

RESEAU FRANÇAIS DE MECANOSYNTHESE

Lettre N° 52

Juillet 1999

155 Groupes de Recherche
(dont 83 à l'étranger / 34 Pays)

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

Sommaire

- 17 Nouvelles Adhésions soient 8 Nouveaux Groupes de Recherches
 - Liste des Congrès Nanomatériaux
 - 2 Nouvelles Soutenances de Thèses
 - 2 Nouvelles Propositions de Post Doc

17 Nouvelles Adhésions

- M. Baron - EMAC - Albi - France
- P. Baviera - Lab. Métal. Phys.- URA CNRS 131 - Poitiers - France
- D. Bernard - TCPP - Azerables - France
- B. Bureau - Eq. Physique de l'Etat Condensé - Univ. du Maine - Le Mans - France
- H.S. Cao - UTBM - Belfort - France
- P. Delcroix - LSG2M- CNRS- Ecole des Mines - Nancy - France
- M. Gasnier - CNRS UPS CPMA - Orsay - France
- V. Gauthier - LRRS - CNRS / Univ. Bourgogne - Dijon - France
- C. Gonzales - Eq. Physique de l'Etat Condensé - Univ. du Maine - Le Mans - France
- H. Guerault - Eq. Physique de l'Etat Condensé - Univ. du Maine - Le Mans - France
- S. Mirales - EMAC - Albi - France
- P. Pareja - Lab. Catlyse Hétérogène - Vandoeuvre les Nancy - France
- M. Rallo - ECM Infra - Four - Seyssinet - France
- C. Roques - Carmes - ENSMM - Besançon - France
- V.V Tuyet - EMAC - Albi - France
- T. Ziller - LSG2M- CNRS- Ecole des Mines - Nancy - France
- Dr. Min Zhu - Dpt Mechano - Electronic Eng. - South China Univ. Technology-Guangzhou-Chine

Le site web du RFM est le suivant

<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.

Le Site WEB du RFM a franchi le seuil des 1000 Connexions

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ANNONCE DE CONGRES ET / OU ECOLES
CONGRESS AND SCHOOL ANNOUNCEMENTS

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4th Int. Conf. on Materials Chemistry

13 - 16 Juillet 1999 - Trinity College _ Univ. Dublin - Irlande
Web Site : <http://www.rsc.org/conferences>

Themes :

Inorganic Nano and Micro Particles
Functional Polymers
Magnetic Materials
Organic Nanostructures
Molecular Crystals and Crystal Engineering
Computational Chemistry and Materials for Electronic

Advanced Materials - Nanostructured Systems

15 - 17 Juillet 1999 - Hong Kong

1st workshop of the new IUPAC series :

"New Directions in Chemistry

Theory, Nanoparticles, Quantum Dots,

Bio - Inspired Structures, Applications to Nanotechnology

Organizing Committee A. El - Sayed - Georgia Tech - Atlanta - USA

J. Portner - President of IUPAC - Tel Aviv - Israel

N. Teng Yu - HKUST - Hong Kong

S. Williams - Hewlett - Packard Co., California USA

Web Site : <http://www.iupac.org/symposia/conferences/wam1>

Gordon Conference on Clusters, Nanocrystals and Nanostructures

24 - 29 Juillet - Connecticut College, CT - USA

Contact : J. Heath : alivis@uclink4.berkeley.edu

NOUVEAU

NATO Advanced Research Workshop

Investigations and Applications of Severe Plastic Deformation

2 - 6 Aout 1999 - Moscou - Russie

E-Mail : TLowe@lanl.gov and Valiev@ippm.rb.ru

IAC - 2

2nd International Alloy Conference

8 - 13 Aout 1999 - Davos - Suisse

Website : www.engfnd.org

"Thermal Spray Processing of Nanoscale Materials II"

15 - 20 Aout, 1999 - Quebec City, Canada

(Contacts: C.C. Berndt, SUNY-Stony Brook, 306 Old Engineering, Stony Brook,

NY 11794-2275, FAX: 516-632-8525, EMAIL: cberndt@notes.cc.sunysb.edu,

WEBSITE: <http://www.engfnd.org>; E.J. Lavernia, EMAIL: lavernia@uci.edu; C.

Moreau, EMAIL: christian.moreau@nrc.ca; M.L. Trudeau, EMAIL:

trudeaum@ireq.ca; and L. Kabacoff, EMAIL: kabacol@onr.navy.mil)

NANO 2000

5th International Conference on Nanostructured Materials

Sendai - 20 - 25 Aout 2000

E-Mail : nano2000@imr.tohoku.ac.jp

RQ10

10th International Conference on Rapidly Quenched and Metastable Materials

Bangalore - Inde - 23 - 27 Aout 1999

Website : <http://www.metalrg.iisc.ernet.in/rqten/>

ISMANAM 99

International Symposium on Metastable Mechanically Alloyed and Nanocrystalline Materials
and Euro Conference on Gas Phase Synthesis of Nanocrystalline Materials.

Org. : L. Schultz, J. Eckert, H. Hahn

Dresden - 30 Aout - 3 Septembre 1999

E-Mail : ISMANAM99@ifw-dresden.de

WebSite: <http://www.ifw-dresden.de/imw/ismanam/>

EUROSOLID

6 - 10 Septembre 1999 - Carry le Rouet - France

SMM14

14th International Conf. on Soft Magnetic Materials
8 - 10 Septembre 1999 Balatonfüred - Hongrie
web site : <http://www.kfki.hu> - Subsite : smm14

EUROMAT 99

20 - 30 Septembre 1999 - Munich- Allemagne
E-Mail : euromat@dgm.de
Web Site : <http://www.euromat.fems.org>

Elaboration et Transformation des Solides Divisés

(Ecole Thématique)

21 - 24 Septembre 1999 Carry le Rouet - France
Renseignements : F. Gruy - ENSM St Etienne
E-Mail : gruyemse.fr

Int. Symp. Cluster and Nanostructure Interfaces (ISCANI)

25 - 29 Octobre 1999 - Richmond USA
website : <http://www.vcu.edu/ISCANI/>

NOUVEAU

**Processing and Properties of Structural Nanomaterials
TMS Fall Meeting**

31 Octobre - 4 Novembre 1999 - Cincinnati - OH - USA
Contact LL Shaw : fax : 703 6960934

J.A. 99 / SF2M

2- 5 Novembre 1999 - ENSAM / Paris - France
Les matériaux pour les microactionneurs et microcapteurs
Les mécanismes de renforcement dans les polymères & élastomères
Matériaux Magnétiques de Grande Diffusion
Tribologie, Contact Métal - Outil lubrifiant en laminage à froid
Surface et Interface Métallique
Surface et Revêtement
Solidification
Les Imageries de l'angstrom au micron
Les Matériaux pour le Bâtiment
Adresse : SF2M - 1 Rue de Craiova - 92024 Nanterre Cedex

EURO PM99

3rd European Conference on Advances in Hard Materials Production
8 - 10 Novembre 1999 - Turin - Italie
Web site : <http://www.epma.cm>

MRS Fall Meeting 99

29 Novembre - 3 Décembre 1999 - Boston MA - USA
Website : <http://www.mrs.org>

Sintering 2000

7th International Conference on Sintering
Sintering Science and Technology beyond 2000AD
22 - 25 Février 2000 - New Delhi - Inde
E-Mail : gsu@iitk.ac.in

NOUVEAU

**Ultrafine Grained Materials
Strengthening, Fracture and Creep of Nanostructured Materials
Symposia**

12 - 16 Mars - TMS Annual Meeting - Nashville TN - USA
Contact : RSMIOSHRA@ucdavis.edu

JRFM'2000

23 - 24 Mai 2000 - Bordeaux France
Wbsite : <http://www.bls.fr/amatech>

4th EUROMECH

26 - 30 Juin 2000 - Metz - France
E-Mail : euromech@lpmm.univ-metz.fr
WebSite : <http://www.lpmm.univ-metz.fr/euromech>

III European Conference on Fluidization

29 - 31 Mai 2000 - Toulouse - France
E-Mail : Progep@ensigct.fr

**Solid State Chemistry 2000
Prague, Czech Republic, in September 2000**

(Information provided by Klara Tkacova)

NOUVEAU

The Institute of Anorganic Chemistry of the Czech Academy of Sciences prepares a conference entitled Solid State Chemistry 2000, which will be held in Prague, Czech Republic, in September 2000. The meeting will continue the tradition established by the previous SSC conferences which took place in 1986 and 1989 in the Czech Republic, and in 1996 in the Slovak Republic, as well as the tradition of the 1st International Conference on Mechanochemistry and Mechanical Alloying held in 1993 in Kosice, Slovakia. In case of adequate number of presentations and participants would the scientific meeting take place as a satellite symposium entitled 3rd International Conference on Mechanochemistry and Mechanical Alloying INCOME. With small number of participants our topic would be discussed in a special section entitled Mechanochemistry.

