



**RESEAU FRANÇAIS DE
MECANOSYNTHESE**

Lettre N°77

Août 2001

**185 Groupes de Recherche
(dont 111 à l'étranger / 33 Pays)**

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

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1 Nouvelle Adhésion

M.-N. Avettand – Fenoel – Lab. Métallurgie Physique & Génie Matériaux – Villeneuve d'Ascq - France

Full Congress Announcements
Congress List (related to Nanomaterials)
Proposal for Cooperation, PhD, new Post Doc proposal, International Relationships)
Books (related to Nanomaterials)
Bibliography

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.

Bulletin d'adhésion 2001 / Subscription Print

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

Eric GAFFET
CNRS UMR5060 « Métallurgies et Cultures »
Thème « Nanomatériaux : Elaboration et Transitions de Phases Hors Equilibre »
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 100FF

The check has to be to the order : Reseau Francais de Mecanosynthese

Lettre RFM N°77 - Août 2001
Corresp. : <mailto:Eric.Gaffet@utbm.fr>

(Please do not use Eurocheck, the taxes do correspond to 40% of the amount of the check).



FRITSCH: Your specialist in the processing laboratory (Press Release)

Contact : Andrea Köhler

FRITSCH GMBH - Laborgerätebau-Industriestrasse 8 - D-55743 Idar-Oberstein – Germany

Tel.: 06784/70-0 - Fax: 06784/70-11 - E-Mail: info@fritsch.de - Internet: <http://www.fritsch.de/>

“pulverisette 4” - Vario Planetary Mill

Fritsch presents the Vario-Planetary Mill “pulverisette 4” on the following exhibition:

22.10. - 24.10. Powder Metallurgy Congress & Exhibition - Nice, FRANCE

Main advantages:

1. In ball mills grinding is performed by friction and/or impact:
 - **high impact energy is released when balls impact vertically onto the interior grinding bowl wall**
 - **high friction is released when the balls approach the interior grinding bowl wall tangentially**
 - **in a centrifugal mill the grinding is performed by rolling the grinding ball along the interior grinding bowl wall**

The proportions of friction and impact in the case of traditional ball mills depends on fixed, pre-set transmission ratio.

In contrast to traditional planetary mills, the innovative Vario Planetary Mill “pulverisette 4” allows the rotational speed of grinding bowls and supporting disc to be adjusted completely independent of one another. By varying the transmission ratio it is possible to control the movements and trajectories of the grinding balls at will so that the balls strike the inner wall of the bowl vertically (high impact energy), approach each other tangentially (high friction) or just roll down the inner wall of the bowl (centrifugal mills). All intermediate levels and combinations of frictional and impact pressures can be set as required.

Thus extremely different mill types can be simulated and the kind of loads they exhibit can be precisely reproduced. Grinding processes can be reproduced and the parameters can be optimally adapted to the sample material.

2. **With 2 powerful motors (4 kW and 1.5 kW) extremely high rpm's (unloaded up to 1000 rpm) can be set, so that very high energy input is possible, permitting a considerable reduction in grinding time**
3. **RS232 interface for programming the grinding parameters using PC software (included in the package), for outputting process data (validation), as well as for controlling the “pulverisette 4”**
4. **Real time display of the rotational speed for monitoring, reproduction or optimising the grinding process**
5. **reversing option (direction of rotation reversed periodically) to improve the grinding results**
6. **simultaneous grinding in up to 4 small (80 ml grinding bowls stackable) or 2 large 500 ml-grinding bowls**
7. **final fineness $\ll 1 \mu\text{m}$, making it visibly finer than that of traditional planetary mills**
8. **“safe-lock” bowl tightening is standard**
9. **use of gas pressure and temperature measuring system (GTM)**
10. **safety standard EN 61010 tested by the German Technical Inspection Agency and CE-mark**

Remarks:

The free selection of the rotational speed of grinding bowls and supporting disc make it possible for the first time to perform mechanical activation as well as mechanical alloying with a single mill by changing the transmission ratio. The main applications are in the field of materials research and, of course, wherever a powerful, innovative planetary mill is required.

Now a gas pressure and temperature measuring system (GTM) is available for measuring the gas pressure and temperature during the grinding process. This system was developed in co-operation with the Fraunhofer Institute for Applied Material Research in Dresden, and enables process values to be measured during the grinding process. A radio transmitter located in the lid of the grinding bowl sends data from the grinding bowl to a receiver, which is connected to a computer. The corresponding WINDOWS™ programme presents the results in the form of tables and graphics.



**Prière de bien vouloir noter la Programmation 2002 et 2003
des Prochaines Journées Annuelles du Réseau Français de Mécanosynthèse**

200
2

Les JRFM'2002 seront intégrées
dans le cadre du Congrès

Matériaux 2002

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

Symposium 1 :
**Poudres et Matériaux Nanostructurés,
du fondamental aux applications industrielles**

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

E_mail : materiaux@materiaux2002.net

200
3

Les **JRFM'2003**

auront lieu à Albi (Mai/Juin 2003)
(Ecole des Mines d'Albi – Contact : M. Baron)

Thème :

Matériaux Avancés, Micro, Nanotechnologie et Mécanosynthèse

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Congress and School Announcements
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International Conference on the Applications of the Mossbauer Effect
Oxford, UK
2-7 September 2001

Abstracts are now invited for for the above meeting, which is the next in the ICAME conference series. You are asked to submit your abstract via the conference website <http://www.iop.org/IOP/Confs/ICAME/> by no later than 1 April 2001.

For further information or enquiries please contact the Conference Office at the Institute of Physics, 76 Portland Place, London W1B 1NT, UK.

E-mail should be directed to: <mailto:rebecca.chapple@iop.org>

International Conference
"FUNDAMENTAL BASES of MECHANOCHEMICAL TECHNOLOGIES"
Novosibirsk, Russia, August 16-18, 2001

Contact: Prof. N. Lyakhov
Institute of Solid State Chemistry
E-mail: <mailto:Conf@solid.nsc.ru>
Fax: , +7 3832 32 28 47

The first circular is available on WEB-Site of the Institute:
<http://www.solid.nsc.ru/>

(IPCM 2001)

La 7eme conference internationale sur les phenomenes d'interface dans les materiaux composites (IPCM 2001) se tiendra au palais des congres d'Arcachon (40 km de Bordeaux) du 11 au 14 septembre 2001.



Lettre RFM N°77 - Août 2001
Corresp. : <mailto:Eric.Gaffet@utbm.fr>

<http://www.arcachoncongres.com/ipcm2001/>



(IWSIS-3)

October, 7-12, 2001.

3rd International Workshop on Surface and Interface Segregation, Island of Porquerolles, French Riviera,
This Workshop is devoted to the study of the segregation phenomenon
in defects of crystallized solids (surface, grain boundary, interface of
interphase...)

INFOS, : <http://www.crmc2.univ-mrs.fr/conf/iwsis>

"VI International Symposium on Self-Propagating High-Temperature Synthesis, (SHS-2001)"

Haifa, Israel . October 14-18, 2001.

More information on the

Web site: <http://www.technion.ac.il/technion/materials>

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>

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>

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



SOUTENANCES DE THESE

Nathalie Bouad

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

Potentialité de la mécanosynthèse d'alliages à base de tellurure 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>

COREE du SUD (19/01/2000)

From, Professor Soon H. Hong
Dept. of Materials Science and Engineering - Korea Advanced Institute of Science and Technology

373-1 Kusung-dong, Yusung-gu - Taejon, 305-701, Korea

E-mail : <mailto:shhong@sorak.kaist.ac.kr> / <mailto:shhong@sorak.kaist.ac.kr>

Fax. : 82-42-869-3310 - Tel. : 82-42-869-3327

We are currently working on the mechanical alloying processes and the characterization of mechanical & thermal properties of nanocrystalline materials and composite materials, such as SiC/Al, WC/Co and W/Cu for structural or thermal management applications. We are very pleased to discuss for international cooperative research on related topics with Members of Mechanosynthesis Group.



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

**Post Doc Position
Dijon/ France (10/07/2001) – From F. Bernard**

The research group (fine grain materials) from the Research Lab UMR 5613 (Laboratoire de Recherche sur la Réactivité des Solides) is seeking for a post doctoral associate with experience in X – ray Diffraction (experimental and numerical approaches, computer simulation, Monte Carlo ...).

The candidate (he or she) has to demonstrate the ability to work independently, contribute to innovative numerical approach, and develop new projects in this area.

The work will be performed in collaboration between three french labs (Dijon – F. Bernard, Belfort – E. Gaffet, Vitry - Y. Champion).

Scientific Field : In spite of a lot a research effort, the mechanism of phase formation during MA is not well understood. It is most often proposed that the process of MA introduces a variety of defects (vacancies, dislocations, grain boundaries, stacking fault,...) which raise the free energy of the system making it possible to produce metastable phases. But there are very few investigations that deal with the characterization and quantification of the defects produced in mechanically alloyed powders. As a primary investigation, the effect of the mechanical activation mode (i.e. the friction or direct shock ones, at least the component ratio of both components) can be assumed on analysing the microstructure of post-mortem milled powders. XRD is really a valuable technique for a characterisation in terms of size and morphology of crystallites and imperfections (microstrains, dislocation, stacking faults,...). Indeed, the ball milling of metals or alloys induces extended variations in the intensity distribution of XRD diagrams and, in particular, in the line profile. Knowledge of the stacking fault density and the twin-fault density is essential to understand the nanomaterials behaviour.

A new line profile analysis method is proposed by Ustinov et al. [123], in order to take into account the dependence of the crystallite size, of the residual strains as well as of the planar defects, on the line profile broadening that may be observed on ball-milled materials. Such a method will allow to understand the influence of ball-milling parameters and for controlling the synthesis of nanostructured materials

Financial Support : Regional Financial Support from Burgundy Region in France

Interested candidates should send **correspondence** to:

BERNARD Frédéric - Université de Bourgogne - UFR Sciences et Techniques
9, Avenue Alain Savary - Laboratoire de Recherches sur la Réactivité des Solides,
UMR 5613 CNRS / Université de Bourgogne - Equipe "Matériaux à grains Fins"
B.P. 47870 - 21078 DIJON CEDEX

fax : 33.3.80.39.61.67 - e-mail : fbernard@u-bourgogne.fr

Please note that this proposal is not available for french students.

**Singapour (11/06/2001) – From Professor L. Lu
Post-doctoral fellow position**

The position requires candidates with an Ph.D. degree materials science. He/she should have a demonstrated track record on synthesis of metallic amorphous materials. Preference will be given to candidates with relevant experience of mechanical alloying.

The application form can be downloaded from the website:

<http://www.nus.edu.sg/NUSinfo/Appoint/RESAPPT.HTML>

Please submit your application to
Associate Professor L. Lu
Dept. of Mechanical Engineering
National University of Singapore
10 Kent Ridge Crescent
Singapore 119260
E-mail: mailto:mpeluli@nus.edu.sg

Brazil (4 / 6 / 2001) – Post Doc Position from Professor Gerardo F. Goya

The Magnetic Materials Group at São Paulo University is seeking a postdoctoral associate with experience in powder synthesis and magnetism to work on nanostructured ceramics. The candidate should demonstrate the ability to work independently, contribute to innovative experimental design, and develop new projects in this area. Background in at least three of the following areas is desirable: Mechanochemical synthesis. Mechanical alloying. Magnetism in nanostructured systems. Transport measurements. Mossbauer Spectroscopy Scanning/Transmission Electron Microscopy

The candidate should send a curriculum vitae, three representative publications (preferably with the candidate as a first author) and the names, addresses, email and phone numbers of two references that can comment on the candidate's capabilities. Position is open for applicants within three years of receipt of Ph.D. The postdoctoral contract will be one+one year, with salary US\$ ~15000 /y.

Applicants should send the information before 15-August-2001.



Interested candidates should send **correspondence** to:

Professor Gerardo F. Goya
Laboratório de Materiais Magnéticos
Instituto de Física - Universidade de Sao Paulo
CP 66318 Sao Paulo
05315-970 SP Brazil
e-mail: goya@macbeth.if.usp.br
Fax: (55) 11 3818 6984
Desk: (55) 11 3818 6885

France (28/05/2001) – from V. Nivoix

Proposition de sujet de thèse du

Laboratoire d'Analyse Spectroscopique et de Traitement de Surface des Matériaux,
UPRES EA 1290, Université de Rouen

Relation structure-propriétés d'oxydes mixtes nanométriques élaborés par différentes voies de synthèse

Les ferrites mixtes manganèse-zinc utilisés dans les composants électroniques sont des ferrites doux toujours très utilisés de nos jours, dont les performances peuvent encore être améliorées.

Les propriétés magnétiques, largement conditionnées par la répartition cationique dans les sites de la phase spinelle, dépendent également de la microstructure et plus particulièrement de la porosité et de la taille moyenne des grains dans le matériau final.

Le procédé industriel actuel par voie céramique ne permet pas d'obtenir une microstructure dense à grains fins pourtant très favorable. De nouvelles voies de synthèse sont actuellement explorées, notamment dans le domaine de la "chimie douce".

Nous nous proposons d'élaborer ces oxydes sous forme de poudres nanométriques par broyage à haute énergie et par voie hydrothermale puis de comparer leurs caractéristiques physiques et structurales.

La synthèse par broyage à haute énergie se fera à l'aide d'un broyeur planétaire nouvelle génération (P4 de Fritsch) permettant d'optimiser le broyage par une meilleure maîtrise des paramètres techniques.

Pour mener à bien la caractérisation complète de ces matériaux nous disposerons de différentes techniques telles que la diffraction des rayons X, la spectrométrie IRTF et la spectrométrie Mössbauer pour la caractérisation structurale, la microscopie électronique à balayage haute résolution ou à transmission et la DRX pour la taille et la morphologie, un SQUID pour les propriétés magnétiques. D'autres méthodes d'analyse pourront être mises en œuvre selon les besoins de l'étude. Le candidat ou la candidate devra avoir des connaissances en chimie des solutions aqueuses et sur la caractérisation des matériaux (diffraction des rayons X, spectrométrie IR, spectrométrie Mössbauer, mesure de magnétisme ...)

Financement : Nous ferons une demande de financement auprès du ministère de la recherche ou de la région Haute-Normandie.

