



**RESEAU FRANÇAIS DE
MECANOSYNTHESE**

Lettre N°84

Mars 2002

**186 (+1) Groupes de Recherche
(dont 112 (+1) à l'étranger / 34 (+1) Pays)**

**Bureau du RFM : E. Gaffet (Président)
G. Le Caër (Secr. Gén.), A.R. Yavari (Trés.)**

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**Une Nouvelle Adhésion
Dr Nadia Bensebaa**
Université d' Annaba - Institut de physique.- Annaba - Algérie

Bulletin d'adhésion 2002 / Subscription Print

(à retourner à l'adresse suivante - to be sent at the following address) :

Eric GAFFET

CNRS UMR5060 « Métallurgies et Cultures »

Nanomaterials Research Group

Site de Sévenans (UTBM) - F90010 - Belfort Cedex - France

Nom/Name :Prénom / First Name :

Adresse complète / Full Address :

Téléphone/ Phone:Télécopie (Fax) :

e_Mel. / e-Mail :

désire adhérer au Réseau Français de Mécanosynthèse / want to become a member of the French Mechanical Alloying Network

Chèque ci joint / Check enclosed in the amount of **20 Euros (20€)**

The check has to be to the order to : Réseau Français de Mécanosynthèse

Le site web du RFM est :

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

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

⇒ vous y trouverez les anciennes lettres du RFM (accessible par Adobe Acrobat)

les statuts du RFM ainsi que les annonces concernant les JRFM'2001

et quelques éléments mis à jour régulièrement concernant les derniers résultats dans ce domaine.

Sommaire

⇒ Thèses / Congrès

⇒ Bibliographie du mois d'Octobre

⇒ Dossiers d'annonces techniques

Congress and School Announcements

Nano 2002

16 - 21 Juin 2002
Orlando, Florida - USA
Website : <http://www.nano2002.com/>

NANO-7 / ECOSS-21

**7th International Conference on Nanometer-Scale Science and Technology
& 21st European Conference on Surface Science**

June 24-28,
in Malmö, Sweden.
Website : <http://www.malmo-congress.com/nano-ecoss.html>
(Deadline for abstract submission is **February 17**)

Workshops

Gordon Research Conference on Granular and Granular-Fluid Flow

Plymouth, NH, USA June 30 - July 5 ,2002
<http://sol.rutgers.edu/~shinbrot/gordon2002/gordon2002.html>

ICSTR

INTERNATIONAL CONFERENCE ON SOLVO-THERMAL REACTIONS
July 22-26, 2002 - Hilton East Brunswick / East Brunswick, New Jersey,
More information on this meeting can be found at <http://www.ICSTR.rutgers.edu/>
or by contacting Professor Richard E. Riman at Rutgers University via
riman@email.rci.rutgers.edu/732-445-4946(v)/732-445-6262 (f).

RQ11

Rapidly Quenched and Metastable Materials
25-30 August 2002
Department of Materials, University of Oxford, UK
Contact: RQ11 Conference Organiser, Beggars Roost, Channels End Road,
Comworth Bedford MK44 2NS, U.K.
Tel: +44 (0) 1234 378862
Fax: +44 (0) 1234 376219
E-mail: mailto:rq11@materials.ox.ac.uk
Website: <http://www.materials.ox.ac.uk/rq11>

10th European Symposium on Comminution

Heidelberg from 2-5 September 2002.
Org. European Federation of Chemical Engineering
Full information available at <http://www.comminution2002.de>

8th ICCPS

8th International Conference on Ceramic Processing Science
Hamburg - Sept. 2nd - 5, 2002.

The conference will focus on novel processing of advanced structural and functional
ceramics and ceramic composites. The program will favor the most recent
developments in this presented in only 10 topical sessions:

1. New Concepts for Economic Production of Powders of High Purity, Reactivity and Ease of Handling
2. Novel Powder Processing and Non-Conventional Shaping (Nanoprocessing, Cellular Structures, etc.)
3. Solution Processing (Thin Film Deposition, Soft Solution, Polymer-Derived, etc.)
4. Biomimetic Structuring (Biotemplates, Biomineralization, etc.)
5. Novel Reaction Forming (Controlled SHS, Reactive Casting, in situ Processing, etc.)
6. Computer-Controlled Shaping and Structuring (Rapid Prototyping, Solid Free-Forming, Controlled Heterogeneities, etc.)
7. Tailoring of Synergy Ceramic Microstructures (LTCC, Self-Sensing Devices, Smart Structures, MEMS, etc.)
8. Grain Boundary Engineering (Grain-Boundary-Free Microstructures, Directed Eutectics, Advanced Electroceramics, etc.)
9. Micromechanics of Composite Synthesis (Transient and Residual Stresses, Constrained Sintering, etc.)
10. COST 528: Chemical Solution Deposition of Thin Films

website : <http://www.tu-harburg.de/gk/8th-ICCPS>

ISMANAM-2002

International Symposium on
Metastable, Mechanically Alloyed and Nanocrystalline Materials
Seoul, Korea, 8-12 September, 2002.
Web site : <http://anu.andong.ac.kr/~ismanam/>



L. A. C. A. M. E – 2. 0. 0. 2
EIGHTH LATIN AMERICAN CONFERENCE
ON APPLICATIONS OF THE MÖSSBAUER EFFECT
PANAMA, 22-27, SEPTEMBER, 2002.
E-mail: <mailto:lacame2000@fisica.ciens.ucv.ve>
<http://www.up.ac.pa/Eventos/lacame2002/inicio.htm>

Matériaux 2002

Tours - France

21- 25 Octobre 2002

Website : <http://www.materiaux2002.net>

E_mail : materiaux@materiaux2002.net

Les JRFM'2002 seront intégrées dans le cadre du Congrès

Matériaux 2002 (Tours – France, du 21 au 25 Octobre 2002)

Poudres et Matériaux Nanostructurés, du fondamental aux applications industrielles

Symposium 1 :

Website : <http://www.materiaux2002.net> : E_mail : materiaux@materiaux2002.net



SOUTENANCES DE THESE

Sophie Soiron

Influence de la mécano-chimie sur les propriétés structurales et catalytiques d'oxydes de structure type pérovskite et spinelle

16 novembre 2001, à Amiens, Amphi Figlarz, à 14h

Jury:

Rapporteurs: M. Jean Mimault (*Université de Poitiers*), M. Edmond Payen (*Ecole de chimie de Lill*)

Examineurs: M. Luc Aymard (*Université d'Amiens*), M. Christian Julien (*CNRS- Université de Paris VI*), M. G-Abbas Nazri (*General Motors R&D*), Melle. Aline Rougier (*CNRS- Université d'Amien*), M. Bechara Taouk (*Université de Compiègne*), M. Jean-Marie Tarascon (*Université d'Amien*)

F. Dore

"Densification de pseudo alliages W - Cu à partir de phases submicroniques"

Université de Grenoble - 13 Novembre 2001

Jury :

E. Gaffet (Rapporteur), J.-L. Jorda (Rapporteur), C. Allibert (Directrice de Thèse),
C. Martin (Co - Dir. De Thèse), J.-F. Lartigue, M. Soustelle

Raphaël JANOT

Université de Nancy I – 24 octobre 2001

Mécanosynthèse en milieu liquide de composés graphite-lithium superdenses, de graphite très anisométrique et de maghémite supportée ou non sur graphite

Jury :

J. Conard (Rapp.), M. Danot (Rapp.), P. Ehrburger, D. Guérard (Dir. Thèse), R. Marassi, A. Rougier

Thierry Girot

"Cinétique et modélisation des transformations de phases induites par broyage à haute énergie dans TiO₂ anatase "

Thèse INPL, 15 Octobre 2001

Jury :

J. Focet, D. Michel (R), J.C. Niepce (R), F. Radjaï, G. Le Caer, A. Mocellin, S. Begin (Dir. Thèse)

Sébastien Lehnard

"Texture, Microstructure et Propriétés d'un Alliage Fe-40 Al à grains fins obtenu par métallurgie des poudres et extrusion : Influence des paramètres du procédé et de traitements thermiques"

Université de Metz - 5 octobre 2001-08-23



Jury :

R. Schwarzer (Rapp.), E. Gaffet (Rapp.), JP Morniroli, V Skrotzi, R. Baccino, A. Hazotte,
F. Wagner (Dir. Thèse), Th. Grosdidier (Co. Dir. Thèse)

Nathalie Bouad

"Mise au point d'un procédé d'élaboration de matériaux thermoélectriques pour thermogénérateur.

Potentialité de la mécanosynthèse d'alliages à base de tellure de plomb"

Montpellier, Université Montpellier II, 10 mai 2001

Jury :

J. Foct, J.C. Niepce, H. Scherrer, R. Griot, A.M. Bouchardy, J. Delallée, Y. Lacrouts-Cazenave, M. Ribes, J.C. Tédénac, R.M. Marin-Ayral (directeur de thèse)

Cooperative Research on Related Areas

France (12/04/2001)

Le portail Internet "France Contact" a été lancé: ce portail s'adresse aux chercheurs étrangers séjournant ou ayant séjourné en France et permettra le suivi et l'animation du réseau que constituent les milliers de chercheurs étrangers ayant effectué un séjour scientifique au sein des établissements et des organismes de recherche français:

Website : <http://www.francecontact.net>

Europe (6/03/2001)

The ESF, on the recommendation of the scientific Standing Committee for Physical and Engineering Sciences (PESC), will support, in fields related to PESC's remit, approximately 10 ESF Exploratory Workshops to be held in 2002.

Each workshop will allow 20-25 leading European scientists to explore novel ideas at the European level with the challenging aim to "spearhead" new and preferably inter-disciplinary areas of research.

In specific terms, PESC's 2001 Call is for workshop proposals on R&D subjects which are NOVEL AND PREFERABLY INTERDISCIPLINARY and which concern emerging fields within any of the following areas: chemistry, physics, mathematics, information sciences, fundamental engineering sciences, materials sciences, and technologies research in these areas.

The PESC Call is available at <http://www.esf.org/physical/WorkshopCalls/Call2001.htm>



**Job Vacancies, Ph D Position and, Post Doc Position
Requests – Proposals**

Proposal from 28/01/2002

The Laboratory for Neutron Scattering (Paul Scherrer Institute and ETH Zuerich) announces three openings for research scientists (physicists, chemists, crystallographers) at the Swiss Spallation Neutron Source 'SINQ' (<http://sinq.web.psi.ch/>). The posts represent excellent opportunities for postdoctoral scientists to develop their expertise, broaden experience and interact with scientists from many countries. We are looking for

A - Responsible for the new SANS-II facility (joint venture between PSI and Risoe National Lab.)

(<http://sinq.web.psi.ch/sinq/instr/sans2.html>)

(at your earliest convenience)

Reference Number: 3302A

B - Co-responsible for the new triple-axis spectrometer Rita-I (joint venture between PSI and Risoe National Lab.)

(<http://sinq.web.psi.ch/sinq/instr/ritai1.html>)

(from 01/07/2001)

Reference Number: 3302B

C - Co-responsible for the triple-axis spectrometer TASP (<http://sinq.web.psi.ch/sinq/instr/tasp.html>)

(from 01/10/2002)

Reference Number: 3303A

Your tasks would be:

- Responsibility for the installation/operation and further development of the instruments, in particular co-operation with guest scientists in their experiments at SINQ
- Performance of neutron scattering experiments at SINQ for your own research projects
- Development and implementation of analytical software for the instruments

For further information (but not applications) please contact for

(A) Dr. S. Janssen, phone: +41-56-310-2875, e-mail: <mailto:stefan.janssen@psi.ch>,

(B) Dr. J. Mesot, phone: +41-56-310-4029, e-mail: <mailto:joel.mesot@psi.ch>

(C) Dr. P. Allenspach, phone: +41-56-310-4029-2527, e-mail: <mailto:peter.allenspach@psi.ch>

Information about the Laboratory for Neutron Scattering and about SINQ can be obtained from the following web pages:

<http://lns.web.psi.ch/>

<http://sinq.web.psi.ch/>

Please send applications with C.V., a list of publications and the names of two academic referees quoting reference 3302A, 3302B or 3303A, no later than by March 15, 2002 to: PAUL SCHERRER INSTITUT, Human Resources, CH-5232 Villigen PSI, Switzerland.