SOUTENANCE DE THESE

D. Cracco

**"Recherche de Nouveaux Alliages Hydrurables de Forte Capacité Massique
Utilisable comme Matériaux d'Électrode Négative d'Accumulateur Ni - MH"**
CNRS - Thiais - France - 25 Juin 1999

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

A. Gentil - Sagot

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

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

Cooperative Research on Related Areas

COREE du SUD (6 May 1999)

From Professor Soon B. Hong
Department of Materials Science and Engineering
Korea Advanced Institute of Science and Technology
373 - 1 Kusung - dong, Yusung - gu
Taejon 305 - 701 Korea

One research topic is entitled "Mechanical Behavior and Wear Resistance of Nanocrystalline WC - Co alloy". We are investigating the fabrication process, sintering and mechanical & wear properties of nanocrystalline WC - Co hard materials. The other research topic is titled "Fabrication Process and Mechanical Properties of Mechanically Alloyed Tungsten Heavy Alloys". We are investigating the mechanical alloying process, sintering behavior and mechanical properties of MA tungsten heavy alloys.

We are very pleased to discuss for international cooperative research on above research topics with Member of Mecanosynthese Group

E-Mail : ShHong@Sorak.kaist.ac.kr

**Ph D Position
and
Post Doc Position
Proposals**

FRANCE (6/06/99)

P. Bracconi (Univ. Dijon) propose une position de Post Doc en Métallurgie des Poudres, de nationalité autre que française (Poste Fléché CNRS)
Contact : pbrac@u-bourgogne.fr

GRECE (11/06/99)

The PEML (Photonics and Electronics Materials Lab) at FORTH, Herklion Crete, Greece, offers two positions available for European Post and Pre Docs, to work within the framework of two TMR networks (HAFAM and MICROSYNC) concerning microfabrication and microassembling"
Contact : Porf. G. Kiriakidis : Kiriakid@iesl.forth.gr

ISRAËL (14/5/99)

A postdoctoral position is available at the High-Pressure group of the School of Physics & Astronomy, Tel Aviv University. This position is available starting with the 1999/2000 academic year for one year, with a possible extension for two years. For additional information, please contact Moshe P. Pasternak by email to hh136@post.tau.ac.il or visit the MEDC web site (www.unca.edu/medc).

Denmark (22/02/99)

One Ph.D. position will be available in the department of Physics at the Technical University of Denmark from 1st April 1999. The candidate will work in the area of Crystallization Kinetics in Bulk Metallic Glasses, which is associated with a Talent Project supported by the Danish Research Council. The position is for three years, and monthly salary is about 20,000 Dkr (3300 USD). Applications including a CV, publication list, and names of three references should be sent as soon as possible to:

Assoc. Prof. Jianzhong Jiang
Department of Physics, Building 307
Technical University of Denmark
DK-2800 Lyngby, Denmark
e-mail jiang@fysik.dtu.dk
fax. +45 45 93 23 99
tel +45 45 25 31 65

Québec (CANADA) (22/01/99)

Institut National de la Recherche Scientifique
Département ...nergie et Matériaux

POST-DOCTORAL POSITION IN Ni-MH BATTERY TECHNOLOGY

Candidates will be interested in developing a research project focused on the study of new materials for use as negative electrode in nickel-metal hydride (Ni-MH) batteries. Mg-based compounds as electrode material and high-energy ball milling as synthesis method will be privileged. Particular efforts will be performed in order to clarify the correlation between the structure, the composition and the morphology of the alloy and its electrochemical performances.

Experience in electrochemistry and materials science is essential, a working knowledge of Ni-MH battery is an advantage.

Applicants must have obtained their Ph-D between July first, 1996 and January first, 2000.

The work will start between June 1st, 1999 and May 31, 2000.

Initial appointment is for one year, renewable for one year. Salary is \$28,000/year, which could be increased with qualifications and experience.

Applicants should send a CV including a list of publications before March 1st, 1999 to:

Pr. Lionel ROUE
INRS- Energie et Matériaux
1650, bd. Lionel Boulet
Varenes, Québec, CANADA
J3X 1S2
E-Mail: HYPERLINK mailto:roue@inrs-ener.quebec.ca / roue@inrs-ener.quebec.ca•

USA (17/12/98)

Rutgers University is seeking a postdoctoral associate with demonstrated expertise in mechanochemistry to work on research focused on biomaterials. The candidate must be able to work on research focused on biomaterials. The candidate must be able to work as part of multidisciplinary team involving industry and academia focused on making biomedical implant devices. 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, address, 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 will be posted until a suitable candidate is identified. Interested candidates should send correspondence to

Professor R.E. Riman
Rutgers University
Department of Ceramic and Materials Engineering
607 Taylor Road
Piscataway, NJ 08854 - 8065
Riman@alumina.rutgers.edu

Bibliographie Récente

Livres ou "Special Issues"

"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 Besterčí, 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)

References

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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 nanométrie) ; les conférences et les forums sur Internet se multiplient où s'échangent des informations sur les avancées scientifiques et technologiques dans ce domaine des matériaux nanostructurés qui se distinguent des matériaux polycristallins conventionnels par la dimension des cristallites les composant ou par la dimension des hétérostructures présentes : ces dimensions sont de quelques dizaines d'angströms, voire de quelques nanomètres. A ces dimensions, les propriétés des matériaux changent radicalement.

Au début des années 90, les japonais ont été les premiers à lancé d'ambitieux programmes de R & D puisque le MITI a consacré aux nanomatériaux près de 200 millions de dollars pour la période 1990 - 2000 et que la Science & Technology Foundation a investi presque la même somme pour co - financer des projets de laboratoires publics et privés. Les Etats Unis puis les pays européens ont investi plus tardivement mais déjà ont obtenu des résultats prometteurs (.....) Certaines applications existent déjà au niveau international, quelque 400 sociétés se partagent aujourd'hui un marché voisin de 1 milliard de dollars mais qui devrait tripler, voire quintupler à l'horizon 2001.(.....)

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

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

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, (.....)

Please send your order to: Book Department - Cambridge International Science Publishing 7 Meadow Walk, Great Abington, Cambridge CB1 6AZ, England Fax: +44 1223 894 539; tel +44 1223 893295, email: orders@cisp.demon.co.uk / Cambridge International Science Publishing <http://www.demon.co.uk/cambsci/homepage.htm>

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)

[50] MICROSTRUCTURAL AND MECHANICAL CHARACTERISATION OF SiC - SUBMICRON TiB₂ COMPOSITES"

C. Blanc, F. Thevenot, D. Goeuriot - J. Europ. Cer. Soc., 19 (1999) 561 - 569

The aim of this work is to characterize ceramic composites SiC - TiB₂. After preparation of dense composites aSiC - TiB₂ (5, 10 and 15 vol. % TiB₂) by reactive pressureless sintering, the materials have been characterized by their microstructure and their mechanical properties. The dispersion of TiB₂ particles is quite homogeneous, observed both by optical and scanning electron microscopies. Image analysis has revealed a majority of submicronic particles. Atomic force and transmission electron microscopies have shown the presence of nanometric TiB₂ particles. Concerning mechanical properties, toughness increases with the TiB₂ content, whereas hardness decreases when the TiB₂ content increases.

[49] NON-THERMAL PRODUCTION OF TUNGSTEN FROM SCHEELITE

Welham NJ. - Materials Science & Technology. 15(4):456-458, 1999

A mixture of scheelite and magnesium was mechanically milled for 100 h in an inert atmosphere. The resultant powder was examined using thermal analysis, isothermal annealing, and X-ray diffraction to determine the effect of milling on the reduction of scheelite. Differential thermal analysis showed that the magnesium was completely consumed during the milling process. It was found that elemental tungsten was formed during the milling stage and the unwanted phases, MgO and CaO, were readily removed by leaching in acid, leaving a fine powder which was similar to 99% W. The major impurities were iron and chromium which were present as a result of abrasion of the mill and balls.

[48] UTILIZATION OF THE BALL MILLING TECHNIQUE IN THE SYNTHESIS OF ELECTROCATALYSTS FOR H₂ OXIDATION IN POLYMER ELECTROLYTE FUEL CELLS

G Lalande, MC Denis, D Guay, JP Dodelet, J Huot, R Schulz - NEW MATERIALS FOR FUEL CELL AND MODERN BATTERY SYSTEMS II, 1997, pp 720-731 - 2ND INTERNATIONAL SYMPOSIUM ON NEW MATERIALS FOR FUEL CELL AND MODERN BATTERY SYSTEMS; MONTREAL, CANADA. JULY 6-10, 1997

The high energy ball milling technique has been used to produce nanocrystalline alloys to perform H₂ oxidation in polymer electrolyte fuel cells. It has been demonstrated that Pt-x-Ru1-x alloys are easily obtained but they lack specific area due to the agglomeration of the nanocrystallites in much larger particles. Attempts to break the agglutinated particles by milling them with carbides (TiC or WC) failed. Better results were obtained by milling the alloys with carbon black but there is still much room for improvement.

[47] PRODUCTION OF BIMETALLIC CARBIDES BY MECHANICAL ALLOYING FOR CATALYSIS

AM Alves, R Rosenthal, VLST daSilva - ADVANCED POWDER TECHNOLOGY (Series: MATERIALS SCIENCE FORUM), 1999, Vol 299-3, pp 121-125

Transition metal carbides have attracted attention as catalysts for hydrogenation, isomerization and hydrotreating reactions. The present work investigates the conditions for the synthesis of bimetallic iron molybdenum carbide using Mechanical Alloying, as a stage of a wider project that also includes producing carbides by the Temperature Programmed Carburization method. The materials were characterized by x-ray diffraction and scanning electron microscopy. Results revealed that an amorphous or nanocrystalline structure resulted for the employed conditions (ball-to-powder ratio and milling time), thus suggesting that this material can be very promising for hydrogenation reactions if a sufficiently high final powder surface area is achieved.

