

RESEAU FRANÇAIS DE MECANOSYNTHESE

Lettre N° 53

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

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

1 Nouvelle Adhésion

L. Beaulieu - Dpt of Physics - Dalhousie University - Halifax (N.S.) - B3H3J5 - Canada

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<http://www.bls.fr/amatech>

Rubrique Pages Sciences et Techniques pour l'Ingénieur (Rubrique Sciences)

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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

JRFM'99

**Le compte rendu
des JRFM'99**

**sera bientôt disponible sur le site web du RFM
Programme définitif
et Résumés des Différentes Contributions**

(100 Participants ont assisté à nos Journées Annuelles)

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ANNONCE DE CONGRES ET / OU ECOLES
CONGRESS AND SCHOOL ANNOUNCEMENTS

=====

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/>

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

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)

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.

**International Workshop on
APPLIED ASPECTS OF INTERFACE SCIENCE (AAIS)
(St.Petersburg, Russia; September 22 - 24, 1999)**

Announcement:

If any one technology can be said to shape the face of today society, it is materials technology. New materials, materials processes and applications are of major importance in boosting other military and civilian technological sectors such as electronics, energy technology and aerospace engineering. The objective of this Workshop is to present current research on advanced nanostructured materials and high temperature superconducting materials (characterized by nano-scaled coherence length) with the special attention being paid to the effect of interfaces on the unique, commonly highly desired properties of such materials as well their applications in various high technology areas. The Workshop aims to assess the current status and to identify future directions of R&D and applications of nanostructured bulk solids, films and coatings as well as polycrystalline superconducting materials. Particular emphasis is placed on developing close interactions and fostering future collaborations among scientists and engineers from the USA, Russia and other countries. We plan to focus to the following topics: Synthesis and processing; Modeling; Characterization and properties (mechanical and transport properties of nanostructured materials, high- and low-current properties of superconducting materials); Applications.

International Organizing Committee:

R.Masumura (Naval Research Laboratory, USA) I.Ovid'ko (Russian Academy of Sciences, Russia)
C.Pande (Naval Research Laboratory, USA) B.Smirnov (Russian Academy of Sciences, Russia)
M.Soto (Office of the US Naval Research, USA)

Local Organizing Committee:

Chairman: I.Ovid'ko (RAS)

Members: D.Grigorieva (RAS), T.Orlova (RAS), A.Reizis (RAS), B.Smirnov (RAS)

Contacts:

Dr.Ilya Ovid'ko - Institute of Problems of Mechanical Engineering, Russian Academy of Sciences
Bolshoj 61, Vas.Ostrov, St.Petersburg 199178, Russia Phone: +(7 812)321 4764
Fax: +(7 812)321 4771 - E-mail: ovidko@def.ipme.ru

List of AAIS invited speakers:

Prof.E.L.Aero (Institute of Problems of Mechanical Engineering, Russia)
Prof.R.A.Andrievskii (Institute of Chemical Problems, Russia)
Dr.U.Balachandran (Argonne National Laboratory, USA)
Prof.N.Browing (University of Illinois at Chicago, USA)
Prof.G.-M.Chow (National University of Singapore, Singapore)
Dr.J.Eastman (Argonne National Laboratory, USA)
Dr.E. Gaffet (CNRS / UTBM - Nanomaterials Research Group, France)
Dr.K.Goretta (Argonne National Laboratory, USA)
Prof.Y.Gogotsi (University of Illinois at Chicago, USA)
Dr.P.Hazzledine (Universal Energy System, USA)
Dr.P. Jena (Virginia Commonwealth University, USA)
Prof.G.Kiriakidis (IESL/FORTH, Greece)
Prof.P.Klimanek (Freiburg Technical University, Germany)
Prof.J.-Y.Laval (CNRS/ESPCI, France)
Prof.S.Majetich (Carnegie-Mellon University, USA)
Dr.R.A.Masumura (Naval Research Laboratory, USA)
Dr.K.Merkle (Argonne National Laboratory, USA)
Dr.T.Orlova (Ioffe Physico-Technical Institute, Russia):
Dr. I.Ovid'ko (Institute of Problems of Mechanical Engineering, Russia)
Dr. C.Pande (Naval Research Laboratory, USA)
Dr.B.B.Rath (Naval Research Laboratory, USA)
Prof.B.Smirnov (Ioffe Physico-Technical Institute, Russia)
Dr.M.Soto (Office of Naval Research, USA)
Prof.R.Valiev (Institute of Advanced Materials, Russia)
Prof.O.G.Vendik (State Electrotechnical University, Russia)

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

Since 1972, hydriding alloys have been studied as materials for Ni - MH batteries. The first generation of alloys, already commercialized, is based on AB₅ compounds. They form hydrides with the following formula : AB₅H₆. Their maximum electrochemical capacity measured was around 330 mAh/g. A second generation is based on AB₂ compounds. Their hydride formula is AB₂H₃. Their maximum electrochemical capacity measured was around 400 mAh/g. The aim of the following work is to synthesize and study alloys which exhibit better properties than the alloys AB₅ on the market.

The results obtained during this work showed the practical interest of milling and mechanical alloying regarding hydrides. First, it was possible to improve the hydrogen absorption properties of an AB₂ alloys, by milling it with Mg₂Ni or Nickel. This treatment enhanced the following absorption properties :

Surface activation,

Hydrogen absorption kinetics,

first cycles discharge capacities.

Furthermore, mechanical alloying, followed by annealing, allowed us to substitute partially zirconium with magnesium in an AB₂ alloy. The elaboration of alloys containing zirconium and a substantial amount of magnesium was a première. the alloy Zr_{0.5}Mg_{0.5}(Mn_{0.6}Ni_{0.6}V_{0.6}Cr_{0.1}) was synthesized and showed interesting characteristics regarding hydrogen absorption. Unfortunately, resistance to electrochemical cycling was bad. Using the same synthesis process, the nanocrystalline alloy Zr_{0.65}Ti_{0.35}Cr₂ was made. It was shown that this compound had an excellent solid - gas capacity, equivalent to 460 mAh/g.

In a second part, the alloys of the Mg - Ni system was studied. Optimising milling time and intensity, the compound Mg₂Ni was elaborated after 4 hours of milling at intensity 10. This nanocrystalline alloy exhibited a discharge capacity of 210 mAh/g whereas polycrystalline Mg₂Ni only discharged 8 mAh/g. This enhancement was attributed to the presence of a disordered phase. We also attempted to synthesize the Mg₅₀Ni₅₀. Unfortunately, we could not obtain such an alloy. Indeed, despite changing the milling parameters, a phase containing Mg₂Ni or MgNi₂ was always observed. It seems that the formation of stable phases instead of the metastable phase was due to excess energy brought by the milling.

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
Requests - Proposals**

***** Looking for Job Position**

(6th July 1999)

I have a Ph.D. in Physics (Mechanical Alloying by Ball Milling) from the Australian National University (Canberra) and extensive experience (more than 17 years and more than 40 publications) in the area of Materials Sciences, Mechanical Engineering and Electronics. The topic of my Ph.D. was Production of Hard Materials by Mechanical Alloying (under the guidance of Dr. A. Calka). I have worked on the production of hard compounds by ball milling such as nitrides, carbides and special alloys. I have completed a post-doctoral fellowship program in Japan (Tokyo University of Technology and The Photon Factory at Tsukuba) and I am currently working as a X-ray Diffraction Officer at CSIRO in Melbourne (Australia). My contract expires and I am looking for a new position. For more information please contact: ph./fax +61-3-95433002 or email: Jonian.Nikolov@Minerals.CSIRO.au

****** Proposals**

FRANCE (2/07/99)

Joindre Pascal Viel , tél 01 69 08 41 47 CEA SRSIM Bt 461, 91191 Gif sur yvette

Proposition de post doc qui débiterait idéalement en septembre 99. Le financement du post doc est acquis (1 an) Le lieu de travail est le centre de Saclay (DSM/DRECAM/SRSIM) Le sujet concerne une étude très appliquée sur la dépollution des eaux de rejets industriels : Mise au point et étude d'un procédé d'élimination des métaux lourds basé sur la fabrication d'un filtre actif (complexation-décomplexation) utilisant la modification de surfaces métalliques par des films polymères électrographés en couches minces.

Conditions: avoir sa thèse depuis moins d'un an, ne pas avoir été salarié depuis sauf pour un an de postdoc à l'étranger et avoir moins de 30 ans

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 MICROSYN) concerning microfabrication and microassembly"

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](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

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 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)

[64] METAL INJECTION MOLDING OF NANOSTRUCTURED

Kim JC. Ryu SS. Lee H. Moon IH. - International Journal of Powder Metallurgy. 35(4):47-+, 1999
Nanostructured W-Cu composite powder was fabricated by mechanical alloying. Compacts were then prepared from the powder in the as-milled conditions, and after a thermal reduction treatment, by metal injection molding using a multicomponent wax/polyethylene/stearic acid binder system. A debinding cycle was identified which resulted in a high surface quality of the bionic parts, shape retention, and complete removal of the binder. An intermediate isostatic repressing step increased the bionic density but did not contribute significantly to densification during sintering. Densities >96% of theoretical were achieved in the injection molded compacts for both powder conditions via liquid phase sintering, notwithstanding the low brown density after debinding. This high sinterability is attributed to grain growth of the W and a double rearrangement process in the powder due to its intrinsic nanostructural characteristics.