From Prof. H.E. Schaefer

(22/01/2002)

In the framework of the 5th European Research and Development Program, the Institut fuer Theoretische und Angewandte Physik, Stuttgart University, Research Group of Prof. H.-E. Schaefer

(<http://www.itap.physik.uni-stuttgart.de/~gsweb/english/index.html>)

has been selected a Marie Curie Training Site and offers a one year position :

Marie Curie Training Site
Doctoral Training / Post-Doc Positions
at
Stuttgart University, Germany
Institute für Theoretische und Angewandte Physik
Research Group Prof. H.-E. Schaefer

in the field of
Nanostructured Materials :
Atomic Transport Properties for the Synthesis and
Characterization of Novel Soft and Hard Magnets
(Contract No.: HPMT-CT-2001-00224)

Applicants are invited for a 12 month term as a research fellow supported by individual fellowships of the Marie Curie fellowship scheme. The successful candidates will be involved in the synthesis and processing of novel nanostructured materials and composites for soft magnets (Finemet-type) and hard magnets (FeNdB-type), as well as with the investigation of their microstructure, magnetic, and diffusional properties. The gas-phase condensation technique with subsequent



compaction under high pressure is used for the production of highly dense nanocrystalline materials. Basic material characterisation will be carried out by x-ray diffraction, differential scanning calorimetry, optical microscopy, and atomic resolution electron microscopy (HRTEM). In addition, several instruments for characterisation of magnetic materials are available. These experimental techniques allow the investigation of a number of phenomena, including: order-disorder transformations, transformation kinetics, phase transitions, and relaxation processes. Furthermore, diffusion studies using the radioactive tracer technique are carried out in order to study the atomic transport properties in nanocrystalline structures.

The candidates have to satisfy the basic criteria of the training scheme as outlined on the Marie Curie Host Fellowship Web site <http://www.cordis.lu/improving/fellowships/home.htm>. As the fellowship forms part of a higher degree project, the candidates should be registered as full-time Ph.D. research students in a well recognized institution of the EC. The research interest of the candidates should be in at least one of the following fields: solid state physics, materials science including synthesis and characterisation of materials, mechanical and magnetic properties of advanced materials, and structural studies.

The group closely cooperates with the Max-Planck-Institut für Metallforschung, Stuttgart. This collaborative character of the research training provides an additional international profile to the education of the fellows increasing their interaction and eventually their active collaboration with research institutions in different European countries.

Applicants, also Post-Docs, should contact us for further information:

e-mail	Prof. H.-E. Schaefer	mailto:schaefer@itap.physik.uni-stuttgart.de
	Dr. W. Sprengel	mailto:sprengel@itap.physik.uni-stuttgart.de
phone:	+49-711-685-5261	+49-711-685-5192
FAX	+49-711-685-5271	+49-711-685-5271
	http://www.itap.physik.uni-stuttgart.de/~gsweb/english/index.html	

De L. Aymard CNRS / Amiens (France)

1/10/2001

Pour la Rentrée Universitaire 2001 - 2002

Le Laboratoire de Réactivité et de Chimie des Solides à Amiens recherche pour la rentrée prochaine :

- un étudiant pour une thèse de 3ème cycle. (Durée 3 ans) à partir de septembre 2001

- un étudiant pour un Stage Post Doc (12 mois prolongement possible) à partir de septembre 2001.

Domaine de Recherche: Stockage d'énergie, Hydrures Métalliques.

Envoyer vos CV avant la fin juillet à L. Aymard LRCS

Email : <mailto:luc.aymard@u-picardie.fr>



Bibliographie Récente

Livres ou "Special Issues"

(21/06/2001)

From Christian Wohlbier (Scientific. Net Webmaster)

This is a service of <http://www.scientific.net>

*** **Materials Science Forum** ***

Materials Science Forum specializes in the rapid publication of international conference proceedings and stand-alone volumes on topics of current interest. It covers all areas of Materials Science, Solid State Physics and Solid State Chemistry. The periodical is indexed in Science Citation Index and covered by all major abstract media.

Volume 246 until 246 [Surface Coatings for Advanced Materials] and

Volume 207 until 209 [Intergranular and Interphase Boundaries in Materials]

<http://www.scientific.net/msf>

*** **Solid State Phenomena** ***

Solid State Phenomena specializes in the rapid publication of international conference proceedings and stand-alone volumes on topics of current interest in the field of solid state physics and its applications to materials science related topics. The periodical is indexed in Science Citation Index and covered by all major abstract media.

Volume 61 until 62 [Contemporary Studies in Condensed Matter Physics],

Volume 59 until 60 [Interfaces and Plasticity] and

Volume 57 until 58 [Gettering and Defect Engineering in Semiconductor Technology]

<http://www.scientific.net/sfp>

(07/06/2001)

« **Strategic and Technological Watch on Nanomaterials** »

by **E. Gaffet** (1998 – 2000) – 4 CD reports (6.000 analysed references)

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

Website : <http://www.innovation128.fr/>

(28/05/2001)

Advanced Ceramic Materials

*** **Key Engineering Materials, Volume 122 until 124** ***

In spite of the very great progress made in ceramic science, and the elegance and excitement of the research which has been performed, the real driving force for developments in ceramics remains their potential applications. The opportunity for dramatic scientific advances was certainly one reason for the "ceramic fever" of a decade ago, but there is also no doubt that the

prediction of an annual market for fine ceramics, amounting to 6 billion Yen played a role. The challenge is to ensure that ceramics can be successfully introduced into the full breadth of applications where their properties have long made them so appealing. The present volume takes a refreshing and firm step towards the realization of this aim. The publication of a book which sets out to present ceramics from the specific point of view of applications is an event greatly to be welcomed. Systematic organization into various types of application ensures that the reader can fully appreciate the outstanding opportunities offered; and the present limitations. Armed with such a survey, the engineer and scientist will be fully alert to possibilities for progress whenever these arise. 1. Introduction. 2. Electrical and Electronic Functions. 3. Magnetic Functions. 4. Chemical and Physical Functions. 5. Mechanical and Thermal Functions. 6. Biological Functions. 7. Nuclear Applications. 8. Ceramic Coatings. 9. Selected Ceramics with Multi-Applications.

<http://www.scientific.net/kem>

(11/2000) **Information from Fritsch (A. Kohler)**

The subject of the sixth forum part, Fritsch Forum Part VI scheduled for September 14/15th, 2000, will be "high-energy fine grinding". Research and Development demand general-purpose grinding processes which simultaneously exactly define the required energy and the type of stress. This is the only way that reliable results can be achieved when determining activation energies or the mechanical alloying. It must be possible to reproducibly adjust all of the grinding parameters affecting the grinding results.

Participants from research, development and industry will report on demands and novel technological solutions in developing innovative milling technologies. One of the highlights of the event will be FRITSCH's new Vario-planetary mill "pulverisette 4". This planetary ball mill can simulate ball mills of conventional construction, precisely copy the types of stresses that occur there, and thus reproduce or optimise grinding processes. Due to the great flexibility when selecting the grinding parameters, it is possible to produce results that are unattainable with other ball mills. It is the ideal mill for mechanical activation and alloying. The main applications are in the area of material research and naturally wherever a powerful, innovative laboratory planetary mill is needed.

An extensive report has been written about this event which details and makes readily available the relevant parts of the lectures and the extensive results of the discussions. Anyone interested can request a copy of the complete report for this forum part VI event on the topic "high-energy fine grinding". Please contact Andrea Köhler, FRITSCH GMBH, Industriestrasse 8, D-55743 Idar-Oberstein, (Phone: 0049/6784/7046, E-Mail: koehler@fritsch.de)

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



Lettre RFM N°84 - Mars 2002
Corresp. : <mailto:Eric.Gaffet@utbm.fr>

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

MACROMOLECULAR MECHANOCHEMISTRY

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

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

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

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

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

Send your preliminary order to <mailto:orders@cisp.demon.co.uk>

(9/06/2000)

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

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

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

Preface

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

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

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

(3/02/2000)

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

1. MECHANICAL ALLOYING - FUNDAMENTALS AND APPLICATIONS <http://www.demon.co.uk/cambsci/book52.htm>

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

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

Energy transfer and energy maps;

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

Modelling mechanical alloying (mechanistic models, deformation, coalescence and fragmentation, evolution of particle size, milling time, powder heating, powder cooling, atomistic model, thermodynamic and kinetic model) Joining of mechanically alloyed materials; Rapid solidification and mechanical alloying; Applications (nickel-based superalloys, Al-based materials, supersaturated solutions, magnetic materials, mechanically alloyed powders for spray coatings, superplasticity, tribological materials, composites, amorphous solids, nanocrystalline materials, solid-state chemical reactions, etc). ISBN 1898326568, 160 pages 234 156 mm, cased, £45.00, 1999

DISPERSION STRENGTHENED ALUMINIUM PREPARED BY MECHANICAL ALLOYING, by M Besterçi - <http://www.demon.co.uk/cambsci/book51.htm>



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Corresp. : <mailto:Eric.Gaffet@utbm.fr>

1. Characteristics of dispersion-strengthened systems 2. **Mechanical alloying** (kinetics and mechanism of preparation of the Al-C system by mechanical alloying; compaction of powders and heat treatment of compacts);
3. Microstructure and quantitative evaluation of parameters of dispersion-strengthened materials (definition and properties of interparticle distance; experimental possibilities of determination of structural objects; models of heterogeneous structures and their evaluation; simulation of model structures; analysis of the spatial distribution of particles in the Al-Al₄C₃ material)
4. Static and dynamic mechanical properties (mechanical properties at elevated temperatures; mechanical properties at 20 °C; effect of interface on the mechanical properties; superplastic properties of the system; thermal stability of the system; creep characteristics; creep-fatigue characteristics)
References - ISBN 189832655X, 90 pages, 234 156 mm, soft laminated cover, £25.00, 1999

"Mechanical Alloying : Fundamentals and Applications"

Prof. P.R. Soni, (1999) - Cambridge International Science Publishing
web site : <http://www.demon.co.uk/cambsi/book52.htm>

"Nanomatériaux"

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

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

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

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

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

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



Patent / Brevet

ONE STEP SYNTHESIS AND CONSOLIDATION OF NANOPHASE MATERIALS

Z.A. Munir, F. Charlot, F. Bernard, E. Gaffet – International patent WO 0112366 (publié le 22.02.2001)

Solid reaction products with a dense nanocrystalline structure are formed from reactant particles with diameters in the nano – scale range by compacting the particles into a green body, then passing an electric current through the body causing Joule heating sufficient to initiate the reaction to form the reaction product while simultaneously applying pressure to the reacting body to densify it to a density approaching the theoretical density of the pure product. Surprisingly, this process results in a reaction product that retains the nanocrystalline structure of the starting materials, despite the fact that a reaction has occurred and the materials have been subjected to highly stringent conditions of electric current, heat and pressure.

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ou encore pour la version brevet d'application

<http://12.espacenet.com/dips/bnsviewer?CY=ep&LG=en&DB=EPD&PN=WO0112366&ID=WO+++0112366A1+I+>

Périodiques

[63] ELECTROCHEMICAL ACTIVITY OF NANOCRYSTALLINE COATING OF TI-RU-FE-O

E Irissou, M Blouin, R Schulz, L Roue, D Guay - ELECTROCHEMICAL AND CHEMICAL REACTIVITY OF AMORPHOUS AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2001, Vol 377, pp 29-37 - 6TH INTERNATIONAL SYMPOSIUM ON CHEMICAL AND ELECTROCHEMICAL REACTIVITY OF AMORPHOUS AND NANOCRYSTALLINE MATERIALS; MT TREMBLANT, CANADA. FEBRUARY 7-9, 2001

Large quantities of nanocrystalline Ti-Ru-Fe-O (2-1-1-2) powder were prepared in a ZOZ attritor. The powder thus obtained contains more than 50 wt.% of hexagonal Ru and Fe₂Ti. When used as starting material for plasma spray deposition, these phases react together to form B2 cubic structures with various lattice parameters, reflecting change in the stoichiometries. The coating obtained by plasma spraying this powder is adherent on a Fe substrate. It exhibits a cathodic overpotential for hydrogen evolution ($i = -250 \text{ mA cm}^{-2}$) in typical chlorate electrolysis conditions Of $\eta(250) = -550 \text{ mV}$.