[46] PROPERTIES OF SILICIDES AND SILICIDE COMPOSITES DENSIFIED BY PRESSURELESS REACTIVE SINTERING

R Scholl, P Tsakirooulos, A Bohm, B Kieback - ADVANCED POWDER TECHNOLOGY (Series: MATERIALS SCIENCE FORUM), 1999, Vol 299-3, pp 165-170

The difficulties of casting high melting silicides and their poor sinterability limit structural and functional applications up to now. Furthermore mechanical properties of single phase silicides (MoSi₂, TiSi₂, Ti₅Si₃,...) are insufficient at room temperature (fracture toughness) and above the brittle to ductile transition (BTDT) (creep). Approaches for advantages are concentrated on silicide composites using different hard phases (SiC, TiB₂, ZrO₂,...), mixtures of silicides (e.g. (Mo,W)Si₂) and introduction of ductile metallic phases (e.g Nb) for strengthening the matrix. Our investigations on MoSi₂-composites using the reactive sintering technique of activated powder mixtures proved a high level of mechanical properties. In particular we found for 4-point bending strength: 500 MPa (RT) and 700 MPa (1100 degrees C); K-IC (crack prolongation method): 5 MPam^{1/2} (0 Vol.% SiC) and 8,4 MPam^{1/2} (30 Vol.% SiC), creep rate: 10⁻⁵ (0% SiC) 10⁻⁶ (30Vol.% SiC). This data are at least as high as reported by other authors but in contrary to the state of art the preparation of complex parts should be possible. Furthermore it was shown that the materials show good corrosion resistance under flue gas atmosphere with high gas pressures and high gas speed.

[45] POWDER METALLURGY PROCESSING OF NI-CO BASE ELECTRODES FOR APPLICATION IN WATER ELECTROLYSIS

OBG Assis, FC Crnkovic, R Machado, LA Avaca, M Ferrante, SAS Machado - ADVANCED POWDER TECHNOLOGY (Series: MATERIALS SCIENCE FORUM), 1999, Vol 299-3, pp 371-375

Electrodes of Co, Ni and Ni₅₀Co₅₀ were produced by powder metallurgy techniques aiming at their application as cathodes for water electrolysis system. Sintering was carried out in vacuum/argon atmosphere at low temperature to keep some residual porosity to enhance surface electroactivity by an increase of the geometric area. The Ni-Co alloying was obtained by mechanical synthesis, making use of the appropriated route. The final electrodes presented porosity around 12% where the electrochemical tests presented increasing in the critical peak when compared with

electroplated flat samples.

[44] EFFECT OF WAX ADDITION ON MONEL SYNTHESIS BY HIGH ENERGY MILLING

CJ Rocha, EG deAraujo, RA Nogueira, F Ambrozio - ADVANCED POWDER TECHNOLOGY (Series: MATERIALS SCIENCE FORUM), 1999, Vol 299-3, pp 457-462

The effects of addition of polyethylene wax on the synthesis of Cu-Ni alloy (Monel) with 50wt%Ni processed in a high energy ball mill were investigated by X-ray diffraction. The Monel powder was characterized by laser particle size measurement and scanning electron microscopy. The results showed that the addition of wax at the beginning of milling causes a delay in alloy formation, and the particle shape obtained was essentially flaky. However, a strong effect was observed on the shape and particle size distribution when wax was added to the Monel powder with a flaky shape previously processed without wax. The particle shape changed from flake to more irregular and the median particle size decreased from 55 μm to 7.5 μm after 4 hours of milling.

[43] COMPACTION OF AISI M2+10%VOL. NbC PROCESSED BY HIGH ENERGY MILL

R Panelli, F Ambrozio - ADVANCED POWDER TECHNOLOGY (Series: MATERIALS SCIENCE FORUM), 1999, Vol 299-3, pp 463-469

A mixture of AISI M2 + 10% vol. NbC was processed in an Attritor high energy mill to obtain a composite powder. The milling time was varied during the process. The equation $\sqrt{\ln(1/(1-D))} = A\sqrt{P} + B$ was used to fit the compacting experimental data from the composite milled powders. The linear correlation coefficients were better than 0.985 for all the composite powders. The parameters A and B from this equation were used to show how the composite powder characteristic changed after attritional milling.

[42] FABRICATION AND HOT EXTRUSION OF MECHANICALLY ALLOYING CU-15WT%CR ALLOY

BR Juan, GC Jorge, MZV dePaul - ADVANCED POWDER TECHNOLOGY (Series: MATERIALS SCIENCE FORUM), 1999, Vol 299-3, pp 470-477

Mechanical alloying (MA) of copper-chromium (Cu-15wt%Cr) elemental powders was carried out using a planetary ball mill with tungsten carbide balls during 60 h, using rotation speed of 200 and 350 rpm. The evolution of element combination in the milling was examined through X-rays diffraction, observing that the saturation grade of chromium depends on the milling time and rotation speed of the mill. The lattice parameter of copper decreases as the chromium enters into solution, reaching a minimum value of 3.599 Angstrom for 50 h. The Vickers hardness of mechanically alloyed powder increased with milling time due to plastic deformation, tending to a value of 594 HV. The mechanically alloyed powders were consolidated by hot extrusion after encapsulation and degassing at 500, 600 and 700 degrees C, using an extrusion ratio of 10:1 and an extrusion speed of about 5 mm(-1). The powder consolidation and the final Vickers hardness depended strongly on the pre-heating temperature. Chemical analysis of the extruded bars showed that it was contaminated with W and Fe. Finally, aging heat treatments at 900 and 1000 degrees C aiming to raise the hardness of the extruded bars were performed.

[41] MG BASED ALLOYS OBTAINED BY MECHANICAL ALLOYING

S Ordonez, G Garcia, D Serafini, A SanMartin - ADVANCED POWDER TECHNOLOGY (Series: MATERIALS SCIENCE FORUM), 1999, Vol 299-3, pp 478-485

In the present work, we studied the production of magnesium alloys, of stoichiometry 2Mg + Ni by mechanical alloying (MA) and the behavior of the alloys under hydrogen in a Sievert's type apparatus. The elemental powders were milled under argon atmosphere in a Spex 8000 high energy ball mill. The milled materials were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM). Only minimum amounts of the Mg₂Ni intermetallic compound was obtained after 22 h of milling time. Most of the material was stuck to the inner surface of the container as well as to the milling balls. Powders milled only for 12 hours transform to the intermetallic at around 433 K. Effects of the MA on the hydrogen absorption kinetics were also studied.

[40] MECHANICAL ALLOYING OF HIGH PERFORMANCE ALUMINUM ALLOYS

RM Callegaro, JL Cesar, L Schaffer - ADVANCED POWDER TECHNOLOGY (Series: MATERIALS SCIENCE FORUM), 1999, Vol 299-3, pp 486-496

The aim of this work; is to present a good technique for preparing high resistant aluminum alloy, and also some results achieved by researches using Al₅Fe₁₂Si alloy in powder metallurgy. This alloy has been studied in order that strength at high temperature applications could be improved, to be used in automotive internal parts. A brief description of the Mechanical Alloying process is done, followed by results after testing.

[39] EFFECT OF MECHANICAL ACTIVATION IN VIBRATION GRINDING ON SINTERING AND PROPERTIES OF MULLITE CERAMICS

Ordan'yan SS. Andreev KP. Stepanenko EK. - Russian Journal of Applied Chemistry. 71(12):2108-2112, 1998

With the use of low-temperature synthesis of mullite 3Al₂O₃ . 2SiO₂ and mechanical activation in vibration grinding, ultradispersed powders of mullite were obtained and fundamental aspects of densification in sintering and properties of mullite ceramics were studied.

[38] MECHANICAL ACTIVATION OF CEMENT WITH ADDITION OF FLY ASH

Sekulic Z. Popov S. Duricic M. Rosic A. - Materials Letters. 39(2):115-121, 1999

Influence of mechanical activation on quality of cement with addition of fly ash was investigated. The experiments of mechanical activation were performed in laboratorial vibro mill with rings. The effects of mechanical activation were determined by the comparative analyses of the starting cement and the same cement treated mechanically for 3, 10 and 30 min. The analyses included determination of the specific surface area, X-ray diffractometric analysis (XRD), differential-thermal analysis (DTA) and thermogravimetric analysis (TGA). During the 30-min comminution period, the specific surface area was increasing, while the microstructural changes of portland cement with fly ash addition (20% wt.) were intensified. DT and TG analyses show that endothermic and exothermic reactions proceed at the lower temperatures, which is an indication of increased reactivity of mechanically activated sample. Physico-

mechanical properties significantly improved after only 3 min activation. After the 3-min activation, i.e., the seven-day solidification, the increase in the compression strength is very well-expressed. However, even after the 28-day solidification, the compression strength of mechanically activated sample was 58% higher, in comparison with inactivated sample of the same composition. Also, after the 28-day solidification, the compression strength of portland cement with addition of fly ash, mechanically activated for 3 min, was approximately the same to the compression strength of portland cement without additives.