[63] HEAT RESISTANCE OF ALUMINIDE COATINGS ON NICKEL OBTAINED BY MECHANOCHEMICAL HEAT TREATMENT

Simonov VN. Khasyanov MA. - Metal Science & Heat Treatment. 40(9-10):402-404, 1998
Mechanochemical heat treatment (MCHT) is a new direction in the creation of protective coatings with a controlled micro- and macrostructure. The use of MCHT provides diffusion coatings with an anomalously large thickness and a structure that is most favorable for the operating conditions. Results of a study of high-temperature aluminide coatings on nickel deposited by an MCHT method are presented.

[62] THE MICROSTRUCTURE AND ITS PROPERTIES OF Ni3Al BASED COMPOSITE

Ye WJ. Feng D. Luo HL. - Materials Research Innovations. 2(6):321-324, 1999
Composites of B-doped ductile Ni₃Al alloy matrix with no-oxide WC ceramic powders were produced by mechanical alloying, half-sintering and buildup welding. WC powders form non-continuous hardening phases, which are distributed in Ni₃Al matrix, wetting well with the matrix. The hardness and the structure stability are retained to temperatures of at least 850 degrees C. After build-up welding, most of the NiAl phase left after sintering was changed into other phases and some graphite was precipitated in the matrix. The sand-laden water wear test showed expected results.

[61] EFFECT OF THE PROCESSING ROUTE ON THE OXIDATION BEHAVIOUR OF A Ni3Al POWDER METALLURGY ALLOY

Perez P. Eddahbi M. Gonzalez-Carrasco JL. Adeva P. - Intermetallics. 7(6):679-687, 1999
This work deals with the oxidation behaviour of a powder metallurgy (PM) Ni₃Al alloy synthesised using rapidly solidified powder particles, which were milled for 20 h before consolidation by hot isostatic pressing. Oxidation kinetics was evaluated in the temperature range of 535 to 1020 degrees C, with special emphasis at 635 degrees C for which tests were performed up to 1000 h. Oxidation rate was lower than that showed by the material made from non-milled powder particles, irrespective of the oxidation temperature and exposure time. This better oxidation behaviour is associated to the smaller average grain size of the substrate induced during the milling stage, which favoured formation of a thin alumina layer.

[60] MECHANICAL PROPERTIES OF SPARK PLASMA SINTERED Nb-AL COMPACTS STRENGTHENED BY DISPERSION OF Nb2N PHASE AND ADDITIONS OF MO AND W

Murakami T. Komatsu M. Kitahara A. Kawahara M. Takahashi Y. Ono Y. - Intermetallics. 7(6):731-739, 1999
Mechanically alloyed and blended Nb-Al-N powders were sintered by the spark plasma sintering process, and their microstructure and mechanical properties were investigated. All of the Nb-Al-N compacts consisted of phases in the Nb-Al system in which the Nb₂N phase was dispersed. The microstructure of blended powder compacts was much coarser than that of mechanically alloyed powder compacts. The compacts obtained by sintering powder produced by crushing blended powder compacts have finer microstructure, higher hardness, and higher fracture toughness than blended powder compacts. The strength of Nb-Al-N compacts increases with increasing the fraction of AlN added to the Nb powder, while their fracture toughness at room temperature decreases. As for the Nb-Al-Mo and Nb-Al-W system, the effect of solid-solution hardening of W was larger than that of Mo, and Nb-15Al-40Mo compact has the highest strength at room temperature and 1273 K among Nb-15Al-xMo compacts.

[59] NANOCRYSTALLINE C-BN SYNTHESIZED BY MECHANICAL ALLOYING

Zhang YF. Tang YH. Lee CS. Bello I. Lee ST. - Diamond & Related Materials. 8(2-5):610-613, 1999 Mar.
Nanocrystalline carbon-boron nitride (C-BN) compounds have been synthesized by solid phase mechanical alloying of C-x(BN)(1-x) powder with various carbon fractions x. Ternary mixing within the structures was found to occur after ball milling and hot-pressing. High temperature recrystallization led to a change in structural configuration, specifically, into alternating layers of graphite and BN. The hardness of the C-BN plates increased when the carbon fraction x increased from 0 to 0.5, and then decreased when x increased thereafter. The composite C-0.5(BN)(0.5) possessed the highest hardness equalling twice that of nanocrystalline graphite and three times that of nanocrystalline BN. The lattice parameters c and a were larger and smaller, respectively, than those of graphite and hBN. The property differences between the initial and resultant materials were attributed to the structural transformation of the original substances into heterogeneously layered C-x(BN)(1-x) compounds. New additional peaks were also observed and matched those of shock-wave compressed BN.

[58] SYNTHESIS OF NIOBIUM ALUMINIDES USING MECHANICALLY ACTIVATED SELF-PROPAGATING HIGH-TEMPERATURE SYNTHESIS AND MECHANICALLY ACTIVATED ANNEALING PROCESS

Gauthier V. Josse C. Bernard F. Gaffet E. Larpin JP. - Materials Science & Engineering A-Structural Materials

Properties Microstructure & Processing. 265(1-2):117-128, 1999

The mechanically activated self-propagating high-temperature synthesis (MASHS) technique and the mechanically activated annealing process (M2AP) were used to produce NbAl₃ intermetallic compound. The MASHS process results from the combination of two steps: first, a mechanical activation of the Nb + 3Al powders mixture; second, a self-propagating high-temperature synthesis (SHS). The M2AP process also results from the combination of two steps: the first is the same; the second consists of the annealing of as-milled powders. Based on X-ray diffraction (XRD) analysis, scanning electron microscopy (SEM) and X-ray energy dispersive spectroscopy (EDXS), the as-milled powders, MASHS, and M2AP end-products were characterized. Various process controlling parameters such as mechanical activation milling conditions have been studied.

[57] STRUCTURAL MODIFICATIONS IN MAGNETIC OXIDES INDUCED BY DRY AND WET MILLING

Kaczmarek WA. - Journal of Magnetism & Magnetic Materials. 197:173-174, 1999

The structural developments in nanostructural Zn- and Ba-ferrites obtained by wet and dry ball milling is discussed.

[56] MAGNETISATION REVERSAL IN MECHANICALLY MILLED EXCHANGE-COUPLED PR_{4.25}Tb₁Fe₇₁Co_{2.5}Cr₃B₁₈ NANOPARTICLES

O'Sullivan JF. Smith PAI. David S. Givord D. Coey JMD. - Journal of Magnetism & Magnetic Materials. 197:182-184, 1999

Mechanically milled Pr_{4.25}Tb₁Fe₇₁Co_{2.5}Cr₃B₁₈ has been annealed to produce a material comprising a (Pr,Tb)₂(Fe,Co)₁₄B hard phase intimately mixed with (Fe,Cr)₂B and alpha-Fe soft phases. Susceptibility and magnetic viscosity measurements have been used to characterise the reversal process in the temperature range 20-300 K.

[55] IONIC DISORDER AND NEEL TEMPERATURE IN ZNFE₂O₄ NANOPARTICLES

Goya GF. Rechenberg HR. - Journal of Magnetism & Magnetic Materials. 197:191-192, 1999

Magnetic properties of ultrafine ZnFe₂O₄ particles obtained from mechanosynthesis of the precursor oxides are presented. The ordering temperature of as-milled sample is T_N approximate to 115(10) K, and extends over a Delta T approximate to 60 K range. This behavior can be explained by oxygen vacancies and local disorder at Fe sites resulting from the milling process. Magnetic coupling between ordered and disordered phases is discussed.

[54] STRUCTURE AND MAGNETIC PROPERTIES OF NANOCRYSTALLINE SOLID SOLUTIONS OF IN IN FE

Smith PAI. Coey JMD. - Journal of Magnetism & Magnetic Materials. 197:199-200, 1999

Body-centred-cubic alloys of Fe and In, elements which are immiscible in equilibrium, have been prepared by mechanical alloying. The variation of Mossbauer hyperfine field distributions deviates from that expected for a random solid solution, indicating some clustering of In atoms. Measurements of saturation magnetisation suggest that In carries no moment and simply dilutes the Fe moment.

[53] STRUCTURAL AND MAGNETIC PROPERTIES OF MECHANICALLY ALLOYED (FEXCU_{1-X})(93)ZR-7 (X=0.5, 0.7) SOLID SOLUTIONS

Multigner M. Hernando A. Crespo P. Stiller C. Eckert J. Schultz L. - Journal of Magnetism & Magnetic Materials. 197:214-215, 1999

Nanocrystalline FCC-(Fe_{0.5}Cu_{0.5})(93)Zr-7 and BCC-(Fe_{0.7}Cu_{0.3})(93)Zr-7 (at%) solid solution, have been obtained by mechanical alloying. Both alloys are ferromagnetic at room temperature. By heating, the alloys decompose into FCC and BCC phases which, with further annealing, evolve into BCC-Fe, FCC-Cu and the intermetallic Fe₃Zr. This process leads to an increase of the coercive field originated by a decoupling of the ferromagnetic grains and to the appearance of the high anisotropy Fe₃Zr phase.