[62] PEROVSKITE-TYPE OXIDES SYNTHESIZED BY REACTIVE GRINDING

S Kaliaguine, V Szabo, A VanNeste, JE Gallot, M Bassir, R Muzychuk - ELECTROCHEMICAL AND CHEMICAL REACTIVITY OF AMORPHOUS AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2001, Vol 377, pp 39-56 - 6TH INTERNATIONAL SYMPOSIUM ON CHEMICAL AND ELECTROCHEMICAL REACTIVITY OF AMORPHOUS AND NANOCRYSTALLINE MATERIALS; MT TREMBLANT, CANADA. FEBRUARY 7-9, 2001

Perovskite type-materials LaCo((1-x))FexO₃ Were prepared from their component oxides using mechanosynthesis, a new method of preparation designated as reactive grinding. When grinding additives are used, the perovskites generated via this process are of unprecedentedly high specific surface area (> 100 m²/g) and their surface chemical properties are of particular interest. The perovskite surface shows a large concentration of surface OH's (due to the low grinding temperature) which upon calcination leads to reformation of the materials lattice. The presence of oxygen during calcination initiates a competitive process, which stabilizes sites with an excess of electronic charge, yielding reduced Co sites upon oxygen desorption (Co²⁺) At high calcination temperature (> 673 K), TPD studies show the release of beta -oxygen originating from the bulk. In this work, the catalytic properties in the n-hexane oxidation reaction of five nanocrystalline LaCo(1-x)FexO₃ perovskites prepared by this method are studied. The low preparation temperature allows to stabilize high surface area (SSA) materials and the adequate control of the calcination conditions allows to tailor the surface properties of these materials. Through a kinetic study of the total oxidation of n-hexane by oxygen, the fine control of the nature and the concentration of the various sites active in oxido-reduction catalysis is demonstrated. Furthermore, the roles of two oxygen species O²⁻ and O⁻ respectively associated with alpha and beta oxygens are discussed.

[61] CHARACTERIZATION OF PD-MG CATALYST PRECURSORS PREPARED BY BALL MILLING AND COMPARISON WITH CU-MG

G Mulas, M Varga, I Bertoti, M Mohai, A Molnar, G Cocco - ELECTROCHEMICAL AND CHEMICAL REACTIVITY OF AMORPHOUS AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2001, Vol 377, pp 57-62 - 6TH INTERNATIONAL SYMPOSIUM ON CHEMICAL AND ELECTROCHEMICAL REACTIVITY OF AMORPHOUS AND NANOCRYSTALLINE MATERIALS; MT TREMBLANT, CANADA. FEBRUARY 7-9, 2001

We have prepared a Pd-MgO sample by self-sustaining reaction between Mg and NO, and two Pd-Mg bicomponent alloys by mechanochemical synthesis induced by ball milling. They were characterized by XRD, DSC and XPS techniques. Further characterization was carried out following controlled oxidation which was necessary to enhance the catalytic performance in the; synthesis of methyl isobutyl ketone, Complex transformations characterized in most cases by the formation of Pd-Mg intermetallics are observed by XRD. A common feature of all samples is the surprisingly low concentration of the metallic components due to oxygen and carbon impurities and the lack of palladium on the surface, High catalytic activities were achieved in catalytic studies but overhydrogenation due to the high hydrogenating ability of Pd resulted in decreased selectivities.



[60] OPTIMIZATION OF THE BALL-MILLING PARAMETERS FOR THE SYNTHESIS OF AMORPHOUS MGNI ALLOY USED AS NEGATIVE ELECTRODE IN NI-MH BATTERIES

S Ruggeri, C Lenain, L Roue, H Alamdari, G Liang, J Huot, R Schulz - ELECTROCHEMICAL AND CHEMICAL REACTIVITY OF AMORPHOUS AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2001, Vol 377, pp 63-70 - 6TH INTERNATIONAL SYMPOSIUM ON CHEMICAL AND ELECTROCHEMICAL REACTIVITY OF AMORPHOUS AND NANOCRYSTALLINE MATERIALS; MT TREMBLANT, CANADA. FEBRUARY 7-9, 2001

The optimization of the ball milling parameters resulted in the synthesis with a milling duration equal to 10 hours of amorphous MgNi having an initial discharge capacity over 520 mAh/g. Further milling results in a partial crystallization of amorphous MgNi into nanocrystalline MgNi₂ and Mg₂Ni, which decreases significantly the electrode performance. This study also shows that it is possible to obtain an amorphous and electroactive material in large scale (1 kg of alloy per batch) using an industrial high-energy attritor. In addition, it was demonstrated that the carbon added at the beginning of the milling to avoid powder welding, in spite of its small proportion (1 wt.%), has a notable influence on the performance of the material. This study also confirms the major loss of activity during cycles. However, the partial substitution of Ti for Mg leads to a remarkable improvement of the cycle lifetime of the alloy.

[59] SYNTHESIS OF NANOCRYSTALLINE CANIR(5)-BASED ALLOYS AND USE FOR METAL HYDRIDE ELECTRODE

G Liang, S Ruggeri, C Lenain, H Alamdari, J Huot, L Roue, R Schulz - ELECTROCHEMICAL AND CHEMICAL REACTIVITY OF AMORPHOUS AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2001, Vol 377, pp 71-75 - 6TH INTERNATIONAL SYMPOSIUM ON CHEMICAL AND ELECTROCHEMICAL REACTIVITY OF AMORPHOUS AND NANOCRYSTALLINE MATERIALS; MT TREMBLANT, CANADA. FEBRUARY 7-9, 2001

The CaNi₅-based alloys were synthesized by mechanical alloying followed by a low temperature annealing. Study shows that both substitution and annealing treatment affect the hydrogen storage capacity. Min substitution for Ca results in an increased plateau pressure. The Zn and Al substitution for Ni leads to a bigger unit cell volume, a lower plateau pressure and a reduced capacity. Annealing treatment of the mechanically alloyed CaNi₅-based alloys is a necessary process for obtaining good hydrogen storage capacity. The synthesized Ca-Mm-Ni-Zn-Al multicomponent nanocrystalline alloys exhibit fast kinetics on gas phase and electrochemical reaction and significantly improved cycle stability on electrochemical reaction.

[58] BIMETALLIC CATALYST EFFECT ON THE SORPTION PROPERTIES OF NANOCRYSTALLINE MGH₂ HYDRIDE

Z Dehouche, J Goyette, TK Bose, G Liang, J Huot, R Schulz - ELECTROCHEMICAL AND CHEMICAL REACTIVITY OF AMORPHOUS AND NANOCRYSTALLINE MATERIALS (Series: MATERIALS SCIENCE FORUM), 2001, Vol 377, pp 77-83 - 6TH INTERNATIONAL SYMPOSIUM ON CHEMICAL AND ELECTROCHEMICAL REACTIVITY OF AMORPHOUS AND NANOCRYSTALLINE MATERIALS; MT TREMBLANT, CANADA. FEBRUARY 7-9, 2001

In this work, we have investigated the hydriding/dehydriding properties and the effect of prolonged cycling done under pure hydrogen on the performance of nanostructured Mg-V-Ti composite synthesised by ball milling. The hydrogen charge and discharge characteristics of the nanocomposite hydride has been tested at 300 degreesC using up to 1000 cycles. The comparison of the hydriding/dehydriding kinetics and the pressure-concentration-isotherms curves, measured before and after 1000 cycles, reveals no significant change in the kinetics and thermodynamic properties of the MgH₂-V-Ti composite. However, a clear improvement in the cyclable hydrogen capacity was observed. SEM, X-ray and BET specific surface area characterisations reveal that the nanocrystalline Mg based composite exhibits an improved decrepitation resistance upon cycling.

[57] DIFFRACTION-LINE PROFILE ANALYSIS - A SIMPLE WAY TO CHARACTERIZE BALL-MILLED MO?

I Lucks, P Lamparter, EJ Mittemeijer - EPDIC 7: EUROPEAN POWDER DIFFRACTION, PTS 1 AND 2 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 378-3, 1&2, pp 451-456 - 7TH EUROPEAN POWDER DIFFRACTION CONFERENCE (EPDIC 7); BARCELONA, SPAIN. MAY 20-23, 2000

Molybdenum powder was ball milled for times ranging from 90min to 180h in a steel vessel with hardened steel balls under Ar atmosphere. The milling products were examined by X-ray diffraction. The methods of Williamson and Hall, Warren and Averbach and an alternative method after van Berkum et al. For the separation of size and strain contributions were applied to the profiles. Differences and similarities between the results of the various methods were discussed.

[56] X-RAY ANALYSIS OF THE DEFECT STRUCTURE IN CU SUBJECTED TO SEVERE PLASTIC DEFORMATION

AR Kilmametov, K Zhang, IV Alexandrov, RM Mazitov, K Lu - EPDIC 7: EUROPEAN POWDER DIFFRACTION, PTS 1 AND 2 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 378-3, 1&2, pp 457-462 - 7TH EUROPEAN POWDER DIFFRACTION CONFERENCE (EPDIC 7); BARCELONA, SPAIN. MAY 20-23, 2000

The X-ray analysis of the nanostructured copper (99.98%) processed by different techniques of severe plastic deformation (SPD) was fulfilled in the temperature range from 85 K to 295 K. The analysis of obtained results in frames of the Debye quasiharmonic theory revealed a considerable decrease in the Debye temperature up to 19-23%, an increase of static and dynamic atomic displacements approximately 1.5 times and linear thermal expansion coefficient more than 3 times in the comparison with the coarse-grained copper. The revealed decrease in the Debye temperature doesn't contradict the results of previous experiments on the measurement of the adiabatic Young and shear modules by the ultrasonic method. The above mentioned changes of thermal characteristics of the SPD nanostructured copper can be caused by special non-equilibrium state of grain boundaries and nearby regions formed as a result of SPD. Results of fulfilled investigations gave a possibility to develop the structural model of SPD nanomaterials.



[55] X-RAY LINE PROFILE ANALYSIS OF NANODISPERSE SILICON NITRIDE CERAMICS

J Gubicza, J Szepevolgyi, I Mohai, T Ungar - EPDIC 7: EUROPEAN POWDER DIFFRACTION, PTS 1 AND 2 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 378-3, 1&2, pp 729-734 - 7TH EUROPEAN POWDER DIFFRACTION CONFERENCE (EPDIC 7); BARCELONA, SPAIN. MAY 20-23, 2000

Nanodisperse silicon nitride powders were produced by different methods of synthesis. The effect of the production routes on the grain-size distribution and the dislocation density in the powders were studied by high-resolution X-ray diffraction. The average grain-size and the dislocation density of the samples were determined by the recently developed modified Williamson-Hall and Warren-Averbach procedures from X-ray diffraction profiles. A new numerical method provided log-normal grain-size distributions from the size parameters derived from X-ray diffraction profiles. It was established that the powder produced by silicon nitridation and milling had lower average grain-size and wider size distribution than the sample crystallized from amorphous silicon nitride powder synthesized in plasma reactor. The grain-size distribution and the area-weighted average grain-size obtained by X-rays were in good agreement with those determined by TEM and from the specific surface area, respectively. The dislocation density was found to be between 10^{14} and 10^{15}m^{-2} .

[54] MECHANOCHEMICAL TREATMENT OF $\text{SrFe}_{12}\text{O}_{19}$ - MICROSTRUCTURAL INVESTIGATION BY NEUTRON DIFFRACTION

M Hofmann, SJ Campbell, E Wu, WA Kaczmarek, M Dahlborg, U Dahlborg - EPDIC 7: EUROPEAN POWDER DIFFRACTION, PTS 1 AND 2 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 378-3, 1&2, pp 765-770 - 7TH EUROPEAN POWDER DIFFRACTION CONFERENCE (EPDIC 7); BARCELONA, SPAIN. MAY 20-23, 2000

Neutron diffraction measurements have been used to track the microstructural changes which take place on milling $\text{SrFe}_{12}\text{O}_{19}$ and on subsequent annealing treatments. Mechanochemical treatment causes $\text{SrFe}_{12}\text{O}_{19}$ to transform progressively to disordered material, incorporating nanostructured and amorphous phases, with chemical reduction to haematite, $\alpha\text{-Fe}_2\text{O}_3$ and SrO . Good agreement is obtained between the behaviour of the recrystallisation process as investigated by room temperature measurements of annealed products, and by separate in situ measurements at high temperatures. The recrystallisation process, which starts at similar to 640 degreesC and is complete by similar to 780 degreesC with mean particles sizes for $\text{SrFe}_{12}\text{O}_{19}$ of d similar to 130 nm at 800 degreesC, follows a similar behaviour to that of the spontaneous magnetisation and magnetic coercivity as reported previously for milled and annealed $\text{BaFe}_{12}\text{O}_{19}$ and $\text{SrFe}_{12}\text{O}_{19}$.