[37] IMPEDANCE SPECTROSCOPY OF N-DOPED (BA,SR)TiO₃ CERAMICS PREPARED BY MODIFIED LOW TEMPERATURE AQUEOUS SYNTHESIS

Viviani M. Nanni P. Buscaglia MT. Leoni M. Buscaglia V. Centurioni L. - Journal of the European Ceramic Society. 19(6-7):781-785, 1999.

Low temperature aqueous synthesis (LTAS) has been applied to the preparation of La or Nb-doped (Ba,Sr)TiO₃ powders. Doping elements have been mixed to precursors, in order to directly obtain crystalline powders with modified composition. Similar powders, doped by conventional wet ball-milling process, have been also prepared for comparison. Ceramic samples have been obtained by isothermal sintering in air at different temperatures. Microstructural characterisation showed the presence of a secondary, phase, identified as a Ti-rich barium titanate compound. Impedance spectroscopy (IS) has been used to study the PTCR behaviour of such materials. IS results are analysed taking into account effect of the secondary phase, to discuss bulk and grain boundary characteristics.

[36] STRUCTURE FORMATION AND DEGRADATION OF PARTIALLY STABILIZED ZIRCONIUM DIOXIDE

Makarenko AN. Belous AG. Pashkova YV.- Journal of the European Ceramic Society. 19(6-7):945-947, 1999

The process of structure formation of partially stabilized zirconium dioxide powders during the heat treatment of precipitated zirconium and yttrium hydroxides and structure degradation in time depending on hydroxide precipitation method (mutual and sequential precipitation) has been investigated. During calcination, the t-->m-ZrO₂ transition takes place in sequentially precipitated hydroxides, and the m-->t-ZrO₂ transition takes place in coprecipitated hydroxides. It was noted that soft readily, destroyable aggregates are formed during the heat treatment of sequentially precipitated hydroxides. This makes it possible to produce fine ZrO₂ powders (d = 50-100 nm) avoiding disaggregation and/or milling. It has been found that the zirconium dioxide degradation process, which is due to the t-->m-ZrO₂ transformation, decreases with increasing amount of Y₂O₃ and cubic modification of ZrO₂ in heat treatment products and in the case of coprecipitation of hydroxides

[35] OXIDE SLURRIES STABILITY AND POWDERS DISPERSION: OPTIMIZATION WITH ZETA POTENTIAL AND RHEOLOGICAL MEASUREMENTS

Vallar S. Houivet D. El Fallah J. Kervadec D. Haussonne JM. - Journal of the European Ceramic Society. 19(6-7):1017-1021, 1999

Slurries stability and powder dispersion are highly dependent on both the pH and their density. The analysis of these parameters allows us to keep the slurries deflocculated, leading to an optimum mix of powders before calcination and reaction. The present work is devoted to the mixing of the different oxide and carbonate components in slurries to be synthesized by solid state reaction of the (Zr,Sn)TiO₄ (ZST) microwave dielectric and the Pb(Mg_{1/3}Nb_{2/3})O-3 (PMN) compositions. Their rheological properties and zeta potential measurements characterized slurries. These techniques allowed tw to optimize the dispersion parameters of the oxide slurries. We have therefore been able to synthesize as well pure PMN and ZST microwave resonators with very high performances.

[34] DIELECTRIC PROPERTIES OF BARIUM-TITANATE SINTERED FROM TRIBOPHYSICALLY ACTIVATED POWDERS

Stojanovic BD. Pavlovic VB. Pavlovic VP. Djuric S. Marinkovic BA. Ristic MM. - Journal of the European Ceramic Society. 19(6-7):1081-1083, 1999

It has been well known that the tribophysical (mechanical) activation could be used as a method for modification of physicochemical properties of dispersed systems such as polycrystalline mixture of oxides. In this study, we consider the properties of BaTiO₃ obtained from tribophysically activated initial powders. The mixture of 50 mol% BaCO₃ and 50 mol% TiO₂ powders was tribophysically activated in high energy vibromill curing 0, 3, 30, 90 and 180 min, calcinated at 800 degrees C for 1h and reaction sintered at 1100, 1200 and 1300 degrees C for 2 h. The surface specific areas, densities (green and sintered), phase compositions and dielectric properties were evaluated.

[33] PHASES IN LA₂O₃ AND NIO DOPED (ZR,SN)TiO₄ MICROWAVE DIELECTRIC CERAMICS

Houivet D. El Fallah J. Haussonne JM. - Journal of the European Ceramic Society. 19(6-7):1095-1099, 1999

Dielectric ceramics with composition in the ZrO₂-SnO₂-TiO₂ system containing La₂O₃ and NiO as sintering aids were prepared and investigated by XRD, XPS, SEM, EDS,; and microwave dielectric measurements. Ceramics prepared with deflocculated slurry using a Dyno-Mill(R), and sintered at 1370 degrees C exhibit very good microwave dielectric characteristics. epsilon = 37 and QF up to 62 000 at 4 GHz. We have observed a matrix phase with at least two or three different secondary phases in function of the employed grinding media (balls of zircon or magnesium stabilised zirconia). So, we have synthesised and characterised these phases.

[32] PREPARATION AND PROPERTIES OF BARIUM-FERRITE-CONTAINING GLASS CERAMICS

Muller R. Ulbrich C. Schuppel W. Steinmetz H. Steinbeiss E. - Journal of the European Ceramic Society. 19(6-7):1547-1550, 1999

Magnetic properties of magnetic ceramics are dependent on the magnetisation processes. They are usually dominated

by Bloch-wall displacements in multi-domain structures of the sintered magnetic material, even if the starting powder material shows single-domain Stoner-Wohlfarth behaviour. Domain wall motion limits in several cases the quality of magnetic materials for special applications like permanent magnets or rf devices. We have developed a new type of magnetic powder preparation which permits the fabrication of compact glass ceramics containing Ba-ferrite maintaining the single-domain behaviour of the starting powder. This technology is based on the glass crystallisation method for the precipitation of single-domain Ba-ferrite particles in a quenched melt of $\text{Fe}_2\text{O}_3\text{-BaO-B}_2\text{O}_3\text{(-SiO}_2\text{)}$. By milling the crystallized melt or leaching out the borate from the SiO_2 -containing glass, a glass ceramic powder containing Ba-ferrite can be produced, which can be sintered at moderate temperatures providing a dense compact glass ceramic containing up to 90 wt% Ba-ferrite. This compact glass ceramic shows the same single-domain behaviour as the starting powders, i.e. it also possesses the high coercivities of hard magnetic material.

[31] EFFECT OF MILLING OF V_2O_5 ON THE LOCAL ENVIRONMENT OF VANADIUM AS STUDIED BY SOLID-STATE V-51 NMR AND COMPLEMENTARY METHODS

Shubin AA. Lapina OB. Bosch E. Spengler J. Knozinger H. - Journal of Physical Chemistry B. 103(16):3138-3144, 1999

Milling of V_2O_5 in a ball mill increased the surface area from 7 to a maximum of 32 m^2/g . Simultaneously, XRD line broadening suggested a reduction of the microcrystallites. A color change during milling indicated oxygen loss, and Raman spectroscopy provided evidence for the formation of shear structures such as V_6O_{13} . The number of V^{4+} ions produced was determined by ESR spectroscopy and that of V^{3+} ions was obtained from magnetic susceptibility measurements. The local environment of the vanadium nucleus in V_2O_5 after milling in a ball mill was characterized by the combination of static (wide line) and MAS V-51 NMR techniques together with theoretical simulations of NMR spectra. Important information on the quadrupole and chemical shielding tensors, including the relative tensor orientation and the distribution of magnetic resonance parameters, is discussed for the vanadium nucleus in V_2O_5 after milling; Special attention was given to the formation of paramagnetic V^{3+} and V^{4+} ions and their influence on V-51 NMR spectra of diamagnetic V^{5+} in milled samples. It was shown that paramagnetic V^{3+} ions are responsible for the loss (up to about 70%) of the intensity in V-51 NMR spectra. The influence of other V^{4+} paramagnetic ions is significantly smaller.

[30] EFFECT OF PARTIAL MECHANICAL ALLOYING ON THE SELF-PROPAGATING HIGH-TEMPERATURE SYNTHESIS OF Ni_3Si

Lagerbom J. Tiainen T. Lehtonen M. Lintula P. - Journal of Materials Science. 34(7):1477-1482, 1999

Self-propagating high-temperature synthesis (SHS) is a new method for economical processing of intermetallic compounds and ceramic materials, as well as composites based on them. On the other hand, mechanical alloying is an effective method for producing highly metastable and, therefore, reactive metal powders. In this paper an overview of partial mechanical alloying is given. The effect of partial mechanical alloying on the self-propagating high-temperature synthesis of Ni_3Si -compounds is studied. The influence of alloying time on powder characteristics, e.g. particle size distribution, is given. The effect of alloying time on the properties of Ni-Si composite powders and on the characteristics of the SHS process, e.g. propagation rate, is reported. Ni_3Si was chosen as the object for this study because of its corrosion and high-temperature oxidation resistance. Like other $\text{L}_1(2)$ -type compounds, the strength of Ni_3Si shows an anomalous behaviour as a function of temperature; therefore, it has potential for high-temperature applications.