[52] MAGNETIC CHARACTERIZATION OF MECHANICALLY ALLOYED FE-30(AL_{1-X}CUX)(70)

de Toro JA. de la Torre MAL. Puche RS. Riveiro JM. - Journal of Magnetism & Magnetic Materials. 197:243-245, 1999

Powder samples of nanocrystalline Fe-30(AL_{1-x}Cux)(70) (x = 0, 0.1, 0.2, 0.3) were prepared by mechanical alloying. AC and DC susceptibility measurements as a function of temperature, frequency and external applied DC field showed the appearance of both a spin-glass-like transition and a superparamagnetic component. The latter decreases as the Cu concentration is increased, and practically extinguishes for x = 0.3.

[51] MAGNETIC PROPERTIES OF NON-STOICHIOMETRIC UFE_X COMPOUNDS OBTAINED BY BALL MILLING

Timko M. Szlaferek A. Zentko A. Kovac J. - Journal of Magnetism & Magnetic Materials. 197:655-657, 1999

The non-stoichiometric UFe_x alloys were synthesized from the starting UFe₂ compounds with the iron excess in a conventional horizontal ball mill. X-ray patterns show that the mechanical alloying (MA) results in the formation of an amorphous iron rich U-Fe phase. The observed values of T_c increase abruptly during the initial 65 h of MA and then more slowly up to 207 K when the milling period rises up to 230 h.

[50] KINETICS OF MECHANOCHEMICAL PROCESSES IN GRINDING MACHINES

Urakaev FK. Boldyrev VV. - Inorganic Materials. 35(4):405-412, 1999

Based on a nonlinear elastoplastic model for collisions between solids, the parameters of impact interaction between grinding media and the material being ground are calculated for various ball mills, a disintegrator, and other grinding machines. Equations are derived for calculating the kinetics and rate constants of mechanical activation and various mechanochemical reactions in grinding machines. All of the calculated values are compared with experimental data.

[49] MECHANOCHEMICAL INTERACTION OF NATURAL ZEOLITE WITH CALCIUM PHOSPHATES [RUSSIAN]

Yusupov TS. Shumskaya LG. Asanov IP. Boldyrev VV. - Doklady Akademii Nauk. 364(4):508-511, 1999

[48] HRTEM AND EELS STUDIES OF DEFECTS STRUCTURE AND AMORPHOUS-LIKE GRAPHITE INDUCED BY BALL-MILLING

Huang JY. - Acta Materialia. 47(6):1801-1808, 1999

Deformation structures and amorphization kinetics of graphite with high crystallinity upon ball-milling (BM) are studied by high-resolution transmission electron microscopy (HRTEM) and electron energy-loss spectroscopy

(EELS). Basal plane stacking disorder, cleavage, delamination cracks; misorientation bands and low-angle (0002) twist boundaries are observed. The basal plane stacking disorder is probably produced by simultaneous shearing of all the lattice planes involved. Other defects such as half Frank loops or interstitial loops and significant bending and buckling of the basal planes are also frequently observed. Sustained shearing of (0002) planes acting in parallel with the increase of the Frank loops finally leads to the breakage of the hexagonal network in a very fine scale until an amorphous-like structure results. Although the amorphous-like phase exhibits amorphous halo diffraction patterns, HRTEM investigations indicate that it consists of highly curled small flakes with average basal plane diameter less than 5 nm and thickness (normal to the basal planes) less than 2 nm. EELS investigations demonstrate that its bonding is essentially sp². The present study clarifies the defect configuration appearing in heavily deformed graphite and also shows evidence that the amorphization of graphite is a defect-controlled process.

[47] SOLID STATE REACTIONS IN AL NI ALTERNATE FOILS INDUCED BY COLD ROLLING AND ANNEALING
Coluzzi B. Biscarini A. Campanella R. Trotta L. Mazzolai G. Tuissi A, Mazzolai FM. - Acta Materialia. 47(6):1901-1914, 1999

Al/Ni composites made of alternate foils having overall composition Al₅₀Ni₅₀ and Al₆₆Ni₃₄ were rolled up to 75 times folding them after every rolling pass to restore approximately the original thickness. It was found that the deformation of the composite is sustained by the Ni with Al acting as transmitting medium. The logarithmic reduction of foil thickness scales with the number of rolling passes. A nanocrystalline slate of the elements, particularly Ni, is progressively reached. No detectable reaction is caused by repeated co-deformation. Reactions in the composites occur on annealing. The sequence of phases obtained at Al/Ni interfaces via nucleation and growth, and identified by X-ray diffraction, transmission electron microscopy, scanning electron microscopy, reproduces that found on annealing deposited multilayers and ball-milled powders. Al₃Ni, Al₃Ni₂, NiAl. All reactions are strongly activated by deformation, i.e. they occur at lower temperature as revealed by continuous heating experiments in a differential scanning calorimeter. The overall set of experimental results is consistent with reaction mechanisms of nucleation and growth with grain-boundary interdiffusion as the rate-determining step. This View is supported by comparison with a collection of data for the activation energy of diffusion, grain growth, and ordering in Al-Ni phases.

[46] MAGNESIUM TETRAHYDROALUMINATE: SOLID-PHASE FORMATION WITH MECHANOCHEMICAL ACTIVATION OF A MIXTURE OF ALUMINUM AND MAGNESIUM HYDRIDES

Dymova TN. Konoplev VN. Sizareva AS. Aleksandrov DP. - Russian Journal of Coordination Chemistry. 25(5):312-315, 1999

It is established that crystalline aluminum and magnesium hydrides are capable of reacting to form magnesium tetrahydroaluminate upon mechanochemical activation of their mixture (molar ratio MgH₂:AlH₃ = 1 : 2; vibratory load: weight of balls 200 g, frequency 23 Hz, vibration amplitude 8-12 mm). Yield of Mg(AlH₄)₂ > 90%. Thermolysis of Mg(AlH₄)₂ proceeds in two steps with the formation of highly reactive MgH₂* and metallic Al* at T = 135-155 degrees C and aluminum- magnesium alloys Al_{0.93}Mg_{0.07} + Al_{0.62}Mg_{0.38} at T = 300 degrees C. Chemical activities of the mechanically activated magnesium hydride synthesized from the elements and MgH₂* generated by Mg(AlH₄)₂ were found to be different. A method is suggested for estimating the extent of reaction of formation of Mg(AlH₄)₂.

[45] INTERACTION OF FERROCENECARBALDEHYDE WITH ZINC TETRAHYDROBORATE AND ITS DERIVATIVES

Makhaev VD. Gorobinskii LV. Kedrova NS. Mal'tseva NN. - Russian Chemical Bulletin. 48(1):212-214, 1999
Ferrocenecarbalddehyde was reduced to methylferrocene upon interaction with zinc tetrahydroborate in THF. The reactions proceeded rapidly (5-10 min) in high yields (ca. 90%). Analogous results were obtained when a mechanically activated mixture of zinc chloride and sodium tetrahydroborate or the complex of zinc tetrahydroborate with dibenzo-18-crown-6, 2Zn(BH₄)₂. DB-18-C-6, was used in the reaction.

[44] IRRADIATION-INDUCED TRANSFORMATION OF MACROMOLECULAR STRUCTURE AND MECHANISMS OF MECHANO-CHEMICAL MICRODESTRUCTION IN AMORPHOUS-CRYSTALLINE POLYMERS

Mateev M. Karageorgiev S. - Radiation Effects & Defects in Solids. 147(4):265-272, 1999.

The microreliefs of the fractured surfaces of non-irradiated low density polyethylene (LDPE) and gamma irradiated high density polyethylene (HDPE) "BULEN" are similar, but are obtained due to different microdestruction mechanisms determined by different interaction mechanisms of the macromolecular chains. The LDPE is characterized by low degree of co-operation of the sublevels of the superstructural molecular organization, high branching and high density entanglements of the macromolecular chains which are uniformly distributed in the amorphous regions and in the intercrystalline amorphous regions. The surface structures in the gamma irradiated HDPE represent areas with homogeneous distribution of the radiation-induced chemical crosslinking density nu(ch) which form lattices build up of macromolecular networks and are distributed predominantly in the intercrystalline amorphous regions. Obtaining such superstructural molecular organization in the irradiated polymer depends strongly on the state and structural organization of the initial non-irradiated polymer.

[43] THE MICROSTRUCTURE AND ITS PROPERTIES OF Ni₃Al BASED COMPOSITE

Ye WJ. Feng D. Luo HL. - Materials Research Innovations. 2(6):321-324, 1999

Composites of B-doped ductile Ni₃Al alloy matrix with no-oxide WC ceramic powders were produced by mechanical alloying, half-sintering and buildup welding. WC powders form non-continuous hardening phases, which are distributed in Ni₃Al matrix, wetting well with the matrix. The hardness and the structure stability are retained to temperatures of at least 850 degrees C. After build-up welding, most of the NiAl phase left after sintering was changed into other phases and some graphite was precipitated in the matrix. The sand-laden water wear test showed expected results.