[53] MICROSTRUCTURAL CHARACTERIZATION AND MECHANICAL BEHAVIOR OF NANO- AND SUBMICRON-GRAINED TITANIUM ALUMINIDE/TITANIUM SILICIDE COMPOSITES PREPARED BY HIGH ENERGY MILLING

R Bohn, G Fanta, T Klassen, R Bormann - POWDER MATERIALS: CURRENT RESEARCH AND INDUSTRIAL PRACTICES, 2001, pp 107-126 - INTERNATIONAL SYMPOSIUM ON POWER MATERIALS: CURRENT RESEARCH AND INDUSTRIAL PRACTICES HELD AT THE 2001 TMS FALL MEETING; INDIANAPOLIS, INDIANA. NOVEMBER 4-8, 2001

This study was launched to systematically investigate the powder-metallurgical synthesis and the mechanical properties of intermetallic/ceramic composites with grain sizes in the nano- and submicron range. As model systems, $\gamma\text{-TiAl}$ -based matrices with varying amounts of finely dispersed Ti_5Si_3 particles were chosen. Three objectives were pursued: (1) The measurement of truly grain size dependent mechanical properties. Not affected by any processing flaws, (2) the coverage of a wide grain size area, extending from several micrometers down to the nanometer range, thus ensuring that the results may be tied to the data available for materials with conventional grain sizes, and (3) the evaluation of the results with respect to the underlying mechanisms. Dense composite material is prepared by high energy milling and subsequent hot isostatic pressing. At room temperature, the grain size dependence of hardness and yield strength can be described by the well-known Hall-Petch relationship. Contrary to the behavior of conventional alloys, the ductility of submicron-grained composites drops if the grain size is further reduced. This may be attributed to arising difficulties evolving for deformation mechanisms based on single dislocation glide. In the high temperature range, the flow stress is strongly reduced. Superplastic deformation becomes feasible already at temperatures less than or equal to 800 degreesC, allowing for easy forming of parts. The silicide particles impede grain growth, but they also promote cavitation during tensile straining. The mechanisms of deformation are similar to those established for coarse-grained materials, though at significantly higher temperatures (> 1000 degreesC).

[52] NANOPHASE AND ULTRAFINE-GRAINED POWDERS PREPARED BY MECHANICAL MILLING: FULL DENSITY PROCESSING AND UNUSUAL MECHANICAL BEHAVIOR

E Ma, D Jia, KT Ramesh - POWDER MATERIALS: CURRENT RESEARCH AND INDUSTRIAL PRACTICES, 2001, pp 257-266 - SYMPOSIUM ON POWER MATERIALS: CURRENT RESEARCH AND INDUSTRIAL PRACTICES HELD AT THE 2001 TMS FALL MEETING; INDIANAPOLIS, INDIANA. NOVEMBER 4-8, 2001

Nanograined powders including elemental metals, intermetallics, and composites have been prepared by mechanical milling. Constrained sinter-forging has been used to consolidate the powders to full density while maintaining nanophase grain sizes, with potential for near net-shape manufacturing. The addition of a second phase, especially when alloyed into the matrix uniformly during milling, facilitates the retention of nanostructures in consolidation. The versatile, solid-state, milling/consolidation approach leads to easy preparation of extremely uniform nanophase composites with multiple components having very different melting points and properties. Under uniaxial compression, very high strength and appreciable elongation to failure have been observed, reflecting the high density and nanocrystalline microstructure in these consolidated samples. Under tensile forces, however, the residual processing flaws may trigger premature catastrophic failure. The high-strength material with little capacity for strain hardening and strain rate hardening is susceptible to plastic



instabilities such as necking in tension and shear banding. In this regard, ultrafine-grained microstructure (e.g., submicron grains), with grain sizes still at least a couple of orders of magnitude finer than conventional materials, can offer a balance for the desired combination of strength and ductility and at the same time be more amenable to full density processing. The shear banding mode as the dominant plastic deformation mechanism is discussed with bcc Fe as an example. Such a shear localization behavior resembles the response of amorphous alloys. It is not only of interest to scientific studies of the dependence of deformation mechanisms on grain size but also of practical value in certain high-rate applications such as kinetic energy penetrators.

[51] MECHANICAL ALLOYING OF THE FE-ZR-B-BASED ALLOYS

A Grabias, M Kopcewicz, D Oleszak - PROCEEDINGS OF THE ALL - POLISH SEMINAR ON MOSSBAUER SPECTROSCOPY (Series: MOLECULAR PHYSICS REPORTS(SERIES)), 2000, Vol 30, pp 66-72 - ALL-POLISH SEMINAR ON MOSSBAUER SPECTROSCOPY; RADOM, POLAND. JUNE 12-14, 2000

- and low-energy ball milling techniques are used to investigate phase transformations occurring in the starting elemental Fe₇₀Zr₁₀B₂₀ and Fe₅₀Co₁₀Ni₁₀Zr₁₀B₂₀ powders. A special care for the possibility of obtaining an amorphous iron-containing phase is taken in the study. The structural and magnetic properties of the powders are studied by X-ray diffraction and transmission Mossbauer spectroscopy as a function of milling time. The Mossbauer results show that certain amount of the iron-containing amorphous phase is obtained only in the Fe₇₀Zr₁₀B₂₀ alloy after high-energy ball milling. No significant amorphization was observed for the Fe₅₀Co₁₀Ni₁₀Zr₁₀B₂₀ alloy, which transforms predominantly to the bcc-FeCo phase.

[50] PREPARATION OF ANTIFERROELECTRIC LEAD ZIRCONATE TITANATE STANNATE CERAMICS BY HIGH-ENERGY BALL MILLING PROCESS

Kong LB. Ma J. Zhang TS. Zhu W. Tan OK. - Journal of Materials Science-Materials in Electronics. 13(2):89-94, 2002
Lead zirconate titanate stannate powders doped with lanthanum and niobium, namely [Pb_{0.99}Nb_{0.02}(Zr_{0.85}Sn_{0.13}Ti_{0.02})(0.98)O₃, or PNZST) and (Pb_{0.9}La_{0.02}(Zr_{0.65}Sn_{0.31}Ti_{0.04})O₃, or PLZST], were synthesized by a high-energy ball milling technique from their corresponding oxide mixtures. The milled powders were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and particle size analysis techniques. The sintering behaviors of the milled powders were investigated by a dilatometer from room temperature to 1100 degreesC. PZST ceramics formed from the high-energy ball milled powders were sintered at temperatures from 900 degreesC to 1200 degreesC. The measured electrical properties of the PZST ceramics were comparable to the results reported in the literature. These results have shown that the high-energy ball milling technique is a promising method to prepare PZST ceramics due to its simple procedure

[49] AL-27 MULTIPLE-QUANTUM MAS NMR OF MECHANICALLY TREATED BAYERITE (ALPHA-AL(OH)(3)) AND SILICA MIXTURES

Ashbrook SE. MacKenzie KJD. Wimperis S. - Solid State Nuclear Magnetic Resonance. 20(3-4):87-99, 2001
Two-dimensional Al-27 multiple-quantum magic angle spinning (MQMAS) NMR experiments are used to study mixtures of bayerite (alpha-Al(OH)(3)) with either silicic acid (SiO₂.nH(2)O) or silica gel (SiO₂) that have been ground together for varying lengths of time. This mechanical treatment produces changes in the Al-27 MAS and MQMAS NMR spectra that correspond to the formation of new Al species. Mean values of the quadrupolar interaction (P-Q) and isotropic chemical shift (delta(CS)) are extracted from the two-dimensional Al-27 NMR spectra for each of these species. The presence of significant distributions of both Al-27 quadrupolar and chemical shift parameters is demonstrated and the effect of grinding duration on the magnitudes of these distributions is discussed.

[48] SOLID-PHASE FORMATION OF CALCIUM HYDRIDOALUMINATES Ca(AlH₄)(₂) AND CaHAlH₄ UPON MECHANOCHEMICAL ACTIVATION OR HEATING OF MIXTURES OF CALCIUM HYDRIDE WITH ALUMINUM CHLORIDE

Mal'tseva NN. Golovanova AI. Dymova TN. Aleksandrov DP. - Russian Journal of Inorganic Chemistry. 46(12):1793-1797, 2001

We established that calcium hydride is capable of solid-phase reaction with aluminum chloride upon heating or mechanochemical activation to yield calcium hydridoaluminates Ca(AlH₄)(₂) and CaHAlH₄. Patterns of the thermolysis and mechanolysis of these hydridoaluminates are proposed

[47] MECHANOCHEMICAL SYNTHESIS AND CHARACTERIZATION OF POLY(VINYL CHLORIDE) -BLOCK- POLY(ACRYLONITRILE-CO-BUTADIENE) COPOLYMERS BY ULTRASONIC IRRADIATION

Fujiwara H. - Polymer Bulletin. 47(3-4):247-253, 2001

Mechanical degradation and mechanochemical reaction in heterogeneous and homogeneous systems of poly(vinyl chloride) and poly(acrylonitrile-co-butadiene) polymer have been studied by ultrasonic irradiation at 30 degreesC. The rates of decrease in the number-average molecular weights of the degraded poly(vinyl chloride) and poly(acrylonitrile-co-butadiene) polymer in the swelled poly(vinyl chloride)-poly(acrylonitrile-co-butadiene) polymer solution were much faster than the homogeneous solution system and the final average chain lengths led to the smaller values than those in the latter system. On the other hand, mechanochemical reaction occurred by polymer radicals produced from the chain scissions of both polymers by ultrasonic irradiation. The changes in the composition of the total block copolymer, the unreacted poly(vinyl chloride), and the unreacted poly(acrylonitrile-co-butadiene) polymer in both reaction systems were obtained.

[46] FABRICATION OF A BULK ICOSAHEDRAL MATERIAL THROUGH MECHANICAL ALLOYING OF THE POWDER MIXTURE Ti₄₁.5ZR₄₁.5NI₁₇



Yi S. Kim KB. Fleury E. Kim WT. Kim DH. - Materials Letters. 52(1-2):75-79, 2002

A bulk material that consists of the stable icosahedral phases (I-phases) in the Ti-Zr-Ni system has been fabricated in a hot press using mechanically alloyed (MA) powders. Considerable amounts of amorphous phase and nanocrystals are formed after MA for more than 30 h in the mixture of elemental powders (Ti, Zr, Ni) with the overall composition of $Ti_{41.5}Zr_{41.5}Ni_{17}$. The amorphous phase transforms into the I-phase in the temperature range of 573-673 K during continuous heating in differential scanning calorimetry (DSC) with the heating rate of 40 K/min. The powders MA for 40 h are hot-pressed for 2 h at 873 K under the applied pressure of 900 MPa to form a bulk I-phase material. The evolution of the Vickers microhardness has been examined in the load range of 0.5-10 N. The indentation size effect is accompanied by a modification of the fracture pattern. At a low value of the load, no crack is observed. As the load increases, the first cracks observed around the indenter are lateral cracks, and then radial cracks are found for load larger than 1 N.

[45] PREPARATION OF ANTIFERROELECTRIC LEAD ZIRCONATE TITANATE STANNATE CERAMICS BY HIGH-ENERGY BALL MILLING PROCESS

Kong LB. Ma J. Zhang TS. Zhu W. Tan OK. - Journal of Materials Science-Materials in Electronics. 13(2):89-94, 2002

Lead zirconate titanate stannate powders doped with lanthanum and niobium, namely $[Pb_{0.99}Nb_{0.02}(Zr_{0.85}Sn_{0.13}Ti_{0.02})(0.98)O_{-3}]$, or PNZST and $(Pb_{0.9}La_{0.02}(Zr_{0.65}Sn_{0.31}Ti_{0.04})O_{-3})$, or PLZST], were synthesized by a high-energy ball milling technique from their corresponding oxide mixtures. The milled powders were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and particle size analysis techniques. The sintering behaviors of the milled powders were investigated by a dilatometer from room temperature to 1100 degreesC. PZST ceramics formed from the high-energy ball milled powders were sintered at temperatures from 900 degreesC to 1200 degreesC. The measured electrical properties of the PZST ceramics were comparable to the results reported in the literature. These results have shown that the high-energy ball milling technique is a promising method to prepare PZST ceramics due to its simple procedure.

