 9TH WORLD ROUND TABLE ON SINTERING (IX WRTCS); BELGRADE, YUGOSLAVIA. SEPTEMBER 1-4, 1998

[67] COPPER MATRIX STRENGTHENING IN Cu-AL₂O₃ SYSTEM BY MECHANICAL ALLOYING AND MILLING OF PURE COPPER AND PREALLOYED COPPER POWDERS

V Rajkovic, S Zec, M Mitkov - ADVANCED SCIENCE AND TECHNOLOGY OF SINTERING, 1999, pp 537-543 - 9TH WORLD ROUND TABLE ON SINTERING (IX WRTCS); BELGRADE, YUGOSLAVIA. SEPTEMBER 1-4, 1998

Mechanical alloying of Cu+3wt.% Al₂O₃ powder mixture and milling of pure and prealloyed copper powder with 3.5wt.%Al (CuAl_{3.5}) have been performed in planetary ball mill in air up to 2oh. All powders have been then treated in H-2 at 400 degrees C for 1h, in order to eliminate formed oxides. Hot pressing was used for compaction (800 degrees C, 3h, Ar, 35 MPa). The copper matrix strengthening was estimated by microhardness measurements. The microhardness of pure copper powder and prealloyed CuAl_{3.5} powder showed 5 times, and Cu+3% Al₂O₃ powder mixture 4 times greater values respectively, in comparison to the pure untreated copper powder. The strengthening effect in Cu compacts is due to grain refinement and increased dislocation density. The hardness of CuAl_{3.5} powder and Cu+3% Al₂O₃ powder mixture is additionally influenced by of Al₂O₃ dispersoids presence.

[66] MECHANICAL ACTIVATION AS INITIATION OF COAL SELF-IGNITION

J Medek, Z Weishauptova - PROSPECTS FOR COAL SCIENCE IN THE 21ST CENTURY, 1999, pp 495-498 - 10TH INTERNATIONAL CONFERENCE ON COAL SCIENCE; TAIYUAN, PEOPLES R CHINA. SEPTEMBER 12-17, 1999

A new theory of the self-ignition of coal was developed, based on the principle of the spontaneous disintegration of coal resulting from the effect of potential energy which is accumulated in the coal mass and causes mechanical stress. This leads to the formation of microcracks where a part of the potential energy is dissipated to heat under formation of microfines as primary centres of burning.

[65] COAL LIQUEFACTION BY USING THE COMMUNUTED BINARY CATALYST OF METAL SULFIDES BY MECHANICAL MILLING

Y Kuriki, K Uchida, S Ohshima, M Yumura, F Ikazaki - PROSPECTS FOR COAL SCIENCE IN THE 21ST CENTURY, 1999, pp 839-842 - 10TH INTERNATIONAL CONFERENCE ON COAL SCIENCE; TAIYUAN, PEOPLES R CHINA. SEPTEMBER 12-17, 1999

Effect was investigated of binary metal sulfide comminuted in a media-agitation mill on hydrogenation of 1-methylnaphthalene and coal liquefaction of Tanito Harum Indonesia coal. Binary metal sulfide such as MoS₂-CoS and MoS₂-NiS was comminuted. The sample prepared by comminuting such binary metal sulfide had hydrogenation and coal liquefaction better than expected from those using comminuted individual metal sulfide, which indicates a synergistic effect in comminution.

[64] MECHANICAL ACTIVATION OF PFBC-ASHES FOR HOT FLUE GAS CLEANING

P Schulle, R Wolf, B Meyer - PROSPECTS FOR COAL SCIENCE IN THE 21ST CENTURY, 1999, pp 1409-1412 - 10TH INTERNATIONAL CONFERENCE ON COAL SCIENCE; TAIYUAN, PEOPLES R CHINA. SEPTEMBER 12-17, 1999

In this paper the investigations using mechanically activated ashes to reduce the alkalis in a hot flue gas, at high pressure up to 1.3 MPa and a temperature of 850 degrees C, will be reported. In a laboratory test program, the potential of several mechanically activated ashes, which are used as additives for the flue gas purification, was tested. The activated ashes were subjected to X-ray diffraction, thermal analysis and determination of the specific surface and adsorption heat. In the case of ashes with a high quartz content, the improvement in reactivity was particularly marked after mechanical activation, with a strong decrease in crystallinity and crystallite size accompanied by a slight increase in crystall lattice distortions. For ashes with higher contents of anhydrite and lime, mechanical activation mainly influence the adsorptive capacity. Besides these studies, a test facility was designed and constructed. In this test plant a simulated gas with the same composition of a PFBC combustion gas, can be produced. The gas cleaning process is carried out in a pressurized entrained flow reactor. The loaded ashes are removed from flue gas by using a back-pulsed ceramic filter candle. The alkali concentration of the ashes is determined by atomic absorption spectrometry. Experiments for alkali removal were carried out in a test facility under PFBC-conditions. The sodium removal efficiency of FBC-ashes could be increased by mechanically activation. Sodium capture efficiencies of activated ashes up to 95 % have been achieved.

[63] SHOCK COMPACTON AND STRENGTHENING OF NANOCRYSTALLINE NiAl

T Chen, NN Thadhani, JM Hampikian - SHOCK COMPRESSION OF CONDENSED MATTER - 1999, PTS 1 AND 2 (Series: AIP CONFERENCE PROCEEDINGS), 2000, Vol 505, pp 733-736 - 11TH CONFERENCE OF THE AMERICAN-PHYSICAL-SOCIETY-TOPICAL-GROUP ON SHOCK COMPRESSION OF CONDENSED MATTER; SNOWBIRD, UTAH. JUNE 27-JULY 2, 1999

The microstructural characteristics and strengthening mechanisms of shock-consolidated nanocrystalline B2-phase NiAl intermetallic are discussed in this paper. Nanophase NiAl powders prepared by mechanical alloying were shock consolidated at a peak pressure of 4-6 GPa, to approximate to 83% theoretical maximum density (T.M.D.). Characterization by transmission electron microscopy (TEM) revealed that the structure of the shock-consolidated sample was retained at the nanoscale. The compacts showed evidence of grain boundary dislocations, subgrains, distorted regions, and shear bands. Investigation of the relationship between microhardness and grain size for nanocrystalline stoichiometric intermetallic NiAl powder compacts, showed that the micro-hardness increased with decreasing grain size. The strengthening mechanism was observed to be consistent with the Hall-Petch behavior dominated by dislocation generation at grain boundary ledges.

REGULAR PAPERS

[62] SMALL ANGLE NEUTRON SCATTERING STUDIES OF THE CRYSTAL AND AMORPHOUS ZIRCONIUM-BASED ALLOYS

Al-Assiri MS. - Physics of Low-Dimensional Structures. 3-4:1-12, 2000

The melt spinning technique has been used to produce metastable crystalline phases of the parent Zr₃Ni, Zr₃Rh and Zr₃Pd samples. X-ray diffraction and small angle neutron scattering (SANS) have been used to investigate the atomic and large scale structural changes of such alloys. Mechanical Milling (MM) technique has been used to transform Zr₃Pd metastable crystalline phase to the amorphous phase. Such amorphous structure of the sample occurred after 8 hours of milling time. SANS measurements indicated that both the Zr₃Rh and Zr₃Pd samples have the same structure and they scatter more strongly than the Zr₃Ni sample. The SANS intensity I(Q) of the crystalline and the amorphous phases of such alloys can often be expressed as a power law of the scattering vector (Q(-D)). The values of D obtained lie in a range which suggests that the volume fractals is a useful concept for describing the inner structure of such alloys.

[61] NANOGANULATION PROCESS INTO MAGNETO-RESISTANT CO-CU ALLOY ON THE ROUTE OF BULK MECHANICAL ALLOYING

Aizawa T. Zhou C. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 285(1-2):1-7, 2000

Nanogranulation process is investigated on the route of our developed bulk mechanical alloying. Cu-Co binary system is employed to describe the alloying process from the elemental powder mixes into the homogenized solid solution and to understand the nano-sized precipitation of Co from the homogenized solid solution in the copper matrix for various annealing conditions. Size and morphology control of Co-particulate in the nanometer order in this

nanogranulation process has close relationship with the materials design of magneto-resistance and magnetic anisotropy in the nanogranular system. Various experimental validation is necessary to construct the nanomechanical methodology as a theoretical model to describe intrinsic properties to nanogranular materials.

[60] AN ELECTROCHEMICAL INVESTIGATION OF MECHANICAL ALLOYING OF MGNI-BASED HYDROGEN STORAGE ALLOYS

Jiang JJ. Gasik M. - Journal of Power Sources. 89(1):117-124, 2000

The electrochemical properties of amorphous MgNi-based hydrogen storage alloys synthesized by mechanical alloying (MA) were evaluated. The results show that these amorphous Mg₅₀Ni₅₀ alloys exhibit a higher discharge capacity and relatively good rate capacity at a suitable grinding time while their cycle life is very poor. In order to improve the cycle life, the surface of the amorphous Mg₅₀Ni₅₀ alloy was coated with Ti, Al and Zr in Spex 8000 mill/mixer and the coating effects were further investigated. Based on experimental results, two kinds of MgNi-based amorphous alloys are designed by substituting part of Mg in MgNi-based alloys by suitable elements. These alloys are then composed of four components. Thus, the cycle life of electrodes consisting of these quaternary amorphous alloys is greatly improved.