[41] SYNTHESIS OF HIGH-SURFACE-AREA COMPLEX ZIRCONIUM PHOSPHATES VIA MECHANOCHEMICAL ACTIVATION ROUTE

Sadykov VA. Pavlova SN. Zabolotnaya GV. Maximovskaya RI. Tsybulya SV. Burgina EB. Zaikovskii VI. Litvak GS. Chaikina MV. Lunin VV. Kuznetsova NN. Roy R. Agrawal DK. - *Materials Research Innovations*. 2(6):328-337, 1999

High-power ball mill activation of the mixture of hydrated zirconium and lanthanum salts (oxonitrates, oxochlorides) with ammonium phosphate followed by hydrothermal treatment at temperatures not exceeding 200 degrees C and a nearly neutral pH was found to yield crystalline dispersed phase of a cubic $\text{NH}_4\text{Zr}_2(\text{PO}_4)_3$ type along with admixtures of disordered orthorhombic compounds of a zirconium orthophosphate type. In the same conditions and at the same Zr/P ratio, hydrothermal treatment of gels obtained by reacting mixed zirconium and lanthanum nitrates solutions with ammonium phosphates yields no crystalline products, and only treatment in acid media generates a phase of the $\alpha\text{-ZrPO}_4(\text{OH})$ type coexisting with the $\text{NH}_4\text{Zr}_2(\text{PO}_4)_3$ phase if polyethylene oxide is present. X-ray powder diffraction, transmission electron microscopy, $^{31}\text{MAS-NMR}$, FTIRS and thermal analysis were applied to elucidate factors affecting crystallization of complex zirconium phosphates in the hydrothermal conditions. The most essential factor appears to be generation of some nuclei of zirconium phosphates under high pressures developed in the course of mixed solids mechanical activation. These nuclei are embedded into matrix of such well-crystallized solid products as ammonium nitrate or chloride. Hence, metastable cubic or orthorhombic structure of the phases obtained via mechanical activation route can be assigned to the nuclei-matrix orientation relationship. Due to easily scaled-up synthesis procedure, these results appear to be very promising for manufacturing of dispersed framework zirconium phosphates as acid catalysts or fast proton conductors.

[40] MECHANOCHEMICAL SYNTHESIS OF NEW AMORPHOUS MATERIALS OF $60\text{Li}(2)\text{S}$ CENTER DOT $40\text{SiS}(2)$ WITH HIGH LITHIUM ION CONDUCTIVITY

Morimoto H. Yamashita H. Tatsumisago M. Minami T. - *Journal of the American Ceramic Society*. 82(5):1352-1354, 1999

New amorphous materials were mechanochemically synthesized by use of crystalline starting materials of Li_2S and SiS_2 . The conductivity of a mechanochemically prepared sample of $60\text{Li}(2)\text{S} \cdot 40\text{SiS}(2)$ (mol%) after a milling for 20 h was around $10^{-4} \text{ S} \cdot \text{cm}^{-1}$ at room temperature. This conductivity was comparable to that of the corresponding glassy powders prepared by twin-roller rapid quenching of melt and then pulverizing. The transport number of Lithium ions in the mechanochemically prepared sample was nearly unity. Mechanochemical synthesis is a promising way to produce new solid electrolytes for solid-state lithium secondary batteries.

[39] MECHANOCHEMICALLY SYNTHESIZED LEAD MAGNESIUM NIOBATE

Wang J. Xue JM. Wan DM. Ng WB. - *Journal of the American Ceramic Society*. 82(5):1358-1360, 1999

Lead magnesium niobate (PMN) of high sintered density has been successfully prepared by a mechanochemical fabrication technique from mixed oxides of PbO , MgO , and Nb_2O_5 . The novel technique skips the phase-forming calcination steps at intermediate temperatures that are almost always required in the most widely employed columbite method. The solid-state reactions among constituent oxides are activated by mechanical energy instead of calcination at elevated temperatures. Nanosized PMN particles of perovskite structure with a minimized degree of particle agglomeration were formed when the oxide mixture was mechanically activated for 20 h. The resulting PMN powder was sintered to a density of similar to 99% theoretical density at 1050 degrees C for 1 h. The sintered PMN exhibited a peak dielectric constant of 18 080 at a frequency of 100 Hz at -11 degrees C.

[38] PROPERTIES AND REACTIVITY OF MECHANOCHEMICALLY SYNTHESIZED TIN SULFIDES

Balaz P. Ohtani T. Bastl Z. Boldizarova E. - *Journal of Solid State Chemistry*. 144(1):1-7, 1999

The paper deals with the study of SnS , Sn_3S_4 ($\text{SnS}+\text{Sn}_2\text{S}_3$), Sn_2S_3 and SnS_2 . The sulfides were prepared by mechanochemical synthesis in a planetary mill, The surface and bulk composition of the sulfides were verified by XPS and XRD methods. The particles of synthesized sulfides are formed by crystallites that are 11-27 nm in size, These crystallites form secondary particles (agglomerates) with 30-50 μm size due to intensive milling, The samples have great reactivity during their dissolution in an alkaline solution of Na_2S . The apparent activation energy of SnS dissolution is 27 kJ mol^{-1} .

[37] SOLID-STATE [4+2] CYCLOADDITION OF FULLERENE C-60 WITH CONDENSED AROMATICS USING A HIGH-SPEED VIBRATION MILLING TECHNIQUE

Murata Y. Kato N. Fujiwara K. Komatsu K. - *Journal of Organic Chemistry*. 64(10):3483-3488, 1999

The solid-state reaction using a high-speed vibration milling (HSVM) technique has been applied to the [4 + 2] cycloaddition of fullerene C-60 with condensed aromatics such as anthracenes, tetracene, pentacene, and naphtho[2,3-a]pyrene. From the reaction with anthracene, [4 + 2] cycloadduct 1 was obtained in better yield than the reaction in solution. Despite such heterogeneous solid-state reaction conditions, an equilibrium state appears to be attained between the reactants (C-60 and anthracene) and products (mono- and bisadducts) since essentially the same product mixture was obtained starting either from the reactants or from the monoadduct after 30 min of the HSVM reaction. The reaction with pentacene gave double C-60 adduct 4 with two C60 cages attached to a pentacene molecule, which cannot be obtained in the reaction in toluene solution, in addition to symmetrical monoadduct 3. The HSVM treatment of C-60 with 9, 10-dimethylanthracene, tetracene, or naphtho[2,3-a]pyrene also gave the corresponding [4 + 2] cycloadducts in fair yields. The redox properties of these adducts were investigated by the use of cyclic voltammetry.

[36] INFLUENCE OF CERAMIC POWDERS OF DIFFERENT CHARACTERISTICS ON PARTICLE PACKING STRUCTURE AND SINTERING BEHAVIOUR

Liu DM. Lin JT. - *Journal of Materials Science*. 34(8):1959-1972, 1999

Yttria-stabilized zirconia powders from different sources were used to investigate powder packing characteristics and subsequent sintering behaviour. Powders of different levels of agglomeration were controlled by varying degrees of ball-milling, followed by shaping through colloidal casting. Experimental findings revealed that the average pore diameter of the powder compacts appears to be a good representative of the whole pore structure of the green compacts. This pore parameter exhibited a well-defined correlation with respect to the initial mean powder size for

each type of powder, irrespective of the difference in particle size distribution and degree of agglomeration, and also correlated soundly with the sintering behaviour of the powder compacts. Critical pore diameter/particle size ratios were experimentally determined, which were shown to be strongly related to the initial particle size by a power-law dependence. This dependence offers an excellent prediction of the critical ratio in alumina powder compacts reported by Zheng and Reed [27], which further supports the feasibility of this relationship.

[35] DYNAMIC SEGREGATION PHENOMENA DURING OXIDATION OF TITANIUM FERRITES

Domenichini B. Perriat P. Merle J. Basset K. Guigue-Millot N. Bourgeois S. - Journal of Materials Chemistry. 9(5):1179-1183, 1999

The cationic composition of three types of titanium ferrite $Fe_{2.5}Ti_{0.5}O_4$ has been analyzed by XPS during their oxidation in order to reveal dynamic segregation phenomena. These samples included two pulverised materials obtained by high energy ball milling followed by a thermal treatment under a well controlled reducing atmosphere (I) and by a ceramic process followed by grinding (II), as well as a compact material obtained by a ceramic process (III). In each case, under pure oxygen and under a linear increase of the temperature, the material was subject to oxidation in the cation deficient phase i.e. without phase transformation below 350 degrees C. During this reaction, an important modification of the chemical composition of the near surface layers has been revealed: the titanium ferrite surface becomes richer in iron and poorer in titanium. For pulverised compounds, if the heating is extended above 400 degrees C, the oxidation in a cation deficient phase can proceed and some titanium can move back to the surface. Then, from this temperature, the amount of titanium detected by XPS increases. For samples obtained by high energy ball milling, this phenomenon can lead to a homogeneous compound. This is not so for the samples obtained by the ceramic process. For these, a phase transformation of the compound appears which generates $\alpha-Fe_2O_3$ at the surface of the material. The segregation phenomenon has been interpreted on the basis of the different mobilities of the species Fe^{2+} , Fe^{3+} , Ti^{4+} and cation vacancies present in the material.