[44] ELECTRON MICROSCOPY AND HYDRIDING PROPERTIES OF $MgYNi_4$ SYNTHESIZED BY MECHANICAL ALLOYING

Kitano Y. Yamada K. Miyamoto M. Orimo S. Fujii H. Aono K. Tanabe E. - Journal of Alloys & Compounds. 330:292-295, 2002

The ternary alloy, $MgYNi_4$, was synthesized by mechanical alloying (MA). Its microscopic structures and hydriding properties were studied. The annealing of the alloy at 773 K was also studied. The alloy consists of nanocrystals of less than 10 nm in diameter. The capacity of the hydrogen atoms in the alloy increases with the increasing size of the nanocrystals. The crystal structure was found to be the C15 type Laves phase structure for the MA-treated alloy, while crystal structure of the as annealed alloy was the ordered C15 type structure which is conventionally called C15b type and is formally called AuBe5 type structure. The hydrogen capacity in the alloy might have a strong relation to the atomic arrangement of this alloy

[43] COMPOSITES FOR HYDROGEN STORAGE BY MECHANICAL GRINDING OF GRAPHITE CARBON AND MAGNESIUM

Imamura H. Tabata S. Shigetomi N. Takesue Y. Sakata Y. - Journal of Alloys & Compounds. 330:579-583, 2002

Novel hydrogen storage Mg/G nano-composites obtained by mechanical grinding of magnesium (Mg) and graphite carbon (G) with organic additives (benzene, cyclohexane or tetrahydrofuran) have been characterized by X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), differential scanning calorimetry (DSC) and temperature programmed desorption (TPD) techniques. The occurrence of various effects as a result of the formation of Mg/G composites ground with benzene, cyclohexane or tetrahydrofuran (designated hereafter as (Mg/G)(BN), (Mg/G)(CH) or (Mg/G)(THP), respectively) is expected. Upon mechanical grinding with benzene or cyclohexane for 4-40 h, new hydrogen-storing sites, other than those due to the magnesium component, were formed in the Mg/G composites and they took up hydrogen reversibly. The cleavage-degraded graphite in the composites plays an important role in such hydrogen uptake and release. The formation of Mg/G composites upon grinding with the organic additives led to not only a drop in the onset temperature of MgH_2 decomposition, but the formation of additional hydrogen uptake sites. In marked contrast to (Mg/G)(BN) and (Mg/G)(CH), the composites ground without any additives (referred as (Mg/G)(none)) did not show such behavior. The effective nano-composites are those in which there are synergetic interactions between magnesium and graphite as a result of mechanical grinding with the organic additives

[42] SYNTHESIS AND HYDROGENATION BEHAVIOR OF $Mg-Ti-Ni-H$ SYSTEMS BY HYDROGEN-INDUCED MECHANICAL ALLOYING

Hong TW. Kim YJ. - Journal of Alloys & Compounds. 330:584-589, 2002

Mg and Mg alloys are attractive hydrogen storage materials because of their lightweight and high absorption capacity. Their range of applications could be further extended if their hydrogenation properties and degradation behavior could be improved. The main emphasis of this study was to find an economic manufacturing method for Mg-Ti-Ni-H systems, and to investigate their hydrogenation properties. $(Mg_{10-x}Ti_x)_{-10}$, 20 mass% Ni systems were prepared by hydrogen-induced mechanical alloying (HIMA) using Mg and Ni chips and sponge Ti. The particles synthesized were characterized by X-ray diffraction, and their morphologies were observed by means of scanning electron microscopy (SEM) with energy dispersive spectrometry (EDS). The absorbed hydrogen capacity (AHC) was measured by using thermogravimetry analysis (TGA) after FEMA. In addition, the crystal structures were analyzed in terms of their bright-field images and the selected area diffraction pattern (SADP) of transmission electron microscopy (TEM). In order to examine hydrogenation behavior, a Sieverts type automatic pressure-composition-isotherm (PCI) apparatus was used and the experiments were performed at



423, 473, 523, 573, 623 and 673 K. The results of TGA reveal that the absorbed hydrogen contents are around 2.5 mass% for (Mg₉Ti₁)-10 mass% Ni. With increased Ni content, the absorbed hydrogen content decreases to 1.7 mass%, whereas the dehydrogenating starting temperatures are lowered by some 70-100 K. The results of PCI on (Mg₉Ti₁)-20 mass% Ni show that its hydrogen capacity is around 5.3 mass% and its reversible capacity and plateau pressure are also excellent at 523 and 573 K. In addition, the reaction enthalpy, $\Delta H(D, \text{plateau})$, is -30.6 ± 5.7 kJ/mol H₂

[41] HYDROGEN ABSORPTION AND ELECTROCHEMICAL PROPERTIES OF MG₂-XNI (X=0-0.5) ALLOYS PREPARED BY BULK MECHANICAL ALLOYING

Kuji T. Nakano H. Aizawa T. - Journal of Alloys & Compounds. 330:590-596, 2002

Thermodynamic properties of hydrides of Mg₂-xNi alloys produced by bulk mechanical alloying were determined from the pressure-composition isotherms for absorption over the temperatures from 623 to 423 K. The van't Hoff plots for the plateau pressures of the isotherms clearly indicated the existence of high- and low-temperature hydrides with different entropy and enthalpy for hydride formation. The phase transition temperature was 525 K for Mg_{2.0}Ni and decreased with increasing value of x. Chemical and electrochemical behaviors of Mg₂-xNi alloys in an alkaline solution were precisely determined. It was found that hydrogen absorption in Mg₂-xNi alloys takes place by only immersing the alloys in 6 M KOH solution. On the other hand, during electrical charging hydrogen content increased with the quantity of charged electricity and reached the maximum value. However, the hydrogen content decreased afterwards because the corrosion of the alloys in the alkaline solution is significant

[40] CHARGE-DISCHARGE CHARACTERISTICS OF TiV_{2.1}Ni_{0.3} ALLOY SURFACE-MODIFIED BY BALL-MILLING WITH Ni OR RANEY Ni

Inoue H. Miyauchi R. Shin-ya R. Choi WK. Iwakura C. - Journal of Alloys & Compounds. 330:597-600, 2002

For the purpose of improving the charge-discharge characteristics of the TiV_{2.1}Ni_{0.3} alloy, it was surface-modified by ball-milling with Ni and Raney Ni which worked as a catalyst for hydriding and dehydriding. The surface modification of the TiV_{2.1}Ni_{0.3} alloy with Ni decreased the maximum discharge capacity, but the cycle durability was greatly improved. On the other hand, the surface modification of the TiV_{2.1}Ni_{0.3} alloy with Raney Ni improved both the discharge capacity and cycle durability. The surface modification of the TiV_{2.1}Ni_{0.3} alloy with Ni and Raney Ni suppressed the positive shift of the plateau potential in the discharge curve and the negative shift of the rest potential. In particular, Ni was more effective than Raney Ni. The high-rate dischargeability was improved by surface modification with Raney Ni.

[39] HYDROGEN ABSORPTION AND DESORPTION IN MECHANICALLY ALLOYED TITANIUM-CHROMIUM COMPOSITES

Fernandez JF. Sanchez CR. - Journal of Alloys & Compounds. 330:601-606, 2002

Ti and Cr composites with different compositions were prepared by mechanical alloying. Morphological, structural and compositional characterisation of the composites was accomplished by scanning electron microscopy, energy dispersive X-ray analysis and X-ray diffraction. The composites showed improved H₂ absorption and desorption kinetics compared to Ti. Improved kinetics is thought to be related to the formation, at the surface of the composite, of a Ti and Cr intermetallic compound with good activation properties

[38] EFFECTS OF SURFACE AND BULK MODIFICATIONS ON ELECTROCHEMICAL AND PHYSICOCHEMICAL CHARACTERISTICS OF MGNI ALLOYS

Iwakura C. Inoue H. Nohara S. Shin-ya R. Kurosaka S. Miyahara K. - Journal of Alloys & Compounds. 330:636-639, 2002

Effects of partial substitution of Mg in MgNi with both Ti and V and subsequent surface modification by ball-milling with graphite on electrochemical and physicochemical characteristics of amorphous MgNi alloys were investigated. It was found from thermogravimetry (TG) that hydrogen desorbability of the MgNi alloys was improved by either partial substitution with both Ti and V or surface modification with graphite, and that the combination of the partial substitution and the subsequent surface modification enhanced the hydrogen desorbability further. In charge-discharge cycle tests, the partial substitution with Ti and V or surface modification with graphite suppressed the decay of discharge capacity with increasing cycle number. Mg_{0.9}Ti_{0.06}V_{0.04}Ni alloy modified with graphite exhibited further improved cycle performance as compared with either unmodified Mg_{0.9}Ti_{0.06}V_{0.04}Ni alloy or MgNi alloy modified with graphite. These results indicate that modification of both bulk and surface of the MgNi alloys is very effective in improving hydrogen desorbability and charge-discharge cycle performance of the alloys

[37] CHARACTERISTICS OF MG₂Ni_{0.75}M_{0.25} (M=Ti, Cr, Mn, Fe, Co, Ni, Cu AND Zn) ALLOYS AFTER SURFACE TREATMENT

Yang HB. Yuan HT. Ji JT. Sun H. Zhou ZX. Zhang YS. - Journal of Alloys & Compounds. 330:640-644, 2002

The ternary Mg₂Ni_{0.75}M_{0.25} (M=Ti, Cr, Mn, Fe, Co, Ni, Cu and Zn) alloys have been successfully synthesized by the ball milling diffusion method (BDM). XRD results show that they all have a hexagonal crystal structure. We show that the alloys after fluorination treatment show good performance: the hydrogen desorption capacity reaches the highest value after only two adsorption-desorption cycles. The ratio of H/M is about 1.18 (approximate to 3.3 wt.%) on desorption at 250°C. The hydrides have monoclinic phases. The larger the unit volume, the more unstable is the hydride. The dissociation enthalpies and temperatures of the hydrides in an open system are decreased with increasing unit cell volume. Replacement of Ni in Mg₂Ni by Cr, Mn and Co has the same effect on Mg₂Ni: they lower the decomposition plateau pressure; Ti and Cu have the opposite effect and Fe and Zn have little effect

[36] DYNAMIC IN SITU X-RAY DIFFRACTION OF CATALYZED ALANATES



Gross KJ. Sandrock G. Thomas GJ. - Journal of Alloys & Compounds. 330:691-695, 2002

The discovery that hydrogen can be reversibly absorbed and desorbed from NaAlH₄ by the addition of catalysts has created an entirely new prospect for lightweight hydrogen storage. NaAlH₄ releases hydrogen through the following set of decomposition reactions: $\text{NaAlH}_4 \rightarrow (1/3)(\alpha\text{-Na}_3\text{AlH}_6) + (2/3)\text{Al} + \text{H}_2 \rightarrow \text{NaH} + \text{Al} + (3/2)\text{H}_2$ These decomposition reactions as well as the reverse recombination reactions were directly observed using time-resolved in situ X-ray powder diffraction. These measurements were performed under conditions similar to those found in PEM fuel cell operations (hydrogen absorption, 50-70°C, 10-15 bar H₂; hydrogen desorption, 80-110°C, 5-100 mbar H₂). Catalyst doping was found to dramatically improve kinetics under these conditions. In this study, the alanate was doped with a catalyst by dry ball-milling NaAlH₄ with 2 mol.% solid TiCl₃ X-ray diffraction clearly showed that TiCl₃ reacts with NaAlH₄ to form NaCl during the doping process. Partial desorption of NaAlH₄ was even observed to occur during the catalyst doping process.

[35] ENGINEERING CONSIDERATIONS IN THE USE OF CATALYZED SODIUM ALANATES FOR HYDROGEN STORAGE

Sandrock G. Gross K. Thomas G. Jensen C. Meeker D. Takara S.- Journal of Alloys & Compounds. 330:696-701, 2002

The hydrogen storage properties of catalyzed NaAlH₄ (and associated Na₃AlH₆) were studied in relation to various practical engineering considerations. Properties measured were cyclic capacity, charging and discharging rates, thermal effects, gaseous impurities, volume changes, low temperature plateau pressures and detailed isothermal desorption kinetics over the temperature range 23-180°C. Two materials were evaluated, one mechanically milled with the liquid alkoxides Ti(OBu)₄ and Zr(OPri)₄ and one milled with dry TiCl₃, as catalyst precursors. The alkoxide-catalyzed materials had low reversible capacities and released significant levels of hydrocarbon impurities during H₂ discharge. These problems were virtually eliminated with the inorganic TiCl₃ catalyst precursor. The NaAlH₄ and Na₃AlH₆ decomposition kinetics of TiCl₃-catalyzed Na-alanate conform to Arrhenius behavior with activation energies of 79.5 and 97 kJ/mol H₂, respectively. Measured absorption and desorption kinetics were surprisingly good and it is shown that 3-4.5 wt.% H₂ can be stored and recovered in reasonable times at 100-125°C. It may even be ultimately possible to use the NaAlH₄ decomposition reaction to provide 3 wt.% H₂ at room temperature for low-rate applications.