[59] DEPENDENCE OF THE MAGNETIC MOMENT OF IRON NITRIDES ON THE PRESENCE OF DEFECT PHASE
Lancok A. Zaveta K. Vratislav S. Bezdzicka P. Paseka I. - Journal of Magnetism & Magnetic Materials. 215(Special Issue SI):30-32, 2000

The samples were prepared by nitriding iron powder in a mixture of H₂ and NH₃ at 700 degrees C, quenching to liquid N₂, milling, and annealing at 130-150 degrees C. They contained alpha Fe, gamma FeN_x, alpha'FeN_x, alpha " Fe₁₆N₂, and the defect phase. Their content was identified by XRD, neutron diffraction, and Mossbauer spectroscopy. The magnetic moments measured in SQUID magnetometer were compared with those evaluated from the Mossbauer spectra.

[58] THE STRUCTURE AND MAGNETIC PROPERTIES OF A POWDER FeCuNbSiB MATERIAL

Kollar P. Kovac J. Fuzer J. Sovak P. Pancurakova E. Konc M. - Journal of Magnetism & Magnetic Materials. 215(Special Issue SI):560-562, 2000

We investigated the influence of milling on the structure and magnetic properties of Fe_{73.5}Cu₁Nb₃Si_{13.5}B₉ alloy as a function of milling time. We found that with an increase of milling time the size of the powder decreases and both the coercivity and the volume fraction of the crystalline phase increase.

[57] HYDROGEN ELECTROSORPTION IN NANOCRYSTALLINE TI-BASED ALLOYS

Roue L. Guay D. Schulz R. - Journal of Electroanalytical Chemistry. 480(1-2):64-73, 2000

The electrochemical behavior in alkaline solution (1 M NaOH) of nanocrystalline Ti:Ru:Fe:O (2:1:1:2) prepared by high-energy ball milling was studied over its whole electroactivity domain, with a particular emphasis on the hydrogen evolution reaction (her). Comparison has also been made with nanocrystalline Ti:Ru:Fe (2:1:1) and a mixture of Ti:TiO:Ru:Fe₂O₃ (3/2:1/2:1:1/2). It was shown by cyclic voltammetry, open circuit potential decay and chronopotentiometry measurements that hydrogen absorption in the electrode material occurs during hydrogen discharge. The electrochemical behavior of nanocrystalline Ti:Ru:Fe:O (2:1:1:2) closely follows that of Ti:Ru:Fe (2:1:1), but differs radically from that of Ti:TiO:Ru:Fe₂O₃ (3/2:1/2:1:1/2). This is due to the fact that the former two compounds contain a significant fraction of B₂ phase (59 and 97 wt.%, respectively), while the latter does not. In steady state conditions, the ratio H/B₂ phase in nanocrystalline Ti:Ru:Fe:O (2:1:1:2) is 0.15, about 1.6 times less than that for the O-free nanocrystalline compound. The coefficient of diffusion of hydrogen in nanocrystalline Ti:Ru:Fe:O (2:1:1:2) is $2.6 \times 10^{-13} \text{ cm}^2 \text{ s}^{-1}$, more than three times less than that in nanocrystalline Ti:Ru:Fe (2:1:1). The difference between the hydrogen absorption characteristics of both nanocrystalline compounds are tracked down to the fact that their B₂ phases have different stoichiometries.

[56] EFFECTS OF TI ON THE CYCLE LIFE OF AMORPHOUS MGNI-BASED ALLOY PREPARED BY BALL MILLING

Han SC. Lee PS. Lee JY. Zuttel A. Schlapbach L. - Journal of Alloys & Compounds. 306(1-2):219-226, 2000

Amorphous MgNi alloys have a critical problem of poor cyclic behavior in spite of their high capacity for the negative electrode of a Ni/MH rechargeable battery. In order to improve the cycle life of the alloys, the degradation mechanism of amorphous MgNi alloy is suppressed. It is found that the surface property of the MgNi alloy is a critical factor for improvement of cycle life because an increase of the thickness of the magnesium oxide layer on the alloy surface results in a decrease of the discharge capacity of the alloy. To prevent further oxidation of magnesium, both titanium surface coating and substitution methods are adopted. Ti substitution is found to be very effective for improving the cycle life while Ti surface coating proves to be hardly effective. Magnesium in the Mg₅₀Ni₅₀ alloy is replaced by titanium. The amount of titanium (x in Mg_{1-x}Ti_xNi_{1.0}) is varied from 0.1 to 0.3. In the case of x = 0.3 (Mg_{0.7}Ti_{0.3}Ni_{1.0}), the alloy shows the best cycle life. To explain this phenomenon, both electrochemical and phenomenological analyses are conducted. Electrochemical impedance spectroscopy (EIS) analysis shows that the charge transfer resistance (R_{ct}) between the Mg_{0.7}Ti_{0.3}Ni_{1.0} alloy and the electrolyte does not increase during charge-discharge cycles. It is also found by Auger electron spectroscopy (AES) that the thickness of the surface oxide layer on the Mg_{0.7}Ti_{0.3}Ni_{1.0} alloy is thinner than that of the oxide layer on the MgNi alloy. At the same time, a nickel-enriched layer is found on the surface of the Mg_{0.7}Ti_{0.3}Ni_{1.0} alloy. X-ray photoelectron spectroscopy (XPS) also shows that magnesium in the Mg_{0.7}Ti_{0.3}Ni_{1.0} alloy exists in metallic state while titanium forms an oxide layer. These results indicate that the titanium oxide layer not only prevents further oxidation of magnesium; but also induces a nickel enriched layer on the alloy surface.

[55] NANOCRYSTALLINE NiSi ALLOY AS AN ANODE MATERIAL FOR LITHIUM-ION BATTERIES

Wang GX. Sun L. Bradhurst DH. Zhong S. Dou SX. Liu HK. - Journal of Alloys & Compounds. 306(1-2):249-252, 2000

Nanocrystalline intermetallic NiSi alloy powders were prepared by high energy ball-milling. A high lithium storage capacity of 1180 mA h g⁻¹ (7050 mA h ml⁻¹) has been achieved for a NiSi electrode in the initial discharge. During the reaction of lithium with the nanocrystalline NiSi alloy electrode, Si atoms acts as active centers, which

react with Li to form Li₂Si alloys. It is demonstrated for the first time that intermetallic alloys with Si as the active element can be used as an anode material for lithium-ion batteries.

[54] STRONG UNI-DIRECTIONAL ANISOTROPY IN DISORDERED NiFe₂O₄

Shi Y. Ding J. Shen ZX. Sun WX. Wang L. - Solid State Communications. 115(5):237-241, 2000
High-energy mechanical milling of spinel NiFe₂O₄ leads to the formation of a disordered wustite-like structure. Cluster glass behavior was found in the Mossbauer study. The investigation suggested ferrimagnetic clusters in an antiferromagnetic matrix. The ferrimagnetic and antiferromagnetic exchange coupling results in a strong uni-directional anisotropy and a coercivity of over 10 kOe after magnetic cooling.

[53] STRUCTURAL AND MAGNETIC PROPERTIES OF LEACHED BCC Ni PRODUCED BY ROD MILLING

Kim HG. Moon JC. Myung WN. - Journal of the Korean Physical Society. 36(6):449-452, 2000
Nanocrystalline Al-60 Ni-40 and Ni have been obtained by rod milling Al and Ni powder mixtures and chemically leaching Al atoms from rod-milled Al₆₀Ni₄₀. The rod-milled alloy powders retained their bcc structure after being treated at 85 degrees C with a 30 wt% KOH solution. On cooling of the specimen from 600 degrees C, spontaneous magnetization sharply increased at about 350 degrees C, indicating that the bcc phase was transformed to fcc phase. DC magnetic susceptibility measurements showed a single transition from the paramagnetic to the spin glass state below 45 K (T-f) The temperature behavior at T-f was quite different from field cooling (FC) and zero-held cooling (ZFC).

[52] SYNTHESIS OF PURE MONOLITHIC CALCIUM, STRONTIUM, AND BARIUM HEXALUMINATES FOR CATALYTIC APPLICATIONS

Di Filippo L. Lucchini E. Sergio V. Maschio S. - Journal of the American Ceramic Society. 83(6):1524-1526, 2000
Calcium, strontium, and barium hexaluminates have been prepared via a chemical route. In light of their interest as catalytic substrates the relation between attrition-milling time and density was studied, with the scope of obtaining fired bodies with suitable pore structure. It was found that all hexaluminates grew in elongated structures; longer milling times resulted in higher aspect ratios for calcium hexaluminate, with less than 75% of the theoretical density. Strontium and barium hexaluminates were prepared with even lower densities, but the reason is the inherent poor densification, and not the growth of mutually interfering elongated structures, as in the case for calcium hexaluminate.

[51] MECHANICAL ALLOYING OF MG-33AT.% Ni UNDER HYDROGEN ATMOSPHERE

Wang AM. Ding BZ. Zhang HF. Hu ZQ. - Journal of Materials Science Letters. 19(12):1089-1091, 2000

[50] ELABORATION OF THE Cu₃Si COMPOUND USING A MECHANICALLY ACTIVATED ANNEALING PROCESS

Souha H. Gaffet E. Bernard F. Niepce JC. - Journal of Materials Science. 35(13):3221-3226, 2000
The mechanically activated annealing process were used to produce Cu₃Si compound. This process results from the combination of two steps, the first is a mechanical activation of the 3Cu + Si powders mixture, the second consists of the annealing of as-milled powders. Based on X-ray diffraction experiment, scanning electron microscopy, the as-milled powders and M2AP end-products were characterized. Various process controlling parameters such as mechanical activation conditions were studied. In the same time, a study of the reactivity of Cu₃Si towards CuCl were performed to compare the M2AP end-products behavior with that of the Cu₃Si reference powder.