[34] SYNTHESIS OF Sm_2Co_{17} ALLOY NANOPARTICLES BY MECHANOCHEMICAL PROCESSING

Liu W. McCormick PG. - Journal of Magnetism & Magnetic Materials. 195(2):L279-L283, 1999

Sm_2Co_{17} alloy nanoparticles of 10-250 nm in size were prepared by mechanochemical processing involving the co-reduction of Sm_2O_3 and CoO with Ca. The crystal structure of the nano-sized Sm_2Co_{17} particles was mainly of the ordered Th_2Zn_{17} -type. When embedded in the CaO matrix the Sm,Co., nanoparticles exhibited a high coercivity of 14.2 kOe. The CaO by-product could be removed by a carefully controlled washing process without significant oxidation of the ultrafine alloy particles. After washing, the cold-pressed powder exhibited a coercivity value of 11.8 kOe and a maximum magnetization of 92.0 emu/g under an applied field of 50 kOe.

[33] GIANT MAGNETOELASTIC RESPONSE IN MNAS

Chernenko VA. Wee L. McCormick PG. Street R. -Journal of Applied Physics. 85(11):7833-7837, 1999

MnAs has been prepared from elemental powders using a modified ball-milling technique, followed by hot pressing and subsequent annealing. Giant magnetic field-induced uniaxial strains of up to 0.7% were observed in a temperature range above the Curie temperature ($T-C = 305$ K). Dilatometric and magnetization measurements showed that the strains were associated with the volume change accompanying a field-induced magnetostructural paramagnetic to ferromagnetic transition above T-C. The occurrence of a tricritical point (TCP) in temperature-field space, where the two low temperature phases and the high temperature NiAs-type phase are identical was shown to be responsible for the decrease of the magnitude of both the maximum strain and magnetization change with increasing temperature while approaching the TCP along the line of the first-order transitions. The coordinates of the TCP are approximately $T-tr = 405$ K and $H-tr = 165$ kOe.

[32] A SOLID-STATE PROCESS FOR FORMATION OF BORON NITRIDE NANOTUBES

Chen Y. Chadderton LT. FitzGerald J. Williams JS. - Applied Physics Letters. 74(20):2960-2962, 1999

The formation of boron nitride (BN) nanotubes via a solid-state process is demonstrated. The nanotubes are produced by first ball-milling hexagonal BN powder to generate highly disordered or amorphous nanostructures, followed by annealing at temperatures up to 1300 degrees C. The annealing leads to the nucleation and growth of hexagonal BN nanotubes of both cylindrical and bamboo-like morphology. Unlike previous mechanisms for nanotube formation, the reordering and solid-state growth process of our nanotubes does not involve deposition from the vapor phase nor chemical reactions.

[31] HIGH TEMPERATURE ELECTRICAL RESISTIVITY AND OXIDATION RESISTANCE OF MO-SI-B PREPARED BY MECHANICAL ALLOYING

BA Cook, S Zelle, MJ Kramer - 22ND ANNUAL CONFERENCE ON COMPOSITES, ADVANCED CERAMICS, MATERIALS, AND STRUCTURES: A (Series: CERAMIC ENGINEERING AND SCIENCE PROCEEDINGS), 1998, Vol 19, Iss 3, pp 429-436

Alloys based on the Mo_5Si_3 -B benchmark were prepared by mechanical alloying (MA) of elemental powders and consolidated by vacuum hot pressing. The effect of two different types of milling media were examined; hardened tool steel and tungsten carbide. The evolution of the milling process was followed by X-ray diffraction and TEM. The intensity of the diffraction peaks corresponding to elemental silicon were reduced to background noise after as little as two hours of milling. Powders subject to MA for as little as one hour exhibited high defect density and showed evidence of diffusion of silicon into molybdenum. After 15 hours, amorphous and crystalline nanophased grains containing Mo and Si were observed. A systematic reduction in the bcc lattice parameter of Mo was observed, suggestive of substitutional diffusion of Si onto Mo sites. Densities greater than 95% of theoretical were obtained on powders consolidated at 1300 degrees C. Electrical resistivity measurements on specimens taken from the consolidated compacts gave results comparable to that of are melted and sintered materials. Results of preliminary oxidation tests at 1100 degrees C indicate that a continuous protective scale is formed but that the presence of contamination From the grinding media affects the chemistry of the scale.

[30] A NOVEL PROCESS FOR SYNTHESIZING NANOSTRUCTURED CARBIDES: MECHANICALLY ACTIVATED SYNTHESIS

RM Ren, ZG Yang, LL Shaw - 22ND ANNUAL CONFERENCE ON COMPOSITES, ADVANCED CERAMICS, MATERIALS, AND STRUCTURES: B (Series: CERAMIC ENGINEERING AND SCIENCE PROCEEDINGS), 1998, Vol 19, Iss 4, pp 461-468

Nanostructured SiC and TiC have been synthesized through a novel process called as mechanically activated synthesis (MAS). The basic form of the MAS process is to mechanically activate reactants (e.g., SiO₂, and C for synthesizing SiC, and TiO₂, and C for synthesizing TiC) at room temperature through high energy ball milling, followed by heating the milled powder mixture to high temperatures to complete the synthetic reaction. The MAS process not only produces nanostructured carbides, but also substantially reduces the time and temperature of carbothermic reduction through which carbides are formed. In this paper, synthesis of nanostructured SiC and TiC through the MAS process is described and the fundamentals for this novel process are discussed.

[29] PRODUCTION OF HIGH-DENSITY FE-16 AT. % AL ALLOY AND FE-16 AT.% AL-15 VOL% AL₂O₃ COMPOSITE BY MECHANICAL SMEARING AND HOT ISOSTATIC PRESSING

S Bandyopadhyay, SK Mukherjee, DS Perera, S Moricca, MV Swain, TJ Bell - FIRST INTERNATIONAL CONGRESS ON ADHESION SCIENCE AND TECHNOLOGY - INVITED PAPERS, 1998, pp 857-866

Fe-16 at.% Al alloy matrix and Fe-16 at.% Al-15 vol% Al₂O₃ composite have been made from iron, aluminium and alumina powder by mechanical smearing at room temperature followed by hot isostatic pressing (HIP) at 1200 degrees C. The HIP compacts possess 97 and 99% theoretical densities respectively, resulting in very good hardness values. Ultra-micro-indentation techniques revealed that the composite has three distinct zones so far as hardness is concerned. These are Al₂O₃-rich grain boundaries, an interface region of functionally graded material, and a matrix of Fe-16 at.% Al. The incorporation of the Al₂O₃ phase significantly improves the hardness. From indentation fracture toughness tests, both materials showed an estimated minimum fracture toughness of 15 MPam^{1/2} which is good for these types of materials.

[28] SHOCK SYNTHESIS OF MO₂SIC/SICW COMPOSITES FROM MECHANICAL ALLOYING PRETREATED PRECURSORS

T Aizawa, NN Thadhani - INNOVATIVE PROCESSING AND SYNTHESIS OF CERAMICS, GLASSES, AND COMPOSITES II (Series: CERAMIC TRANSACTIONS), 1999, Vol 94, pp 273-284

The shock reactive synthesis is one of the most promising methodologies to yield non-traditional materials. Use of the pretreated powders by the mechanical alloying enables us to make shock reactive synthesis with full reactivity. Furthermore, various intermediate phases can be synthesized during the shock induced reaction. Mo-Si system is employed here to investigate the effect of pretreatment on the synthesis of MoSi₂. Of great importance is that amorphous MoSi₂ can be synthesized as an intermediate phase. Both the SiC particles and whiskers are mixed with the mechanical alloying pretreated powders for shock experiment to discuss the fast quenching effect on the shock induced reaction process. MoSi₂ - SiCp/SiCw composites are successfully synthesized into full-dense billets. Morphological difference between SiC particle and whisker reflects on the local interfacial reaction; no reactions are seen between the SiC particle and the synthesized MoSi₂ matrix, while the tertiary phase was formed on the interface of thin, long SiC whisker. Through close microstructure observation, mass mixing mechanism during shock loading is discussed.

[27] MECHANOCHEMICAL SYNTHESIS OF REFINED AG- AND ZN-COMPOSITE POWDERS STARTING FROM OXIDES

SD DelaTorre, KN Ishihara, PH Shingu, D RiosJara, H Miyamoto - INNOVATIVE PROCESSING AND SYNTHESIS OF CERAMICS, GLASSES, AND COMPOSITES II (Series: CERAMIC TRANSACTIONS), 1999, Vol 94, pp 287-294

De-oxidation of Ag₂O and ZnO powders has been accomplished by the mechanochemical process (MCP) using Si and Fe as reducing agents. Exception is the reduction reaction of ZnO with Fe since it involves a positive enthalpy, and so even after 300 h milling its reduction does not take place. A solid solution of nanosized Fe₂O₃ into an amorphous-like ZnO matrix is developed instead. In contrast to reduced products prepared by conventional thermal decomposition of oxides, a number of advantages are conferred into the MCP-synthesized Ag-SiO₂, Ag-Fe₂O₃, Zn-SiO₂ and ZnO-Fe₂O₃ submicrometre composite products, such as a homogeneous dispersion of active oxides into the reduced metal matrix and nanometre tailored particles.