[34] HYDRIDING KINETICS OF NANO-PHASE COMPOSITE HYDROGEN STORAGE ALLOYS PREPARED BY MECHANICAL ALLOYING OF MG AND MMNi(5-X)(COAlMn)(X)

Zhu M. Gao Y. Che XZ. Yang YQ. Chung C. - Journal of Alloys & Compounds. 330:708-713, 2002

Nano-phase composite hydrogen storage alloys were prepared by mechanical alloying of Mg and MmNi(5-x)(CoAlMn)(x). The hydrogen absorption kinetics of the nano-phase composite and melted MmNi(5-x)(CoAlMn)(x) alloys were measured under isobaric conditions at different temperatures. The obtained hydrogen absorption kinetic curves were fitted using various rate equations to reveal the mechanism of the hydriding reaction process. The results showed that the hydriding reaction kinetics of the nano-phase composite and melted MmNi(5-x)(CoAlMn)(x) were different. For the nano-phase composite, the hydriding reaction was in agreement with an auto-catalysis process. For the melted MmNi(5-x)(CoAlMn)(x), the hydride reaction was in agreement with a nucleation and growth process. The difference in hydriding kinetics of the two alloys is discussed based on the fitting results and the microstructural characteristics of the alloys

[33] HYDROGEN DESORPTION KINETICS OF NANOSTRUCTURED MGH₂ COMPOSITE MATERIALS

von Zeppelin F. Reule H. Hirscher M. - Journal of Alloys & Compounds. 330:723-726, 2002

In order to investigate the microscopic mechanism of hydrogen desorption we have studied ball-milled magnesium-composite materials with a relatively large volume fraction of additives. After ball milling the microstructure typically shows additive particles covered with a film of MgH₂ as investigated by scanning electron microscopy and energy-dispersive X-ray microanalysis. Depending on the milling time and material used as additive, the desorption of hydrogen takes place at a far lower temperature than in pure MgH₂ as studied by thermal desorption spectroscopy. In the next step we developed a new method to use only small volume fractions of additives in order to minimize the amount of additive material and maximize the percentage of hydrogen storage material. Therefore, MgH₂ was sputter-deposited by palladium and successively ball milled to achieve a composite material with finely dispersed metallic additives. The desorption kinetics could be strongly improved even though only small amounts of palladium were added. The new method of ultra-fine dispersion may lead to MgH₂ composite materials with a high storage capacity of hydrogen accessible at moderate temperatures.

[32] EFFECT OF REACTIVE MECHANICAL GRINDING ON CHEMICAL AND HYDROGEN SORPTION PROPERTIES OF THE MG+10 WT.% CO MIXTURE

Bobet JL. Chevalier B. Darriet B. - Journal of Alloys & Compounds. 330:738-742, 2002

Reactive mechanical grinding (MG under H₂) of magnesium powder improves the hydrogen sorption properties. The hydrogenation of Mg starts in situ during the milling process that allows suppressing the activation procedure generally requested for Mg. The addition of Co, which acts as a catalyst for the dissociation of H₂, also leads to an improvement of the hydrogen sorption properties (but a strong dependence upon the milling time is reported). The hydriding is determined to be a two-step process: nucleation and diffusion. A direct relationship exists between the nucleation duration and the specific surface. A critical milling time exists below which the diffusion process is improved and above which no more improvement is observed (the maximum internal stress in the powder is also reached at this critical time). The diffusion is controlled by the number of crystallites per particle which can be decreased by increasing the milling time up to 10 h. However, the sorption properties of Mg-Co mixtures are a little under those reported for MgH₂-metal mixtures



[31] EFFECT OF MECHANICAL GRINDING UNDER AR AND H-2 ATMOSPHERES ON STRUCTURAL AND HYDRIDING PROPERTIES IN LANI5

Fujii H. Munehiro S. Fujii K. Orimo S. - Journal of Alloys & Compounds. 330:747-751, 2002

The effects of mechanical grinding (MG) under argon and hydrogen gas atmospheres on the hydrogen storage properties of a LaNi₅ alloy were studied in detail. During MG under Ar atmosphere, a crystallite size reaches a similar to 20 nm in grinding time of 60 min and reduces to approximately half this size after 600 min without any dissociation. The pressure-composition isotherm (P-C) in LaNi₅ at 293 K indicates an increase in hydrogen in zero offset region (trapping site region), a lowering of plateau pressure and a narrowing of the width of the pressure plateau by MG. On the other hand, in reactive MG (RMG)-LaNi₅ under H₂ atmosphere, a nanocrystalline LaNi₅H_{0.15} and an amorphous phase coexist when the grinding time is less than 180 min. For much longer RMG times than 180 min, the nanostructured LaNi₅H_{0.15} phase disappears and the remaining amorphous phase dissociates into nanocrystalline Ni+amorphous LaNi₅-H_z (y<5). The P-C isotherm indicates no plateau for the LaNi₅ produced by RMG longer than 60 min and the hydriding properties become worse and worse with increasing RMG times. From the above results, we conclude that the hydriding properties cannot be improved by structural modifications in systems containing metals with a strong affinity for hydrogen like rare earth metals

[30] A STUDY ON THE ELECTRODE CHARACTERISTICS OF Zr-BASED ALLOY SURFACE-MODIFIED WITH Ti-BASED ALLOY BY BALL-MILLING PROCESS AS AN ANODE MATERIAL FOR Ni-MH RECHARGEABLE BATTERIES

Lee SM. Kim SH. Lee JY. - Journal of Alloys & Compounds. 330:796-801, 2002

In order to improve the kinetic properties of the Zr-based hydrogen storage alloy electrode, the ball-milling process is applied to the Zr-based alloy using the Ti-based alloy powder as a surface modifier. While the Zr-based alloy electrode is not fully activated before 50 cycles, the ball-milled Zr-based alloy electrode using Ti-based alloy as a surface modifier is fully activated within only four cycles. In order to analyze the strikingly improved kinetic characteristics after ball-milling, the microstructure of ball-milled alloy is examined by transmission (TEM), scanning electron microscopy (SEM), and energy dispersive spectroscopy (EDS). It is observed that there is a surface-alloying region at the contact points between the two alloy powders from the TEM bright-field image. Furthermore, the local quantitative analysis by EDS clearly reveals that the atomic concentration of the constituting elements in the surface-alloying region is gradually changed between the two alloy powders. From the above results, it is suggested that the high kinetic energy applied in the ball-milling process causes cold-welding or surface alloying at the points of impact where Zr-based alloy particles collide with Ti-based alloy particles by the action of steel balls at high speed. The SEM analysis demonstrates that the particle size is decreased as the ball-milling time increases, which implies an increase in the surface area of Zr-based alloy particles touching Ti-based alloy particles. Eventually, it can be suggested that Ti-alloy powder serves as a window for hydrogen to penetrate into the Zr-based alloy, which leads to easy absorption/desorption of hydrogen and also to improvement in the kinetic properties of the Zr-based alloy electrode at initial cycles

[29] DEGRADATION OF AMORPHOUS MgNi ELECTRODE AND EFFECT OF HEAT TREATMENT IN AR

Hatano Y. Tachikawa T. Mu DB. Abe T. Watanabe K. Morozumi S. - Journal of Alloys & Compounds. 330:816-820, 2002

The mechanism underlying degradation of an amorphous MgNi electrode was studied. Amorphous MgNi powder was prepared by mechanical alloying, and a charge/discharge cycle test was carried out in 6 M KOH solution. The amount of hydrogen absorbed in the charge process was determined by vacuum hydrogen extraction. The discharge capacity decreased rapidly with progress of the cycle test. The results of the hydrogen extraction indicated that almost all hydrogen absorbed in the charge process was released in the discharge process. X-ray diffraction analyses showed that Mg(OH)₂ was formed on the surfaces of MgNi particles after the cycle test. It was deduced that the reduction in the discharge capacity was mainly due to the degradation in absorption capability in the charge process caused by retardation of electron transfer by Mg(OH)₂ layer. The cyclic stability of the discharge capacity was improved by heat treatment in Ar at 573 K.

[28] CHARACTERISTICS OF SURFACE-MODIFIED METAL HYDRIDE ELECTRODE WITH FLAKE Ni BY THE BALL-MILLING PROCESS

Lee SM. Yu JS. Lee PS. Lee JY. - Journal of Alloys & Compounds. 330:835-840, 2002

In order to improve the electrochemical cyclic durability of the Ti-based hydrogen storage alloy electrode, the ball-milling process was applied to the Ti-based alloy using flake Ni as a surface modifier. Flake Ni powder is a very useful material to modify the surface of metal hydrides. While the as-cast Ti-based alloy electrode is completely degraded within ten cycles, the ball-milled Ti-based alloy electrode using flake Ni as a surface modifier shows only 8% of capacity decay even after 180 cycles. It is also noticeable that the ball-milled alloy electrode shows just a little decreased discharge capacity (429 mAh/g) by ball-milling with flake Ni powder. These results are closely related to the higher strain and higher coverage area of flake Ni powder. The residual strain of flake Ni powder can promote the diffusion reaction in the impact event of ball-milling. Consequently, the flake Ni as a new surface modifier can modify the alloy surface without changing the bulk properties of the alloy, resulting in an effective surface coating within a shorter ball-milling time.

[27] IMPROVEMENT OF ELECTRODE PERFORMANCES OF Mg₂Ni BY MECHANICAL ALLOYING

Han SS. Lee HY. Goo NH. Jeong WT. Lee KS. - Journal of Alloys & Compounds. 330:841-845, 2002

Nanocrystalline and amorphous Mg₂Ni-based hydrogen storage alloys for Ni-MH batteries had been synthesized by mechanical alloying. The surface modification and Zr addition had also been carried out for improvement of its electrode performance. In comparison with the arc-melted polycrystalline one, the nanocrystalline Mg₂Ni phase showed a higher discharge capacity. By increasing milling time from 120 to 160 h, the grain size of Mg₂Ni phase was more refined and the



discharge capacity was raised from 180 to 370 mAh g⁻¹). The discharge capacity of the 160-h milled Mg-Ni-Zr amorphous alloys also reached 530 mAh g⁻¹). XPS (X-ray photoelectron spectroscopy) analysis showed that Mg2p spectra were shifted to lower binding energy implying the enhancement of the hydrogen diffusion and charge transfer reaction, and resulted in increasing the discharge capacity in amorphous alloys. To prevent the rapid degradation, the alloy powders were also coated with Ni and graphite by additional ball milling. The Ni and graphite protected the Mg from oxidation, and the coated powders showed a better cyclic stability. After 50 cycles, the degradation of bare electrode was 94% of maximum capacity, but that of coated electrode with Ni and graphite was 45 and 76% of maximum capacity, respectively

[26] RADICALS IN THE MECHANOCHEMICAL DECHLORINATION OF HAZARDOUS ORGANOCHLORINE COMPOUNDS USING CAO NANOPARTICLES

Ikoma T. Zhang QW. Saito F. Akiyama K. Tero-Kubota S. Kato T. - Bulletin of the Chemical Society of Japan. 74(12):2303-2309, 2001

For the first time, we detected paramagnetic products generated during the grinding of 3-chlorobiphenyl (BP-Cl) with calcium oxide (CaO) nanoparticles by a ball mill method, which is one of the promising ways to detoxify hazardous chlorinated organic compounds. Those products were assigned to oxygen-centered aromatic radicals coming from BP-Cl and trapped electrons in oxygen vacancies on the surfaces of the CaO reactants using high-frequency and pulsed electron paramagnetic resonance spectroscopies. The observed good correlation between the dechlorination efficiency and the radical yield suggests that a radical mechanism plays an important role in the destruction of organochlorine compounds. The mechanochemical dechlorination could be interpreted by the following mechanism. First of all, the mechanical stressing induces the electron transfer from O²⁻ sites on the surface of the CaO particle to the organic compounds. The produced organic anion radicals then undergo the effective self-dissociation of a chlorine-carbon bond

[25] HYDROGEN IN MECHANICALLY PREPARED NANOSTRUCTURED h-BN: A CRITICAL COMPARISON WITH THAT IN NANOSTRUCTURED GRAPHITE

Source Applied Physics Letters. 80(2):318-320, 2002

Nanostructured h-BN was prepared by mechanical milling under hydrogen atmosphere. The hydrogen concentration reaches up to 2.6 mass% after milling for 80 h, and this value corresponds to ca. 35% of that of nanostructured graphite as was previously reported. In addition to the hydrogen desorption starting at about 570 K, nitrogen desorption was also detected at about 700 K. There was no recrystallization phenomenon at least below 1173 K. The dissimilarities on the (de-)hydriding properties between nanostructured h-BN and graphite might be due to the different local electronic structure near the specific defects

[24] DEVELOPMENT OF THE ELABORATION PROCESS AND JOINING TECHNOLOGIES OF RAFM ODS STEELS

Revol S. Launois S. Baccino R. Le Marois G. Rigal B. - Fusion Engineering & Design. 58-9:761-765, 2001

The development of the production process and the joining technologies of oxide dispersion strengthened reduced activation ferritic martensitic (RAFM ODS) steels aims at raising the operating temperature of fusion reactors from 550 up to 650 or even 700 degreesC. Our laboratory is involved in the development of the entire Eurofer ODS steel production route, from the atomisation of the base alloy powder to the consolidation of the reinforced material powder and the joining of consolidated samples through mechanical alloying. For this, the atomised powder is milled with a small amount of yttrium oxide powder, and then consolidated by hot isostatic pressing (HIP). This technique is the principal route for manufacturing large net-shape or near net-shape parts for structural applications, as well as for joining these parts by diffusion welding. Samples of Eurofer ODS steels with different mechanical alloying parameters have been produced and some characterizations of the microstructure have been done. Some preliminary results were obtained by TEM investigation regarding the different particles present after consolidation.