[49] THE INFLUENCE OF ALUMINA DISTRIBUTIONS UPON SCAVENGING HIGHLY RESISTIVE GRAIN-BOUNDARY PHASE OF 8 MOL% YTTRIA-STABILIZED ZIRCONIA

Lee JH. Mori T. Li JG. Ikegami T. Komatsu M. Haneda H. - Electrochemistry. 68(6):427-432, 2000
The effect of concentration, size, and distribution of Al₂O₃ upon scavenging siliceous phase at grain boundary, was studied in 8 mol% yttria-stabilized zirconia (8YSZ) containing 0.2-1 mol% Al₂O₃ prepared from ball milling and ultrasonic dispersion. The grain-boundary resistivity at 400 degrees C decreased prominently with adding Al₂O₃ to 8YSZ, which reflects the scavenging of siliceous grain-boundary phase. The uniform distribution of fine Al₂O₃ particles (0.3 μm) was more beneficial than that of coarse one (4-10 μm). However, the difference was not significant when the density is high, that is, when the connectivity between grain boundaries is good enough. As density decreases, the uniform distribution of fine Al₂O₃ particles becomes more important to decrease the grain-boundary resistivity. It explains that the scavenging reaction is assisted by rapid grain-boundary diffusion. From the Si-28(-) concentration map using imaging scanning secondary ion mass spectroscopy (SIMS), it was proved that the coarse Al₂O₃ scavenges the siliceous phase mainly by interfacial reaction.

[48] EFFECT OF PROCESS PARAMETERS ON THE ELECTROCHEMICAL ACTIVITY OF C₈CrO₃ ELECTRODE IN LITHIUM INTERCALATION IN ORGANIC ELECTROLYTES

Ol'shanskaya LN. Popova SS. - Russian Journal of Applied Chemistry. 73(1):59-62, 2000
The effect of the dispersity of active material components and the time of their mechanical mixing, grinding, and homogenization with ultrasound on the electrochemical activity of the C₈CrO₃ electrode in a solution of LiClO₄ in a 1:1 mixture of propylene carbonate and dimethoxyethane was studied.

[47] NITROGEN-INDUCED POWDER FORMATION OF TITANIUM ALUMINIDES DURING MECHANICAL ALLOYING

Hashimoto H. Abe T. Sun ZM. - Intermetallics. 8(7):721-728, 2000
The authors formerly found that introduction of nitrogen gas into milling atmosphere improved drastically production yield of gamma-based TiAl powder by mechanical alloying of a powder mix of Ti and Al. In this report, the role of nitrogen gas on the improvement in production yield of gamma-based TiAl powder was investigated by three series of milling experiments. As a result, the authors found that. (1) nitrogen gas affects the formation of TiAl powder after milling in argon for a certain period; (2) TiAl powder formed by mechanical alloying has the crystal structure of Al supersaturated alpha-Ti phase; (3) the content of nitrogen gas in the milling atmosphere has a great influence on the yield; (4) concerning the mechanism, it is presumed that a layer of Al supersaturated alpha-phase was formed during milling in argon, and transformed into a solid-solution with nitrogen during milling under the existence of nitrogen gas, and thereby became brittle and was broken into the fine powder.

[46] HYDROGEN ABSORPTION PROPERTIES OF A ZR-AL ALLOY BALL-MILLED WITH NI POWDER

Lee SM. Park YJ. Lee HY. Kim KC. Baik HK. - *Intermetallics*. 8(7):781-784, 2000 Jul.

The preparation of the Zr 84 wt%-Al 16 wt% non-evaporable getter alloy by means of mechanical alloying and its hydrogen absorption characteristics were investigated. Scanning electron microscopy and energy dispersive X-ray analysis revealed that the mechanical ball-milling with Ni was successfully employed to coated nickel particles on the surfaces of the Zr-Al getter alloy. The resulting composite particles with pure nickel on the surface of the Zr-Al getter compound show good gettering performance and fast sorption kinetics without any activation process.

[45] HARDENING AND SOFTENING OF FEAL DURING MILLING AND ANNEALING

Amils X. Noguees J. Surinach S. Baro MD. Munoz-Morris MA. Morris DG. - *Intermetallics*. 8(7):805-813, 2000

Changes in hardness of Fe-40Al powders during milling and during subsequent annealing have been examined and related to the many structural changes occurring. During milling, the material becomes significantly disordered with a small domain size and many vacancies, and refines to a nano-scale grain size, while on subsequent annealing at progressively higher temperatures these structural defects are lost as the material re-orders, loses point defects and as the grains grow to large sizes. The increase in hardness during milling can be explained by the combined contributions of vacancy hardening, ordered domain/particle hardening, and by disorder hardening within the ordered regions. Softening during annealing occurs as the domain/particle hardening, disorder hardening and vacancy hardening are successively lost. Dislocation-induced work hardening and grain size hardening are believed to play only minor roles in affecting the material hardness.

[44] FABRICATION OF A THICK SURFACE LAYER OF AL₃Ti ON Ti SUBSTRATE BY REACTIVE-PULSED ELECTRIC CURRENT SINTERING

Matsubara T. Shibutani T. Uenishi K. Kobayashi KF. - *Intermetallics*. 8(7):815-822, 2000

The thick intermetallic compound Al₃Ti layer was formed by reactive-pulsed electric current sintering (PECS) of mechanically alloyed powders set on the Ti substrate to improve the wear and oxidation properties. During heating by PECS, Al and Ti in MA powders reacted to form an Al₃Ti layer, and simultaneously reacted with the Ti substrate to achieve joining between the surface layer and substrate. The densification behavior of the synthesized surface layer, and the reaction kinetics between the surface layer and substrate were investigated. By holding at 1100 K for 180 s under 40 MPa, a fully dense and homogeneous Al₃Ti surface layer with a thickness of about 1600 μm was obtained. However, a higher temperature (1210 K) or longer holding time over 1800 s was required to eliminate voids on the Al₃Ti/Ti interface. The obtained Al₃Ti layer exhibited almost the same properties as cast bulk Al₃Ti for hardness, wear and oxidation.

[43] A MOSSBAUER STUDY OF MECHANICALLY ACTIVATED MGFE₂O₄

Sepelak V. Baabe D. Litterst FJ. Becker KD. - *Hyperfine Interactions*. 126(1-4):143-147, 2000

The structural and magnetic evolution in magnesium ferrite (MgFe₂O₄) caused by high-energy milling is investigated by Mossbauer spectroscopy.

[42] EFFECT OF MECHANICAL ACTIVATION ON THE SINTERING OF A TITANIUM-NICKEL ALLOY AND A BIOCERAMIC TINI COMPOSITION

Itin VI. Terekhova OG. Ul'yanova TE. Kostikova VA. Shevchenko NA. Berdnikova DV. - *Technical Physics Letters*. 26(5):436-438, 2000

Mechanical activation strongly influences the sintering of pressed articles made of a powdered titanium-nickel alloy and its compositions with dental porcelain. Preliminary treatment of the powdered mixtures in a planetary ball mill suppresses the martensite transformation and removes anisotropy in the variation of linear dimensions of the pressings. The resulting samples exhibit, instead of a volume expansion related to the rupture of contacts between particles in the course of the reverse martensite transformation, a sintering-induced shrinkage whose extent increases with the sintering temperature and the duration of mechanical activation treatment.

[41] OPTIMIZATION OF CALCINATION CONDITIONS ON THE Bi-2223 KINETIC FORMATION AND GRAIN SIZE

Garnier V. Monot I. Desgardin G. - *Superconductor Science & Technology*. 13(5):602-611, 2000

The effects of the calcination conditions (time, temperature, intermediate milling) on the kinetics of formation of Bi-2223 and on the grain size were studied using a powder precursor synthesized by the polymer matrix method. The samples were characterized by XRD and SEM analysis. The grain size and the kinetics of formation of Bi-2223 strongly depend on the calcination conditions and thus on the phase assemblages formed at the end of the calcination that determines the reactivity during sintering. The higher the calcination temperature is the larger the grain size. We have observed that 24 h of calcination at 820 degrees C without intermediate milling allows one to obtain 79% of Bi-2223 phase in 60 h of sintering at 835 degrees C.

[40] FIELD AND TEMPERATURE DEPENDENCE OF MAGNETIZATION IN FECU-BASED AMORPHOUS ALLOYS

Crespo P. Multigner M. Castano FJ. Casero R. Hernando A. Escorial AG. Schultz L. Kaul SN. - *Physical Review B*. 61(21):14346-14349, 2000

In this paper, the production of FeCu-based FeCuZr amorphous alloys by ball milling is reported. The thermal dependence of magnetization for the (Fe_{0.5}Cu_{0.5})(85)Zr-15 (at. %) amorphous alloy has been found to show a dramatic field dependence of the kink point of the magnetization. This kink corresponds to a temperature different from the Curie temperature, above 400 K, of the ferromagnetic phase, which, according to spin waves fitting, can be induced by applying external fields. Just above 235 K, the thermoremanence increases sharply, and this feature strongly suggests an increase of the ferromagnetic ordering under zero field heating. Neutron diffraction experiments seem to confirm the enhancement of spin alignment. The thermal expansion above the compensation temperature is proposed to be the origin of the thermoremanence enhancement through the anti-Invar effect as might be explained within the framework of recent ab initio calculations [M. van Schilfgarde et al., *Nature* (London) 400, 46 (1999)].