Contacts : Virginie NIVOIX ou Malick JEAN - Université de Rouen - LASTSM-IUT
76821 Mont Saint Aignan Cedex
tel 02 35 14 63 59 fax 02 35 14 63 58
email : malick.jean@univ-rouen.fr

SPAIN (03/04/2001)- From J.J. Suñol (joan josep.sunyol@udg.es)

Postdoctoral researchers required Universitat de Girona

Soft magnetic materials obtained by mechanical alloying and rapid solidification: thermal and structural characterization. Analysis of nanocrystallization process.

The research position will involve aspects of: materials processing by mechanical alloying, thermal and structural characterization by DSC, TG., XRD, SEM, TEM, TMS; kinetic modeling.

The position will begin with effect from september 2001 to september 2002.

Interested candidates should send correspondence to: Dr. J.J. Suñol.

Department of Physics., EPS (P II). Girona University. E-17071. Girona, Spain. Fax: 34-972418098.

E-mail: <mailto:joanjosep.sunyol@udg.es>

(From Paul J. Warren – 6/03/2001)

A Research Training Network on nanostructured Aluminium alloys is urgently looking for researchers. There are opportunities for Post-doctoral and Pre-doctoral researchers available immediately in Oxford(UK), Grenoble(F), Turin(I), Stockholm(S), Madrid(E), Waterford(IRL), Ioannina(G), Warsaw(PL), and Bratislava(SK). Please pass this information to anyone who may be interested.



Lettre RFM N°77 - Août 2001
Corresp. : <mailto:Eric.Gaffet@utbm.fr>

For further details please visit the website <http://www.materials.ox.ac.uk/nano-al>.

Post Doctoral Fellow Positions - University of Toronto (5/03/2001)

The University of Toronto is seeking three postdoctoral fellows for a major new initiative in laser nanofabrication, material diagnostics, and nano-optics fabrication. State-of-the-art laser-processing facilities and optical material fabrication and diagnostic infrastructure are funded from various government and industrial sources. The research is centered in the Photonics Group of the Department of Electrical and Computer Engineering, and directed by Professor Peter R. Herman.

1 - F2-Laser Nanofabrication Facility The post doctoral fellow will drive the development of precise optical tools and nanofabrication processes in one of the world's forefront facilities for F2-laser nanofabrication research. The record short-wavelength light of 157 nm drives strong interactions in challenging materials at sub-micron feature sizes that are attractive for widespread application in fabricating photonic components, optical circuits, lab-on-a-chip systems, and wireless electronic circuits. Responsibilities include co-supervision of graduate students, coordination of research activities with scientists from academia and industry, and co-management of the novel facility. The \$1 million program also includes establishing state-of-the-art optical communication diagnostics. The research goals are new micromachining and photosensitivity processes for fabricating and/or tuning optical circuits, 3-D photonic devices, photonic bandgap structures, and binary optics. These studies are to be integrated with related program in ultrafast laser processing.

2 - Photonic-Bandgap Integrated Optical Circuits An outstanding candidate is sought for a collaborative project to integrate photonic bandgap structures into functional optical circuits. Three-dimensional diffractive optical structures offer in theory, powerful capabilities in managing light in optical circuits. The goal is to practically harness this capability within the structure of our existing photonics fabrication program (the F2-laser nanofabrication facility and ultrafast-laser processing laboratory) and through collaboration with a leading photonic-bandgap group at the University of Toronto. The project is suited to a technically strong and creative individual motivated to revolutionize the future manufacturing of photonic circuits.

3 - Laser-Induced Breakdown Spectroscopy of Aluminum A laser-spectroscopy specialist is required to drive an industrially sponsored research program in laser-induced breakdown spectroscopy of recycled aluminum. The goal is to develop novel laser and diagnostic technology for collecting accurate assays of aluminum metal for a future large-scale pilot project in automobile recycling by Alcan International. One project is the study of a new laser interaction - invented at the University of Toronto - that entails high-repetition 'bursts' of ultrafast laser pulses. This approach promises to cleanly remove surface oxides and precisely probe the underlying bulk aluminum within a single burst. Research centers on fundamental laser interactions and defining laser processing windows in cooperation with our industrial partner.

The research positions entail extensive academic collaboration within the Engineering Faculty, the Department of Physics, the Department of Chemistry, and Photonics Research Ontario (www.pro.on.ca) and with other academic research centers: Laser Laboratorium, Goettingen, Germany; National Research Council, Canada; and Optical Fibre Technology Centre, Australia. Research also includes close interaction with world-leading photonics and manufacturing companies in Canada (i.e. JDS Uniphase, Mitel, Raytheon Elcan Optical Technology, Alcan International) and internationally (i.e. Photonics Integrated Research, Lambda Physik, MicroLas). Our principle goals are forefront science and engineering research for public dissemination in high-quality journals and the generation of intellectual property. Successful candidates will lead one of the following three research areas.

Required qualifications for all three positions include a Ph.D. in experimental Physics, Engineering Science, or Electrical Engineering, and experience with several of the following areas: F2 or excimer lasers, ultrafast lasers, optical and opto-mechanical design, photonic devices for optical communications, optical waveguide fabrication and modelling, optical communication diagnostics, photonic bandgaps, laser-matter interaction physics, spectroscopy, and material diagnostics (SEM, FTIR, AFM, SEM, EDX, ESR). An independent and highly motivated person with good technical and communication skills is required. Each position entails a supervisory role with graduate students and other researchers. The successful candidate will also be responsible for coordination and administration of research involving visiting scientists and industrial partners in the local booming Photonics industry.

The postdoctoral positions are available immediately and remain open until filled. Provide a CV, relevant publications, three references, and recent university transcripts by mail, electronically, or by fax:

Professor Peter R. Herman

10 King's College Rd. Tel: 416-978-7722 - Dept. of Electrical and Computer Engineering Fax: 416-971-3020

University of Toronto, Toronto, ON hermanp@ecf.utoronto.ca - M5S-3G4, CANADA

The University of Toronto is Canada's top university, located in the center of Canada's largest and most dynamic city.

Toronto is home to a large and diverse immigrant population and has low-crime rates. See more at:

<http://www.utoronto.ca/toronto.htm>

Further Employment Information: http://www.ecf.utoronto.ca/~hermanp/job_available.htm

From B. Mhohamed – UK – (10/01/2001)

Marie Curie Training Fellowships

Applications are invited for 3-12 month research fellowships supported by the Marie Curie Training Sites scheme. The successful candidates will be involved with the processing of alloys, intermetallics, nanostructures, or composites for high-temperature, biomedical and/or energy-storage applications. Processing techniques and facilities include ball milling, mechanical alloying, reaction synthesis, tape casting, slurry powder metallurgy, and vacuum cold/hot pressing. Materials characterisation will be carried out by TG/DTA, DSC, MS, optical microscopy, X-Ray, and SEM/TEM techniques. Complementary modelling activities for materials-design, processing, microstructural evolution, and/or property predictions may also be involved as part of the fellowship training programme. Modelling methodologies range from *ab initio* atomistic simulations to finite-element methods. The candidates must satisfy the basic criteria of the training scheme as outlined under <http://www.cordis.lu/improving>. As the fellowship forms part of a higher degree project, the candidates should be a registered full-time PhD research student in a well-recognised institution, working on materials synthesis, characterisation,



and/or computer modelling of materials, of an EU nationality (non-UK) and under 35 years of age. Deadline for application: 30 February 2001.

For further details, please contact: Professor Z. Xiao Guo, phone: 0044-20-7882-5569; e-mail: x.guo@qmw.ac.uk; or visiting: <http://www.metallicmaterials.com/>.

QMW / University of London is an equal opportunity employer.

From A.R. Yavari - France (8/01/2001)

EU Postdoc/Ph.D. positions in fields of Nanostructured Materials and Bulk

Metallic Glasses are available immediately in France and several other EU

States. Please check the following web page

<http://www.inpg.fr/BMG-RTN/>

and contact the Coordinator A.R. Yavari at <mailto:euronano@ltpcm.inpg.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/ssp>

(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 Riecan sky Publisher



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

MACROMOLECULAR MECHANOCHEMISTRY

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

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

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

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

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

Send your preliminary order to <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 Besteri -

<http://www.demon.co.uk/cambsci/book51.htm>



Lettre RFM N°77 - Août 2001
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.

L'adresse du site web où trouver le texte complet du brevet...

<http://164.195.100.11/netacgi/nph-Parser?Sect1=PTO2&Sect2=HITOFF&p=1&u=/netahtml/search-bool.html&r=1&f=G&l=50&co1=AND&d=ft00&s1=gaffet.INZZ.&OS=IN/gaffet&RS=IN/gaffet>

ou encore pour la version brevet d'application

<http://12.espacenet.com/dips/bnsviewer?CY=ep&LG=en&DB=EPD&PN=WO0112366&ID=WO+++0112366A1I+>

Périodiques

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

[90] CHARACTERIZATION OF NIMESULIDE/BETA-CYCLODEXTRIN COMPOSITE OBTAINED BY SOLID STATE ACTIVATION

L Magarotto, S Bertini, C Cosentino, G Torri - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 643-648 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

The mechano-chemical activation was used to obtain a new composite of a anti-inflammatory drug (Nimesulide) and a polysaccharide carrier (beta -cyclodextrin). The original industrial process for activation, patented by Eurand Int. S.p.A., permits an improvement of the physico-chemical properties of the drug, which reaches a higher thermodynamic state (activation state). This work wants to demonstrate that the interaction between the drug and the carrier as a composite causes the thermodynamic activation of the Nimesulide as a new physical compound.

[89] TUNGSTEN CARBIDE IRON ALUMINIDE HARDMETALS: NANOCRYSTALLINE VS MICROCRYSTALLINE

A Mosbah, D Wexler, A Calka - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 649-654 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

In this work an attempt has been made to replace the soft cobalt binder phase in WC hardmetals by harder and tougher Fe60Al40. Both WC hard phase and FeAl binder was made nanostructural by controlled ball milling of the particular microcrystalline starting material. Consolidation properties of microcrystalline powders with blended precursor powders of the same composition with both the WC and FeAl in nanocrystalline form were studied. Densification was achieved using uniaxial hot pressing. Powder blends containing 80 wt.% WC and 20 wt.% Fe60Al40 were liquid phase sintered to near full density using either nano-sized or micron-sized precursor powders. Consolidated material from blended nanostructural precursor powders showed significantly refined microstructures and improved properties. Pressure was found to be essential in densifying WC-FeAl composites when nano-sized binder was used. For ultrafine and nanostructural precursor WC powders pressure assisted sintering at 1450 degreesC resulted in segregation within the binder and the formation of an additional Al-rich phase. The reasons for this are unclear. Hardness values of the materials with nano precursor powders were higher than those of both conventional WC/FeAl and conventional WC-Co hardmetals. Results indicate that blended iron aluminide and nanostructural WC shows great potential for hot pressing into hardmetals with particular advantages over Co-WC. Characterisation methods used in this work included XRD, SEM, thermal analysis and bulk density measurements.

[88] SYNTHESIS OF FEAL-TIN NANOCOMPOSITE BY MECHANICAL ALLOYING OF AL-FE-TI POWDER MIXTURE UNDER NITROGEN ATMOSPHERE

M Krasnowski, H Matyja - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 433-438 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Mechanical alloying process has attracted much attention as a method to synthesize alloys through not only a solid-solid reaction, but also a solid-gas reaction. In the present work, the mechanical alloying process was used for synthesizing a FeAl-TiN nanocomposite. A powder mixture of Al-Fe-Ti (equal proportions) was milled in a high energy ball mill under a nitrogen atmosphere. The phase evolution occurring in the powder during the milling was monitored by the X-ray diffraction method. Transmission electron microscopy was used for examining the microstructure of the milling product. The results obtained show that the process leads first to amorphisation of the material and then to transformation of the amorphous



phase into disordered FeAl phase and TiN nitride, both nanocrystalline. In order to check the thermal stability of the product phases, the milled powder was subjected to heat treatment and then examined by X-ray diffraction (XRD) and transmission electron microscopy (TEM). No phase changes, except an ordering of the FeAl phase, were found in the XRD pattern of the powder after the heat treatment. It was observed that for the heat treated powder, all the peaks in the XRD pattern were sharper than those obtained for the untreated powder. This effect is due certainly to a stress release and some crystallite growth. The latter was confirmed by TEM observations.

[87] REACTIVE MILLING UNDER HYDROGEN OF A ND-FE-B POWDER

G KHELIFATI, JM LEBRETON, L AYMARD, F MACHIZAUD, J TEILLET - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 439-444 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Mechanical millings of a Nd-Fe-B powder under reactive hydrogen and inert argon atmospheres were performed. The influence of milling atmosphere and hydrogen pressure on milling products was investigated by X-ray diffraction (XRD) and transmission Mossbauer spectrometry (TMS). Milling under argon leads to a mixing of alpha -Fe, Nd-rich regions and amorphous Fe-B. Upon milling under hydrogen, Nd reacts with hydrogen to form NdH_{2+x}. Alpha -Fe and amorphous Fe-B. The dissociation of the elements is more important during HEBM under hydrogen and increases with hydrogen pressure. The differential scanning calorimetry analysis of a powder milled under hydrogen show a two-step desorption of hydrogen from NdH_{2+x}, at temperatures similar to those observed during the DDR process. Powders milled under reactive hydrogen atmosphere were annealed for 0.5 hour, at temperatures between 500 degreesC and 600 degreesC. The Nd₂Fe₁₄B phase was found to recombine at 520 degreesC. The presence of secondary phases such as neodymium oxides, alpha -Fe and Nd_{1.1}Fe₄Be₄ after annealing, shows that the recombination reaction is incomplete, likely due to oxygen contamination during the process.

[86] RELAXATION KINETICS OF MECHANICALLY ALLOYED POWDERS. FE-NI-SI-P: A CASE STUDY

MT ClavagueraMora, JJ Sunol, N Clavaguera - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 459-466 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Powders obtained by mechanical alloying show a relaxation or recovery of the stresses, which are related to the milling process, when submitted to heat treatment. The kinetics of relaxation of the as milled 'state' is studied. The analysis is performed by the coupling of experimental data obtained by differential scanning calorimetry under different working conditions: continuous heating and isothermal heat treatments. The modelling of the kinetics of the relaxation process is grounded on the consideration of a wide spectrum of relaxation times or activation energies. It is shown that two different mechanisms can be distinguished for the relaxation process. Evidence is facilitated by the experimental set-up employed: i) isothermal annealing allows the relaxation of processes whose relaxation time is lower than the annealing time or equivalently, whose activation energy is restricted to values that depend on annealing temperature and time. ii) Continuous heating on the contrary, allows the relaxation of processes that have activation energy progressively increasing with temperature. In general, since relaxation processes occurs in a large temperature interval, they are overlapped with other thermally activated transformations of the powdered materials. Examples related to the relaxation processes in Fe-Ni-Si-P alloys are presented. The as-milled powdered materials contain a mixture of nanocrystals and amorphous phase, whose relative amounts vary with milling time and alloy composition. Depending of the heat treatment applied to the sample, relaxation is overlapped with both growth of nanocrystals and nucleation of new crystalline phases. The several contributions of all these transformations in the overall calorimetric signal are discussed.