[29] EFFECTS OF SURFACE AREA, POLYMER CHAR, OXIDATION, AND NiO ADDITIVE ON NITRIDATION KINETICS OF SILICON POWDER COMPACTS

Bhatt RT. Palczar AR. - Journal of Materials Science. 34(7):1483-1492, 1999

The oxidative stability of attrition-milled silicon powder under reaction-bonding processing conditions has been determined. The investigation focused on the effects of surface area, polymer char, preoxidation, nitriding environment, and a transitional metal oxide additive (NiO) on the nitridation kinetics of attrition-milled silicon powder compacts tested at 1250 and 1350 degrees C for 4 h. Silicon powder was wet-attrition-milled from 2 to 48 h to achieve surface areas (SAs) ranging from 1.3 to 63 m^2/g . A silicon powder of high surface area (63 m^2/g) was exposed for up to 1 month to ambient air or for up to 4 days to an aqueous-based solution with the pH maintained at 3, 7, or 9. Results indicated that the high-surface-area silicon powder showed no tendency to oxidize further, whether in ambient air for up to 1 month or in deionized water for up to 4 days. After a 1-day exposure to an acidic or basic solution, the same powder showed evidence of oxidation. As the surface area increased, so did the percentage nitridation after 4 h in N_2 at 1250 or 1350 degrees C. Adding small amounts of NiO significantly improved the nitridation kinetics of high-surface-area powder compacts, but both preoxidation of the powder and residual polymer char delayed it. Conversely, the nitridation environment had no significant influence on the nitridation kinetics of a high-surface-area powder. Impurities present in the starting powder, and those accumulated during attrition milling, appeared to react with the silica layer on the surface of silicon particles to form a molten silicate layer, which provided a path for rapid diffusion of N_2 and enhanced the nitridation kinetics.

[28] SYNTHESIS AND CHARACTERIZATION OF SUBMICRON ZIRCONIA-12 MOL% CERIA CERAMICS

Muccillo ENS. Avila DM. - Ceramics International. 25(4):345-351, 1999

Zirconia powders containing 12 mol% ceria have been prepared by the coprecipitation technique. The aim of the present work was to obtain nanosized powders with suitable sinterability and reduced grain size in the sintered

ceramic by means of this solution technique in its simplest route, that is, without using any milling or other special procedure. To accomplish those requests some processing variables have been systematically studied. For comparison purposes, specimens of the same composition have been prepared by the powder mixing technique. The optimization of some processing variables allowed for obtaining sintered specimens with apparent densities of 98% of the theoretical value, 100% of tetragonal phase, and 500 nm of average grain size.

[27] ULTRA GRAIN REFINING OF STEELS AND DISSOLUTION CAPACITY OF CEMENTITE BY SUPER-HEAVY DEFORMATION [JAPANESE]

Hidaka H. Kimura Y. Takaki S. - *Tetsu to Hagane-Journal of the Iron & Steel Institute of Japan*. 85(1):52-58, 1999
Mechanical milling using high energy planetary ball mill was applied to Fe-C alloy powders with (ferrite+cementite) two-phase structures to give an ultimate large strain into the powders. Dissolution behavior of cementite during mechanical milling was investigated in relation to ultra grain refining of ferrite matrix, and dissolution capacity of cementite was discussed in terms of carbon content in the powders. Ultra grain refining of ferrite matrix to about 10nm results in full dissolution of cementite in the powders with carbon up to 2 mass% C. Most of carbon, which has been rejected from decomposed cementite, is suggested to segregate at grain boundary to form amorphous layer. Thus, it was proposed that the dissolution limit of cementite depends on both volume fraction of the grain boundary amorphous layer and carbon concentration therein. For example, as the maximum carbon content of the grain boundary amorphous layer was to be about 4.2 mass% C, the dissolution limit of cementite was estimated at 30 vol% from the mass balance for carbon content in the case ferrite grains were refined to around 10 nm. This volume fraction of cementite is just correspondent to that in Fe-2mass%C alloy.

[26] ULTRA GRAIN REFINING AND DECOMPOSITION OF OXIDE DURING SUPER-HEAVY DEFORMATION IN OXIDE DISPERSION FERRITIC STAINLESS STEEL POWDER

Kimura Y. Takaki S. Suejima S. Uemori R. Tamemuro H. - *ISIJ International*. 39(2):176-182, 1999
Mechanical milling using a high energy planetary ball mill was applied to the powder mixtures of iron, chromium and yttria (Y₂O₃) (Fe-24mass%Cr-0-15mass%Y₂O₃) to introduce a very large strain into the iron-base matrix, and microstructural changes during mechanical milling were investigated in relation to decomposition behavior of Y₂O₃ particles. Mechanical milling of more than 36 ks was long enough to allow the mechanical alloying of iron and chromium powders. After the milling of 36 ks, ultrafine bcc crystalline grains of 10 to 20 nm were formed within Fe-24mass%Cr-15mass%Y₂O₃ powder mixture and 15 mass% of Y₂O₃ particles were almost decomposed. The resultant powder mixture markedly hardened to about 1 000 Hv. The decomposition of Y₂O₃ particles can be explained as being due to the formation of an amorphous grain boundary layer where yttrium and oxygen atoms are enriched. As a result, it is proposed that, for the dissolution of Y₂O₃, bcc crystalline grains should be refined to a nanometric size to provide a sufficient volume fraction of the grain boundary layer, and that Y₂O₃ particles should be crushed to several nanometers to produce the driving force for the decomposition of Y₂O₃ particles.

[25] NATURE OF THE ANOMALOUS DISPERSION OF PARTICLES IN FULLERITE-ALKALI-METAL-HALIDE COMPOSITES

Reznikov VA. Sukhanov AA. - *Technical Physics Letters*. 25(4):313-314, 1999
The occurrence of a solid-phase reaction between powders of the fullerite C-60 and potassium halides under the conditions of mechanical grinding is established. The electrostatic potential and surface-active properties of C-60 molecules are considered as causes of the anomalously high degree of dispersion of the particles within a composite.

[24] FRICTION INDUCED MECHANOCHEMICAL AND MECHANOPHYSICAL CHANGES IN HIGH PERFORMANCE SEMICRYSTALLINE POLYMER

Li TQ. Zhang MQ. Song L. Zeng HM. - *Polymer*. 40(16):4451-4458, 1999
By using X-ray photoelectron spectroscopy (XPS) and Fourier transform Raman (FT-Raman) spectroscopy, the worn specimens of polyetheretherketone (PEEK) tested under unlubricated sliding friction and wear conditions at a constant sliding speed were investigated in order to reveal mechanochemically and mechanophysically induced structural changes of polymer as well as wear mechanisms on a molecular scale. Chain scission was found on the worn surface layer. The results suggest that oxidation was the major mechanochemical reaction that followed the chain scission on the top surface. Evidence for chain branching or even crosslinking in bulk materials was also presented. Moreover, two-stage loading dependencies were found for both surface and subsurface in the bulk as revealed through wear rate measurement and wear debris analysis. It was proved that a thermo-activation of polymer segments may be responsible for the transition in the dependence of structure on load. The results of the present work also provide a method (based on spectral analyses) that can be used for studying micromechanisms accounting for shear deformation and failure.

[23] ENHANCEMENT OF SELF-SUSTAINING REACTION BY MECHANICAL ACTIVATION: CASE OF AN FE-SI SYSTEM

Gras C. Gaffet E. Bernard F. Niepce JC. - *Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing*. 264(1-2):94-107, 1999
Mechanical high energy ball milling of an Fe + 2Si elemental powders mixture was used to activate a self sustaining combustion reaction or so-called self-propagating high-temperature synthesis (SHS) to form iron disilicide, a reaction for which the thermodynamic criterion is not favorable. A complete characterization of the milled powders before reaction was performed with energy dispersive X-ray spectrometry, specific surface measurements and X-ray diffraction profile analysis. Thermal and structural information describing the combustion front initiated by heating up a sample to 400 degrees C in a Fe-Si system is communicated. In order to isolate the phases involved in the gasless reaction, a time-resolved X-ray diffraction experiment was designed to study in situ the formation of silicide phases (FeSi and beta-FeSi₂) produced by the new process called MASHS (mechanically activated self-propagating high-temperature synthesis).

[22] LOW TEMPERATURE PRESSURELESS SINTERING OF SOL-GEL DERIVED MULLITE

Amutharani D. Gnanam FD. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 264(1-2):254-261, 1999

Mullite of stoichiometric composition has been synthesized by sol-gel process. Aluminium nitrate and ethyl silicate are used as the starting materials to prepare the single phase mullite powder. DTA shows that the mullitization starts at 971 degrees C. It is observed from XRD analysis that the amorphous mullite completely transforms to orthorhombic crystalline mullite at 1300 degrees C. Wet milling operation has enhanced the sintering density of pure mullite by 12% more than that of dry milling. The naturally available raw material, clay has been identified as one of the suitable sintering aids for mullite. The addition of clay to the mullite powder enhances the density up to 95% of TD even at a very low temperature, 1450 degrees C by pressureless sintering. A viscous flow sintering mechanism during densification is one of the main criteria to achieve good densification.