[39] MECHANICALLY INDUCED OXIDATION OF SRFeO₃-DELTA

Schmidt M. - *Materials Research Bulletin*. 35(2):169-175, 2000

The nonstoichiometric strontium-iron oxide SrFeO₃-delta (2.5 less than or equal to 3 - delta less than or equal to 3.0)

exhibits richness of equilibrium states over a broad range of temperatures and oxygen partial pressures. Its oxidation induced by ball milling was investigated. The mechanical treatment of the material at room temperature in oxygen atmosphere was found to cause the reaction. However, the product differs from compounds obtained by annealing, due to crystal imperfections introduced by milling: The changes in the oxygen stoichiometry, the crystal structure, and the morphology of the powder are discussed.

[38] SIZE EFFECT AND GAS SENSING CHARACTERISTICS OF NANOCRYSTALLINE $x\text{SnO}(2)-(1-x)\alpha\text{-Fe}_2\text{O}_3$ ETHANOL SENSORS

Tan OK. Zhu W. Yan Q. Kong LB. - *Sensors & Actuators B-Chemical*. 65(1-3):361-365, 2000

Non-equilibrium nanocrystalline $x\text{SnO}(2)-(1-x)\alpha\text{-Fe}_2\text{O}_3$ powders have been prepared using the mechanical alloying technique. The thick film screen printing technology is then employed to fabricate these ethanol gas sensors. Their particle size and structural properties are systematically characterized using X-ray diffraction (XRD) and transmission electron microscopy (TEM). The gas sensing characteristics are also measured. Based on the experimental results, we have observed that particle size of the powders is drastically milled down to about 10 nm after 24 h of high-energy milling. A very high gas sensitivity value of 845 for 1000 ppm of ethanol gas in air has been obtained. Our proposed new structural model for these non-equilibrium nanocrystalline $x\text{SnO}(2)-(1-x)\alpha\text{-Fe}_2\text{O}_3$ materials explains both the lattice expansion of these high energy mechanically alloyed powders as well as the charge neutrality in terms of additional oxygen dangling bonds at the nano-sized particle surfaces. It is those enormous oxygen-dangling bonds at the particle surfaces that give rise to the high gas sensitivity. The sensors are found to be 32.5 times more selective to the ethanol gas compared to CO and H₂ gases.

[37] RECOMBINATION OF THE $\text{Nd}_2\text{Fe}_{14}\text{B}$ PHASE AFTER REACTIVE MILLING UNDER HYDROGEN OF A Nd-Fe-B POWDER

-Khelifati G. Le Breton JM. Aymard L. Teillet J. - *Journal of Magnetism & Magnetic Materials*. 218(1):42-48, 2000
Mechanical milling under reactive hydrogen atmosphere of a Nd-Fe-B powder, followed by vacuum annealing up to 600 degrees C, was achieved. Both as-milled and annealed powders were investigated by X-ray diffraction and Mossbauer spectrometry. During reactive milling, the $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase disproportionates into neodymium hydrides ($\text{NdH}_{2+/-x}$) poorly crystallized $\alpha\text{-Fe}$ and an amorphous Fe-B phase. Upon annealing, hydrogen desorbs from the neodymium hydride and this is followed by the recombination of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase. The recombination temperature was found to be 520 degrees C. The recombination reaction is incomplete, likely due to oxygen contamination, as neodymium oxides, $\alpha\text{-Fe}$ and $\text{Nd}_1.1\text{Fe}_4\text{B}_4$ phases are detected in the powder annealed at 600 degrees C.

[36] MICROSTRUCTURE AND MAGNETIC PROPERTIES OF MECHANICALLY MILLED NANOGRAINED $\text{Pr}_x\text{Co}_{100-x}$ ($x=15.4-20.5$) POWDERS

Chen ZM. Zhang Y. Hadjipanayis GC. - *Journal of Applied Physics*. 88(3):1547-1551, 2000

The structure, microstructure, and magnetic properties of high-coercivity PrCo₅-based Pr_xCo_{100-x} ($x=15.4-20.5$) powders synthesized by mechanical milling and subsequent annealing were systematically studied as a function of Pr content. While the magnetization decreases monotonously with the Pr content, the coercivity increases, reaching a maximum of 24.1 kOe in Pr₁₉Co₈₁, and then decreases for higher Pr content. As a result, the maximum energy product goes through a broad peak of about 11.3-11.8 MGOe at the 16.7-18.0 at. % Pr content. Microstructural studies reveal that a uniform nanoscale PrCo₅/Pr₂Co₁₇ microstructure with an average grain size of about 15-30 nm is developed in powders with Pr content up to 19 at. %. The volume fraction of the Pr₂Co₁₇ decreases with Pr content and a nearly single PrCo₅ structure is obtained in Pr₁₉Co₈₁ powders. Further increase in the Pr content leads to the presence of the less-hard Pr₂Co₇ phase in the form of large grains, resulting in lower coercivities. Evidence of intergranular exchange coupling is found in all the samples. The results suggest that the observed magnetic hardening originates from the high anisotropy field of the PrCo₅ phase and the uniform nanoscale microstructure developed by the processing used.

[35] EFFECT OF PAN-MILLING STRESS ON CRYSTAL STRUCTURES OF HIGH DENSITY POLYETHYLENE

Huang H. - *Chinese Journal of Polymer Science*. 18(4):363-367, 2000

A detailed study was performed on the crystal structures of pan-milled high-density polyethylene (HDPE) using differential scanning calorimetry (DSC) and X-ray diffraction. The crystallinity of HDPE first decreased slightly, followed by a gradual increase with increasing milling times. Monoclinic crystals appeared after 4 cycles of milling. With increasing times of milling, the proportion of monoclinic crystals increased significantly while the proportion of orthorhombic crystals decreased gradually. With increasing times of milling, the crystallite size of orthorhombic form decreased greatly, while the size of monoclinic crystallites kept almost constant during milling.

[34] SOLID-PHASE SYNTHESIS OF IRON(III) ACETYLACETONATE UPON MECHANICAL ACTIVATION [RUSSIAN]

Petrova LA. Borisov AP. Aleshin VV. Makhaev VD. - *Zhurnal Neorganicheskoi Khimii*. 45(3):390-394, 2000

[33] ELECTRO-DISCHARGE CONSOLIDATION APPLIED TO NANOCRYSTALLINE AND RSP/MA POWDERS

Okazaki K. - *Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing*. 287(2):189-197, 2000

Electro-discharge consolidation (EDC) was applied to consolidate nanocrystalline intermetallic powders and RSP Al alloy powders. Because EDC employs a high-voltage, high-density current pulse discharged from a capacitor bank to powders under external pressure, one can take advantage of the basic characteristics of EDC to preserve the microstructure inherent in the starting powder, remove oxide films on the prior powder particle surface to enhance the bonding, densify to the bulk and thereby improve the mechanical properties. Mechanically alloyed Nb-23 at.% Al was consolidated to a density of 99% theoretical, and the nanocrystalline state (less than 35 nm grain size) was preserved. Because of such a small grain size, the EDC consolidates exhibit a negative Hall-petch relation. Another example is presented for RSP and MA powders of an Al alloy, in which the magnitude of ductilities exhibited by EDC bulks clearly indicates that the oxide film on the powder particle surface has been completely removed.

[32] MECHANICAL ACTIVATION-ASSISTED SYNTHESIS OF $\text{Pb}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3$

Ang SK. Wang J. Wan DM. Xue JM. Li LT. - Journal of the American Ceramic Society. 83(7):1575-1580, 2000
Perovskite $\text{Pb}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3$ (PFW) was prepared via a mechanical activation-assisted synthesis route from mixed oxides of PbO , Fe_2O_3 , and WO_3 . The mechanically activated oxide mixture, which exhibited a specific area of $>10 \text{ m}^2/\text{g}$, underwent phase conversion from nanocrystalline lead tungstate (PbWO_4) and pyrochlore ($\text{Ph}_2\text{FeWO}_6.5$) phases on sintering to yield perovskite PFW, although the formation of perovskite phase was not triggered by mechanical activation. When heated to 700 degrees C, $>98\%$ perovskite phase was formed in the mechanically activated oxide mixture. The perovskite phase was sintered to a density of similar to 99% of theoretical density at 870 degrees C for 2 h. The sintered PFW exhibited a dielectric constant of 9800 at 10 kHz, which was similar to 30% higher than that of the PFW derived from the oxide mixture that was not subjected to mechanical activation.

[31] EFFECTS OF DRY GRINDING ON THE STRUCTURAL CHANGES OF KAOLINITE POWDERS

Sanchez-Soto PJ. de Haro MDJ. Perez-Maqueda LA. Varona I. - Perez-Rodriguez JL. - Journal of the American Ceramic Society. 83(7):1649-1657, 2000

The present study examined the effects of dry grinding, using ball-milling, on the structure of reference well-crystallized (KGa-1) and poorly crystallized (KGa-2) kaolinite powders from Georgia. Grinding produced a strong structural alteration, mainly along the c axis, resulting in disorder and total degradation of the crystal structure of the kaolinite and the formation of an amorphous product. The surface area increased with grinding time, mainly in KGa-2 (maximum value $50.27 \text{ m}^2/\text{g}$), a result associated with particle-size reduction. These particles became more agglomerated with grinding, and the surface area decreased after 30 min, as confirmed by scanning electron microscopy and particle-size-distribution analysis. There was a limit to particle-size reduction with grinding time. When grinding time was increased, the original endothermic differential thermal analysis (DTA) effects of dehydroxylation in both samples shifted to lower temperatures, decreased in intensity, then disappeared completely after 120 min of grinding. The temperature of the characteristic first exothermic effect shifted slightly to lower temperatures with grinding, although the DTA effects did not increase with grinding time in either kaolinite sample, at least up to 325 min. The amorphous, mechanically activated kaolinite converted into low-crystalline mullite nuclei at a lower temperature than did the unground samples, as deduced by thermal and X-ray observations. This effect was especially important for the KGa-2 sample. Grinding did not seem to influence the formation of silicon-aluminum spinel from kaolinite. The present results may explain why ground kaolinite samples prepared via different routes-e.g. with differences in grinding-behave differently during high-temperature transformations, as reported in the related literature.