[85] CRYSTALLISATION OF PARTIALLY AMORPHIZED NI-MO ALLOY

R MartinezSanchez, SD delaTorre, F EspinosaMagana, LB Gomez, JG CabanasMoreno - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 487-492 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

The Ni-Mo system processed through mechanical alloying (MA) technique has been widely investigated. Complete or partial amorphisation in this binary system mainly depends on the: concentration of the elements. A new microstructural phase, called "Y" has been reported upon crystallisation of the powder. Although the "Y" phase formation has been attributed to the presence of C, O or N contaminants, its identification and characteristics remains unclear. In this work, an oxycarbide (Ni₆Mo₆(C,O)(1.06)) has been identified in samples in the as-heated condition and has been found as the crystallised phase in major amount in the final product. The XRD pattern of this oxycarbide is very similar to that reported before for a new phase called "Y". The simulated XRD pattern of oxycarbide (Ni₆Mo₆(C,O)(1.06)) is in good agreement with that experimentally found in this work. The activation energy required to induce crystallisation (-336 kJ/mol) matches well to the values reported in literature.

[84] NANOMETRIC ARCHITECTURE OF SMC05 RELATED MAGNETIC PROPERTIES

Y Champion, J Zhang, G Youhui, L Bessais - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp



513-518 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

SmCo₅ samples, initially melted in a cold crucible, were melt-spun on a Cu-(Co,Be) wheel, leading to the formation of thin chemically homogenous flakes. Sample composition is within the single phase domain SmCo_{5+x}, with -0.05 < x < 0.21. Series of 2 g of flakes were ball milled for a short time with a planetary miller, for which vials and plate rotations (omega/Omega) can be set independently. The coherent domain size and the strain rate were estimated from X-ray line broadening. Magnetic measurements were carried out at room temperature using a superconducting quantum interference device. X-ray diffraction experiments showed a typical decrease in grain size and increase in distortion rate with respect to the milling energy (proportional to omega.Omega (2)). They also revealed that the grain shape is strongly dependent on the milling conditions (omega.Omega). The magnetic hysteresis loop, the coercivity, the magnetisation and the remanence ratio of these powders are seen to be very sensitive to the nanostructure and the milling conditions applied.

[83] OXIDATION BEHAVIOUR OF ND-FE-B NANOCOMPOSITE POWDERS

JM LeBreton, G Khelifati, J Jakubowicz, M Jurczyk - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 519-524 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

The oxidation behaviour of several Nd-Fe-B based nanocomposite powders is investigated by X-ray diffraction and Mossbauer spectrometry. The powders are nanocomposite two-phase Nd-2(Fe,Co,Al,Cr,Zr)(14)B / alpha -Fe powders with different volume fractions of alpha -Fe, and nanostructured single-phase Nd-2(Fe,Co,Al,V,Cr)(14)B powders. They were prepared by High Energy Ball Milling (HEBM) and annealed for 30 minutes at 630 degreesC. The microstructural investigations revealed that the powders are composed of particles with a very broad size distribution (1 to 50 mum) For the oxidation experiments, the powders were oxidized in ambient air at 300 degreesC for times up to 72 hours. Then, the oxidation kinetics were followed using room temperature Mossbauer spectrometry. The results showed that the oxidation rate of the Nd₂Fe₁₄B phase decreases as the volume fraction of alpha -Fe increases, in agreement with the fact that alpha -Fe is more corrosion resistant than Nd₂Fe₁₄B (as it contains no rare earth). A nanocomposite two-phase powder appears thus to be intrinsically more corrosion resistant than a single-phase powder. Furthermore, the positive influence of addition elements (Cr and V) was confirmed.

[82] MOSSBAUER SPECTROSCOPY STUDY OF THE CRYSTALLISATION BEHAVIOUR OF FE-NI-SI-P AMORPHOUS POWDERS PREPARED BY BALL MILLING

JJ Sunol, T Pradell, N Clavaguera, MT ClavagueraMora - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 525-530 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Crystallisation of ball milled amorphous powders with the overall composition Fe₄₀Ni₄₀P_{20-x}Si_x (x=6, 10, 14) are studied by X-ray diffraction and transmission Mossbauer spectroscopy. Structural relaxation and growth of preexisting phases appear at temperatures below 473K. Further annealing at higher temperature results in the complete crystallization of the material. Different amorphisation ability of the resulting amorphous powders is determined as a function of the Si/P content. The crystallisation products determined - Fe(Si) bcc and Fe-Ni fee phases, Ni rich silicides and Fe-Ni phosphides- are the same as those obtained after crystallization of melt spun amorphous alloys of the same composition. The collapse of the magnetic hyperfine field, characteristic of INVAR Fe-Ni fee phases with compositions in the 30-40at% Ni range, is observed. However, this magnetic collapse is not obtained in the corresponding amorphous alloys obtained by melt spinning which, on the contrary, show the precipitation of both ferromagnetic and paramagnetic Fe-Ni fee phases. Although this behaviour seems to indicate the precipitation of Fe-richer phases, the mechanical stresses introduced in the material are also known to be able to induce such different magnetic behaviour.

[81] NANOCRYSTALLINE FE₇₈B₁₃SI₉ ALLOY POWDERS PREPARED BY BALL MILLING

M Jachimowicz, VI Fadeeva, H Matyja, A Grabias - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 537-542 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Nanocrystalline Fe₇₈B₁₃Si₉ alloy powders were prepared by ball milling of amorphous and crystallized ribbons as well as by mechanical alloying of elemental powders mixture. Ball milling produces nanocrystalline supersaturated alpha -Fe(Si,B) solid solution for all the materials used. Mechanical crystallization of the amorphous phase is sufficient for the nucleation of the crystalline phase, even after short milling. Further milling is responsible for an increase in the crystalline phase fraction and grain growth processes. In the case of milling crystalline materials (mixture of crystalline powders or crystallized ribbon), continuous refinement of the microstructure was observed. The grain refinement to the nanometer range is accompanied by an increase in atomic-level strain.

[80] THE MAGNETIC PROPERTIES OF COMPACTED FE_{73.5}CU₁NB₃SI_{13.5}B₉ ALLOY IN AMORPHOUS AND NANOCRYSTALLINE STATE

P Kollar, E Fechova, J Fuzer, A Zelenakova, V Kavcansky, J Kovac, M Konc - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM),



2001, Vol 360-3, pp 559-563 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Bulk samples were obtained from a powder of crushed amorphous alloy ribbon of standard Finemet composition by uniaxial cold compacting. The structure of the powder was determined using X-ray diffraction. The Curie and the crystallization temperatures of powder and bulk samples were determined from thermomagnetic curves measured with a vibrating sample magnetometer (VSM). Uniaxial compacted ring cores were produced. The hysteresis loops of these bulk samples were measured with a.c. hysteresisgraph with a storage oscilloscope. We have found that the ring core prepared by 7 GPa uniaxial stress of powder exhibits a coercivity of 180 A/m after annealing at 540 degreesC.

[79] MAGNETIC PHASE DIAGRAM OF THE FEXMN0.60-XAL0.40 (0.20 <= X <= 0.60) ALLOYS MECHANICALLY ALLOYED FOR 48 HOURS

C Gonzalez, G Medina, JM Greneche, GAP Alcazar, S Surinach, JS Munoz, MD Baro - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 565-570 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Alloys of the system $\text{Fe}_x\text{Mn}_{0.60-x}\text{Al}_{0.40}$, 0.20 less than or equal to x less than or equal to 0.60, were prepared by mechanical alloying for 48 hours. The Mossbauer spectra were fitted with either a single line and a quadrupolar doublet or a single line, a quadrupolar doublet and a hyperfine field distribution (HFD), according to their hyperfine structures. Alloys with x less than or equal to 0.40 show a paramagnetic character at RT while those with x greater than or equal to 0.45 are ferromagnetic with some paramagnetic sites due to the disorder. At 77 K, one can distinguish three types of alloys which display different magnetic behaviors: (i) antiferromagnetic with lower Fe concentration, (ii) paramagnetic with intermediate Fe contents and (iii) ferromagnetic with high Fe content. In the 0.40 less than or equal to x less than or equal to 0.60 composition region of the ac magnetic susceptibility χ Vs T, Superparamagnetic clusters appear inside the ferromagnetic matrix, as a consequence of the high Fe content and the disordered character of the samples.

[78] THE STRUCTURE AND MAGNETIC PROPERTIES OF POWDER FINEMET MATERIALS

E Fechova, J Kovac, P Kollar, J Fuzer, M Konc - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 577-581 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

The aim of this work was to prepare powder samples by milling of amorphous and partially nanocrystalline ribbons of Finemet type material and to investigate the influence of milling time on the structure and magnetic properties of powder Finemet material. The magnetic moments and the ratio of amorphous parts of the samples were measured by a vibrating sample magnetometer (VSM) as a function of milling time. The coercivity was determined from the hysteresis loops traced by the VSM, We found that magnetic properties significantly depend on milling time and the structural state of the ribbons, The coercivity increases to its maximum for amorphous and partially nanocrystalline samples up to 800 hours of milling time and then decreases. This maximum is corresponding with maximum of the volume fraction of crystalline phase in the sample.

[77] PRECURSORS FOR FE-BASED MAGNETIC NANOCRYSTALLINE PHASES IN AG MATRIX

A Jianu, V Kuncser, R Nicula, R Rohlsberger, M Stir, E Burkel - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 589-593 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

A new approach for obtaining Fe-based magnetic nanocrystalline phases in a paramagnetic Ag matrix is presented. $\text{Ag}_{85}\text{Sm}_{15}$ alloys were obtained by the single-roll melt-spinning method. The Ag-Sm ribbons were ball-milled together with fine-grained Fe powder in a RETSCH-4000 high-energy ball-mill for more than 80 hours. The diffusion of Fe into the Ag-based matrix is almost complete after approx. 50 hours. The evolution of the resulting Ag-Sm-Fe solid solution vs. The milling time was monitored by synchrotron radiation powder diffraction and by Mossbauer spectroscopy.

[76] MECHANICALLY MILLED NANOCRYSTALLINE Ni_3Sn_4 AND FeSi_2 ALLOYS AS AN ANODE MATERIAL FOR LI-ION BATTERIES

JH Ahn, GX Wang, HK Liu, SX Dou - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 595-602 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Nanocrystalline Ni_3Sn_4 and FeSi_2 have been synthesized by high energy ball-milling and their electrochemical properties have been examined with respect to their performance as new anode materials for lithium-ion batteries. A comparison was made between the mechanically milled nanocrystalline and the subsequently annealed microcrystalline electrodes. High energy ball-milled nanocrystalline powder exhibits a large first discharge capacity of 1515 mAh/g and 1129 mAh/g for Ni_3Sn_4 and FeSi_2 , respectively. The irreversible capacity loss happens primarily during the first charge/discharge cycle in the nanocrystalline powders. Although initial capacity decreases rapidly in the early stage of charge/discharge cycling, the nanocrystalline FeSi_2 exhibits higher irreversible lithium capacities than the annealed microcrystalline powders.

[75] NANOCRYSTALLINE MG-BASED HYDRIDES: HYDROGEN STORAGE FOR THE ZERO-EMISSION VEHICLE



T Klassen, W Oelerich, R Bormann - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 603-608 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Nanocrystalline light metal hydrides are interesting for hydrogen storage in zero-emission vehicles. In this paper, recent detailed results on the sorption behaviour of nanocrystalline Mg and Mg-based alloys are presented. Different transition metals have been added to Mg to achieve a thermodynamic destabilisation of the hydride, thus lowering the desorption temperatures to about 230 degreesC. A breakthrough in sorption kinetics was achieved by using nanocrystalline materials produced by high-energy milling. Additionally, new catalysts based on metal oxides have been added to further enhance the sorption reaction. As a result, hydrogen is absorbed and released within minutes instead of hours as in conventional Mg hydrides. The new materials fulfill the current requirements for the desired application with respect to both, capacity and kinetics.

[74] EFFECTS OF REACTIVE MECHANICAL GRINDING ON BOTH CHEMICAL AND HYDROGEN SORPTION PROPERTIES OF MG-M (M=CO, NI AND FE) MIXTURES

JL Bobet, B Darriet - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 609-614 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Reactive Mechanical Grinding (RMG) under H₂ Of magnesium powder allows to obtain a partial conversion of Mg to MgH₂. The ratio MgH₂/Mg can be improved by increasing the milling time or by adding a 3d-element M (with the same initial particle size). X-ray diffraction was applied successfully to determine the quantity of MgH₂ formed. The RMG allows to suppress the activation procedure generally requested for magnesium. 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. It is demonstrated that RMG for short time (2 hours) is a effective way to strongly improve the hydrogen storage properties of both magnesium and (Mg + 10 wt.%M) mixtures.

[73] REDUCTION IN GRAIN SIZE OF DOPED PZT CERAMICS FOR MINIATURE SENSORS AND ACTUATORS BY BALL MILLING TECHNIQUE

L Lu, BW Chua, MO Lai, RM Johar - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 615-622 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Piezoelectric ceramics Pb(Zr_{0.52}Ti_{0.48})O₃ (PZT) doped with Bi₄Ti₃O₁₂ were investigated via ball-milling technique. It was found that the doped PZT ceramics not only had sub-micron grain size without appreciable degradation to their piezoelectric properties, but also enhanced fracture toughness value. Thus, it is feasible to produce fine grain miniature sensors and actuators by ball milling technique.

[72] PRODUCTION AND SINTERING OF MGO-MGFE₂O₄ POWDER CERAMICS BY MECHANICAL ALLOYING

A Huerta, HA Calderon, M Umemoto - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 631-636 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Ceramic materials consisting of magnetic particles (MgFe₂O₄) in an insulating matrix (MgO) with a nanocrystalline structure have been produced by mechanical alloying and sintering techniques. Initial mixtures of MgO and Fe₂O₃ were milled in high-energy mills and characterized after different milling times. Increasing amounts of magnesiowustite (Mg_xFe_{1-x}O) form as a function of milling time. Sintering was performed from 673 to 1273 K in vacuum. Sintering at low temperatures allows retention of nanosized grains containing a fine dispersion of magnetic particles in a magnesiowustite matrix. Measurement of magnetic properties is used to determine both the nature of the developed phases and the grain sizes.