[23] DISTRIBUTION OF CATIONS IN MAGNETITE PREPARED BY MECHANOCHEMICAL SYNTHESIS

Novikov SI. Lebedeva EM. Scholtz AK. Yurchenko LI. Tsurin VA. Barinov VA. - Physics of the Solid State. 44(1):124-132, 2002.

The distribution of iron cations in the crystal lattice of the Fe_{3-v}O₄ (v = 0.153) cation-deficient spinel produced by mechanical dispersion of alpha-Fe₂O₃ hematite in water is investigated using x-ray diffraction and Mossbauer spectroscopy. Analysis of the Mossbauer data shows that the Fe_{2.847}O₄ magnetite prepared by mechanochemical synthesis is a chemically heterogeneous compound. The crystal structure of Fe_{2.847}O₄ is characterized by local environments of the (Fe_{2.5+})(0) cations at v(0) less than or equal to 0.1, v(1) congruent to 0.12, v(2) congruent to 0.18, and v(3) congruent to 0.26, which are responsible for a broad distribution of magnetic hyperfine fields with the P(H) probability maxima near 37.0, 36.0, 34.0, and 30.0 MA m(-1).

[22] MAGNETIC PROPERTIES OF NANOCRYSTALLINE FE: ROLE OF THE DISORDERED GRAIN BOUNDARY

Del Bianco L. - Physics of Metals & Metallography (English Translation of Fizika Metallov i Metallovedenie). 91(Suppl 1):S93-S99, 2001.

Ball-milled Fe samples with grain sizes in the 8-25 nm range have been analyzed by a combination of Mossbauer spectroscopy, X-ray diffraction, and magnetization measurements. The nanocrystalline material exhibits a two-component Mossbauer spectrum due to the presence of the crystallites and of the grain boundary region. In particular, the grain boundaries give rise to a distribution of hyperfine magnetic fields typical of an amorphous-like configuration. The thermal stability of the interfaces has been investigated. In high-energy ball-milled Fe, thermally induced rearrangement of the grain boundaries may result in the formation of a new phase with a hyperfine field of 21 T and a magnetic order-disorder transition temperature of about 500 K. This phase was found to possess a fcc crystal structure. The disorder at the grain boundaries is



proposed to determine the unusual low-temperature magnetic behavior of nanocrystalline Fe. The temperature, dependence in different magnetic fields of the field-cooled and zero-field-cooled magnetization in the 5-250 K temperature range is consistent with a transition between two different dynamic regimes occurring at a temperature of about 70 K: the high-temperature regime has a ferromagnetic character, whereas the low-temperature regime is a disordered frozen magnetic state which is erased by applying an external field $H = 1000$ Oe, favoring the ferromagnetic order. The effect has been interpreted in terms of the temperature variation of the efficiency of interfaces as an exchange interaction transmitter.

[21] THE EFFECT OF COMPOSITION AND MILLING CONDITIONS ON THE STRUCTURE OF MECHANICALLY ALLOYED MG-AL BASED ALLOYS

Hazelton LE. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 32(12):3099-3108, 2001
Mg-Al based alloys were mechanically alloyed under varying conditions. Elemental reaction times correlated with known diffusion coefficients and elemental hardness, but milling temperature had almost no effect over a 200 degreesC range. Increasing impact energy caused the steady-state level of crystallinity to increase. Alloys with up to 6 at. pct of Ti, Y, Ca, Zr, V, Er, or Pr yielded amorphous alloys near the composition Mg₄₀Al₆₀. Certain phases were suppressed by mechanical alloying, while others became more dominant than in the equilibrium phase diagram. These effects are explained by differential scanning calorimetry (DSC) results, which indicate they are growth rate controlled during mechanical alloying. Hard elements such as Cr and Mo with positive free energy of mixing did not react completely even after relatively long milling times

[20] GRAIN GROWTH OF NANOCRYSTALLINE NI POWDERS PREPARED BY CRYOMILLING

Lee J. Zhou F. Chung KH. Kim NJ. Laverina EJ. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 32(12):3109-3115, 2001

Grain growth of nanocrystalline Ni powders with an average grain size of similar to 22 nm prepared by cryogenic mechanical milling (or cryomilling) was investigated by using X-ray diffraction (XRD) and transmission electron microscopy (TEM). A dispersion of NiO and Ni₃N particles with a size less than 5 nm was formed in the cryomilled powders. The Ni₃N particles decomposed at 773 K. It was found that at 0.56 homologous temperature (T/T-M), Ni grains were retained at similar to 150 nm even after long annealing times (e.g., 4 hours). For 0.45 to 0.62 T/T-M, the time exponent n deduced from $D-1/n - D_0(1/n) = kt$ was 0.16 to 0.32, tending toward 0.5 as T/T-M increased. The activation energy for grain growth in the Ni sample was determined to be 113 kJ/mol, which is close to the activation energy for grain boundary self-diffusion in polycrystalline Ni. The observed high grain size stability was attributed primarily to a grain boundary pinning mechanism arising from the NiO particles as well as impurity segregation

[19] EFFECT OF TRANSITION METALS ON THERMAL STABILITY OF AMORPHOUS PHASES IN AL(7)0FE(2)5TM(5) (TM=CU, NI, ZR, TI) COMPOSITE POWDERS PREPARED BY MECHANICAL ALLOYING

ZZou Y. Saji S. Kusabiraki K - Journal of Materials Science Letters. 20(22):2039-2041, 2001.

[18] STRUCTURE OF MECHANOCHEMICAL REACTION PRODUCTS IN THE SYSTEMS PBO-SB2O5 AND PBO-SB2O3

Zyryanov VV. - Inorganic Materials. 37(12):1278-1284, 2001

Mechanochemical reactions in 2PbO + Sb₂O₅ and 2PbO + Sb₂O₃ oxide mixtures treated in a high-energy planetary mill were studied by x-ray diffraction. The reactions were found to lead to rapid formation of crystalline products with the pyrochlore and fluorite structures, in line with the reaction-zone model. The temperature ranges of the structural and chemical transformations involved were established, and the compositions of the resulting phases were accurately determined. Heating in air leads to the reduction of the sample containing the pyrochlore phase, with the formation of one crystalline product having a variable lattice parameter because of the chemical inhomogeneity of the material. Under the same conditions, the sample containing the fluorite phase oxidizes in air, yielding four phases with different Ph : Sb ratios: PbSb₂O₆, pyrochlore, a new compound Pb_{1/3}Sb_{2/3}O₂ with the fluorite structure, and Pb₃Sb₂O₈. The Pb: Sb ratio is found to vary over a wide range, from 0.5 to 1.5. The morphology of the powders produced by mechanical processing at above-threshold rates is considered in terms of nonequilibrium thermodynamics: Since the powder is a dissipative structure, its inhomogeneity cannot be fully eliminated, which correlates with the formation of an intermediate, dynamic state during mechanochemical reactions

[17] PREPARATION OF NANOCRYSTALLINE HIGH-NITROGEN STAINLESS STEEL POWDERS BY MECHANICAL ALLOYING AND THEIR HOT COMPACTION

Miura H. Ogawa H. - Materials Transactions. 42(11 Special Issue SI):2368-2373, 2001

Mechanical alloying (MA) was performed, with Fe-N alloy powders as nitrogen source. on stainless steels of Fe-Cr-Ni-N (Cr-Ni type) and Fe-Cr-Mn-Mo-N (Cr-Mn type) systems in an Ar atmosphere using a planetary ball mill. In the MA processing, high nitrogen nanocrystalline stainless steel powders with 0.45-0.90 mass%N were readily manufactured after about 504-700 ks of processing. Nitrogen highly enhanced austenitizing of the powder products. The onset temperature (Md) of strain-induced martensite formation in nitrogen-free 19Cr-11Ni stainless steel MA powders was estimated to be about 100 K or more higher than those obtained under conventional plastic deformation processes. Compaction of the MA powders at 1173 K using a spark-plasma sintering (SPS) process still retained their nanostructure. When 5 vol% of dispersion particle agents AlN or NbN were added to the MA powders, grain growth in the compacts was greatly suppressed during SPS processing. On SPS processing, nitrogen in the Cr-Mn type system was completely retained but about 10-15 mass% of nitrogen in the Cr-Ni type powder samples was observed to escape, depending on the composition. However, when Mn, Nb or Ti was added to the samples or Cr content was increased, the nitrogen loss was greatly decreased. Furthermore, by simultaneous addition of these elements, such nitrogen loss was almost completely prevented. The nitrogen retention



depending upon the chemical composition during SPS processing and the marked raising of Md temperature due to the MA processing are thermodynamically explained

[16] ESTIMATION OF EXTRACTION RATE OF YTTRIUM FROM FLUORESCENT POWDER BY BALL MILLING

Mio H. Lee JY. Nakagawa T. Kano J. Saito F. - Materials Transactions. 42(11 Special Issue SI):2460-2464, 2001

Red fluorescent powder containing yttrium (Y) which is one of the rare earth elements (REEs), was milled in air using a small-scale planetary ball mill to investigate the relation between the extraction rate of Y and the impact energy of the balls calculated from computer simulation based on the Discrete Element Method (DEM) under various conditions. Milling improves the extraction yield, and extraction rate increases with an increase in mill rotational speed, whereas the rate is independent of ball diameter. The same trend is observed in the relation between the specific normal impact energy of the balls and rotational speed. The relation between the extraction rate and the specific normal impact energy can be expressed as a straight line, irrespective of the milling conditions, and it is applicable to estimation of the extraction rate using a large-scale planetary ball mill. Therefore, the extraction rate of Y would be estimated by the specific normal impact energy of the balls calculated from the computer simulation

[15] MECHANICAL PROPERTIES OF ULTRA FINE GRAINED STEELS

Takaki S. Kawasaki K. Kimura Y. - Journal of Materials Processing Technology. 117(3 Special Issue SI):359-363, 2001

Mechanical milling (MM) of iron powder is one of the useful techniques to achieve ultra grain refining to nanosize. For instance, MM treatment using high energy ball-mill makes the grain refining to 20-30 nm possible and such an ultra grain refining results in marked hardening of iron powder to around Hv 9.5 GPa. On the annealing of the powder with MM treatment (MM powder), very fine oxide particles (Fe₃O₄) play an important role to keep the grain size fine through the effect of grain boundary pinning. Thus, MM powder with sufficient initial hardness can be successfully consolidated at around 950 K with keeping the grain size below 1 μm. Tensile strength of consolidated bulk iron increases with grain refining and Hall-Petch relationship was realized to about 0.2 Min as to 0.2% proof stress ($\sigma(0.2)$, (MPa) = 100 + 0.6d(-1/2) d (in)). In the test pieces with the grain size less than 1 μm, uniform elongation completely disappears due to the marked strengthening. On the hardness, Hall-Petch relationship was also confirmed to around 0.1 Min at least (Hv (MPa) = 330 + 2.0d(-1/2)). In the grain size range below 0.1 μm, however, experimental data tended to deviate toward lower side from the Hall-Petch line and hardening was seemed to level off at around Hv 12 GPa. Ductile brittle transition temperature was lowered with grain refining (DBTT (K) = 330 - 0.33d(-1/2)) but upper shelf energy becomes smaller when the grain size was refined below 5 μm

[14] PREPARATION OF ULTRAFINE V2O5-TiO2 COMPOSITE OXIDE CATALYST BY HIGH-ENERGY BALL MILLING METHOD [CHINESE]

Lin M. Fan YN. Liu L. Xu BL. Hu Z. Chen Y. - Chinese Journal of Catalysis. 22(6):585-588, 2001

The ultrafine V₂O₅-TiO₂ composite oxide particles have been prepared by the high-energy ball milling method and characterized by X-ray diffraction, transmission electron microscopy, laser Raman spectroscopy, temperature-programmed reduction, and microreactor testing. It has been shown that the milling process induces the formation of ultrafine V₂O₅-TiO₂ composite oxide particles with dispersed vanadium oxide on the surface of anatase TiO₂, accompanied by a decrease in particle size of V₂O₅ and TiO₂. The TPR results indicate that the strong interaction between dispersed V-O species and TiO₂ increases the reducibility of the vanadium oxide. The catalytic properties of the catalysts for the selective oxidation of o-xylene were evaluated. Under the similar o-xylene conversion (58%), the ultrafine V₂O₅-TiO₂ composite oxide catalyst exhibits a higher selectivity for phthalic anhydride (44%) than the catalyst prepared by the conventional impregnation method (23%), and the catalyst composition has a great influence on the catalytic properties

[13] MICROSTRUCTURE AND MECHANICAL PROPERTIES OF MECHANICALLY SYNTHESIZED NiAl/HfB2 COMPOSITE [CHINESE]

Yang FB. Guo JT. Zhou JY. - ACTA METALLURGICA SINICA. 37(5):483-487, 2001

Mixed elemental powders of Ni, Al, Hf and B were ballmilled to reaction synthesize NiAl/HfB₂ composite. The formation mechanism can be attributed to self propagating reaction induced by mechanical collision. The microstructure and compressive properties of densified bulk prepared by hot pressing and isostatically pressing have been investigated. The results show that the fine HfB₂ reinforcing particles formed by in situ mode distributed mainly in matrix. The yield strength of NiAl/HfB₂ composite is much stronger than that of NiAl, furthermore, it also possess good compressive ductility at low temperature. The yield strength of NiAl/HfB₂ composite at high temperature is dependent on strain rate. The stress exponent n as well as activity energy Q calculated by standard power law are much higher than NiAl and similar to NiAl composite with a higher volume fraction of TiB₂ dispersoids.