[30] STRUCTURAL AND MAGNETIC PROPERTIES OF ULTRAFINE $\text{La}_{0.7}\text{Ca}_{0.3}\text{MnO}_3$ POWDERS PREPARED BY MECHANICAL ALLOYING

Muroi M. Street R. McCormick PG. - Journal of Solid State Chemistry. 152(2):503-510, 2000

Ultrafine $\text{La}_{0.7}\text{Ca}_{0.3}\text{MnO}_3$ powders with controlled oxygen stoichiometry have been synthesized by mechanical alloying at ambient temperature. It is found that high-energy ball milling of the starting materials, La_2O_3 , CaO , MnO_2 , and Mn_3O_4 mixed in the stoichiometric cation ratio, yields single-phase $\text{La}_{0.7}\text{Ca}_{0.3}\text{MnO}_3$ powders having crystallite sizes of about 10 nm and various oxygen content (2.68 less than or equal to z less than or equal to 3.35), adjustable by changing the $\text{MnO}_2/\text{Mn}_3\text{O}_4$ ratio. Magnetic measurements show that the spontaneous magnetization (M-s) of the as-milled powder depends less on the nominal Mn valence ($\nu(\text{Mn})$) than in bulk $(\text{La,Ca})\text{MnO}_3$ and that the maximum M-s, observed for $\nu(\text{Mn})$ similar to 3.3, is much smaller. Annealing in air at temperatures above 500 degrees C increases M-s for all the samples, but a marked increase in M, toward the bulk value (similar to 90 emu/g) occurs in a lower temperature range for lower $\nu(\text{Mn})$ than for higher $\nu(\text{Mn})$. These observations are discussed in terms of magnetic disorder resulting from defects induced by high-energy milling and surface effects dominant in small crystals.

[29] MICROSTRUCTURAL MODELLING OF NANOSTRUCTURED FLUORIDE POWDERS PREPARED BY MECHANICAL MILLING

Guerault H. Greneche JM. - Journal of Physics-Condensed Matter. 12(22):4791-4798, 2000

Nanostructured fluoride powders were prepared by the high-energy ball milling route. A combination of suitable techniques with complementary spatial scales was used for the first time to investigate both structural and microstructural properties: x-ray diffractometry, magnetic measurements and local probes such as Fe-57 Mossbauer spectrometry and F-19, Ga-69 and Ga-71 NMR. The set of data allows us to describe these nanostructured powders on the basis of nanocrystalline grains and grain boundaries. The relevant hyperfine data support then modelling of the microstructure in terms of pseudo-cubic and random packing of corner sharing octahedral units corresponding to nanocrystalline grains (similar to 15 nm diameter) and disordered grain boundaries (a few nanometres thick), respectively. Their different cationic topologies are consistent with the antiferromagnetic and speromagnetic behaviours, respectively, as evidenced by in-field Mossbauer spectrometry. They also support the temperature dependence of the magnetic properties which reveal a progressive magnetic decoupling of grains when the temperature increases, originating a superparamagnetic behaviour above a blocking temperature which is dependent on the thickness of the grain boundaries, i.e. the milling conditions.

[28] IN SITU XAS AND XRD STUDIES ON THE FORMATION OF MO SUBOXIDES DURING REDUCTION OF MOO_3

Ressler T. Jentoft RE. Wienold J. Gunter MM. Timpe O. - Journal of Physical Chemistry B. 104(27):6360-6370, 2000

Reduction of MoO_3 with hydrogen (5-100 vol %) in the temperature range from 573 to 833 K was studied by in situ X-ray diffraction and X-ray absorption spectroscopy. The experiments performed focused on elucidating phase composition and evolution with time under isothermal as well as temperature-programmed reduction conditions. At reaction temperatures below 698 K, the reduction of MoO_3 to MoO_2 is a one-step process without the formation of crystalline intermediates. At reduction temperatures above 723 K and H₂ concentrations higher than 10 vol %, Mo metal is the final product of the reduction of MoO_3 . In addition, at temperatures higher than 698 K, the formation of Mo_4O_{11} was observed. However, Mo_4O_{11} is not an intermediate in the reduction of MoO_3 but is being formed in a

parallel reaction from MoO₃ and MoO₂ at temperatures above 698 K. It is shown that Mo₄O₁₁ can be obtained from a reaction of MoO₃ and MoO₂ at temperatures above 773 K, affording the same phase ratio of monoclinic and orthorhombic Mo₄O₁₁ as the reduction of MoO₃ with hydrogen. Quantitative XRD analysis reveals a sigmoidal shape of the evolution of the MoO₃ and MoO₂ phases during reduction of MoO₃ and an increase in the crystallite size of the phases present. This Oswald ripening indicates that a nucleation-growth kinetic mechanism governs the reduction of MoO₃ under the conditions studied. The results presented in this work clearly demonstrate the potential of a combined application of in situ XRD and XAFS to reveal phase composition and kinetics of solid-state reactions.

[27] PT-BASED NANOCOMPOSITES PRODUCED BY HIGH ENERGY BALL MILLING AS ELECTROCATALYSTS IN POLYMER ELECTROLYTE FUEL CELLS

Lalande G. Denis MC. Gouerec P. Guay D. Dodelet JP. Schulz R. - Journal of New Materials for Electrochemical Systems. 3(3):185-192, 2000

Ball milling of Pt powder with powders of WO₂ WO₃, MoO₂ or MoO₃ has been performed to synthesize CO-tolerant nanocomposite anode electrocatalysts for polymer electrolyte membrane fuel cells. In order to increase the specific surface area of the final products and to prevent sticking during milling MgH₂ was added to the powders as a dispersing agent. After milling, MgH₂ was leached away in IM HCl (lixiviation step). The specific surface areas of the new catalysts range from 12.4 to 33.5 m²/g. X-ray diffraction indicates that WO_x-based catalysts are true nanocomposites while MoO_x-based systems display only the Pt structure. Catalysts obtained by milling Pt+WO₃ are made of Pt nanocrystals and crystallites of WO₃ ·H₂O, H_{0.12}WO₃ ·2H₂O and H₂WO₄ ·H₂O, while catalysts obtained by milling Pt+WO₃ are made of Pt nanocrystals and crystallites of WO₃ and WO₃ ·H₂O. For the Pt+MoO_x systems, the ball milled Mo oxides decompose into Mo-based species and are leached away during the lixiviation step. X-ray Photoelectron Spectroscopy of Pt+MoO_x indicates that some Mo remains in these catalysts and that it is in solid solution into the Pt structure. In fuel cell tests with H₂ + 100 ppm CO at the anode and O₂ at the cathode, Pt+WO_x catalysts and commercial PtRu black display comparable CO-tolerance while Pt+MoO_x powders exhibit lower performances. Pt+WO₃ catalysts lack, however long term stability, their current density at 0.5V decreasing at about 3%/100 hours.

[26] FORMATION OF ACTIVE SITES FOR SELECTIVE TOLUENE OXIDATION DURING CATALYST SYNTHESIS VIA SOLID-STATE REACTION OF V₂O₅ WITH TiO₂

Bulushev DA. Kiwi-Minsker L. Zaikovskii VI. Renken A. - Journal of Catalysis. 193(1):145-153, 2000

Interaction of V₂O₅ with TiO₂ during the preparation of V/Ti-oxide catalysts via solid-state reaction has been studied by means of in situ FT-Raman spectroscopy, HRTEM and XPS. This interaction results in the formation of monomeric vanadia species with vanadium in tetrahedral coordination. The bridging oxygen in the V-O-Ti bond is suggested to be responsible for the catalytic activity during the partial oxidation of toluene. The formation of the monomeric vanadia species correlates with the improved catalyst performance, characterized by reaction rate and selectivity to benzaldehyde and benzoic acid. Mechanical activation by intensive grinding of V₂O₅/TiO₂ mixture via ball milling was necessary for the interaction of the oxides during the calcination. The monomeric species formation was observed at a temperature as low as 523 K. The dynamics of V₂O₅/TiO₂ interaction strongly depends on the presence of moisture during the calcination. In dry oxidative atmosphere, a part of the monomeric species is rapidly formed. Then, the process slows down and becomes diffusion-controlled. During the calcination in humid oxidative atmosphere, quick amorphization of bulk crystalline V₂O₅ was observed followed by slow formation of the monomeric vanadia species.