[71] HYDROGEN SORPTION PROPERTIES OF COMPOSITES MIXTURES MG PLUS YNi SUBMITTED TO MECHANICAL GRINDING

Nakhl, JL Bobet, B Chevalier, B Darriet - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 637-642 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Ball milling was used to make composite Mg + x wt.% YNi powders (x = 10, 25 and 50). The intermetallic YNi crystallizes with an orthorhombic symmetry. For the first time we have studied its hydriding properties. YNi absorbs, at 296K under 1.1MPa of hydrogen pressure, approximately 3 H.mol⁻¹ to form a stable hydride. Its crystallographic characteristics, studied by the aid of Transmission Electron Microscopy and X-ray diffraction, correspond to a monoclinic symmetry. The Mg-based materials were prepared using a planetary ball-mill. SEM micrographs showed that 3h of ball milling allow a homogeneous distribution of fine particles of YNi on the surface of the magnesium. X-ray diffraction revealed that this mixture is constituted by the same initial two phases : Mg and YNi. This mixture is not stable upon high temperature (573K) hydriding and dehydriding cycling. It was transformed to a new Mg + YH_x + Mg₂Ni composite, identified by X-ray diffraction, which is stable upon further cycling. The new composite has good hydrogen absorption kinetics at 573K



(compared to Mg). The best absorption capacity reaches 4.5 wt.% with the starting mixture Mg + 25 wt.% YNi milled for 3h. Its hydrogen desorption kinetics, under 0.1MPa at 573K were also significantly improved compared to Mg but not as much as the absorption one.

[70] ALLOYING IN A SYSTEM WITH POSITIVE HEAT OF MIXING: MD SIMULATIONS OF AG-CU

HW Sheng, E Ma - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 275-281 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Based on molecular dynamics (MD) simulation results, we present two schemes to enhance solid solubility in positive-heat-of-mixing (+ ΔH) systems at low temperatures. The Ag-Cu system, which is virtually immiscible below about 600 K, is used as a model for systems in which the relatively large atomic size mismatch (13%) between the two constituent elements contributes significantly to the + ΔH of the crystalline solid solution. We show two cases where the atomic configurations involved lead to easier accommodation of the alloying atoms such that the magnitude of the + ΔH can be significantly reduced. The first strategy is to go to free surfaces with under-coordinated atoms such that in the surface layers the apparent + ΔH is less positive relative to its value in the bulk. A solubility enhancement is thus observable at moderately elevated temperatures (greater than or equal to 600K), representing an extension of the experimentally known phenomenon of surface alloying at very low coverages. The second approach takes the system through the liquid state that also costs less strain energy for alloying compared with the crystalline phase. Our approach differs from the well known melt quench processes, however, in that it is an interdiffusional mixing reaction between two elemental melts induced mechanically through high strain rate deformation of crystals at relatively low temperatures.

[69] INFLUENCE OF MILLING TEMPERATURE ON THE MECHANICAL ALLOYING AND MAGNETIC PROPERTIES OF CU-CO AND CU-FE SYSTEMS

MA Uimin, AY Korobeinikov, AY Yermakov, VI Khrabrov, AK Shtolz - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 289-294 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

In this work the influence of temperature during mechanical alloying (from 80 K up to 525 K) on the structural state and magnetic properties of alloys based on immiscible Cu-Co and Cu-Fe alloys with 3d-metal contents of up to 30 at. % was investigated. It was established that mechanical alloying of Cu-Co and Cu-Fe forms a heterogeneous nanophase structure, in a temperature range from 80 up to 400 K containing clusters or particles with various contents of a ferromagnetic element. The reversibility of structural changes for Cu-Co alloy was observed; in this case the final structural state does not depend on the sample prehistory, and is determined by the milling temperature for given parameters of the milling equipment. The mechanism for a change of critical temperature for magnetic phase transformation in the alloys is discussed taking into account the magnetic interaction between particles.

[68] A STUDY OF THE EVOLUTION OF PARTICLE SIZE AND GEOMETRY DURING BALL MILLING

A Calka, D Wexler - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 301-310 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

This paper describes some simple investigations of the effects of milling condition and milling technique on powder particle size distribution and morphology. Experiments were performed on brittle alumina and ductile Al. Three types of ball-mill were used; a vibrational mill containing one large ball, a planetary Fritsch mill, and a horizontal Uni ball-mill with external magnets used to control ball movement and milling mechanism. For alumina, dry and wet milling was carried out, while Al was milled in vacuum, Ar, He, H₂, NH₃ and under wet conditions. Milling of alumina powder in a dry atmosphere using vibratory, planetary and magneto techniques periods was found to result in the evolution of similar particle size distributions, all tending to bimodal for extended milling times. Compared with controlled milling in a dry atmosphere, wet milling of alumina powder under the same conditions was found to result broadening of the size distribution curves, including the formation of nanoparticles. High energy milling of Al powder, using vibratory, planetary and magneto-milling under impact, was found to result in excessive cold welding preventing particle evolution. Magneto-milling of Al powder under He using low energy shearing mode resulted in the evolution of spheroidal Al particles. The latter process involved, 1, the shearing and flattening of irregular particles leading to the formation of flakes, 2, bending of flakes and, 3, subsequent formation via cold welding and compaction of spheres. Increasing the pulling force on the balls resulted, firstly, in finer deformed flakes comprising the spheres while further increases in pulling force resulted in the formation of hollow spheres. Low energy magneto-milling Al powder under Ar and under wet milling conditions both resulted in the production of Al flakes, however, there was no tendency for spherodisation.

[67] ON THE COUPLING BETWEEN MECHANICAL BEHAVIOR AND PHASE STABILITY IN MECHANICALLY MILLED ALLOY POWDER

M Atzmon, J Xu, HH Tian - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 311-316 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000



Previous results on phase evolution during mechanical attrition are reviewed. These are interpreted using the effective-temperature concept proposed by Martin (G. Martin, Phys. Rev. B30 (1984), p. 1424). The observed non-monotonic decomposition behavior is attributed to coupling between the evolving microstructure and phases. Previous experimental results and model of nanograin-size evolution in elemental Fe powder are employed to predict the dependence of the "ballistic" and thermal diffusion coefficients on the milling intensity and deformation behavior of the powder. These results lead to predictions on the dependence of the effective temperature, and therefore of alloy solubilities, on the hardness and milling intensity.

[66] PARTICLE SIZE EVOLUTION IN NON-ADHERED DUCTILE POWDERS DURING MECHANICAL ALLOYING

J GuerreroPaz, FC RoblesHernandez, R MartinezSanchez, D HernandezSilva, D JaramilloVigueras - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 317-322 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

The interaction among events as deformation, cold-welding and fracture, occurring during the mechanical milling of powders is unclear and controversial. We believe that the understanding of such interaction can be deduced from particle size evolution studies. It is well known that the elemental ductile powders adhere to the milling media. However when some of these powders are combined to form an alloy by milling, the adherence phenomenon is not observed. Systems which include ductile powders, such as, Cu-15at.%Al, Co-68at.%Al and Ni-25at.%Al were processed with not adherence to the milling media, thus allowing to follow up the particle size evolution during the complete milling process. The particle size was measured by the sedimentation-photometry technique. Those results were supported by scanning and transmission electron microscopy. The results showed a high proportion near 95% in number of particles of submicrometric size at early milling times for the three systems. However its particle size evolution for each system was different. Such findings can be important to understand some mechanisms as the grain size refinement, the alloy formation and the microstructural evolution. In the studied systems, the particle size measurements are presented based on volume or mass, area, line and number of the particles. The particle size results based on volume and line or number of the particles can give an idea of the evolution of the biggest particles and the finest ones respectively during the milling. Also the behavior of the complete particle system can be deduced from the results based in the area of the particles. Results of particle size as well as observations by microscopy helped to suggest the particle size and shape evolution of the studied systems. Such findings were employed to previously propose a grain size refinement mechanism for ductile powder systems non-adherent to the milling media during the mechanical alloying.

[65] THE ROLE OF NUCLEATION IN SOLID STATE REACTIONS DURING MECHANICAL ALLOYING AND HEAT TREATMENT OF HIGH ENERGY MECHANICALLY MILLED POWDERS

DL Zhang, DY Ying - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 323-328 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

One of the salient features of powder processing using high energy mechanical milling is the creation of a large area of interfaces between different phases through formation and refinement of a composite structure in each powder particle. When there exists a thermodynamic driving force for the reactions of the different phases in contact, they tend to occur. The kinetic requirements by nucleation and growth of the new phase to be formed by the reaction dictate the reaction paths. With increasing interface area, the nucleation plays increasingly important role in controlling the reaction path This paper shows that using the relatively simple classical nucleation theory, a great insight can be achieved on the factors influencing the selection of first phase to be formed and the dependence of reaction temperature on the interface area during mechanical alloying and solid state reactions during heat treating of the mechanically milled powders.

[64] MECHANICAL ALLOYING OF AN Fe_{0.30}Cr_{0.70} ALLOY FROM ELEMENTAL POWDERS

P Delcroix, T Ziller, C Bellouard, G LeCaer - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 329-336 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Magnetization measurements and Mossbauer spectrometry have been used among other techniques to characterize the steady-state structure of Fe_{0.30}Cr_{0.70} powders ground in a high-energy planetary mill and their evolution by subsequent annealing. The as-milled state is a b.c.c heterogeneous alloy with intragranular composition fluctuations of similar to 0.1 in amplitude on a scale of a few nanometres. Isothermal annealing at 623 K results in a coarsening of Fe-rich clusters.

[63] A QUANTITATIVE DESCRIPTION OF THE AMORPHISATION BEHAVIOUR BY MECHANICAL ALLOYING

F Delogu, L Schiffini, G Cocco - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 337-342 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

In the belief that further progress requires an adequate characterisation of both the milling dynamics and the reactive behaviour of mechanically induced co-deformation processes, we attempted to gain a quantitative description of the transformation behaviour as a function of the energy intensity of the mechanical treatment. Structural evolution of pure



transition metals, binary mixtures and intermetallic compounds was studied. Sigmoidal curves characterised the conversion of elemental mixtures leading to either amorphous or crystalline phases, while a progressively decreasing transformation rate was observed in the case of the disordering behaviour of intermetallics. The variation of the milling intensity only affected the reaction rates and the transformation processes were found to be isokinetic as a function of the mechanical work performed on the system. A macrokinetic model was developed in order to relate the experimental findings to the impact sequence registered in the course of die milling treatment. Based on a statistical approach, the model provides a set of differential equations solutions to which describe the evolution of the product phase in terms of conventional rate expressions. The model allows one to reproduce the different kinetic behaviours by means of a single, unifying mathematical formalism. The experimental trends for both the comminution and phase transformation processes are well reproduced and the conventional best-fitting procedures permits to quantify the conversion rate through suitable kinetic constants, related to the fraction of the powder charge actually cold-worked at each impact. The rate constants are found to depend linearly on the average impact energy. In the case of Ti-based alloys with metals belonging to the first transition period, their values correlate with the Poisson's ratio of the metal partner of Ti.

[62] STRUCTURAL STUDIES OF HF50CO50 ALLOYS PREPARED BY MECHANICAL ALLOYING

A AlHajry, M AlAssiri, S Enzo, J Hefne, S AlHeniti, N Cowlam, A AlShahrani - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 343-348 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

This study concerns the structural transformations in Mechanically Alloyed (MA) Hf-Co system. This system, by analogy with the Hf-Pd system, is supposed to exhibit a fast solid state "amorphization" reaction. A series of MA specimens separated by very short MA time were prepared. X-ray diffraction (XRD) was used to characterise the fast structural transformations during the amorphization reaction in these specimens. Almost complete amorphous HfCo phase formed after 3 3/4 h. This unstable amorphous phase transforms to the more stable intermetallic HfCo phase after MA time of 5 h. DSC measurements showed that this intermetallic phase is stable up to 600 degreesC. The Rietveld's profile refinement method was used to analyse the structural transformations and to evaluate crystallite sizes and strain effects as a function of MA time.

[61] STUDY OF THE SYNTHESIS AND SINTERING OF NANOPHASE FE73AL27 OBTAINED BY MECHANICAL ALLOYING

G Gonzalez, L D'Angelo, J Ochoa, L Donofrio - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 349-354 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

In the present work the effect of the ball to powder relation (BPR) and the different milling media, for the initial stages of milling from 15 min to 5 tl, was studied for elemental powders of Fe- 27 at % Al. Long time milling periods were also carried out in order to obtain nanophase particles and further sintering was performed for the samples mechanically alloyed (MA) during 5 h and 50 h. The evolution of the phases formed, for the different stages of milling was followed by X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and Mossbauer spectroscopy. The formation of the alloy was found to be more effective for BPR 8:1, in which case the complete formation of the alloy was achieved after 3 h of milling. Nanophases with particle size of 7 nm were obtained after 50 h of milling. The alpha -Fe solid solution was also obtained by sintering at the different temperatures.

[60] EFFECT OF THE MILLING MEDIA ON THE PHASES OBTAINED IN MECHANICALLY ALLOYED EQUIATOMIC FE-CO

G Gonzalez, A Sagarzazu, R Villalba, J Ochoa, L Donofrio - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 355-360 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

In the present work the effect of the milling media on the phases obtained in the Fe-Co equiatomic mixture of mechanical alloyed elemental powders is studied. The Fe-Co powders were milled in a high energy mill for different periods of time, employing two different set of milling tools, WC and steel. The evolution of the phases Formed, for the different stages of milling, was followed by X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and Mossbauer spectroscopy. A remarkable difference between both milling media was found, with respect to phases formed and homogeneity of the samples with milling time. Contamination is observed in both cases, which seems to affect the phases formed. For long milling periods (49 h) nanophase particles with mean particle size of 6 nm were obtained.