[21] THERMAL AND MAGNETIC PROPERTIES OF BULK GLASS FORMING FE-AL-P-C-B-(GA) ALLOYS

Schlorke N. Eckert J. Schultz L. - Journal of Physics D-Applied Physics. 32(8):855-861, 1999

High-energy ball milling of pre-alloyed ingots, rapid quenching and mechanical alloying of elemental powder mixtures are used to prepare amorphous Fe-Al-P-C-B(Ga) alloys, which are known to have interesting soft magnetic properties combined with a good glass-forming ability, promising the formation of bulk soft magnetic materials. Pieces of the melt-spun ribbon with a coercivity of 7 A m(-1) are ball milled to investigate the influence of the milling process on the thermal and magnetic properties. X-ray diffraction analysis reveals the amorphous nature of all samples after milling. The samples exhibit a supercooled liquid region $\Delta T_x (= T_x - T_g)$ varying in the range 44-53 K between the glass-transition temperature T_g and the crystallization temperature T_x . Annealing of the powders was performed in this temperature region to reduce the high stresses induced during milling while maintaining the amorphous structure. The lowest coercivities H_c after annealing of the powders are found to be correlated to the oxygen content. Ball milling of the melt-spun ribbon leads to a slight decrease of the thermal stability, an oxygen content of 0.21 at% and a high H_c of 1620 A m(-1) which drops to 60 A m(-1) upon subsequent annealing above T_g . Mechanically alloyed powders with $\Delta T_x = 44$ K and 1.2 at% oxygen exhibit a coercivity of 170 A m(-1) and a saturation polarization $J(s)$ of 1.02 T after heating above T_g to 748 K. The coercivity of 77 A m(-1) found for ball milling of crystalline ingots (0.16 at% oxygen, $J(s) = 1.2$ T) after heating to 703 K is of the same order of magnitude as that of cast amorphous material, which was reported to be 12.7 or 82 A m(-1). Nonmagnetic inclusions are supposed to lead to the difference in H_c between the as-spun ribbon on the one hand and the ball-milled, mechanically alloyed or cast samples on the other.

[20] ANNEALING EFFECTS ON THE COERCIVITY OF SMC05 NANOPARTICLES

Chowdary KM. Giri AK. Pellerin K. Majetich SA. Scott JHJ. - Journal of Applied Physics. 85(8 Part 2A):4331-4333, 1999

The enhanced coercivity of ball milled nanoparticles of SmCo5 has been attributed to the reduced particle size, but the milling process introduces additional strain which can also affect the coercivity. Here the effects of size and strain are decoupled from each other using a combination of x-ray diffraction, electron microscopy, and coercivity measurements. Ball milled SmCo5 nanoparticles were annealed under different temperature, time, and atmospheric conditions to minimize the strain without chemically altering the sample, and with minimal grain growth and sintering. X-ray diffraction was used to determine if phase changes had occurred, and to monitor the average grain size and strain. From scanning electron microscopy the log-normal size distributions were found and no evidence of sintering was revealed. The sample coercivities were measured by superconducting quantum interference device following annealing.

[19] X-RAY ABSORPTION, NEUTRON DIFFRACTION, AND MOSSBAUER EFFECT STUDIES OF MNZN-FERRITE PROCESSED THROUGH HIGH-ENERGY BALL MILLING

Fatemi DJ. Harris VG. Chen MX. Malik SK. Yelon WB. Long GJ. Mohan A. - Journal of Applied Physics. 85(8 Part 2A):5172-5174, 1999

MnZn-ferrite has been prepared via high-energy ball milling of elemental oxides MnO, ZnO, and alpha-Fe2O3. Neutron diffraction measurements suggest a high density of vacancies in a spinel structure. The spinel phase appears to comprise 99.8 wt% of the material in the sample milled for 40 h, with the remainder attributable to unreacted alpha-Fe2O3. The x-ray absorption near-edge structure was analyzed to provide an understanding of the charge state of the constituent Fe ions. This analysis reveals about 2/3 of Fe cations to be trivalent, increasing to about 3/4 after a 5 h anneal at 450 degrees C. The heat treatment is also observed to induce a cation redistribution in the ball-milled ferrite toward that of a standard processed via ceramics methods. Results from Mossbauer spectroscopy determine the average hyperfine fields in the sample milled 40 h to be 289 and 487 kOe at 295 and 78 K, respectively. The average isomer shift is 0.32 mm/s at 295 K and 0.46 mm/s at 78 K, values which are typical of iron (III) in a spinel oxide lattice. As expected for a cubic-like environment, the quadrupole shifts are very small, ranging from 0.07 mm/s at 295 K to 0.00 mm/s at 78 K.

[18] HIGHLY COERCIVE SMC05 MAGNETS PREPARED BY A MODIFIED HYDROGENATION-DISPROPORTIONATION-DESORPTION-RECOMBINATION PROCESS

Kubis M. Handstein A. Gebel B. Gutfleisch O. Muller KH. Schultz L. - Journal of Applied Physics. 85(8 Part 2B):5666-5668, 1999

The hydrogenation-disproportionation-desorption-recombination (HDDR) process was applied to SmCo5 using extreme conditions, namely high hydrogen pressures and reactive milling under hydrogen. Investigations on the hydrogen absorption behavior of SmCo5 by differential scanning calorimetry under hydrogen pressures between 1 and 7 MPa showed absorption events due to an interstitial absorption at about 100 degrees C and a disproportionation reaction at about 600 degrees C. X-ray diffraction showed the disproportionation of SmCo5 into Sm hydride and fcc-Co. A favorable effect of high hydrogen pressures on the disproportionation reaction was observed which can be explained by a decrease of the free enthalpy of the samarium hydride for increasing hydrogen pressures. Reactively

milled SmCo₅ showed also the products of the disproportionation reaction. The recombination to the original SmCo₅ phase on hydrogen desorption in a subsequent heat treatment in vacuum was successful for both methods. However, Sm₂O₃, Sm₂Co₁₇, and Sm₂Co₇ were detected as minor phases. Maximum coercivities $\mu_0 H(C)$ of 2.1 and 4.7 T were achieved for high pressure and reactively milled HDDR powders, respectively. The high coercivities originate from the high anisotropy field of the SmCo₅ phase in combination with the grain refinement due to the HDDR treatment.

[17] AMORPHIZATION OF ZR-AL SOLID SOLUTIONS UNDER MECHANICAL ALLOYING AT DIFFERENT TEMPERATURES

Sheng HW. Lu K. Ma E. - Journal of Applied Physics. 85(9):6400-6407, 1999

The effects of temperature on the amorphization of Zr-Al solid solutions have been investigated by ball milling Zr_{100-x}Al_x powder blends at different temperatures. At low milling temperatures, the Zr-Al solid solutions amorphized under the polymorphic constraint imposed by intensive external forcing. At elevated temperatures, the solid solution and the amorphous phases coexisted in an obvious two-phase region, signaling a transition approaching two-phase metastable equilibrium. The Al concentration needed for the complete amorphization of Zr-Al increased with increasing milling temperature. These observations, and in particular, the apparent reentrant liquidus, can be explained in terms of the temperature dependence of the external forcing effects brought in by the nonequilibrium milling process in this dynamic driven system.

[16] MECHANICAL ACTIVATION OF THE REACTIVE SINTERING OF TITANIUM CARBIDE

Chernikova ES. Timofeeva IL. Uvarova IV. Bykov AI. Skorokhod VV. Smirnov VP. - Powder Metallurgy & Metal Ceramics. 37(5-6):265-269, 1998

This paper describes the mechanical alloying of Ti and C powders by intensive grinding in a planetary mill. The effect of milling conditions and initial composition on the compositional homogeneity and properties of powders and samples consolidated by hot-pressing was studied by x-ray diffraction, scanning electron microscopy, and surface area measurement. The diffraction patterns indicated that a powder mixture ground for 24 hours transforms to TiC after sintering for only one minute at 600 degrees C under a pressure of 3 GPa.

[15] POLYMERIZATION OF BUTADIENE WITH ORGANOMETALLIC COMPOUNDS PREPARED BY THE REACTION OF BENZENE FUNCTIONAL DERIVATIVES AND LANTHANIDES UNDER THE CONDITIONS OF MECHANICAL ACTIVATION [RUSSIAN]

Yakovlev VA. Tinyakova EI. Gol'shtein SB. Sokolova VL. Bondarenko GN. - Vysokomolekulyarnye Soedineniya Seriya a & Seriya B. 41(3):556-559, 1999

Polymerization of butadiene initiated by organolanthanide compounds prepared by the reaction of lanthanides with the benzene functional derivatives conducted using mechanical activation (ball milling) was studied. It was shown that the stereospecificity of the organolanthanide compounds under investigation is determined by the nature of the metal and the functional groups of benzene derivatives.