[25] PLASMA SPRAYING OF STAINLESS-STEEL PARTICLES COATED WITH AN ALUMINA SHELL

Ageorges H. Fauchais P. - Thin Solid Films. 370(1-2):213-222, 2000

The effect of an alumina coating, obtained by mechanofusion, on stainless-steel particles used in plasma spraying has been studied by examining sprayed particles in mid-flight and their resulting splats and coatings. The mean size of the injected powders is about 65 μm and the thickness of the alumina shell 4 μm. The results show that without preheating the substrate the splats of both types of powder are extensively fingered and become circular when the substrate surface is preheated over 200 degrees C. For the case of the stainless steel/alumina composite splats, Energy dispersive spectroscopy (EDS) analysis of the distribution of the various elements shows that the alumina is either spread exactly on the stainless-steel splat or is dispersed in pieces and frozen over the surface of the stainless-steel splat. The first case corresponds to well molten particles where, after their flight in the plasma jet, all the alumina shell has flowed to the tail of the particle; the second case is related to particles which have still an alumina shell uniformly distributed around the stainless-steel core. Finally, a composite stainless steel/alumina coating sprayed on a rough (R_a similar to 6.7 +/- 0.3 μm) stainless-steel substrate preheated to 400 degrees C is compared with a pure stainless-steel coating. Both hardness and cohesion are found to improve for the alumina-coated particles.

[24] SYNTHESIS AND SINTERINGS OF CHROMIUM BORIDES VIA SOLID-STATE REACTIONS AND MECHANOCHEMICAL PROCESSES OF CHROMIUM-AMORPHOUS BORON MIXED POWDER [JAPANESE]

Iizumi K. Kudaka K. - Nippon Kagaku Kaishi. (6):369-380, 2000

As a basic study for the development of chromium boride materials which are expected as refractory materials or hard materials, syntheses and sinterings of the chromium borides via solid state reactions and mechanochemical processes of chromium-amorphous boron mixed powders were studied. The single phases of Cr₂B, CrB and CrB₂ were obtained by the solid state reaction in the temperatures of 1400-1500 degrees C. These borides were so unsinterable that could be consolidated by pressure sintering only. The chromium boride of the highest relative density and hardness among the ceramics obtained was CrB₂. By mechanochemical processes, the single phases of CrB₂ and CrB were obtained by milling of Cr-B (molar ratio, 1 : 2A) and Cr-B(1 : 1) mixed powders by using a planetary ball mill for 20-40 h, followed by annealing at 900 and 1000 degrees C, respectively. As-milled Cr-B(1 : 1) mixed powders were consolidated by both of pressureless and pressure sinterings. These are a reaction sintering because the sinterings are accelerated by the exothermic reaction heat of the unreacted Cr-B in the as-milled mixed powders. The unsinterable powder could be consolidated by pressureless sintering via mechanochemical process. At pressure sintering via mechanochemical process, the sintering temperature lowered and the hardness of ceramics obtained increased.

[23] FACE-CENTERED-CUBIC TO HEXAGONAL-CLOSE-PACKED TRANSFORMATION IN NANOCRYSTALLINE NI(SI) BY MECHANICAL ALLOYING

Datta MK. Pabi SK. Murty BS. - Journal of Materials Research. 15(7):1429-1432, 2000

An allotropic transition from face-centered-cubic (fcc) to hexagonal-close-packed (hcp) Ni(Si) solid solution in Ni₉₅Si₅ and Ni₉₀Si₁₀ during nanocrystallization by mechanical alloying is reported. The transformation was identified as a defect-induced melting accompanied by a volume expansion of 8.6% and was observed when fine Ni(Si) reached a critical crystallite size of 10 nm. Calculation based on equation of state showed that a 37% reduction in tetragonal shear modulus and a negative pressure of about 8.7 GPa were generated at the onset of transformation.

[22] INVESTIGATION ON STRUCTURE TRANSITION OF FULLERENE DURING MECHANICAL ALLOYING AND SUBSEQUENT TREATMENTS

Liu ZG. Ohi H. Masuyama K. Tsuchiya K. Umemoto M. - Journal of Materials Research. 15(7):1528-1537, 2000

Mechanical milling of fullerene (soot containing C-60/C-70 fullerenes in a 8:2 molar ratio) was investigated through various characterization methods. It was found that mechanical milling would not destroy the molecular structure of fullerene C-60(C-70), while the long-range order of the face-centered-cubic crystalline structure was easily modified and transformed into amorphous phase, a mixture of fullerene C-60(C-70) polymers and monomers. Differential scanning calorimetry analysis revealed a recovery of polymers to pristine fullerene molecules at 678 K, which is much higher than the reported depolymerization temperature of fullerene polymers induced by photo irradiation and by high-pressure-temperature processes. It is suggested that the contaminated Fe acts as a catalyst in the polymerization process.

[21] STRUCTURAL CHARACTERIZATION OF CERIA-ZIRCONIA POWDER CATALYSTS PREPARED BY HIGH-ENERGY MECHANICAL MILLING: A NEUTRON DIFFRACTION STUDY

Enzo S. Delogu F. Frattini R. Primavera A. Trovarelli A. - Journal of Materials Research. 15(7):1538-1545, 2000

Neutron diffraction measurements were carried out on samples of CeO₂-ZrO₂ powder catalysts prepared by high-energy mechanical milling. The formation of solid solution was evidenced across the entire composition range examined. Quantitative phase evaluation by the Rietveld method indicated formation of tetragonal structure for low CeO₂ content, whereas cubic solid solutions were the stable form at high CeO₂ loading. In addition, a pseudocubic or tetragonal t " cell with axial ratio of unity and with internal deformation of the oxygen sublattice was observed at intermediate composition (50 mol% CeO₂). Thermal annealing up to 1000 degrees C showed expansion of the unit cell parameters; an increase in the degree of tetragonality at the expense of cubic and monoclinic phase was observed for composition C_xZr_{1-x}O₂ (x < 0.5).

[20] THE EFFECTS OF MECHANICAL ACTIVATION IN SYNTHESIZING ULTRAFINE BARIUM FERRITE POWDERS FROM CO-PRECIPIATED PRECURSORS

Liu X. Wang J. Ding J. Chen MS. Shen ZX. - Journal of Materials Chemistry. 10(7):1745-1749, 2000

Well dispersed and fine barium ferrite (BaFe₁₂O₁₉) powders have been successfully prepared by mechanically activating co-precipitated precursors, followed by calcination at 700 and 800 degrees C. When mechanically activated in a sodium chloride matrix for 20 hours, nanocrystallites of BaFe₁₂O₁₉, alpha-Fe₂O₃ and a spinel (gamma-Fe₂O₃) phase of < 10 nm in size were triggered in the co-precipitated precursor. Single phase barium ferrite platelets 50-100 nm in size were developed upon subsequent calcination at 800 degrees C for 1 hour. The resulting barium ferrite powder exhibits an intrinsic coercivity (H-i(c)) of 436.7 kA m(-1) and a saturation magnetization (M-s) of 67.8 A m(2) kg(-1). These magnetic properties compare favorably with those of the materials derived from conventional calcination of the co-precipitated precursor without prior mechanical activation, which led to very poor powder characteristics.

[19] BLENDING ADDITIONS OF COBALT TO ND₁₆FE₇₆B₈ MILLED POWDER TO PRODUCE SINTERED MAGNETS

Mottram RS. Williams AJ. Harris IR. - Journal of Magnetism & Magnetic Materials. 217(1-3):27-34, 2000

A blending process involving the mixing of powders of NdFeB and pure cobalt has been developed. This process has been shown to be an effective and simple way of adding cobalt to the composition. This allows the composition and hence properties of the finished magnets to be adjusted subsequent to the casting and milling of the basic alloy. After standard sintering treatments, the cobalt substitutes into the matrix phase, causing a linear increase in the Curie temperature of 11 degrees C per at% Co in the range 0-10 at% Co. As the amount of cobalt increases, so the remanence is improved, but there is a corresponding decrease in the coercivity. With increasing cobalt content, the proportion of grain boundary phase decreases and cobalt-containing phases are observed. The increase in remanence is attributed to the increased proportion of Nd-2(Fe, Co)(14)B and the decrease in coercivity is attributed to the reduced magnetic isolation of the grains and to the introduction of ferromagnetic, cobalt containing grain boundary phases.

[18] XPS SURFACE STUDY OF NANOCRYSTALLINE TI-RU-FE MATERIALS

Sunol JJ. Bonneau ME. Roue L. Guay D. Schulz R. - Applied Surface Science. 158(3-4):252-262, 2000

The surface properties of Ti:Ru:Fe (2 -x:1 + x/2:1 + x/2) (with x = 0, 0.25, 0.5, 0.75, and 1) and Ti:Ru:Fe:O (2:1:1:w) (with w = 0.0, 0.5, 1.0, 1.5, and 2.0) have been determined by X-ray photoelectron spectroscopy (XPS) in both their as-milled state and after being in contact with a chlorate oxidizing solution. The O surface concentrations of both sets of samples are almost identical, indicating that the O-free samples readily react with air. All samples in their as-milled state have an elemental Ti, Ru and Fe surface contents that closely follow that expected from their bulk composition, indicating that there is no surface enrichment in any of the elements. In the as-milled state, more than 90% of Ti and Fe atoms are in the highest possible oxidation state, while Ru is in the metallic state. Following immersion of the samples in an oxidizing chlorate electrolyte, the Ru surface concentration decreases by a factor of two. This is also accompanied by an increase in the oxidation state of the Ru atoms left at the surface from 0 to +4. From a comparison between the Ru 3d(5/2,3/2) core level spectra of the electrodes with those of crystalline and hydrated RuO₂, it is postulated that dissolution and re-deposition of Ru in the form of hydrated RuO₂ occurs at open circuit potential in the chlorate electrolyte. The consequences of these findings for the electrocatalytic activity of the

electrodes in chlorate electrolyte are finally discussed.