[59] ALLOY FORMATION AT BALL MILLING OF CU50CR50 AND FE86.5CU13.5 COMPOSITIONS

VV Tcherdyntsev, SD Kaloshkin, IA Tomilin, EV Shelekhov, VN Serdyukov - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 361-366 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Two compositions of immiscible systems - Cu50Cr50 and Fe86.5Cu13.5 - were selected for study of phase composition and crystalline structure transformation kinetics during ball milling of powder mixtures. Samples were milled in planetary ball mill in hermetic vials using two different rotation velocities of vials and two ball's diameters. The curves of the phases content vs. Milling time were sigma -shaped for Cu50Cr50 composition. Complete transformation of alloy into b.c.c, phase



was not observed for this composition, a steady-state ratio between phases amounts was reached after certain milling time. On the contrary, for Fe_{86.5}Cu_{13.5} composition a complete transformation of f.c.c. Phase into b.c.c. At mechanical alloying was observed. The curve of b.c.c. Phase content vs, milling time includes two periods corresponded to the different transformation rates. Particular attention has been given to the initial part of phase transformation curves. The discussion of the results is based on the model of thermodynamic driving forces of mechanical alloying process.

[58] CHARACTERISATION OF THE BALL-MILLED CU-80(Fe0.3Co0.7)(20) COMPOUND AND EFFECTS OF THE MILLING CONDITIONS ON ITS NANOSTRUCTURE

S Galdeano, L Chaffron, MH Mathon, E Vincent, CH DeNovion - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 367-372 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

The aim of this work is the study of the milling conditions (milling temperature T and milling intensity I) and the influence of post-milling heat treatments on the microstructure of the Cu-80(Fe_{0.3}Co_{0.7})(20) compound, the giant magnetoresistance (GMR) properties of which strongly depend on the size of the magnetic particles and their spatial distribution. Batches of Cu₈₈Fe₁₂ and Cu₇₂Co₂₈ powders were prepared by mechanical alloying (MA) elemental powders at T = 300 K and I = 1000 m/s². The ternary compound was produced by ball milling a mixture of these two binary compounds in the milling temperature range 300 K - 473 K with a milling intensity ranging from 1000 m/s² to 4500 m/s². Annealing was then performed under vacuum at 773 K over a time range of 15 min - 24 h. The compounds were characterised by neutron powder diffraction, Small Angle Neutron Scattering experiments and magnetic measurements. The results showed that the majority of the Fe and Co atoms are probably in f.c.c. Fe-Co superparamagnetic particles after milling. The remaining atoms seem to be in solid solution in the Cu-rich matrix or in very small superparamagnetic germs. After annealing, the particles coarsen and transform partly to b.c.c. Phase. Their ferromagnetic volume fraction increases with the annealing time. Effects of the milling conditions have been proved.

[57] EXPERIMENTAL MEASUREMENT AND THEORETICAL COMPUTATION OF MILLING INTENSITY AND TEMPERATURE FOR THE PURPOSE OF MECHANICAL ALLOYING KINETICS DESCRIPTION

LY Pustov, SD Kaloshkin, VV Tcherdyntsev, IA Tomilin, EV Shelekhov, AI Salimon - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 373-378 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

The kinetics of phase formation in the Fe₅₀Mn₅₀ composition was investigated at different conditions of planetary and vibratory mills. Milling intensity and temperature were measured experimentally using specially developed techniques. The theoretical computation of milling intensity and temperature using computer simulation was conducted for comparison in order to verify the main assumptions of a model. Energy input was calculated, which is necessary for phase transformation in the system under investigation. Good agreement of the gamma-(Fe-Mn) phase formation rate with the milling process energy parameters was found.

[56] STRUCTURAL STUDIES OF THE AMORPHISATION REACTION OF ZRRH ALLOYS PREPARED BY MECHANICAL ALLOYING

M AlAssiri, A AlHajry, J Hefne, S Enzo, N Cowlam, A AlShahrani, AE Alsalami - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 379-384 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Mechanical alloying (MA) has been used to study the amorphisation reaction of ZrRh system. A two of MA alloys of Zr₇₅Rh₂₅ and Zr₅₀Rh₅₀ were prepared. X-ray diffraction was used to investigate the structure of these specimens. During the investigated MA time, Zr₇₅Rh₂₅ alloy is partially amorphised while Zr₅₀Rh₅₀ alloy is completely amorphised by EIIA technique. The amorphous phase of Zr₅₀Rh₅₀ system occurred in a very short MA time. This amorphous phase was confirmed by the reduced radial distribution function G(r) obtained by Fourier transform of the structure factor S(Q). The thermal stability of the amorphous Zr₅₀Rh₅₀ phase was investigated by DSC measurements to determine the crystallisation temperature of such a system.

[55] SYNTHESIS OF POWDER ALLOYS IN NI-AL-NB-C SYSTEM BY MECHANICAL ALLOYING

K Krivorouchko, T Kulik, H Matyja, VI Fadeeva, VK Portnoy - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 385-390 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Structure and phase composition of the mechanically alloyed nanocrystalline Ni-Al-Nb-C alloys with a constant content of Nb and C (5 at. % of each element) and various contents of Ni and Al were investigated. It was shown that in the alloys with C-Ni/C-Al = 1 divided by 1.6, the interaction between Ni and Al, and Nb and C occurs independently because of the high negative formation enthalpies of NiAl and NbC. As a result, stable phases NiAl_{1-x} (B2 structure) and non-stoichiometric carbide NbC, were formed during milling. When the ratio C-Ni/C-Al greater than or equal to 2.5, a supersaturated solid solution Ni(Al, Nb, C) with the fcc- structure was formed during the MA. Further heating up to 700 degreesC leads to the dissociation of the solid solution; Ni₃AlC_x phase (L1(2)) and non-stoichiometric carbide NbC, appear after the heating. The



DSC curve of this alloy exhibits an exothermic effect, which corresponds to the dissociation process. Hardness was measured in pressed and sintered (700 MPa, 1200 degreesC, 2 hours) samples; the dependence between the hardness and structure of the intermetallic matrix was found.

[54] COMPARISON OF MA PROCESSES AND PRODUCTS FROM Ni(OH)(2) WITH Al OR Al(OH)(3)

S Sekino, M Senna, T Isobe, T Shinohara, F Wagatsuma - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 409-413 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Ni(OH)(2) was milled either with Al metal or with Al(OH)(2) particles to compare the process of compounding and the properties of the products. Although the Knight shift, K-27, of Al-27-NMR spectrum of Al(OH)(3) is about 0ppm, it exceeds 10000ppm after milling with Ni(OH)(2) for 12h. This shift increases to above 20000ppm at lower temperature. In metallic Al system, K-27 under the same condition was ca. 1600ppm and slightly decreases with decreasing temperature. In the latter system, the average conduction electron density of metallic Al is reduced by Ni species in the surface oxide layer and reduces the Fermi contact interaction at Al nuclei to change the Knight shift. In contrast, in Al(OH), system, Al(m) species are surrounded by Ni(II) so that 27K is directly influenced by the local magnetic field of Ni(II). From the viewpoint of materials synthesis, using Al(OH), is more beneficial due to much more homogeneous structure close to that of NiAl₂O₄. We further elucidated the change in the state of Al by heating the as-milled product to give well-crystallized NiAl₂O₄.

[53] MECHANISM OF H-2 ACTIVATION ON METALS UNDER MECHANOCHEMICAL TREATMENT

OS Morozova, AV Leonov, TI Khomenko, VN Korchak - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 415-420 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

It has been found that the maximum efficiency of graphite hydrogenation by H-2 under high-energy impact milling action is provided by the presence of hydride-forming metals. This effect is due to activation of H₂ through the formation and decomposition of metal hydrides. The hydrogenation of graphite was carried out under flow conditions. The kinetic parameters of the process were studied for a graphite - Zr system. Kinetic and XRD measurements showed that H₂ sorption during the first 15 - 20 min of milling form a highly dispersed tetragonal ZrH_x (x = 1.8 - 2) phase. The specific surface area of the graphite - Zr sample changed from 0.6 to similar to 58 m²/g. At this stage, the Zr metal content in the sample was small. The next stage can be characterized as a steady state for the sorption - desorption of hydrogen. The formation of CH₄ stopped because of coating the Zr microcrystals by carbon. We found that concurrent decomposition of tetragonal ZrH_x and sorption of H-2 by graphite followed the steady-state condition. Further mechanochemical treatment led to a "stratified" composition that contained phases of graphite, cubic ZrH₂, tetragonal ZrH, and Zr with a hcp lattice partly distorted and disordered.

[52] MECHANICAL ALLOYING OF FINE STRUCTURED Ti-AL-Nb ALUMINIDES INVOLVING Ti-HYDRIDE

M Bououdina, Z Luklinska, ZX Guo - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 421-426 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

In order to produce high-quality aluminide alloys, a comparative study was carried out on the Mechanical Alloying (MA) characteristics of two powder mixtures, Ti-22Al-23Nb and TiH₂- 22Al-23Nb(at.%), respectively. It was noted that for the Ti-22Al-23Nb mixture MA led to the formation of a bcc (Nb,Ti,Al) solid solution with some degree of amorphisation under the experimental conditions. The use of Ti-hydride in the TiH₂-22Al-23Nb mixture led to the formation of an amorphous (Nb,Ti,Al)H-2 solid solution. The presence of hydrogen in the powder mixture largely enhances the amorphisation process. TEM diffraction patterns confirmed the formation of both solid solutions. EDS analysis showed that chemical compositions of the bcc and of the amorphous solid solutions were close to the starting values.

[51] THE EFFECT OF INERT ADDITIVES ON THE ACTIVATION PERIOD OF MECHANICALLY-INDUCED SELF-PROPAGATING REACTIONS

L Takacs, V Soika - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 427-432 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

The effect of inert additives on the mechanically-induced self-propagating reaction in equimolar Zn-S powder mixtures has been investigated. The addition of Al₂O₃ or ZrO₂ does not change the ignition time substantially, but more than about 25 vol.% renders the reaction gradual. If NaCl is added to the Zn-S mixture, the ignition time increases linearly and it almost triples before the reaction becomes gradual. The effect of Ni addition was also studied. Ni reacted with S itself, therefore it is not entirely inert in the Zn-S system. These observations are very different from the steady increase of the ignition time usually assumed.

[50] MODELING AND OPTIMIZATION OF JET MILLING

Eskin DI. Dorokhov IN. Vasil'kov OI. Voropaev SN. - Theoretical Foundations of Chemical Engineering (English Translation of Teoreticheskie Osnovy Khimicheskoi Tekhnologii). 35(2):188-195, 2001



The energy efficiencies of the acceleration nozzles of ejector and pneumatic jet mills are compared. Mathematical models are proposed for the acceleration of a two-phase mixture in the nozzle and the jet, and a procedure is put forward for optimizing subsonic nozzles and open-loop jet mills. The gas-dynamic perfection of the nozzles of pneumatic jet mills is assessed, and the initial particle-size range where the mill efficiency is highest is determined

[49] SYNTHESIS OF ENGINEERED PARTICULATES WITH TAILORED PROPERTIES USING DRY PARTICLE COATING [REVIEW]

Pfeffer R. Dave RN. Wei DG. Ramlakhan M. - Powder Technology. 117(1-2 Special Issue SI):40-67, 2001

Dry particle coating is used to create new-generation materials by combining different powders having different physical and chemical properties to form composites, which show new functionality or improve the characteristics of known materials. Materials with relatively large particle size (1-200 μm) form a core and these core (host) particles are mechanically coated with fine submicron (guest) particles; no liquid of any kind (solvents, binders or water) is required. A number of different devices used to achieve dry particle coating are reviewed. The fundamental mechanisms by which these devices achieve coating are discussed, and many examples of coated particles produced by these methods in our laboratory, as well as by other researchers, are described. Attempts to model some of these processes, so as to be able to predict suitable operating conditions and processing times for different host and guest particle properties, are also described. A theoretical predictive capability is necessary, not only to determine which of the devices would give the best results in a specific application, but also for scale-up and optimization. Based on our research, we believe that dry coating is a viable alternative to wet coating and can be used successfully for certain applications where wet coating processes are not feasible

[48] AS AND SB LEACHING FROM POLYMETALLIC SULFIDE CONCENTRATES

Balaz P. Kammel R. Villachica C. - Metall. 55(4):196-200, 2001.

The leachability of antimony and arsenic from Peruvian complex sulfide concentrates (Casapalca, Huaron) were studied by alkaline Na_2S leaching. The experiments have shown that by a proper mechanical activation of the difficult to treat concentrates 99% Sb and 99% As from Casapalca and 63% Sb and 80% As from Huaron can be eliminated. The chemical composition of CuPbZnAg concentrates after Sb and As elimination may improve the further pyrometallurgical treatment from the technological as well as environmental aspects

[47] BULK METALLIC GLASSES AND COMPOSITES IN MULTICOMPONENT SYSTEMS

Eckert J. Deledda S. Kuhn U. Reiger-Leonhard A. - Materials Transactions. 42(4):650-655, 2001

Single-phase (Zr/Ti)Cu-Al-Ni metallic glasses were synthesized by copper mold casting and mechanical alloying of elemental powder mixtures. In addition, nanostructured metallic glass composites were produced by annealing of as-cast specimens and by mechanical alloying of elemental powder mixtures blended with different volume fractions of ZrC particles. For cast specimens, the effect of composition on the thermal stability and the microstructure after partial crystallization was investigated by X-ray diffraction and differential scanning calorimetry. Ti additions lead to a lower overall thermal stability of the material and induce primary crystallization. The grain size of the precipitates after the first crystallization stage becomes progressively finer with increasing Ti content, yielding grain sizes as small as about 2 nm for $\text{Zr}_{54.5}\text{Ti}_{7.5}\text{Cu}_{20}\text{Al}_{10}\text{Ni}_8$. For mechanically alloyed $\text{Zr}_{55}\text{Cu}_{30}\text{Al}_{10}\text{Ni}_5$ powders containing up to 30 vol% nanoscale ZrC particles, stoichiometry variations of the glass due to particle dissolution in the glassy matrix during the mechanical alloying process were observed, which affect the thermal stability of the glassy matrix phase. Nevertheless, the composites maintain an extended supercooled liquid region before crystallization

[46] ELECTRICAL PROPERTIES OF COMPOSITE MATERIALS OBTAINED BY MECHANOCHEMICAL CHARGE-TRANSFER BETWEEN BEDT-TTF AND GOLD-DERIVED ACCEPTORS

Graja A. Brau A. Farges JP. Golub M. Tracz A. Jeszka JK. - Synthetic Metals. 120(1-3 Special Issue SI):753-754, 2001