[14] ULTRA-FINE GRAINED BULK STEEL PRODUCED BY ACCUMULATIVE ROLL-BONDING (ARB) PROCESS

Tsuji N. Saito Y. Utsunomiya H. Tanigawa S. - Scripta Materialia. 40(7):795-800, 1999

Much attention has been directed recently to ultra-grain refining of metallic materials, where the grain size is reduced to less than 1 μ m. It is expected that submicrometer grained structure would result in high strength and toughness at ambient temperature [1] as well as high strain rate or low temperature superplasticity at elevated temperatures [2,3]. It has been reported recently that ultra-fine grains can be obtained by intense plastic straining [4]. Several special intense straining processes, such as Torsion Straining (TS) [5-10], Equal-Channel Angular Pressing (ECAP) [8-12] and Mechanical Milling (MM) of powder metals [13-15], have succeeded in producing ultra-fine grains. On the other hand, the disadvantage of these processes is that they are not applicable to large bulk materials. Not small functional materials but large structural materials do require high strength and toughness. From this critical viewpoint, we have recently developed a novel intense straining process for bulk materials, named Accumulative Roll-Bonding (ARB) [16]. We firstly tried to apply ARE to the aluminum alloys, and the bulk sheets with ultra-fine grains whose grain sizes are several hundred nano-meters were successfully produced [17,18]. The ARBed aluminum alloys with ultra-fine grains showed large strength at ambient temperature, which is up to 3.7 times larger than that of the starting materials [17,18]. Further, it has been also clarified that the ARBed Al-Mg alloy sheet with submicrometer grains shows low-temperature superplasticity at 473 K which is half the melting temperature of the material [19]. The purpose of the present study is to clarify whether or not it is possible to produce the bulk steel sheets with ultra-fine grains by ARE process. Because steel is the most useful structural material, the ultra-grain refining of steel is greatly desired. The ultra-grain refining and resulted strengthening of steels could largely reduce the weight of any constructions, and the strengthening without alloying elements would be preferable for recycling. However, no investigation concerning the intense straining of bulk steels has been carried out by now possibly due to the difficulty in processing, although limited results about small materials, such as grain refining by powder metallurgical process (MM) [13-15] or TS of thin discs [7], have been reported.

[13] TEXTURE DEVELOPMENT IN S200-D, -E AND P31664 BERYLLIUM BLOCKS FROM NEUTRON DIFFRACTION SPECTRA

Bennett K. Varma R. Von Dreele RB. - Scripta Materialia. 40(7):825-830, 1999

The mechanical behavior of beryllium is important in a range of Department of Energy (DOE) weapons applications. Its unique high elastic modulus, modulus-to-density ratio and strength-to-density ratio make beryllium an ideal material for aerospace vehicles and weapons components (1-3). Because of its unique properties and the range of important applications, beryllium has already been extensively investigated. Strain rate sensitivity, work hardening, temperature-dependent viscosity, anisotropic fracture, grain size effects and oxide content are a few physical and chemical factors that have been measured and collated to create a database allowing prediction of its constitutive properties (4-8). Finite element codes have been used to simulate stress during crack propagation in metals that may be applied to beryllium (9). However, successful utilization of beryllium depends on an accurate description of its

microstructure as a function of fabrication and forming history. Texture, preferred crystallographic orientation, is an important microstructural parameter that we use in this paper to characterize structure as a function of manufacturing process for three archived beryllium structural grades. Los Alamos National Laboratory (LANL) maintains a stock of various vintages of structural (nuclear grade) beryllium which has been made by a variety of fabrication processes (3). Texture in beryllium has been observed qualitatively in previous studies, however it has never been investigated systematically or quantitatively (10,8). In this study we determine quantitatively the texture as a function of processing history in specimens cut from three vacuum hot pressed (VHP) block beryllium grades S200-D, -E, and P31664 which have been used in various components over the years. Hot pressing of metal powders has been known to produce preferential alignment of crystals during formation (11). Moreover, it has been shown that the standard production method of beryllium powder creates anisomorphic grain shape and preferred alignment of crystals (3). The vintages in this study were hot-pressed from processed beryllium powder, and therefore texture was anticipated. Texture is directly linked to anisotropic physical properties of (12). For predicting material anisotropic fracture behavior and flow in beryllium, a knowledge of texture is critical. It has long been recognized that single crystals of beryllium exhibit highly anisotropic behavior (for a through review see (13)). The deformation of beryllium at ambient temperature occurs by motion of dislocations with burgers vectors of $(1\ 1\ 2)$ over bar 0, the direction of closest atom packing. Such dislocations can move on the (0001) plane, which is the closest packed plane. They can also move on $(10\ 1)$ over bar 0 prism planes, often at much higher resolved shear stress. The operation of $(1\ 1\ 2)$ over bar 0 $(10\ 1)$ over bar 0, temperatures. Characterizing beryllium texture quantitatively performance prediction of components made from it. temperature. of one or the ether systems, $(1\ 1\ 2)$ over bar 0 (0001) or 1 will depend on which system is most favorably oriented to the direction of applied stress (14). The brittleness of polycrystalline beryllium results from two circumstances. First, the number of independent slip systems is not sufficient to satisfy the Von Mises requirement of 5 independent slip systems in order re, maintain grain-to-grain compatibility. Coincidentally, at ambient temperature there are no mobile dislocations with a c-axis component. Secondly, cleavage fracture is $1\ 1$ also common in beryllium because the stress for cleavage on basal (0001) planes is quite low at ambient temperatures. Characterizing beryllium texture quantitatively is thus crucial for mechanical analysis and Processing of vacuum cast beryllium into powder has been historically carried out by Braun-type attrition methods when a machined beryllium swarf is ground between two opposed beryllium disks to a powder that is then mechanically sieved inside a vacuum. Particles less than 44 micron diameter are then used for consolidation. This method was used for both S200-D and -E vintages. This method produces flattened powder particles because basal cleavage is favored. In the 1970's the method for processing of vacuum cast beryllium into powder was changed in order to increase the mechanical isotropy of the material (by producing more isomorphic grains, thus decreasing the difference between longitudinal and transverse properties). New methods such as impact grinding and ball milling were introduced to beryllium powder processing. During impact grinding a beryllium swarf is Do-ground, usually by pin milling, and pre-ground particles are accelerated through a gas stream, impacting on a beryllium target. Particles less than 44 microns in diameter are collected by a cyclone separator for consolidation. While impact grinding and Braun-type attrition produced similar grain-size beryllium powders, they resulted in very different grain morphologies. The latter method produced flat pancake-like grains and the former produced round grains (3). The three vintages in this study were all vacuum hot pressed (VHP). In this procedure beryllium powder is compacted under uniaxial pressure at high In this paper we present a quantitative texture analysis of 14 specimens cut from either S200-D -E or P31664 vintage billets from the LANL archive. The billets were sectioned for time-of-night (TOF) neutron diffraction measurements to allow comparison of microstructural differences between vintages. Neutron diffraction is ideal for investigating structural prop ties in beryllium because the neutron absorption of beryllium is negligible and bulk (2 cm(3)) specimens can therefore be investigated. Moreover, the polychromatic TOF radiation in this study provides significant statistical advantages for texture measurements compared to the traditional method of monochromatic x-ray diffraction. Even order harmonic coefficients are extracted from a combination of 50 - 60 TOF neutron diffraction spectra containing a total of 40,000-60,000 data points via a Rietveld refinement method (15). Selected pole figures are subsequently calculated from these harmonic coefficients.

[12] SYNTHESSES OF Zn(BH₄)₂ AND RbBH₄ CENTER DOT 18C6 USING MECHANOCHEMICAL ACTIVATION

Mal'tseva NN. Kedrova NS. Gorobinskiy LV. Petrova LA. Makhaev VD. -Russian Journal of Coordination Chemistry. 25(4):246-248, 1999

Mechanochemical activation (MCA) in the syntheses of the thermally unstable Zn(BH₄)₂ and RbBH₄ . L (L = crown ether) compounds was shown to be promising. The optimum parameters are determined for the synthesis of Zn(BH₄)₂ by the metathesis reaction between mechanochemically activated zinc chloride and sodium tetrahydroborate. MCA is shown to be applicable in the synthesis of the complexes by the reaction RbBH₄ + L = RbBH₄ . L, where L = 18C6. The synthesis and physicochemical properties of the RbBH₄ . 18C6 complex are described.

[11] A RESEARCH ON DETONATION GUN COATING WITH FE-SiC COMPOSITE POWDERS MECHANICALLY ACTIVATED

Jia CC. Li ZC. Xie ZZ. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 263(1):96-100, 1999

The Fe-SiC composite powder prepared by the mechanical activation process has been used for coating on materials with the detonation gun (D-gun) machine in order to develop a new way for coating. It has been found that the coating layer has fine, homogeneous, dense structure and good wear resistance. The results of SEM and X-ray diffraction (XRD) show that some reactions happened between Fe and SiC during the D-gun coating, the Fe-Si compounds formed and SiC strength the coating layer. It has been proved that the technology combined mechanical alloying with D-gun coating is a new method for surface modification.

[10] POLY(BUTYLENE TEREPHTHALATE) POLY(ETHYLENE TEREPHTHALATE) MIXTURES FORMED BY BALL MILLING

Font J. Muntasell J. Cesari E. - Materials Research Bulletin. 34(1):157-165, 1999

Mixtures of poly(butylene terephthalate) (PET) and poly(ethylene terephthalate) (PET) polymers were formed using ball milling technique and characterized by means of differential scanning calorimetry (DSC) and X-ray diffraction (XRD). PET with different degrees of crystallinity was used in the solid-state mechanical milling process. Results suggest that PET greatly influences the crystallization of amorphized PET by milling.