[17] STRUCTURAL CHANGES IN POLY(ETHYLENE TEREPHTHALATE) INDUCED BY MECHANICAL MILLING
Bai C. Spontak RJ. Koch CC. Saw CK. Balik CM. - Polymer. 41(19):7147-7157, 2000

Poly(ethylene terephthalate) (PET) has been subjected to high-energy ball milling (mechanical milling, MM) at three different temperatures. The resulting milled powder is characterized by molecular weight measurements, differential scanning calorimetry and wide-angle X-ray scattering. Regardless of the initial degree of crystallinity or milling temperature employed, MM apparently yields an "oriented amorphous" PET morphology in which the PET chains are locally aligned but rotationally disordered. This conclusion is based on the persistence of the (100) peak in otherwise amorphous x-ray patterns from milled PET. Thermograms of milled PET exhibit a small, broad crystallization exotherm and a large melting endotherm. The unusually small crystallization enthalpy is attributed to the local orientation of PET molecules present in the oriented amorphous morphology. Only minor rotations and translations of oriented PET molecules are needed to put the chains into register and, hence, the crystalline state. Evidence is also presented to suggest that extended-chain crystals of PET are produced upon crystallization of mechanically milled PET. **[16] MECHANOCHEMICAL TREATMENT OF MG-FERRITE POWDER [SPANISH]**

Rabanal ME. Varez A. Levenfeld B. Torralba JM. - Boletín de la Sociedad Española de Cerámica y Vidrio. 39(3):277-280, 2000

Powder of MgFe₂O₄, previously prepared by solid state reaction, was mechanochemically treated in a centrifugal ball mill in air atmosphere. Different milling conditions have been employed varying ball/powder mass ratio. After low milling time a strong reduction on crystallite size has been detected while the strain lattice increase slowly. The presence of alpha-Fe₂O₃ was also observed at different milling time, depending on milling conditions, by means of X-Ray diffraction experiments. Magnetic transition temperatures were evaluated using a thermogravimetric analyser, equipped with a small permanent magnet. An attempt of dependence of magnetic properties with crystallite size has been established.

[15] HIGH SPEED STEELS REINFORCED WITH CARBIDES BY MECHANICAL ALLOY [SPANISH]

Ruiz-Roman JM. Cambronero LEG. Suarez JC. Corpas F. Ruiz-Prieto JM. - Boletín de la Sociedad Española de Cerámica y Vidrio. 39(3):281-283, 2000

In this research work we have studied the efficiency of using an advanced technique, as mechanical alloying, on the metal matrix composites fabrication process by powder metallurgy. From the analysis of several composites, based on M2 high speed steel reinforced with WC, we inferred that mechanical alloying is an effective method to manufacture these materials, and they present higher mechanical properties than high alloy steels used in structural applications.

[14] MECHANOCHEMICAL SYNTHESIS OF LITHIUM-MANGANESE SPINEL, A CATHODE MATERIAL FOR LITHIUM POWER CELLS

Kosova NV. Uvarov NF. Devyatkina ET. Avvakumov EG. Solomentsev SY. - Russian Journal of Applied Chemistry. 73(3):436-440, 2000

Highly dispersed LiMn₂O₄ spinel was synthesized mechanochemically from various starting compounds of manganese (MnO₂, Mn₂O₃, MnO) and lithium (LiOH, LiOH · H₂O, Li₂CO₃). The effect of the oxidation state of manganese and the crystal structure and mechanical properties of the starting lithium compounds on the mechanism and kinetics of mechanochemical reactions of LiMn₂O₄ synthesis was studied. Also, the influence of the temperature of the subsequent annealing on the composition and lattice parameters of LiMn₂O₄ was investigated. The method of complex impedance was used to study the conductivity of the synthesized samples in relation to the nature of the starting lithium compounds, annealing temperature, and compaction pressure. The synthesized samples were tested as cathodes in an electrochemical cell.

[13] STRUCTURE AND MAGNETIC PROPERTIES OF MECHANOSYNTHESIZED IRON-TUNGSTEN ALLOYS

Jartych E. Zurawicz JK. Oleszak D. Pekala M. - Journal of Magnetism & Magnetic Materials. 218(2-3):247-255, 2000

X-ray diffraction, Mossbauer spectroscopy and magnetization measurements were used to study the mechanical alloying processes in the Fe-W system. Two solid solutions of W in Fe lattice [Fe(W)] and Fe in W lattice [W(Fe)] were produced during the early stage of the mechanical alloying. The results are inconsistent with those obtained by the calculations of phase diagram (CALPHAD) method. It was assumed that Vegard law and simple dilution model were approximately realized in the Fe-rich and W-rich regions. On the basis of this assumption and Mossbauer measurements, Fe(W) solid solution was recognized as a ferromagnetic phase while W(Fe) solid solution as a paramagnetic one. Nearest neighborhood of Fe-57 atoms was determined using the hyperfine interactions parameters.

[12] EFFECT OF CELLULOSE CRYSTALLINITY ON THE PROGRESS OF THERMAL OXIDATIVE DEGRADATION OF PAPER

Junior JLP. - Journal of Applied Polymer Science. 78(1):61-66, 2000

Dynamic differential scanning calorimetry (DSC) using paper samples of different compositions has evidenced varying degrees of endothermic activity prior to the exothermic, oxidative decomposition of cellulose under static air. Whereas cotton cellulose papers displayed a significant endothermic activity, wood-pulp papers showed a much less pronounced effect or no activity at all. DSC measurements using microcrystalline cellulose submitted to different extents of milling indicated that the observed endotherm is related to the degree of crystallinity of cellulose. Crystallinity decrease is accompanied by a decrease in the area of the endothermic peak. Thermal disruption of cellulose crystalline domains is therefore believed to be the reason for the appearance of an endotherm in the thermograms of paper. The higher degree of crystallinity of cotton cellulose in comparison to wood-pulp cellulose accounts for the more pronounced endothermic activity observed for cotton papers.

[11] EFFECT OF MECHANOCHEMICAL ACTIVATION ON THE THERMAL REACTIONS OF BOEHMITE (GAMMA-ALOOH) AND GAMMA-AL₂O₃

MacKenzie KJD. Temuujin J. Smith ME. Angerer P. Kameshima Y. - Thermochemica Acta. 359(1):87-94, 2000

Boehmite (gamma-AlOOH) and gamma-Al₂O₃ were ground for 60 min and the changes in their structure and thermal reactions were monitored by X-ray powder diffraction, thermal analysis and Al-27 MAS NMR. Grinding does

not cause the boehmite to dehydrate, but causes a decrease in the intensity of its X-ray reflections, and the formation of an amorphous phase containing tetrahedral Al and another Al site with a resonance at 36 ppm (sometimes attributed to pentacoordinated Al). The ground material thermally transforms to corundum at 940 degrees C via gamma-Al₂O₃, by contrast with the unground control, which forms corundum at 1195 degrees C via gamma and delta-Al₂O₃. Grinding gamma-Al₂O₃ decreases the intensity of its X-ray reflections and induces the formation of some corundum without further heating. Complete transformation to corundum occurs at 927 degrees C, compared with 1193 degrees C in the unground material which transforms via delta-Al₂O₃. The relationship between the mechanochemical development of the 36 ppm Al-27 NMR resonance in aluminas and their hydroxyl and adsorbed water content is discussed.

[10] ON THE B2 -> FCC TRANSFORMATION OF FE-RH DURING DEFORMATION

Yavari AR. Navarro E. Mori H. Yasuda H. Hernando A. Botta WJ. - Philosophical Magazine A-Physics of Condensed Matter Defects & Mechanical Properties. 80(8):1779-1793, 2000

It has been previously reported that deformation of the Fe-Rh intermetallic with B2-type ordered bcc structure results in a B2-to-fcc phase transformation and that the Fe phase exhibits a low-temperature magnetic behaviour typical of a spin glass or a cluster glass. Using transmission electron microscopy in conjunction with X-ray diffraction, Mossbauer spectroscopy and other previously published data, we show that the B2-to-fcc transformation occurs before grain refinement and deformation-induced nanocrystallization is completed anti long before the low-temperature magnetic properties show spin-glass-like behaviour. Not only is a B2-to-bcc solid-solution transformation not observed but evidence is found for localized transformations from the B2 phase to ordered fct L1(0), and it is concluded that the metastable fee phase must contain strong chemical short-range order (CSRO), which then is progressively reduced by continued deformation in this case by ball milling. The continued reduction in the degree of CSRO is related to the development of the spin-glass-like behaviour observed after long milling times.

[9] IMPROVED LIGHT STABILITY OF COLORED SiO₂ COATINGS CONTAINING ORGANIC AND METALORGANIC DYE MOLECULES

Diaz-Flores LL. Perez-Bueno JJ. Ramirez-Bon R. Espinoza-Beltran FJ. Vorobiev YV. Gonzalez-Hernandez J. - Journal of Vacuum Science & Technology A-Vacuum Surfaces & Films. 18(4 Part 2):1579-1583, 2000

Organic and metalorganic dyes have been incorporated into SiO₂ coatings using the sol-gel method. It is found that the coatings prepared from sol-gel starting solutions, which were subjected to a ball milling process, have improved light stability. Using optical absorption, atomic force microscopy and microluminescence measurements, it is found that the ball milling process of the starting solutions not only produces better dye dispersion, but there are also some indications that it produces a structure with fewer and smaller pores. These results are in agreement with previous studies, which indicate that the photostability of dyes in metal oxide matrices can be improved when the dye is better dispersed and trapped in more rigid cages.