Conducting organic composites obtained by direct solid-solid charge-transfer reaction between bis(ethylenedithio)tetrathiafulvalene (BEDT-TTF or shorter ET) and iodides AuI or AuI_3 present metallic behaviour for appropriate stoichiometry and appropriate grinding and annealing conditions. X-ray diffractograms, Raman spectra and d.c. electrical conductivity measurements of these composites are discussed and compared with those of (ET) $_2$ I-3. The complex structure of the investigated composites is suggested

[45] WEAR BEHAVIOR AND NANO-STRUCTURE OF SURFACE LAYERS OF HADFIELD STEEL UNDER IMPACT LOADING

Xu YH. Chen YM. Zhu JH. - Progress in Natural Science. 11(6):447-453, 2001

Under high energy impact contact loading, a turning on the weight loss-impact cycle (W-L-N) curve has been found, which results from the formation of nano-sized grains embedded in amorphous in the surface layers of Hadfield steel. As the impact energy increases, the wear rate, $dW(L)/dN$, first increases then decreases. The nanostructure due to the accumulation of plastic deformation is composed of nano-sized grains spanning 10 similar to 50 atoms and amorphous structures

[44] DEFORMATION AND DISINTEGRATION OF SOLID POLYPROPYLENE PARTICLES DURING THEIR MECHANOCHEMICAL MODIFICATION

Zelenetskii AN. Bunina LO. Volkov VP. Obolonkova ES. - Polymer Science (English Translation of Vysokomolekulyarnye Soyedineniya - Series A). 43(5):542-548, 2001

The effect of a mechanochemical modification of PP with maleic anhydride, maleic acid, and sodium maleate on the character of the deformation and disintegration of PP particles was studied by scanning electron microscopy. It was shown that the deformation and disintegration processes start from fine PP particles to spread to bigger ones as the duration and intensity of mechanochemical modification of PP increase. In this case, changes in the morphology and structure of PP particles are accompanied by an increase in the specific surface area, especially, at the final stage. Grafting a comonomer onto the polymer occurs via their joint deformation in all the particles present both at their surface and in the bulk

[43] ON THE SIMILARITY OF MACROMOLECULAR RESPONSES TO HIGH-ENERGY PROCESSES: MECHANICAL MILLING VS. IRRADIATION

Smith AP. Spontak RJ. Ade H. - Polymer Degradation & Stability. 72(3):519-524, 2001.

Recent efforts to blend and compatibilize intrinsically immiscible polymers in the solid state by high-energy methods have shown that macromolecules may undergo scission, crosslinking or amorphization, depending on the chemical nature of the repeat unit, the processing temperature and the initial degree of polymer crystallinity. Identical process-induced molecular



and structural modifications have been previously observed in polymers exposed to large doses of electron and gamma radiation, suggesting that the responses of polymers to high-energy processes may be mechanistically similar. In this work, we explore a variety of similarities between mechanically-milled and irradiated polymers in terms of molecular characteristics, process temperature and polymer crystallinity, and we demonstrate that these similarities provide predictive guidance for the selection of polymers to be subjected to solid-state processing

[42] THERMAL AND ELECTRICAL BEHAVIOR OF GAMMA-Bi₂VO_{5.5} AND F-Bi₂VO₅ OXIDES OBTAINED FROM MECHANOCHEMICALLY ACTIVATED PRECURSORS

Millan P. Rojo JM. Pardo L. Ricote J. Castro A. - Materials Research Bulletin. 36(7-8):1277-1286, 2001

The mechanochemical activation method has been used in order to reduce the synthesis times and temperatures for oxides in the system Bi-V-O-2. This new synthesis procedure yields to the formation of amorphous powders of 2Bi(2)O(3):V₂O₅ and Bi₂O₃:VO₂ compositions. Annealing at low temperatures results in the stabilization at room temperature of the high temperature phase gamma -Bi₂VO_{5.5} and fluorite-like f-Bi₂VO₅ not obtained by the traditional solid-state reaction method. Thermal and electrical behavior of these phases has been studied by DTA, XRD with temperature and impedance spectroscopy measurements. Both gamma -Bi₂VO_{5.5} and f-Bi₂VO₅ transform to well-known alpha -Bi₂VO_{5.5} and alpha -Bi₂VO₅, respectively, on heating above 500 degreesC. The conductivity and activation energy values of the phases are characteristic of good ionic conductor materials. The sintering protocol seems to be an essential point to improve their electrical properties

[41] THE MECHANISMS OF COMBUSTION AND CONTINUOUS REACTIONS DURING MECHANICAL ALLOYING

Calos NJ. Forrester JS. Schaffer GB. - Journal of Solid State Chemistry. 158(2):268-278, 2001

Some materials exhibit a combustion event during mechanical alloying, which results in the rapid transformation of reactants into products, while others show a slow transformation of reactants into products. In this paper, the continuous W + C --> WC reaction is compared to the Ti + C --> TiC combustion reaction. Rietveld refinement of X-ray diffraction patterns is used to show that these particular reactions proceed through different pathways, determined by crystallographic factors of the reactants. When a crystallographic relationship exists between the reactants and the products, such as that between W and WC, the product forms slowly over a period of time. In contrast, insertion of C into the Ti structure is associated with atomic rearrangements within the crowded lattice planes and the subsequent catastrophic failure of the reactant lattices results in combustion to form TiC

[40] INFLUENCE OF POLYMERIC SURFACTANTS ON PESTICIDAL SUSPENSION CONCENTRATES: DISPERSING ABILITY, MILLING EFFICIENCY AND STABILIZATION POWER

Haas S. Hasslin HW. Schlatter C. - Colloids & Surfaces A-Physicochemical & Engineering Aspects. 183(Special Issue SI):785-793, 2001

This paper deals with the influence of different polymeric surfactants respectively dispersants on three crucial aspects for the formulation of pesticides as water based suspension concentrates: the dispersing ability of the polymeric surfactant for the solid active ingredient, the support of the wet milling process leading to a better milling efficiency and the stabilization of the particles against agglomeration and Ostwald ripening upon storage. Therefore a wide range of polymeric surfactants respectively dispersants with different chemistry and/or molecular weight has been tested in our laboratories with regard to these three parameters. As expected, the chemical nature and the molecular weight of the polymeric surfactant respectively dispersant plays an important role for all three points of interest as well as the physico-chemical parameters of the used pesticidal active ingredient

[39] KINETIC PATHS FOR B2 ORDER IN NANOCRYSTALLINE FeCo-Mo: A MOSSBAUER SPECTROSCOPIC STUDY

Sarkar S. Bansal C. - Acta Materialia. 49(10):1789-1792, 2001

The kinetic evolution of B2 order in nanocrystalline FeCo-Mo alloys was studied at different temperatures using Mossbauer spectroscopy. The kinetic paths in the space spanned by the two short-range order parameters alpha (Co/Fe) and alpha (Mo/Fe) were found to be independent of temperature within experimental errors. This behavior is to be compared with that observed in coarse-grained FeCo-Mo alloys, where the paths were not the same at different temperatures. This can be attributed to the presence of more grain boundary regions in the nanophase alloys, which provide short-circuited diffusion paths for atom movements

[38] CRYSTALLOCHEMICAL ASPECTS OF SOLID STATE REACTIONS IN MECHANICALLY ALLOYED AL-CU-FE QUASICRYSTALLINE POWDERS

Salimon AI. Korsunsky AM. Shelekhov EV. Sviridova TA. Kaloshkin D. Tcherdyntsev VS. Baldokhin YV. - Acta Materialia. 49(10):1821-1833, 2001

A number of elemental mixtures having initial compositions close to Al₆₅Cu₂₃Fe₁₂ were mechanically alloyed at different energy intensities using a planetary mill. Laboratory X-ray diffraction analysis, differential scanning calorimetry and Mossbauer spectroscopy were used for characterization of the phase and structural state of mechanically alloyed powders after different periods of milling and annealing. Several exothermic effects were found, and these were ascribed to specific solid-state reactions. Quantitative phase analysis was applied in order to identify the mechanism of solid-state reactions taking place in the vicinity of the quasicrystalline phase domain in the Al-Cu-Fe system as a result of mechanical and thermal excitation and homogenization. Metastable intermetallics were identified which possess certain structural and topological elements identical to those found in quasicrystals

[37] SOLID-STATE SYNTHESIS OF MAGNESIUM BASE ALLOYS

T. Aizawa - MAGNESIUM ALLOYS 2000 (Series: MATERIALS SCIENCE FORUM), 2000, Vol 350-3, pp 299-310 - 1ST NAGAOKA INTERNATIONAL WORKSHOP ON MAGNESIUM PLATFORM SCIENCE AND TECHNOLOGY 2000; NAGAOKA CITY, JAPAN. JULY 27-29, 2000

Magnesium alloys are expected to be used for light-weighted structural members or parts and to improve their functionality per a unit weight. Since conventional processing are often inadequate or incompatible to synthesis and microstructure control solid-state synthesis is proposed as a promising methodology for magnesium alloy design, and developed on the route of the bulk mechanical alloying. Among various magnesium alloys, Mg-Ni system is employed here to demonstrate the validity and effectiveness of the present solid-state synthesis via bulk mechanical alloying. Single-phase Mg₂Ni is successfully



synthesized by the present method. In addition, both chemical modification by the third element addition and formation of nonequilibrium Mg₂Ti phases are performed in the present solid-state synthesis, aiming for further advancement of original functionality for Mg₂Ni alloys.

[36] SOLID-STATE SYNTHESIS OF Mg₂Si VIA BULK MECHANICAL ALLOYING

T Aizawa, KI Ichige - MAGNESIUM ALLOYS 2000 (Series: MATERIALS SCIENCE FORUM), 2000, Vol 350-3, pp 333-338

1ST NAGAOKA INTERNATIONAL WORKSHOP ON MAGNESIUM PLATFORM SCIENCE AND TECHNOLOGY 2000; NAGAOKA CITY, JAPAN. JULY 27-29, 2000

Solid state synthesis via bulk mechanical alloying is proposed to fabricate Mg₂Si alloy bulk samples. Starting from the powder mixture of flaky large-grained powders and silicon, Mg₂Si was reactively synthesized by the bulk mechanical alloying. Through XRD and TEM, the synthesized Mg₂Si was characterized with respect to phase and structure. Although total reactivity is strongly dependent on mechanical behavior during bulk mechanical alloying, single phase Mg₂Si can be successfully synthesized from locally reacted embryo by low-temperature annealing of the BMA samples.

[35] TI-BASED BULK AMORPHOUS AND QUASICRYSTALLINE MATERIALS PREPARED BY WARM PROCESS

KB Kim, S Yi, HK Lim, SH Kim, WT Kim, DH Kim - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 21-28 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Amorphization and crystallization behaviors of Ti₇₀Ni₁₅Al₁₅ and Ti_{41.5}Zr_{41.5}Ni₁₇ powders during mechanical alloying (MA) and subsequent heat treatments are studied. Fully amorphous powders are prepared by MA of the elemental powder mixture of the nominal composition of Ti₇₀Ni₁₅Al₁₅. During continuous heating of the amorphous powders in DSC, two exothermic events are observed. The first exothermic event corresponds to the formation of a supersaturated α-Ti phase of hexagonal close-packed structure. The growth kinetic of the α-Ti phase is sluggish forming nanostructured α-Ti matrix. The second exothermic event is associated with the solid-state transformation of the meta-stable α-Ti into the equilibrium phases, Ti₂Ni and Ti₃Al. Amorphous powders are prepared after MA for more than 30 hours in the mixture of elemental powders with the nominal composition of Ti_{41.5}Zr_{41.5}Ni₁₇. The amorphous powder transforms into the quasicrystalline phase in the temperature range of 300-400 degreesC during continuous heating in DSC. Based on the observed amorphization and crystallization behavior Ti₇₀Ni₁₅Al₁₅ and Ti_{41.5}Zr_{41.5}Ni₁₇ powders, bulk materials consisting of amorphous phase with small fraction of nanocrystals in the Ti-Ni-Al system and quasicrystalline phase in the Ti-Zr-Ni system can be fabricated in a hot press using MA powders.

[34] NANOCRYSTALLINE CAO AND ZrC AS A SECOND PHASE IN AMORPHOUS Zr-CU-AL-NI MATRIX COMPOSITES

Authors S Deledda, J Eckert, L Schultz - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 85-90 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

This contribution focuses on the case of a metallic glass matrix with nominal composition Zr₅₅Cu₃₀Al₁₀Ni₅ blended with increasing volume fractions of CaO or ZrC particles. A comparison with the particle-free metallic glass by means of X-ray diffraction and differential scanning calorimetry analysis shows that the addition of second phase particles does not affect the glass forming ability of the system. The thermal stability of the glass, i.e. The glass transition temperature T_g and the crystallization temperature T_c are only slightly affected. Nevertheless, continuous milling is likely to promote dissolution to a limited extent of the ceramic phase into the matrix. This can result in a change of composition and, eventually, in the suppression of the glass transition. Finally, an analysis of isothermal annealing data is presented in order to evaluate the effect of the nanoscaled particles on the kinetics of crystallization.

[33] THE EVOLUTION OF CRYSTALLINE PRECURSORS DURING THE FORMATION OF AL-CU-FE QUASICRYSTALLINE INTERMETALLICS IN MECHANICALLY ALLOYED POWDERS

Authors AI Salimon, AM Korsunsky, SD Kaloshkin, VV Tcherdyntsev, EV Shelekhov, TA Sviridova - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 137-142 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Mechanical alloying (MA) of elemental powders in the Al-Cu-Fe system was carried out using two types of laboratory equipment (vibratory and planetary mills) to produce single phase, stable icosahedral quasicrystals. The sequence and nature of the solid state reactions during milling and subsequent annealing were studied in detail using qualitative and quantitative laboratory X-ray analysis and differential scanning calorimetry. The effect of milling parameters (energy intensity, temperature, milling time) on the course of transformations was elucidated. Specific reactions were identified which took place during annealing in different temperature ranges. The evolution of crystalline precursors of the quasicrystalline phase was established.

[32] CRYSTALLISATION OF AL₉₀FE₅ND₅ AMORPHOUS ALLOY

KR Cardoso, AG Escorial, WJ Botta, M Lieblich - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp



161-166 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

The crystallisation sequence of Al₉₀Fe₅Nd₅ amorphous alloy obtained by three different methods, melt-spinning, atomisation and mechanical alloying, is studied. In the three cases the crystallisation is multiple stage and new stable and metastable phases are formed. The first step is always a primary crystallisation of Al, giving rise to a nanostructured state consisting of fcc-Al plus the remaining amorphous matrix. In the rapid solidified materials, melt-spun and atomised, the remaining amorphous matrix crystallises in a ternary metastable phase, which at higher temperatures transforms into the equilibrium phases. Differently, in the mechanically alloyed material the remaining amorphous matrix crystallises directly into the equilibrium phases.