[9] PREPARATION AND CHARACTERIZATION OF PEROVSKITE CERAMIC POWDERS BY GELCASTING

Wang HT. Xie S. Lai W. Liu XQ. Chen CS. Meng GY. - Journal of Materials Science. 34(6):1163-1167, 1999

Perovskite $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.8}\text{Fe}_{0.2}\text{O}_{3-\delta}$ (LSCF) powders have been successfully synthesized from oxide and carbonates based on the principle of gelcasting. Phase-forming temperature is very dependent on the ball-milling process during the suspension preparation. As the ball-milling time is increased, the temperature of phase formation decreases, therefore the perovskite powder obtained has a larger Brunauer-Emmett-Teller (BET) specific surface area. The grain sizes were around 1 μm at 1000 degrees C and 2 μm at 1100 degrees C from scanning electron microscopy (SEM) photographs. The perovskite powders have good sinterability: the sintering densities of ceramic bodies shaped with as-prepared powders were investigated. SEM photos show that sintered ceramics exhibit a well defined morphology in the packing and sintering of particles. The oxygen permeance of disc shaped samples, with a thickness ranging from 1.02 to 1.98 mm was 6.39×10^{-8} - 1.99×10^{-8} mol cm^{-2} s^{-1} at 900 degrees C indicating that LSCF ceramics have high oxygen permeation. It can be concluded that gelcasting is a simple and effective method for preparing practical multicomponent perovskite powders.

[8] NANOCRYSTAL FORMATION, AMORPHIZATION AND SUPERCONDUCTIVITY IN YNi2B2C

Ledig L. Hough D. Oertel CG. Eckert J. Skrotzki W. - Journal of Alloys & Compounds. 285(1-2):27-36, 1999

The effect of heavy plastic deformation during ball milling on phase formation, thermal stability and superconducting properties of the YNi₂B₂C intermetallic compound has been investigated by X-ray diffraction, transmission electron microscopy, thermal analysis and ac-susceptibility measurements. Depending on the ball milling parameters either nanocrystalline YNi₂B₂C or an amorphous phase is observed after extended milling. Crystallization of the amorphous phase reproduces the YNi₂B₂C intermetallic compound. The superconducting properties deteriorate upon milling and no superconductivity is found for the amorphous phase. However, annealing at 893 K recovers superconductivity. This is discussed with respect to the nanocrystalline or amorphous state of the powders and deviations from stoichiometry caused by impurities introduced during milling.

[7] HYDRIDING AND DEHYDRIDING CHARACTERISTICS OF AN AMORPHOUS Mg2Ni-Ni COMPOSITE

Iwakura C. Nohara S. Zhang SG. Inoue H. - Journal of Alloys & Compounds. 285(1-2):246-249, 1999

Hydriding and dehydriding characteristics of a Mg,Ni-Ni composite prepared by ball-milling Mg,Ni alloy with 70 wt. % Ni (Mg₂Ni/Ni=1/1.28 in mole ratio) were investigated. As a result, it was found that the Mg₂Ni-Ni composite showed a marked increase in the hydriding rate and the dehydriding commenced at much lowered temperature or even at room temperature, compared with Mg,Ni and ball-milled Mg,Ni alloys. Such a marked improvement in the hydriding and dehydriding characteristics, as well as the charge-discharge characteristics, by ball-milling the Mg₂Ni alloy with Ni is probably due to the appearance of the homogeneous amorphous structure over the whole alloy particles.

[6] METAL HYDRIDE ELECTRODES PREPARED BY MECHANICAL ALLOYING OF ZrV2-TYPE MATERIALS

Jurczyk M. Rajewski W. Wojcik G. Majchrzycki W. - Journal of Alloys & Compounds. 285(1-2):250-254, 1999

The structural and electrochemical properties of a range of alloys, including: ZrV₂ and ZrV₂/Ni (10 wt% Ni), which have the cubic C15 type structure and Zr_{0.35}Ti_{0.65}V_{0.85}Cr_{0.26}Ni_{1.30}, which has the hexagonal C14 type structure, have been investigated. These alloys have been prepared using mechanical alloying (MA) followed by annealing. The amorphous phase forms directly from the starting mixture of the elements, without other phase formation. Heating the MA samples at 1070 K for 1 h resulted in the creation of ordered ZrV₂-type alloys. In mechanically alloyed, amorphous ZrV₂, ZrV₂/Ni materials and Zr_{0.35}Ti_{0.65}V_{0.85}Cr_{0.26}Ni_{1.30} material discharge capacities of 0, 38 and 90 mAhg⁻¹, were obtained, respectively. Annealed nanocrystalline powders have greater capacities than the amorphous parent materials.

[5] NEW APPROACH FOR SYNTHESIZING MG-BASED ALLOYS

Sun D. Enoki H. Gingl F. Akiba E. - Journal of Alloys & Compounds. 285(1-2):279-283, 1999

Mg₂Ni, Mg₂Cu and MgZn₂ were synthesized by means of ball-milling plus solid-state reactions. Firstly, the mixtures of the two elemental powders, corresponding to the compositions of Mg₂Ni, Mg₂Cu and MgZn₂, were subject to ball-milling for 20 min. Then, they were pressed into pellets and sintered at temperatures below the melting points of the components. X-ray diffraction experiments show that the yields of the desired products are above 97 wt.%, higher than those prepared by the conventional metallurgical method. The hydrogenation properties of the Mg₂Ni sample synthesized by solid-state reaction are in agreement with the previously published results on alloys prepared by the conventional methods.

[4] ENERGY DISTRIBUTION OF HYDROGEN SITES FOR MGNI(0.86)ML(0.03) (ML=CR, FE, CO, MN) ALLOYS DESORBING HYDROGEN AT LOW TEMPERATURE

Tsushio Y. Enoki H. Akiba E. - Journal of Alloys & Compounds. 285(1-2):298-301, 1999

The energy distribution of hydrogen sites for MgNi(0.80)M₁(0.03), (M₁=Cr, Fe, Co, Mn) alloys prepared by the ball milling technique was investigated. From the results of DSC, TG analysis and the calculation of the enthalpy of hydrogenation, we derived that the Cr and Fe-substituted alloys have a broad energy distribution and the averaged enthalpy of hydrogenation is about -82 kJ mol⁻¹ H₂. Because of the broad distribution, the hydride of larger enthalpy decomposes at low temperature. However, the Co and Mn-substituted alloys have sharp energy distributions though the averaged value of hydrogenation is about -67 kJ mol⁻¹ H₂. Therefore, these alloys do not desorb hydrogen at low temperature. In order to develop Mg-based alloys that desorb hydrogen at low temperature, the dependence of substitution element on the energy distribution should be considered and substitution elements that make the energy distribution broad must be selected.

[3] THE ELECTROCHEMICAL EVALUATION OF BALL MILLED MGNI-BASED HYDROGEN STORAGE ALLOYS

Han SC. Jiang JJ. Park JG. Jang KJ. Chin EY. Lee JY. - Journal of Alloys & Compounds. 285(1-2):L8-L11, 1999
The electrochemical properties of amorphous MgNi-based hydrogen storage alloys synthesized by ball milling were evaluated. In order to improve the cycle life, the surface of the amorphous Mg₅₀Ni₅₀ alloy was coated with Ti, Al and Zr by ball milling. Among them Ti was very effective to improve cycle life. And two kinds of MgNi-based amorphous alloys were designed by the substitution of Ti and other elements for Mg of MgNi-based alloys, which were composed of four components. Thus, the cycle life of electrodes with these quaternary amorphous alloys was strongly improved.

[2] ACTIVE/INACTIVE NANOCOMPOSITES AS ANODES FOR LI-ION BATTERIES

Mao O. Turner RL. Courtney IA. Fredericksen BD. Buckett MI. Krause LJ. Dahn JR. - Electrochemical & Solid-State Letters. 2(1):3-5, 1999

Composites consisting of "active" grains which can alloy with lithium and "inactive" grains which cannot have been made by mechanical alloying of elemental powders. The inactive grains act as a matrix to hold the active grains as they repeatedly alloy with lithium during the operation of a lithium battery. A microscopic mixture of 25% Sn₂Fe (active) and 75% SnFe₃C (inactive) shows a volumetric capacity for Li which is more than twice that of the graphite materials which are now the anode of choice for the Li-ion battery. Unlike pure Li-Sn and Li-Al alloys, the composite retains this capacity for many charge-discharge cycles suggesting that materials like this will be the anodes of choice for the next generation of compact, high energy, Li-ion batteries.

[1] NANOCRYSTALLINE AL₈₇NI_{8.7}Y_{4.3} AND AL₉₀FE₅GD₅ ALLOYS THAT RETAIN THE LOCALIZED CORROSION RESISTANCE OF THE AMORPHOUS STATE

Sweitzer JE. Scully JR. Bley RA. Hsu JWP. - Electrochemical & Solid-State Letters. 2(6):267-270, 1999

The localized corrosion properties of Al₈₇Ni_{8.7}Y_{4.3} and Al₉₀Fe₅Gd₅, were investigated in the amorphous, high strength partially nanocrystalline, and fully crystalline states. The critical pitting potentials and pit growth behaviors of amorphous and nanocrystalline alloys were improved compared to high purity, polycrystalline Al in 0.6 M NaCl solution. Transition metal (TM) and rare earth additions (RE) were retained in solid solution in amorphous alloys and the remaining amorphous matrix of partially nanocrystalline alloys. Substantial TM and RE solute rejection occurred from nano- and microcrystals. Notably, the improved resistance to pitting was retained in the partially nanocrystalline condition, but was completely lost in the fully crystalline alloys. The opportunity exists to create partially nanocrystalline Al-TM-RE alloys of great strength with corrosion resistance equal to their amorphous counterparts.

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