[26] CERAMIC OXIDE (MeO₂) SOLID SOLUTIONS OBTAINED BY MECHANICAL ALLOYING

F Bandioli, M Romagnoli, L Barbieri, T Manfredini - INNOVATIVE PROCESSING AND SYNTHESIS OF CERAMICS, GLASSES, AND COMPOSITES II (Series: CERAMIC TRANSACTIONS), 1999, Vol 94, pp 295-304

Cerium and zirconium oxide powders (MeO₂) doped with 10 mol% of praseodymium were prepared by mechanosynthesis in a high energy ball mill to investigate whether mechanical alloying is possible in these ceramic systems industrially used as inorganic pigments. The structure variation of the powders was followed by X-ray diffraction (XRD) and scanning electron microscopy (SEM) and to confirm the effective formation of the solid solution, leaching tests were performed. The results prove that mechanosynthesis occurs in both ceramic systems.

[25] BALL MILLING DRIVEN FORMATION OF INTERFACES IN POWDERS OF SUPER ALPHA(2)-Ti₃Al ALLOY

T Kehagias, P Komninou, JG Antonopoulos, T Karakostas, G Nouet, V Pontikis - INTERGRANULAR AND INTERPHASE BOUNDARIES IN MATERIALS, IIB98 (Series: MATERIALS SCIENCE FORUM), 1999, Vol 294-2, pp 333-336

Powders of the multiphase super alpha(2)-Ti₃Al alloy are processed in a vibrating frame; ball mill, in native atmosphere. The structure of the powder particles is investigated by Transmission Electron Microscopy, as a function of the duration of the ball milling process. The phenomena associated with this process include an order-disorder transformation and a gradual amorphisation of the alpha(2)-Ti₃Al phase, that later recrystallises into new structures, the first belonging to the Rhombohedral system, with lattice parameters close to those of alpha-alumina and the second belonging to the Cubic system with an f.c.c. Unit cell that is very rich in Ti. This is explained by a mechanism of oxygen incorporation driven by the continuous deformation of the powder particles, that resembles to

high temperature oxidation of Ti₃Aluminides, The new phases continue to deform by mechanical twinning, forming (0001) and (1 1) over bar 1) twins that exhibit a defected character.

[24] PHYSICAL MECHANISMS OF THE NON-EQUILIBRIUM PHASE TRANSITIONS IN AMORPHOUS SOLIDS

TV Ischenko, SV Demishev - INTERGRANULAR AND INTERPHASE BOUNDARIES IN MATERIALS, IIB98 (Series: MATERIALS SCIENCE FORUM), 1999, Vol 294-2, pp 481-484

A theoretical approach to the non-equilibrium phenomena in disordered solids including explosive crystallization and solid state amorphization is proposed. We consider the special excited metastable states at the phase boundary as a "driving" force of the non-equilibrium phase transition. The decay of these metastable states leads to the appearance of the fundamental scales hierarchy in the sample structure that can be verified experimentally. A possible schemes of the formalisation of the proposed physical mechanisms in a different models are discussed.

[23] GRAIN BOUNDARY EFFECTS IN NANOCRYSTALLINE COPPER

NA Krasilnikov, GI Raab - INTERGRANULAR AND INTERPHASE BOUNDARIES IN MATERIALS, IIB98 (Series: MATERIALS SCIENCE FORUM), 1999, Vol 294-2, pp 701-706

The results of investigations of nanostructured copper with a mean grain size of 75 nm processed by a new method of severe plastic deformation via consolidation of ball-milled powders are given in the paper. The investigations show that the processed copper has a rather stable nanostructure during annealing and elevated strength properties. A significant influence of deformation temperature on a growth of grain size and a decrease in values of internal stresses was also revealed. Evidently, this is connected with realization of recovery processes on grain boundaries and beginning of recrystallization processes already at low homological temperatures. The obtained results show that a state of grain boundaries in nanostructured copper is a very important parameter determining properties of nanostructured materials. To explain the experimental data, the structural model of R.Z. Valiev was taken as a basis. According to this model a structure of nanostructured material consists of ultra fine elastically aligned grains characterized by high density of grain boundary dislocations and disclinations. With all that, during annealing a recovery of these defects occurs.

[22] HIGH-PERFORMANCE DRY MECHANOCHEMICAL POLISHING OF SI WAFER USING SOFT BAC₃ ABRASIVE

N Yasunaga, M Takashina, T Inoue, S Tanaka, Y Yamamoto - PROCEEDINGS OF: SILICON MACHINING, 1998, pp 24-27

[21] ON THE CUTTING PERFORMANCE OF THE 3Y-ZP/AL₂O₃ NANOCOMPOSITE TOOL INSERTS

JS Cheng, H Wang - PROGRESS OF CUTTING AND GRINDING, 1998, pp 147-152 - 4TH INTERNATIONAL CONFERENCE ON PROGRESS OF CUTTING AND GRINDING (ICPCG 98); URUMQI, PEOPLES R CHINA. OCTOBER 5-9, 1998

A series of 3Y-TZP/Al₂O₃ composites were manufactured via attrition milling, pressure casting, and pressureless sintering. Nanocomposites, with alumina content up to 10 vol. % can be obtained. The Vickers hardness increases with increasing alumina content and decreasing sintering temperature, while K-IC increases with increasing alumina content and increasing sintering temperature. When machining high-strength steel like AISI 4340 at lower feed rate, cutting tool inserts made of these composites showed a comparable magnitude of cutting forces yet a somewhat worse surface finish compared with some Al₂O₃-based ceramic inserts.

[20] SYNTHESIS OF COPPER BASED CU-TI BINARY ALLOYS BY MECHANOCHEMICAL SYNTHESIS

S Ghosh, EG Baburaj, FH Froes - SYNTHESIS OF LIGHTWEIGHT METALS III, 1999, pp 35-42 - SYMPOSIUM ON SYNTHESIS OF LIGHTWEIGHT METALS III, AT THE 1999 TMS ANNUAL MEETING; SAN DIEGO, CALIFORNIA. FEBRUARY 28-MARCH 4, 1999

[19] SYNTHESIS OF TI-AL INTERMETALLICS BY MECHANOCHEMICAL PROCESSING

S Ghosh, D Linch, EC Baburaj, FH Froes - SYNTHESIS OF LIGHTWEIGHT METALS III, 1999, pp 43-50 - SYMPOSIUM ON SYNTHESIS OF LIGHTWEIGHT METALS III, AT THE 1999 TMS ANNUAL MEETING; SAN DIEGO, CALIFORNIA. FEBRUARY 28-MARCH 4, 1999

The light weight intermetallics, Ti₃Al and TiAl are potential candidates for applications in aerospace systems and automobiles because of their low density and high temperature strength properties. In spite of attractive combinations of properties, the use of these intermetallics is limited because of the poor ductility and high cost. The ductility of these alloys is known to increase when the grain size is reduced into the nanoscale range. Mechanochemical processing (MCP) using CaH₂ and CaH₂/Mg mixture has been used in the past to synthesize Ti- Al alloys in ultrafine powder form. However, complete alloying of Ti with Al was difficult to attain in those experiments. In the present work the reasons for the absence of complete alloying were investigated and a method for increasing the level of alloying by MCP is presented.

[18] EFFECT OF PROCESSING CONDITIONS ON THE PROPERTIES OF MECHANICALLY MILLED ALUMINUM ALUMINA COMPOSITES

G Fisher, DA Lee, PG McCormick - SYNTHESIS OF LIGHTWEIGHT METALS III, 1999, pp 113-120 - SYMPOSIUM ON SYNTHESIS OF LIGHTWEIGHT METALS III, AT THE 1999 TMS ANNUAL MEETING; SAN DIEGO, CALIFORNIA. FEBRUARY 28-MARCH 4, 1999

Al/Al₂O₃ composites containing up to 40% Al₂O₃ have been synthesized by mechanical milling. Mechanical milling was found result in a homogeneous dispersion of 200 nm Al₂O₃ particles in an aluminum matrix. Consolidation via drop forging resulted in sample densities exceeding 95% of theoretical. Measurements of the effect of the alumina content and heat treatment on the resulting mechanical properties were carried out. A significant improvement in properties was obtained through a two-stage milling process, which enabled enhanced cold welding of the milled particles through variation of the process control agent.