[31] FORMATION OF NANOCRYSTALLINE FERRITE THROUGH ROLLING AND BALL MILLING

M Umemoto, ZG Liu, XJ Hao, K Masuyama, K Tsuchiya - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 167-174 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Nanocrystalline ferrite has been produced in carbon steels with carbon content up to eutectoid composition through heavy work of rolling and ball milling. It was found that nanocrystallization of ferrite occurred at wavy band region with 0.1 μm thick and several μm long in heavy cold rolling and ball milling. During milling, the volume fraction of nanocrystalline band increases with increasing deformation degree until the complete nanocrystallization. The nanocrystalline ferrite was observed in all specimens irrespective of carbon content (0.03-0.89 mass% C) and starting microstructures (pearlite, ferrite or martensite). Together with the nanocrystallization of ferrite, the dissolution of cementite was observed in specimens with either pearlite or spheroidite structures. For the specimen with spheroidite structure, the formation of nanocrystalline ferrite around cementite particles seems to destabilize them and induced their dissolution. By aging the deformed specimens, recrystallization was observed by the appearance of large recrystallized grains (a few μm) in the work hardened regions, while in the nanocrystalline ferrite region only a slight grain growth to several tens of nanometers was observed instead of recrystallization. The fine grain of aged nanocrystalline ferrite is enhanced probably by the pinning effect of cementite particles precipitated at nanocrystalline ferrite grain boundaries.

[30] NANOSTRUCTURE FORMATION AND MECHANICAL ALLOYING IN THE WHEEL/RAIL CONTACT AREA OF HIGH SPEED TRAINS IN COMPARISON WITH OTHER SYNTHESIS ROUTES

M Djahanbakhsh, W Lojkowski, G Burkle, G Baumann, YV Ivanisenko, RZ Valiev, HJ Fecht - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 175-182 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Mechanical attrition and mechanical alloying have been developed as a versatile alternative to other processing routes in preparing nanophase powder materials with a broad range of chemical compositions and atomic structures. This internal refining process results in a reduction of the average grain size to nanometer scales by the creation and self-organization of small-angle and high-angle grain boundaries within the powder particles. Similar observations have been made on heavily worn surfaces. Due to the large shear stresses at the rail/wheel contact area of high speed trains, a transformation of the pearlitic steel to a nanocrystalline state and the dissolution of carbides is observed. As a consequence, the microhardness is increased by a factor of five corresponding to about 12 GPa. This non-equilibrium material favors the development of corrugations causing considerable acoustic noise and the formation of microcracks leading to a steady devastation of the rail as well as the wheel. A comparison is being made between the physical properties of the nanostructured surface layers of the rail, ball milled powder particles and severely plastically deformed bulk samples with identical composition.

[29] STRUCTURAL CHANGE AND FE-57 MOSSBAUER SPECTROSCOPY OF MECHANICALLY ALLOYED FE₃C AND FE₅C₂

K Tokumitsu, M Umemoto - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 183-188 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

TGA/Book No. BS10Q

The structural change of Fe₃C and Fe₅C₂ by mechanical alloying of their elemental powders was studied with X-ray diffraction and Fe-57 Mossbauer spectroscopy. X-ray diffraction patterns showed the orthorhombic phase Fe₃C in the first stage of milling, but the hexagonal phase in the second stage. Amorphous phase was not obtained in both compositions. Fe-57 Mossbauer spectra changed from α -Fe, H-int.=330kG, to a ferromagnetic sextet, H-int.=205kG, in the first stage and split into two ferromagnetic ones, H-int.=165 and 210kG in the second stage.

[28] PHASE EVOLUTION IN FE-CR-MN-MO POWDERS PROCESSED BY MECHANICAL ALLOYING IN N₂ ATMOSPHERE

H Mancha, G Mendoza, S Belmares, MM Cisneros, JI Escalante, H Lopez, M Mendez - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 189-194 - INTERNATIONAL SYMPOSIUM ON METASTABLE,



MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Nanostructured Fe-18Cr-11Mn-5Mo-xN alloys were prepared by attrition milling of elemental powders under N-2 atmosphere for different times. It was found that the amount of nitrogen present in the powder mixture increased up to 1.6 wt% after 192 h. As-milled powders were encapsulated in quartz tubes and heat treated at 1173 and 1473 K to promote austenisation. Different phases were identified posterior to crystallization namely, ferrite, austenite and Cr₂N, which occurred in different amounts depending on the nitrogen content and the heat-treatment temperature. No nitrogen desorption was perceptible after the crystallization annealing treatment. The results showed that it is feasible to obtain a fully austenitic structure by controlling the processing parameters.

[27] DISORDERING OF B2 INTERMETALLICS BY BALL MILLING, WITH PARTICULAR ATTENTION TO FeAl

DG Morris, X Amils, S Surinach, MD Baro, MA MunozMorris - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 195-202 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Our understanding of the disordering, partial or complete, of B2 intermetallics during intense milling is reviewed choosing the FeAl intermetallic as an example, and making especial effort to interpret the changes occurring in terms of the deformation mechanisms during milling and the crystalline and structural changes produced. The ease of disordering can be correlated with the relative energies of possible point defects produced during milling, or by the corresponding types of dislocations. Attempts at modelling structural changes produced by intense deformation appear in good agreement with detailed structural analyses. Many aspects of the processes occurring are still poorly understood, however, and there remains scope for further investigation.

[26] STRUCTURAL ANALYSIS OF AMORPHOUS GE-SE ALLOYS PREPARED BY MECHANICAL MILLING

T Nasu, F Araki, O Uemura, T Usuki, Y Kameda, S Takahashi, K Tokumitsu - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 203-210

INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

The amorphizing process and short-range structure on amorphous GeSe₂, Ge₃Se₄ and Ge₄Se₅ prepared by the mechanical milling technique (MM) have been investigated through X-ray diffraction and Raman scattering measurements. GeSe₂ becomes amorphous in a shorter milling time. However, a much longer milling time is needed to amorphize Ge-enriched alloys such as Ge₄Se₅. Amorphous GeSe₂ and Ge₃Se₄ prepared by the MM have a similar local order to the corresponding ones prepared by quenching the liquid (LQ), that is, the covalent 4(Ge)-2(Se) folded structure. This was confirmed by the EXAFS measurement at Ge and Se K-edges for amorphous GeSe₂ from the MM and LQ. Amorphous Ge₄Se₅, which cannot be prepared by the LQ, has also the 4-2 folded structure, although its composition is nearer to GeSe, the crystalline structure of which is a distorted NaCl-type with the partial ionicity. The Raman spectrum in amorphous Ge₄Se₅ supported its covalent feature. Bond-lengths of Ge-Se and Ge-Ge involved in the first coordination shell in these alloys were estimated by the least squares fit to the observed diffraction function to be 2.36 Angstrom and 2.48 Angstrom, respectively, which are both composition-independent.

[25] THE IMPACT OF MICROSTRUCTURE OF HIGH ENERGY BALL MILLED POWDERS ON THE REACTION KINETICS

DY Ying, DL Zhang - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 211-216 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

The impact of microstructure on the reaction kinetics during heating of high energy ball milled powders was investigated, using an Al-Ni powder as a typical ductile-ductile system and Al-TiO₂ powder as a typical ductile-brittle system. It was found that with increasing milling time the microstructure of the Al-Ni system developed from Ni particles entrapped in Al matrix structure to Al/Ni multilayer structure; while the microstructure of the Al-TiO₂ system retained the TiO₂ particles entrapped in Al matrix structure. Upon subsequent heating, noticeable reactions occurred only after the melting of the Al rich phase in the entrapped particle structures in both Al-Ni and AlTiO₂ systems, while low temperature reactions occurred in the Al/Ni multi-layer structure. The reaction between Al and TiO₂ occurred in two stages with different mechanisms. The first noticeable reaction between Al and TiO₂ occurred when Al started melting, forming Al₃Ti and Al₂O₃. The formation of solid Al₃Ti and Al₂O₃ hindered the reaction between molten Al and solid TiO₂. Further reaction between Al and TiO₂ only occurred at higher temperatures and the process is likely to be controlled by the diffusion through the solid Al₃Ti and Al₂O₃.

[24] THE RECOVERY AND RECRYSTALLISATION OF A MECHANICALLY ALLOYED ODS-FE3AL ALLOY

J Ritherdon, AR Joens, IG Wright - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 217-222 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Few metallic microstructures evolve to such a great extent during the manufacturing process as do those of mechanically alloyed (MA) materials. From the as-MA powder condition, a primary recrystallised microstructure forms on annealing or



during high temperature consolidation, and subsequent annealing can induce the technologically desirable, coarse secondary recrystallised grain structure. The as-MA and primary recrystallised microstructures are often considered transient and of little importance to most high temperature end applications where the material is usually in the secondary recrystallised condition. However, in any mechanically alloyed material, life-limiting artefacts and defects in the final, consolidated microstructure may originate from the earliest stages of manufacture. The current work involved a microstructural investigation of an oxide dispersion strengthened (ODS) intermetallic Fe₃Al alloy from the as-alloyed, micro-wrought state through recovery and recrystallisation to the primary microstructure. X-Ray Diffractometry (XRD), Transmission Electron Microscopy (TEM) and Scanning Electron Microscopy (SEM) are used to elucidate the microstructural evolution on annealing. The ODS-Fe₃Al powder is seen to begin recovery at approximate to 300 degreesC and to recrystallise at 570 degreesC. There is little grain growth during recovery and XRD data suggests that relaxation of a residual strain of 0.012 occurs on annealing. Comparisons are made between the recrystallisation behaviour and microstructures of the ODS-Fe₃Al alloy and a commercial, Fe-based ODS alloy, PM2000. The microstructure in the commercial PM2000 alloy is generally more homogeneous and contains fewer defects than are apparent in the developmental ODS-Fe₃Al powder, as might be expected, and this highlights aspects of the microstructure and processing of the ODS-Fe₃Al powder which might be altered to achieve enhanced alloy performance.

[23] MECHANICAL PROPERTIES OF NANOCRYSTALLINE TiAl-X AND TiAl₃-X PREPARED BY MECHANICAL ALLOYING AND SINTERING

HA Calderon, V GaribayFebles, A Cabrera, JG CabanasMoreno, M Umemoto - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 229-234 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Production of intermetallic materials in the systems TiAl-X and TiAl₃-X (X = Cr, Mn, Fe) has been achieved by means of mechanical milling and sintering techniques. The produced materials have grain sizes in the nano and microscale depending on the material and processing variables. The average grains size range between 30 and 280 nm. Sintered materials in the TiAl-X system are constituted by the gamma and the alpha (2) phases while the Al₃Ti-X alloys are formed only by the cubic L1(2) phase. Compression tests are performed to evaluate their mechanical properties as a function of temperature and grains size. In all cases yield stresses higher than 1 GPa are obtained together with a ductility that depends upon temperature and grains size. No ductility is found for the smallest grains sizes tested (30 nm).

[22] MECHANICALLY ALLOYED NANOCRYSTALLINE INTERMETALLIC MATRIX COMPOSITES REINFORCED WITH ALUMINA

D Oleszak, M Krasnowski - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 235-240 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

The milling processes were performed for starting powder mixture of composition Fe₂O₃ + 4 Al in order to study the phase transformations leading to the "in situ" formation of a composite 2 FeAl + Al₂O₃. Fritsch and Spex ball mills were applied for the experiments. For the process conducted in a Fritsch mill the reduction reaction of hematite takes place after 20 h of milling. However, the reaction product, beside Al₂O₃, is not ordered FeAl intermetallic compound, but disordered bcc Fe(Al) solid solution, characterized by mean crystallite size of 10 nm and lattice parameter of 0.2931 nm. Heating this sample in the DSC results in the ordering process and formation of FeAl intermetallic compound with mean crystallite size of 40-50 nm. The TEM observations show that the size of crystallites inside the powder particles is not uniform, changing from several tens of nanometers to a few hundreds of nanometers. In the case of milling experiment performed in a Spex mill, the reduction reaction of hematite occurs already after 30 min. Of processing. The obtained disordered bcc Fe(Al) solid solution is characterized by similar values of average crystallite size and lattice parameter as the one formed during milling in a Fritsch device, however the processing time is much shorter.

[21] CONSOLIDATION OF DISPERSION STRENGTHENED NANOSTRUCTURED COPPER VIA HOT EXTRUSION

JB Correia, MT Marques, MM Oliveira, P Matteazzi - METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS, ISMANAM - 2000 (Series: MATERIALS SCIENCE FORUM), 2001, Vol 360-3, pp 241-246 - INTERNATIONAL SYMPOSIUM ON METASTABLE, MECHANICALLY ALLOYED AND NANOCRYSTALLINE MATERIALS (ISMANAM 2000); OXFORD, ENGLAND. JULY 9-14, 2000

Nanostructured copper-based powders were prepared by mechanical synthesis. The powder particle size distribution was characterised by sieving. The microstructure was characterised with optical and scanning electron microscopy. The grain (crystallite) size was determined with X-ray diffraction, using the Scherrer method. The mechanical properties were assessed with microhardness measurements. The stability of the nanostructure was tested with heat treatment up to 800 degreesC. Both the X-ray determination of grain size and the microhardness measurements indicate negligible grain coarsening up to 800 degreesC. Powders were vacuum encapsulated in copper, and extruded at temperatures in the range of 500 to 600 degreesC. The microstructure was again characterised after extrusion. The prior particle boundaries of the powders were usually not seen and porosity was very small. The extrudates showed hardness values of up to 193 VHN. The viability of consolidation of nanostructured copper-based powders by hot extrusion, without significant grain coarsening, was demonstrated. The detailed analysis of the nanostructure, namely with transmission electron microscopy, is still on the way.