[12] REACTION OF TI WITH BN DURING HIGH ENERGY BALL MILLING PROCESS [CHINESE]

Li JL. Hu K. Zhou Y. - ACTA METALLURGICA SINICA. 37(5):547-550, 2001

Reaction mechanism of Ti with BN during high energy ball milling has been studied. The microstructure development of the powder mixtures was monitored by X-ray diffraction, XPS, scanning electron microscopy and transmission electron microscopy. Nanosized spherical particles with an average size of 100 nm are formed after 10 h of milling, which was attributed to that Ti particles were wrapped by BN slices. After 30 h ball milling BN and elemental Ti powder mixture at ambient temperature TiN formed. After 40 h milling, the sphere-like nanosized composite particles are consisting of TiN particles, amorphous B, the residual BN and unreacted Ti

[11] INFLUENCE OF OXYGEN ON MECHANICAL ALLOYING OF A MO-SI SYSTEM [CHINESE]

Liu L. - ACTA METALLURGICA SINICA. 37(9):1001-1004, 2001



In this paper, the influence of oxygen contamination on mechanical alloying of Mo-Si powder mixture (MO33Si67) has been investigated by X-ray diffraction and transmission electron microscopy. It is found that a complete Mo/Si reaction with the formation of both low temperature phase alpha -MoSi₂ and high temperature phase beta -MoSi₂ can take place if oxygen is kept under control. However, if the milling is proceeding in an atmosphere containing oxygen, oxygen pick-up can strongly inhibit the Mo/Si solid state reaction, and even cause the destabilization of the already formed intermetallic compounds. The influence of oxygen contamination has been discussed from both thermodynamic and kinetic aspects

[10] HIGH-ENERGY MECHANICAL ALLOYING OF THERMOPLASTIC POLYMERS IN CARBON DIOXIDE

Cavaliere F. Padella F. Bourbonneux S. - Polymer. 43(4):1155-1161, 2002

High-energy ball milling was performed on low density polyethylene (LDPE) and isotactic polypropylene (iPP) as well as on 20/80 binary mixture of both polymers. Mechanical alloying was carried out at high pressure with carbon dioxide for a short period. The presence of CO₂ avoids oxidative mechano-chemical degradation of polymers and enhances the effectiveness of the milling. The effects of the mechanochemical treatment on the molecular and physical properties of both single polymers and blends of intrinsically incompatible polymers were explored by FTIR spectroscopy, thermal analysis, intrinsic viscosity determination and solvent fractionation. Structural changes on PP and PP/LDPE blend were observed and have a strong dependence on the milling time. Mechanical tests confirm an overall improvement in blend properties by mechanical alloying. Experimental evidences are presented to suggest that CO₂ high-energy ball milling causes a self-compatibilization of the blend LDPE-iPP by breaking iPP polymer chains and allowing them to recombine with the neighboring LDPE chains

[9] MECHANICAL PROPERTIES OF CU+0.5 WT % AL₂O₃ NANOCOMPOSITE MATERIAL PRODUCED BY SEVERE PLASTIC DEFORMATION

Amirkhanov NM. Islamgaliev RK. Valiev RZ. - Physics of Metals & Metallography (English Translation of Fizika Metallov i Metallovedenie). 92(5):518-525, 2001

Severe plastic deformation (SPD) by torsion was used to produce nanostructured specimens of Cu + 0.5 wt % Al₂O₃ metal/ceramic composite material. The electron-microscopic and X-ray diffraction methods were employed to determine the average sizes of matrix grains, inclusions, and the root-mean-square values of microdistortions. It was shown that the nanocomposite specimens had both a high room-temperature ultimate strength (to 690 MPa) and a high thermal stability. A relative elongation (to 70%) and a considerable strain-rate sensitivity of the flow stress upon tension of the specimens at elevated temperatures were found. Using the differential scanning calorimetry (DSC), two exothermic processes were observed in the temperature ranges of similar to 200-450 degreesC and similar to 420-600 degreesC. The activation energies for these processes and also that for the plastic deformation of the nanocomposite were determined.

[8] ELECTROCHEMICAL AND KINETIC CHARACTERIZATION

Ong TS. Yang H. - Journal of the Electrochemical Society. 149(1):A1-A8, 2002

The electrochemical lithium intercalation into natural graphite milled in various atmospheres was investigated. The atmosphere in which the natural graphite was milled has a strong influence on its final morphology and microstructure. These physical properties in turn have a pronounced effect on the electrochemical lithium intercalation process. The lithium storage capacity increases with the amount of disorder present, but it is also associated with a more pronounced discharge and charge potential hysteresis. Although a milled sample with a larger specific surface area is more susceptible to first cycle irreversible reactions, it has a smaller charge-transfer resistance. The chemical diffusion coefficient of intercalated lithium decreases when a large amount of disorder has been introduced into the sample by the mechanical milling process.

[7] ENHANCED ELECTROCHEMICAL PROPERTIES OF BALL-MILLED Mg₂Ni ELECTRODES

Niu H. Northwood DO. - International Journal of Hydrogen Energy. 27(1):69-77, 2002

The characteristics and mechanisms of enhanced electrochemical properties for a Mg₂Ni alloy produced by a ball-milling treatment were investigated by electrochemical measurements and XRD analysis. Their particle size decreases, the internal strain increases, and the alloy gradually becomes amorphous on ball-milling. With the accompanying increased diffusion rate of hydrogen into the ball-milled Mg₂Ni alloy, the limiting current density, *i*(*l*), is decreased and the exchange current density, *i*(0), is increased. As a result, the specific discharge capacity is significantly increased compared to that of the as-received Mg₂Ni alloy and reaches a maximum for a ball-milling time of 25 h

[6] EFFECTS OF QUARTZ ADDITION ON THE MECHANOCHEMICAL DECHLORINATION OF CHLOROBIPHENYL BY USING CAO

Zhang QW. Saito F. Ikoma T. Tero-Kubota S. - Environmental Science & Technology. 35(24):4933-4935, 2001

Grinding a mixture of 3-chlorobiphenyl (BP-Cl) and CaO with or without the addition of quartz was conducted in air by a planetary ball mill to investigate the mechanochemical dechlorination of BP-Cl. The dechlorinating reaction proceeds with an increase in grinding time, and over 99% of BP-Cl is decomposed at 360 min. Washing the ground sample with different solvents results in different products. Addition of quartz to the grinding mixture facilitates dechlorination efficiency, especially in the case of a high weight ratio of BP-Cl to CaO.

[5] BALL MILLING INDUCED MICROSTRUCTURE EVOLUTION OF AlN [CHINESE]

Yang ZQ. He LL. Jin ZX. Ye HQ. - ACTA METALLURGICA SINICA. 37(3):230-234, 2001

AlN particles are drastically refined by ball milling, from an original particle size of 40 μm to the range of 30-200 nm. High-resolution electron microscopy (HREM) observations demonstrate that the grain size refinement is governed by plastic deformation via creation, motion and rearrangement of dislocations. During the processing of grain size refinement, crystalline lattice distortion may occur, which results in the disappearance of intrinsic character for AlN

[4] MAGNETIC PROPERTIES OF ZN-SN-SUBSTITUTED BA-FERRITE POWDERS PREPARED BY BALL MILLING



Mendoza-Suarez G. Johal KK. Mancha-Molinar H. Escalante-Garcia JI. Cisneros-Guerrero MM. - Materials Research Bulletin. 36(15):2597-2603, 2001

The present investigation reports the preparation of $BaFe_{12-2x}Zn_xSn_xO_{19}$ ferrite powders via ball milling and subsequent heat treatment. Starting powders were milled during 50 and 100 It, and then heat treated at 950 and 1000 degreesC to induce crystallization and reaction of the starting materials. XRD results showed that it is possible to obtain single-phase barium ferrite powders if processing conditions are carefully chosen. The powders were also characterized for magnetic properties, namely saturation magnetization and intrinsic coercivity. It was found that coercivity could easily be controlled by varying the dopants content, while maintaining a relatively high magnetisation

[3] MECHANOCHEMICAL PREPARATION AND PROPERTIES OF A CELLULOSE-POLYETHYLENE COMPOSITE

Zhang FR. Qiu WL. Yang LQ. Endo T. Hirotsu T. - Journal of Materials Chemistry. 12(1):24-26, 2002.

Ball milling of crystalline cellulose with maleated polyethylene (MPE) yields a novel composite with ester bonds formed by reaction of the hydroxy groups of the cellulose with the maleic anhydride groups of MPE; the composite exhibits much improved toughness and ductility compared with the product formed by melt- mixing, probably because of the formation of an interphase of MPE chains bonded on cellulose particles

[2] THE EFFECT OF QUARTZ CONTENT ON THE MECHANOCHEMICAL ACTIVATION OF KAOLINITE

Mako E. Frost RL. Kristof J. Horvath E. - Journal of Colloid & Interface Science. 244(2):359-364, 2001

The mechanochemical activation (dry grinding) causes destruction in the crystal structure of kaolinite by the rupture of the O-H, Al-OH, Al-O-Si, and Si-O bonds. The major mineral constituents of natural kaolins are kaolinite and quartz. In this study, the attention was mainly directed to the role of quartz content (4, 25, 50, and 75 wt%) in the mechanochemical amorphization of kaolinite. Grinding experiments were carried out for 1, 2, 3, and 4 h in a planetary mill. The rate of destruction of the kaolinite structure was followed by X-ray diffraction, thermal analysis, and Fourier transform infrared (DRIFT) spectrometry. The distortion and rupture of the kaolinite structure induced by grinding was reflected in line broadening, increases in mean lattice strain, and reduction of peak areas (intensities). The increased quartz content resulted in acceleration of the mechanically induced amorphization of the kaolinite structure. The crystalline order of kaolinite was completely destroyed after grinding the sample containing 75 wt% quartz for 4 h. On the other hand, 4 h of grinding was sufficient only to cause some increase in the defect density of kaolinite in the case of samples with lower quartz contents (25 and 4 wt%). The results indicate that quartz grains act as grinding bodies during the intensive dry grinding of kaolinite.

[1] PROCESS OF AMORPHIZATION INDUCED BY MECHANICAL ALLOYING OF IRON WITH TUNGSTEN AND NIOBIUM

Jartych E. Oleszak D. Zurawicz JK. - Acta Physica Polonica A. 100(5):731-736, 2001

Mechanical alloying method was used to synthesise powders of iron with tungsten and niobium. Mossbauer spectroscopy and X-ray diffraction have been applied to monitor the progress in solid-state reactions. In the case of Fe-W system, exhibiting a positive heat of mixing, no trace of amorphization was observed for 20 and 33 at.% of W, as the calculations of phase diagram (CALPHAD) method suggest. During the mechanical alloying process, two solid solutions Fe(W) and W(Fe) were obtained. Mossbauer measurements allowed to recognise the Fe(W) solid solution as a ferromagnetic phase, while the W(Fe) solid solution as a paramagnetic one. In the case of Fe-Nb system, exhibiting a negative heat of mixing, single phase amorphous alloys were synthesised during mechanical alloying of iron with 48 and 64 at.% of Nb. For both investigated compositions, the final products of mechanical alloying processes were amorphous paramagnetic alloys



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the “pulverisette 5” carries the CE mark for proven safety. (for example: the mill cannot be opened during operation, nor started up with the lid in the open position).

no disturbing contamination because grinding bowls and balls are available in 9 different materials

**reproducible grinding and full recovery in sealed grinding bowls
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membrane keypad with digital display of operational parameters, including rotational**

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Integral fan located in the grinding chamber for additional cooling permits longer grinding process

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special lids with valves available for milling in an inert atmosphere suitable for mechanical alloying

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