[17] THE PRODUCTION OF MAGNESIUM-TITANIUM ALLOYS BY MECHANICAL ALLOYING

SB Dodd, S Morris, M WardClose - SYNTHESIS OF LIGHTWEIGHT METALS III, 1999, pp 177-184 -

SYMPOSIUM ON SYNTHESIS OF LIGHTWEIGHT METALS III, AT THE 1999 TMS ANNUAL MEETING; SAN DIEGO, CALIFORNIA. FEBRUARY 28-MARCH 4, 1999

In this paper the development of a mechanical alloying (MA) production technique for novel magnesium titanium binaries will be discussed. The aim of the work was to develop complete solid solution of titanium in a magnesium matrix for use as a structural corrosion resistant lightweight alloy. The alloy range under investigation ranged from 0 to 20 wt% Ti. Previous work((1,2)) using physical vapour deposition (PVD) had demonstrated the potential of the alloy system for corrosion resistance and indicated that alloys under 20Wt% Ti provide a good balance between corrosion resistance and density. Traditionally the use of magnesium alloys, especially in aerospace applications, has been limited by the perceived poor corrosion resistance. The addition of titanium produces both a self-healing corrosion layer and reduced galvanic potential. The boiling point of magnesium is lower than the melting point of titanium and only novel processing routes such as MA and PVD can be successfully utilised to produce alloys of magnesium and titanium. This paper will describe the work carried out to develop an MA process route for solid solution Mg-Ti alloys. The influence of various process control agents on the synthesis of Mg-Ti solid solutions will be discussed. In addition, this paper will detail some of the material properties obtained from these novel materials.

[16] SYNTHESIS OF NANOCRYSTALLINE TiC AND ITS DISPERSION IN A METAL MATRIX

EG Baburaj, SK Menon, D Linch, S Ghosh, FH Froes -SYNTHESIS OF LIGHTWEIGHT METALS III, 1999, pp 231-238 -SYMPOSIUM ON SYNTHESIS OF LIGHTWEIGHT METALS III, AT THE 1999 TMS ANNUAL MEETING; SAN DIEGO, CALIFORNIA. FEBRUARY 28-MARCH 4, 1999

Microcrystalline metal carbides, in bulk quantities, are currently manufactured by comminution of commercially available carbides and hence the cost of production increases in proportion to the reduction in particle size. Fine metal carbides have also been produced by plasma processing, mechanical alloying, carbothermal reduction of ultrafine oxides and chemical vapor deposition techniques. The challenge with all these techniques is to successfully scale up production to commercial volumes of nanocrystalline materials with properties and economies that allow their wide spread use. The present work is an attempt to synthesize ultrafine titanium carbide by mechanochemical processing and to disperse the carbide in a copper matrix. The titanium carbide was produced by the chemical reaction $2TiCl_4 + CaC_2 \rightarrow 2TiC + CaCl_2 + 3C$ induced by mechanical alloying. The process has been modified to avoid liberation of free carbon by the addition of Mg. The reaction product after leaching and drying is TiC powder with a size distribution in the range of 10 to 300 nm. Detailed chemical analysis of the individual crystals by PEELS analysis using TEM does not show the presence of any impurities. The nanocrystalline carbide powder has been dispersed in a copper matrix by mechanical milling. Sintering of the composites results in phase separation of the carbide and copper phases.

[15] THE SYNTHESIS AND PROPERTIES OF NANOCRYSTALLINE MAGNESIUM PRODUCED BY BALL MILLING

S Hwang, PG McCormick - SYNTHESIS OF LIGHTWEIGHT METALS III, 1999, pp 239-246 - SYMPOSIUM ON SYNTHESIS OF LIGHTWEIGHT METALS III, AT THE 1999 TMS ANNUAL MEETING; SAN DIEGO, CALIFORNIA. FEBRUARY 28-MARCH 4, 1999

Nanocrystalline magnesium powder with a grain size of approximately 40 nm was prepared by mechanical milling under an inert atmosphere using a modified SPEX 8000 mixer mill. A modified milling operation markedly reduced the adhesion of the powder to the vial and balls compared to the normal milling process, enabling up to 90% of the initial charge mass to be recovered without the addition of the lubricant. The effect of milling parameters, including ball size and charge ratio, on the evolution of the grain size and internal strain during milling was investigated. The milled samples were vacuum cold pressed to form fully dense cylindrical specimens. Compression tests exhibited remarkably high values of ductility and a significant improvement in yield strength after sintering.

[14] IN SITU SYNCHROTRON CHARACTERIZATION OF MECHANICALLY ACTIVATED SELF - PROPAGATING HIGH - TEMPERATURE SYNTHESIS APPLIED IN MO - SI SYSTEM

Ch. Gras, F. Charlot, E. Gaffet, F. Bernard, JC Niepce - Acta Materialia, 47(7) (1999) 2113 - 2123

An original experiment was designed to monitor structural and thermal evolutions during the MASHS (Mechanically Activated Self - Propagating High - Temperature Synthesis) process in the Mo - Si system. Time - Resolved X - ray Diffraction (TRXRD- coupled with an infrared imaging technique was performed to study, in situ, the formation of the alpha MoSi₂ phase in the combustion front. Despite a temporal resolution of 50 ms between two consecutive diffractograms, no intermediate phase was observed during the passage of the combustion front. The only reaction responsible for the self - sustentation is $Mo + 2Si \Rightarrow MoSi_2$ in the primary zone inside the combustion wave. The mechanical activation was found to influence Self - propagation High - Temperature Synthesis (SHS) parameters such as the propagation front velocity (> 13 mm/s), the maximal combustion temperature and the local thermal gradient. After the MASHS process, the alpha - MoSi₂ compound is nanostructured (DMoSi₂ : 88 nm) and some explanations are expressed in order to understand why the nanostructure of the as - milled powders can be maintained during the combustion reaction.

[13] DECOMPOSITION OF NO₃- BY LEAD PELLETS WITH WET BALL MILLING

Uchida M. Kikuchi K. Okuwaki A. - Waste Management. 19(3):213-219, 1999.

The reduction of an ammonium nitrate solution by lead pellets during wet ball milling was studied, as a method of removing concentrated NO₃- from wastewater. Metal oxides forming on the surface of lead pellets in a ball mill reactor are continuously removed by contact between the pellets. At temperatures between 25 and 80 degrees C and a ball mill rotation speed, omega, of 80-180 rpm, initial concentrations of NH₄NO₃ between 0.05 and 0.1 M, NO₃- rapidly reduce to NO₂-, and then to N₂ and NH₃. Depending on the pH of the solution, the precipitation of basic lead (II) nitrate and basic lead (II) nitrite occurs simultaneously with the reduction of NO₃- and NO₂-. As the temperature increases, the rate of reduction of NO₃- increases and the proportion of NH₃ formed increases, as that of N₂ decreases. The rate of reduction of NO₃- is governed by the rate of wear of the outer layer of the Pb pellets, which is determined by the speed of rotation of the ball mill. In this system, there are three competitive reactions: the

reduction of NO₃⁻ and NO₂⁻ by Pb, the reduction of NO₂⁻ by NH₄⁺ and the precipitation of basic lead (II) salts.

[12] CHEMO-MECHANICAL INTERACTION IN SOLID-SOLID REACTIONS

Bielenberg JR. Viljoen HJ. - AICHE Journal. 45(5):1072-1084, 1999

Deflagrations of solid reactants have been widely studied in the context of self-propagating high-temperature synthesis. Propagating velocities of the order of sound speed or faster have not been contemplated due to the belief that the processes determining thermal conduction and diffusion cannot support those rates. Experiments, by Enikolopyan, Gogulya and others, however; disproved those notions. A model for solid-solid reactions, which describe deflagration and detonation, is presented. The fluctuation energy is large and the system requires significant preheating before ignition occurs. Based on experimental observations, the activation energy of a compressed system is lowered by the amount of elastic work done during compression. This phenomenon is included in the model. Ignition by impact and external thermal sources are investigated and detonations develop only in the case of impact initiation. Interactions between the shock front and the reaction front are also investigated.

[11] PREPARATION OF ANHYDROUS PRASEODYMIUM ACETYLACETONATES BY SOLID-STATE MECHANOCHEMICAL METHOD [RUSSIAN]

Zaitseva IG. Kuz'mina NP. Martynenko LI. Makhaev VD. Borisov AP. - Zhurnal Neorganicheskoi Khimii. 43(5):805-807, 1998

[10] SYNTHESIZING 0.9PZN-0.1BT BY MECHANICALLY ACTIVATING MIXED OXIDES

Xue JM. Tan YL. Wan DM. Wang J. - Solid State Ionics. 120(1-4):183-188, 1999

0.9Pb(Zn_{1/3}Nb_{2/3})-0.1BaTiO₃ (0.9PZN-0.1BT) of perovskite structure has been successfully prepared by mechanically activating mixed oxides of PbO, ZnO, Nb₂O₅, BaO and TiO₂. The novel mechanochemical technique skips the phase-forming calcination step at an intermediate temperature that is always required in both the conventional solid state reaction and chemistry-based precursor routes. Ultrafine 0.9PZN-0.1BT particles of perovskite structure were formed when the constituent oxides were mechanically activated for more than 10 h. The powder was sintered to a density of 96% theoretical density at 1100 degrees C for 1 h. The sintered 0.9PZN-0.1BT exhibits perovskite structure and a peak dielectric constant of 8800 at the Curie temperature of 60 degrees C when measured at a frequency of 100 Hz.