[8] PREPARATION OF MIXED CO AND CU OXIDES VIA THERMAL DECOMPOSITION OF THEIR OXALATES, AND STUDY OF THEIR CATALYTIC PROPERTIES

Donia AM. Radwan NRE. Atia AA. - Journal of Thermal Analysis. 61(1):249-261, 2000

Mixed oxides were prepared by the thermal decomposition of the oxalates of cobalt(II) and copper(II) coprecipitated from aqueous solution or made by mechanical mixing. The compositions and structures of the oxides were confirmed by means of TG and X-ray powder diffraction spectroscopy. The catalytic behaviour of the oxides obtained was studied by using the decomposition of H₂O₂ as a model reaction. The results were compared with those on the oxides produced from the thermal decomposition of mechanically mixed oxalates. The catalytic activities of the mixed oxides were found to be lower than that of pure cobalt oxide, but higher than that of copper oxide. This result was interpreted in terms of the relative standard reduction potential of the catalyst as compared with that of H₂O₂. The catalytic activity of the mixed oxides obtained from the coprecipitate was found to be lower than that of the oxides obtained from the mechanical mixture at the same temperature. As the temperature of preparation was increased, the catalytic activities of the oxides obtained decreased. This was attributed to the solid-solid interactions, which gave a new phase with lower catalytic activity than those of the interacting phases.

[7] ELECTROCHEMICAL CHARACTERISTICS OF MG-NI ALLOYS AS ANODE MATERIALS FOR SECONDARY LI BATTERIES

Kim H. Park B. Sohn HJ. Kang T. - Journal of Power Sources. 90(1):59-63, 2000

The electrochemical characteristics of Mg and several Mg-Ni alloys were studied as alternatives to anode materials for secondary Li batteries. Li was alloyed and dealloyed reversibly with Mg at very low voltage region (below 100 mV vs. Li/Li+), and the initial capacity obtained was approximately 3070 mA h/g. Alloys of Mg₅₀Ni₅₀, Mg₆₇Ni₃₃, and Mg₇₅Ni₂₅, were prepared by mechanical alloying and characterized using X-ray diffraction (XRD) and Auger electron spectroscopy (AES). Mg₅₀Ni₅₀ and Mg₆₇Ni₃₃ were found amorphous and crystalline, respectively, while Mg₇₅Ni₂₅ was a mixture of Mg and Mg₇₅Ni₂₅ phases. Electrochemical tests with these Mg-Ni alloys demonstrated that only Mg₇₅Ni₂₅ reacted significantly with Li at room temperature while Mg₅₀Ni₅₀ and Mg₆₇Ni₃₃ reacted with Li at high temperature. Mg₇₅Ni₂₅ showed enhanced cycle performance compared to that of pure Mg.

[6] THERMAL SPREADING OF MOO₃ ONTO SILICA SUPPORTS

Braun S. Appel LG. Camorim VL. Schmal M. - Journal of Physical Chemistry B. 104(28):6584-6590, 2000

The thermal spreading of MoO₃ onto silica was studied in comparison with the thermal spreading onto alumina using different characterization techniques. X-ray diffraction results showed that MoO₃ crystals were transformed into Mo amorphous species on alumina and silica supports by thermal treatment. Laser Raman spectroscopy results also evidenced the transformation of MoO₃ bulk into small Mo clusters and/or dispersed Mo species, which are highly distorted, interacting with alumina and silica supports, X-ray photoelectron spectroscopy and diffuse reflection spectroscopy (DRS) results gave better evidence of the presence of higher amounts of Mo species at the surface of both supports when compared with the respective physical mixtures. Fourier transform infrared spectroscopy also provided good evidence that a surface reaction may have occurred between MoO₃ and hydroxyl groups on both supports. It was possible to infer that the original MoO₃ crystal lattices were destroyed by reacting with the support surface as a result

of thermal treatment and transformed into small Mo clusters, dispersed Mo species, or both. Moreover, Raman spectroscopy showed nicely that the interaction of Mo species was higher on alumina than silica surfaces, Raman and DRS also provide insight into Mo species dispersion on alumina and silica samples, whereas DRS provided better evidence of the presence of dispersed Mo species on these supports. It was possible to infer that all calcined physical mixtures exhibited dispersed Mo species and small Mo clusters together with a small quantity of bulk MoO₃ that remained after thermal treatment. Therefore, similar results obtained on both supports demonstrated that the thermal spreading of MoO₃ also occurred on silica and the same mechanism was observed as on alumina. However, the Mo dispersion and some Mo species were different on the supports that can be attributed to the different surface properties of silica and alumina.

[5] EFFECT OF GELATION AND BALL MILLING ON THE OPTICAL ABSORPTION AND EMISSION OF MACRILON YELLOW DYE MOLECULES TRAPPED IN SILICA PREPARED BY THE SOL-GEL METHOD

Diaz-Flores LL. Perez-Bueno JJ. Espinoza-Beltran FJ. Perez-Robles JF. Ramirez-Bon R. Vorobiev YV. Gonzalez-Hernandez J. - *Microelectronic Engineering*. 51-2:659-666, 2000

Organic dye molecules (macrilon yellow) were embedded into silicon dioxide matrix by the sol-gel method. Optical measurements at room temperature of absorption and emission were performed at different steps of the gelation process, starting when the suspension was liquid and finishing when it got the solid state. Optical absorption spectra show the dependence with gelation process and with the milling process. The intensity of the absorption band at 420 nm increases with the viscosity of the suspension. This effect is more important for the milled suspension, working with the same pigment concentration. The gelation and the milling processes also affect the emission spectra. The emission spectra has two bands centered at about 520 and 560 nm. During gelation the relative intensity of the band at about 560 nm increases respect the band at about 520 nm. The emission band of the sample made with milled suspension is three times more intense than that of the not milled suspension.

[4] EFFECT OF MECHANICAL ACTIVATION ON THE SINTERING OF A TITANIUM-NICKEL ALLOY AND A BIOCERAMIC TINI COMPOSITION

Itin VI. Terekhova OG. Ul'yanova TE. Kostikova VA. Shevchenko NA. Berdnikova DV. - *Technical Physics Letters*. 26(5):436-438, 2000

Mechanical activation strongly influences the sintering of pressed articles made of a powdered titanium-nickel alloy and its compositions with dental porcelain. Preliminary treatment of the powdered mixtures in a planetary ball mill suppresses the martensite transformation and removes anisotropy in the variation of linear dimensions of the pressings. The resulting samples exhibit, instead of a volume expansion related to the rupture of contacts between particles in the course of the reverse martensite transformation, a sintering-induced shrinkage whose extent increases with the sintering temperature and the duration of mechanical activation treatment.

[3] OPTIMIZATION OF CALCINATION CONDITIONS ON THE BI-2223 KINETIC FORMATION AND GRAIN SIZE

Garnier V. Monot I. Desgardin G. - *Superconductor Science & Technology*. 13(5):602-611, 2000

The effects of the calcination conditions (time, temperature, intermediate milling) on the kinetics of formation of Bi-2223 and on the grain size were studied using a powder precursor synthesized by the polymer matrix method. The samples were characterized by XRD and SEM analysis. The grain size and the kinetics of formation of Bi-2223 strongly depend on the calcination conditions and thus on the phase assemblages formed at the end of the calcination that determines the reactivity during sintering. The higher the calcination temperature is the larger the grain size. We have observed that 24 h of calcination at 820 degrees C without intermediate milling allows one to obtain 79% of Bi-2223 phase in 60 h of sintering at 835 degrees C.

[2] FIELD AND TEMPERATURE DEPENDENCE OF MAGNETIZATION IN FECU-BASED AMORPHOUS ALLOYS

Crespo P. Multigner M. Castano FJ. Casero R. Hernando A. Escorial AG. Schultz L. Kaul SN. - *Physical Review B*. 61(21):14346-14349, 2000

In this paper, the production of FeCu-based FeCuZr amorphous alloys by ball milling is reported. The thermal dependence of magnetization for the (Fe_{0.5}Cu_{0.5})(85)Zr-15 (at. %) amorphous alloy has been found to show a dramatic field dependence of the kink point of the magnetization. This kink corresponds to a temperature different from the Curie temperature, above 400 K, of the ferromagnetic phase, which, according to spin waves fitting, can be induced by applying external fields. Just above 235 K, the thermoremanence increases sharply, and this feature strongly suggests an increase of the ferromagnetic ordering under zero field heating. Neutron diffraction experiments seem to confirm the enhancement of spin alignment. The thermal expansion above the compensation temperature is proposed to be the origin of the thermoremanence enhancement through the anti-Invar effect as might be explained within the framework of recent ab initio calculations [M. van Schilfgarde et al., *Nature (London)* 400, 46 (1999)].

[1] MECHANICALLY INDUCED OXIDATION OF SRFeO₃-DELTA

Schmidt M. - *Materials Research Bulletin*. 35(2):169-175, 2000

The nonstoichiometric strontium-iron oxide SrFeO₃-delta (2.5 less than or equal to 3 - delta less than or equal to 3.0) exhibits richness of equilibrium states over a broad range of temperatures and oxygen partial pressures. Its oxidation induced by ball milling was investigated. The mechanical treatment of the material at room temperature in oxygen atmosphere was found to cause the reaction. However, the product differs from compounds obtained by annealing, due to crystal imperfections introduced by milling: The changes in the oxygen stoichiometry, the crystal structure, and the morphology of the powder are discussed.

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