[9] MAGNETIC PROPERTIES OF MECHANICALLY ALLOYED SM₂FE₁₇-XGAXCY

Ding J. McCormick PG. Street R. - Physica Status Solidi A-Applied Research. 172(2):469-475, 1999

Alloys of Sm₂Fe_(17-x)Ga_xC_(y) have been prepared by mechanical alloying, and their magnetic properties have been determined. High coercivities of 15 to 16 kOe were found in samples with Ga concentrations of 1.5 to 2. The Sm₂Fe_{15.5}Ga_{1.5}C alloy had a remanence of 65 emu/g and a maximum energy product of 10 MGOe using the theoretical density. Study of irreversible magnetization and viscosity exhibited behavior similar to that found in other rare-earth permanent magnets with fine-crystalline structure.

[8] STACKING DISORDERING IN HEXAGONAL BN INDUCED BY SHEARING UNDER BALL MILLING

Huang JY. Jia XB. Yasuda H. Mori H. - Philosophical Magazine Letters. 79(5):217-224, 1999

Significant stacking-disordering within basal planes, such as heavy stacking faults, basal plane twinning or even entirely disordered stackings and shearing of lattice planes, delamination and kink bands were frequently found in most hexagonal BN (h-BN) grains after milling. It is evident that most of these high-energy defect structures are created by simultaneous shearing rather than by dissociation of a perfect dislocation. As a result of frequent shearing, the unit-cell shape of the [11 (2) over bar 0] lattice image changes from a rectangle to a rhombi. Nonbasal plane defects such as half Frank loops, and interstitial and vacancy loops with high-energy stacking configurations were also frequently found. These deformation structures stem inherently from the structural characteristic of h-BN and are significantly different from those observed in fee metals such as copper.

[7] SHOCK-INDUCED REACTION SYNTHESIS OF ISOMORPHOUS (CU-NI) AND IMMISCIBLE (CU-NB) COMPOUNDS

Advani AH. Thadhani NN - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 30(5):1367-1379, 1999

Shock compression of Cu-Ni and Cu-Nb elemental powder mixtures was investigated to study the shock-induced chemical reaction behavior of material systems with very low heats of formation and to synthesize isomorphous, as well as otherwise-immiscible, compounds. Shock loading experiments were performed using a 12-capsule plate-impact recovery fixture, with explosive loading at 0.9 to 1.6 km/s impact velocities. The Cu-Ni powder mixtures revealed formation of an isomorphous Cu-Ni solid-solution alloy with a fine dendritic microstructure, formed via a mechanism involving intense mechanical mixing and melting of elemental reactants. The extent of the reaction was dependent on the shock strength, and the chemistry of the product was observed to depend on the morphology of the powders due to its effect on the crush strength. In the case of Cu-Nb powder mixtures, submicron-scale mechanical mixing was observed between the reactants, with possible dissolution of as much as 10 wt pct solute in each component. Complete alloying of copper and niobium was also observed, as indicated by transmission electron microscopy (TEM)/energy-dispersive X-ray (EDS) and X-ray diffraction (XRD) analysis; however, the alloyed region showed the presence of Mo and other impurity elements from the stainless steel capsule. These additives may, in fact, be responsible for stabilizing the ternary compound, in an otherwise immiscible Cu-Nb system.

[6] CORE-LEVEL AND VALENCE BAND PHOTOEMISSION STUDY OF LA_{1-x}Sr_xMnO₃ PEROVSKITE OXIDE POWDERS SYNTHESIZED BY MECHANICALLY AND THERMALLY ACTIVATED SOLID-STATE REACTION

Santoni A. Speranza G. Mancini MR. Padella F. Petrucci L. Casadio S. - Journal of Physics-Condensed Matter. 11(16):3387-3393, 1999

High-resolution core-level and valence band x-ray photoemission spectroscopy measurements were performed on La_{1-x}Sr_xMnO₃ perovskite oxide powders synthesized for applications in solid-oxide fuel cells by high-temperature solid-state reaction (x = 0.3 and 0.19) and by room-temperature mechanical activation of the precursors (x = 0.3). A structure in the valence band at about 1 eV below the Fermi level was clearly observed and assigned to the emission

from the Mn 3d-derived e(g)(1)up arrow states, thereby allowing the extraction of information about correlation effects in this type of material. Both the core-level and valence band spectral features were found to be independent of the choice of synthesis route. This finding indicates that mechanical activation, due to its lower synthesis temperature, can represent a valid alternative method of synthesis allowing a better control of the microstructure.

[5] ANOMALOUS MAGNETIC PROPERTIES OF THE NANO-SIZE RESIDUAL AMORPHOUS PHASE IN NANOCRYSTALS

Kaptas D. Kemeny T. Balogh J. Bujdosó L. Kiss LF. Pusztai T. Vincze I. - Journal of Physics-Condensed Matter. 11(17):L179-L185, 1999

The composition dependence of the Curie temperature and the magnetic moment of the nano-size residual amorphous phase in partially crystallized Fe_{92-x}Zr_{7B}Cu₁ (2 less than or equal to x less than or equal to 23) amorphous alloys were determined by Fe-57 Mossbauer spectroscopy. Both quantities show broad minima and for increasing relative Zr content (i.e. for decreasing B concentration) surpass the usual monotonic decrease observed for the bulk counterparts. The deviation does not scale with the characteristic size of the residual amorphous regions, which was found to be constant, ruling out explanations based on interphase exchange interaction. A magnetovolume origin of the observed anomalous composition dependence and the improved soft-magnetic characteristics of these nanocrystals is proposed.

[4] STRUCTURAL STUDY OF THE MECHANICALLY ALLOYED FE-CO-CU NANOCRYSTALLINE SYSTEM

Prieto C. de Bernabe A. Gay-Sanz N. Vazquez M. Yu SC. - Journal of Non-Crystalline Solids. 246(3):169-176, 1999

The local order around Fe, Co and Cu atoms has been investigated at room temperature by X-ray absorption spectroscopy (XAS) and the long range order has been studied by X-ray diffraction (XRD) in Fe-Co-Cu nanocrystalline alloys prepared by mechanical alloying. In order to determine the time evolution of the alloying process, samples have been studied after several processing times. The joint analysis of XAS and XRD data shows a picture for the alloy formation in which, Co alloys with Cu in the first stages of the process but the iron enters the alloy phase much later than the cobalt does. On the other hand, a refinement of the XRD data permits the identification of a residual iron phase previously detected by Mossbauer spectroscopy.

[3] MECHANICALLY ALLOYED ZR55AL10CU30NI5 METALLIC GLASS COMPOSITES CONTAINING NANOCRYSTALLINE W PARTICLES

Eckert J. Kubler A. Schultz L. - Journal of Applied Physics. 85(10):7112-7119, 1999

Composites based on the Zr₅₅Al₁₀Cu₃₀Ni₅ bulk metallic glass forming alloy, containing up to 17.5 vol % W particles were synthesized by mechanical alloying. Milling produces a metallic glass matrix with a homogeneous dispersion of nanoscale W particles. The composites exhibit almost the same thermal stability and no reduction of the supercooled liquid region compared to the particle-free metallic glass despite some small amount of dissolution of W into the glassy matrix. The viscosity in the supercooled liquid increases with increasing volume fraction of particles. This will be discussed with respect to the contribution of the particles as well as to changes in matrix composition and in the free volume of the material in the framework of the free volume model for viscous flow. Independent of the W content, the samples behave as moderately strong glasses. The viscous flow of the supercooled liquid is used to consolidate dense bulk samples. The Vickers hardness, H-V, of the composites increases with increasing volume fraction of particles. It is suggested that both the matrix and the nanocrystalline particles contribute to the overall hardness of the composites.

[2] NANOPOROUS CARBON PRODUCED BY BALL MILLING

Chen Y. Gerald JF. Chadderton LT. Chaffron L. - Applied Physics Letters. 74(19):2782-2784, 1999

A nanoporous structure was produced in the samples of graphite after ball milling at ambient temperature. The specific internal surface area of the micropores, as determined using the t-plot method, is higher than the external surface area of particles and mesopores. Phase transformations from hexagonal to turbostratic, and to amorphous structures were investigated using x-ray diffraction analysis and transmission electron microscopy. Formation of the nanoporous structure is associated with that of the disordered carbon. The disordered and nanoporous structure is probably fullerene-like in nature.

[1] THERMOELECTRIC PROPERTIES OF MECHANICALLY ALLOYED BI-SB ALLOYS

Martin-Lopez R. Dauscher A. Scherrer H. Hejtmanek J. Kenzari H. Lenoir B. - Applied Physics a (Materials Science Processing). 68(5):597-602, 1999

Homogeneous polycrystalline Bi_{100-x}Sb_x (x = 12, 15, 22) alloys were synthesized by mechanical alloying. The transport coefficients (electrical resistivity, thermal conductivity, and thermopower) were measured, in the 77-300 K temperature range, on samples consolidated either by sintering or extrusion. The thermoelectric figure of merit was deduced from the three coefficients. The temperature dependences are discussed as a function of the alloys' microstructures taking into account the qualitative effect of potential barriers. Extrusion leads to better performing thermoelectric materials than does sintering. The highest figure of merit is reached for temperatures around 150 K, a temperature at which no reliable thermoelectric material of long service life is available until now.

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