[20] THE EFFECTS OF DISLOCATIONS AND GRAIN BOUNDARIES ON THE COARSENING OF AL₄C₃ DISPERSOIDS AT 600 DEGREES C IN TWO MECHANICALLY ALLOYED AL-TI-O-C BASED MATERIALS

Barlow IC. Jones H. Rainforth WM. - Scripta Materialia. 44(7):1089-1093, 2001

[19] MIXED SPINEL STRUCTURE IN NANOCRYSTALLINE NiFe₂O₄

Chinnasamy CN. Narayanasamy A. Ponpandian N. Chattopadhyay K. Shinoda K. Jeyadevan B. Tohji K. Nakatsuka K. Furubayashi T. Nakatani I. - Physical Review B. 6318(18):4108, 2001

Nanocrystalline NiFe₂O₄ spinel has been synthesized with various grain sizes by high-energy ball milling. From the high-field magnetization studies and extended x-ray-absorption fine-structure, and Mossbauer measurements in an external magnetic field of 5 T applied parallel to the direction of gamma rays, we could observe that Ni²⁺ ions occupy tetrahedral sites on grain-size reduction due to milling. The Fe³⁺ spins have a canted structure and the canting angle increases with grain-size reduction. It is possible that the core Fe³⁺ spins are also canted because of the magnetocrystalline anisotropy introduced by the occupancy of the Ni²⁺ ions in the tetrahedral sites

[18] MAGNETIC PROPERTIES OF ULTRAFINE MnFe₂O₄ POWDERS PREPARED BY MECHANOCHEMICAL PROCESSING - ART. NO. 184414

Muroi M. Street R. McCormick PG. Amighian J. - Physical Review B. 6318(18):4414, 2001

Ultrafine MnFe₂O₄ powders have been synthesized via the displacement reaction 2FeCl₃+4MnO --> MnFe₂O₄ + 3 MnCl₂ activated by high-energy ball milling. Single-phase MnFe₂O₄ powders having crystallite sizes ranging between 9.5 and 40 nm have been obtained after removing the by-product (MnCl₂) and unreacted (FeCl₃ and MnO) phases from the as-milled or annealed powders by washing and magnetic separation. Magnetic measurements show that the spontaneous magnetization decreases with crystallite size; that the coercivity increases with decreasing temperature; and that below about 50 K the temperature dependence of coercivity becomes very strong, and the hysteresis loop becomes asymmetric with the remanent magnetization and the coercivity both enhanced in the upper branch when the sample is cooled in the maximum field. These observations are discussed in terms of a model in which it is assumed that each crystallite consists of a ferrimagnetic core surrounded by a spin-glass shell and the spins in the core and shell an exchange coupled

[17] HYDROGEN DESORPTION BEHAVIOR FROM MAGNESIUM HYDRIDES SYNTHESIZED BY REACTIVE MECHANICAL ALLOYING

Gennari FC. Castro FJ. Urretavizcaya G. - Journal of Alloys & Compounds. 321(1):46-53, 2001

The beta- and gamma -phases of MgH₂ were synthesized by reactive mechanical alloying (RMA) at room temperature under hydrogen atmosphere. The structural and desorption properties of the products obtained were examined by X-ray diffraction (XRD). differential scanning calorimetry (DSC), thermogravimetric analysis (TG) and scanning electron microscopy (SEM). Reactive mechanical alloying of Mg leads to the formation of about 50 wt.% of MgH₂ (beta- and gamma -phases) after 50 h of milling. Different thermal behaviors are observed depending on the milling time. Hydrogen thermal desorption shows a sharp endothermic peak for MgH₂, milled up to 30 h associated with beta -MgH₂. For longer milling times, two endothermic peaks (or a double peak) are observed and are associated with hydrogen desorption from the gamma -MgH₂-beta -MgH₂ mixture and the beta -MgH₂ phase. The presence of gamma -MgH₂ destabilizes the beta -MgH₂ phase, reducing its desorption temperature. The results of this study are consistent with hydrogen desorption from gamma -MgH₂ prior to the gamma --> beta -MgH₂ transformation

[16] MECHANICAL ALLOYING AND HYDROGEN STORAGE PROPERTIES OF CaNi₅-BASED ALLOYS

Liang G. Huot J. Schulz R. - Journal of Alloys & Compounds. 321(1):146-150, 2001

CaNi₅-based hydrogen storage materials have been synthesized by mechanical alloying of the elemental Ca and Ni powder blend, followed by an isothermal annealing. It was found that high energy ball milling of a Ca and Ni blend does not result in the formation of the CaNi₅ intermetallic compound. However, DSC measurements show that the CaNi₅ phase forms upon heating the mechanically milled powders to 395 degreesC. Replacement of Ca by Ce or Mm (Mischmetal), and of Ni by Zn leads to direct formation of nanocrystalline alloy phases with CaCu₅ structure. The as-milled nanocrystalline powder does not absorb much hydrogen. A post-milling isothermal annealing at 640 degreesC leads to grain growth and release of microstrain, and to an improvement of the hydrogen storage properties. Replacement of Ca by Ce or Mm increases the plateau pressure, while replacement of Ni by Zn reduces the desorption plateau pressure

[15] EFFECT OF BALL-MILLING ON 3-V CAPACITY OF LITHIUM-MANGANESE OXOSPINEL CATHODES

Kang SH. Goodenough JB. Rabenberg LK.- Chemistry of Materials. 13(5):1758-1764, 2001

Ball-milling for a sufficient period of time of lithium-manganese oxide spinels, prepared either by a sol-gel method or by solid-state reaction, gave cathode materials of a lithium cell with excellent capacity retention in the 3-V range at room temperature and elevated temperatures despite an apparent Jahn-Teller distortion. In a 50-cycle test with 0.5 mA/cm², a sample ball-milled for 1 h gave a constant capacity of 122 mAh/g at a constant voltage 2.9 V versus lithium. X-ray diffraction and transmission electron microscopy were performed on the ball-milled spinel powders; they were found to contain significant strain and to consist of nanometer-scale grains. Furthermore, partial oxidation of manganese ions took place during the ball-milling process.

[14] EFFECT OF ANTIMONY OXIDE ON MAGNESIUM VANADATES FOR THE SELECTIVE OXIDATION OF HYDROGEN SULFIDE TO SULFUR

Li KT. Chi ZH. - Applied Catalysis B-Environmental. 31(3):173-182, 2001

The effect of antimony oxide addition to MgV₂O₆ and Mg₃W₂O₈ was studied in the selective oxidation of hydrogen sulfide to sulfur. Significant improvements in sulfur selectivity and yield were observed for the uncalcined mechanical mixtures of magnesium vanadates with alpha -Sb₂O₄. Calcination of the mechanical mixtures resulted in the much stronger synergy in catalytic activity and sulfur selectivity. For the uncalcined samples, XRD, TPR and XPS studies indicated that antimony reduction behaviors in the mechanical mixtures differed very much from those in alpha -Sb₂O₄ alone, suggested that their selectivity improvements might be due to the interactions (probably oxygen transfer) between alpha -Sb₂O₄ and magnesium vanadates. For the calcined samples, XRD results indicated that their better catalytic performances in H₂S oxidation were primarily attributed to the formation of VSbO₄ compound from antimony oxide and magnesium vanadates

[13] CA-10(PO₄)₆(OH)₂ SYNTHESIS ASSISTED BY MECHANOCHEMICAL ACTIVATION



Zakharov NA. Orlovskii VP. Kluev VA. Toporov YP.- Russian Journal of Physical Chemistry. 75(5):852-854, 2001
The effect of a preliminary mechanochemical activation of the components in the $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ solid-phase synthesis from the $\text{CaO} + \text{CaHPO}_4$ and $\text{CaCO}_3 + (\text{NH}_4)_2\text{HPO}_4$ was studied. The crystallographic parameters of the products, their spectroscopic and dielectric characteristics were determined. The effect of the composition of the initial components on the rate of the synthesis was examined

[12] EXAFS STUDY FOR MECHANICAL ALLOYED FE80C20

Yang DS. Yoo YG. Yu SC. - Journal of the Physical Society of Japan. 70(4):1002-1005, 2001

The local structure has been examined by the extended X-ray absorption fine structure (EXAFS) for metastable Fe80C20 alloys produced by the mechanical alloying process. The order between Fe atoms in the alloys decreased continuously as the process time increased. The substantial changes of the local structure appears in about G hours milling time and the amorphization occurs between 6 and 12 hours milling time. The magnetization was interpreted by the variation of the local structure.

[11] INFERENTIAL MEASUREMENT OF SAG MILL PARAMETER'

Apelt TA. Asprey SP. Thornhill NF. - Minerals Engineering. 14(6):575-591, 2001

Semi-autogenous (SAG) mill total load and ball load have a marked influence on mill performance. Direct measurement of these inventories is difficult, hence inferential measurement is an attractive option to open the way to their control. Advances have been made in this area based on combined state and parameter estimation formulations, though there is still scope for further soft sensor development. This work investigates the use of mill powerdraw and weight simulation models combined with the corresponding measured variable (to form residual equations) to obtain estimates of total volumetric load fraction, $J(b)$ and ball charge volumetric fraction, $J(b)$, by the application of a constrained nonlinear optimisation technique. The sensitivity of the estimates to model parameters and the uncertainty in the estimates are investigated. Results show that, although all estimates illustrated good agreement with nominal conditions, the estimates from the weight-based models contained the least uncertainty. The inclusion of a mill finer wear model in combined state and parameter estimation formulations is recommended due to the notable contribution of the liner weight to the mill weight measured variable

[10] TREATMENT OF A REFRACTORY GOLD-COPPER SULFIDE CONCENTRATE BY COPPER AMMONIACAL THIOSULFATE LEACHING

Alymore MG. - Minerals Engineering. 14(6):615-637, 2001

The potential for leaching gold directly from a refractory Au-Cu sulfide concentrate, to replace the current treatment of roasting and cyanidation, was investigated using ammoniacal thiosulfate with copper. Leaching the concentrate with a solution containing 0.8M $\text{S}_2\text{O}_3^{2-}$, 0.05M Cu^{2+} and 4M NH_3 resulted in over 90% gold and 60% silver recovery. Evidence of dissolution of gold tellurides was observed and contributed to the high gold extraction from the ore. However the leaching of gold tellurides was slow and high concentrations in a concentrate would be expected to reduce recovery. There was no evidence of partial alteration of pyrite in the leach which would suggest that gold inclusions present in pyrite grains will not be recovered until some pretreatment process is carried out to liberate these inclusions. Pre-treating ore by ultra fine milling followed by thiosulfate leaching resulted in 94% gold and 60% silver recovery after 48 hours. Thiosulfate leaching of roasted concentrate resulted in 98% gold and 93% silver recovery after 24 hours. Under the conditions used in this study, reagent consumption was high ranging from 36 to 70 kg/t for thiosulfate, similar to 5 kg/t for ammonia and 1 to 2 kg/t for copper after leaching for 24 hours. Extended leaching resulted in higher reagent consumption and loss of gold and silver from solution. Preliminary investigation into generating thiosulfate in-situ were undertaken using sulfur dioxide, together with sulfur and ammonia to leach gold directly from concentrate or roasted products. Up to 1.2 M of thiosulfate was generated with gold extraction of 80% for highly refractory concentrate and > 98% for roasted samples. The cost of high reagent consumption observed could be overcome by using sulfur dioxide gas produced from the roaster sulfur and ammonia to leach gold directly from ore or roasted products

[9] A NEW METHOD SYNTHESIZING THE ENCAPSULATED ZRC WITH GRAPHITIC LAYERS

Dong J. Shen WC. Liu X. Hu XF. Zhang BF. Kang FY. Gu JL. Li DS. Chen NP. - Materials Research Bulletin. 36(5-6):933-938, 2001

A new method has been found to synthesize the filled carbon particles, which is different from other methods for synthesizing the encapsulated carbides. It involves high energy milling the carbon with ZrO_2 ball and succedent heat treatment in 1800 degreesC. In addition, a particle with sunken surface is observed while surface of others are convex

[8] GRAIN GROWTH AND KINETICS FOR NANOCRYSTALLINE MAGNESIUM ALLOY PRODUCED BY MECHANICAL ALLOYING

Cao P. Lu L. Lai MO. - Materials Research Bulletin. 36(5-6):981-988, 2001

A study on the kinetics of grain growth of an Mg-12.1 wt%Cu alloy produced by mechanical alloying was carried out. The grain sizes of as-mechanically alloyed powder and of cold-compacted annealed powder were determined from the broadening of X-ray lines. The grain size decreases initially due to recrystallization and then increases gradually, and finally ceases to reach an ultimate value regardless of annealing time. From isothermal anneals, the grain growth kinetics can be described by $D - n - D - 0(n) = ct$, where n ($n = 5$ to 8) is a constant essentially dependent on the annealing temperature. The activation energy for grain growth Q has been determined to be 118 kJ/mol, which is longer by 26 kJ/mol than that for pure magnesium. Second-phase intermetallic particle Mg_2Cu produced during ball-milling influences not only on activation energy but also on exponent of the kinetic equation

[7] ONE-STEP SYNTHESIS AND CONSOLIDATION OF NANOPHASE IRON ALUMINIDE



Bernard F. Charlot F. Gaffet E. Munir ZA. - Journal of the American Ceramic Society. 84(5):910-914, 2001

The simultaneous synthesis and densification of nanophase iron aluminide is investigated. Elemental nanophase reactants produced by mechanical activation were reacted by field activation with the simultaneous application of uniaxial pressure. The process was demonstrated in this work by the synthesis of dense nanometric FeAl. Iron and aluminum powders were co-milled in a specially designed planetary mill to obtain nanometric reactants and to avoid formation of any product phases. These powders were then subjected to high AC currents (1250-1500 A) and pressures in the range of 70-106 MPa. Under these conditions, a reaction was initiated and completed within a short period of time (2.5-3.5 min). XRD and energy dispersive analyses showed the product to be Fe_{0.515}Al_{0.485}. The relative density of the product ranged from 98.7% to 99.7%. The crystallite size of the "FeAl" intermetallic was determined by XRD line-broadening analysis using the Williamson-Hall method. The size ranged from 32 to 89 nm and showed a dependence on lattice direction

[6] THE USE OF METAL HYDRIDE POWDER BLENDING IN THE PRODUCTION OF NDFEB-TYPE MAGNETS

Mottram RS. Davis B. Yartys VA. Harris IR. - International Journal of Hydrogen Energy. 26(5):441-448, 2001

The ability to vary the composition of NdFeB-type magnets, during or after the milling stage offers a number of advantages. To this end, powder blending processes have been developed. In the present work, the sintering characteristics of a Nd₁₃Fe_{80.5}B_{6.5} alloy have been improved by powder blending with neodymium hydride. A blending addition of 1 at% of neodymium in the form of Nd-hydride, consistent with a final composition of Nd₁₄Fe_{79.6}B_{6.4}, was sufficient to reduce the required sintering temperature from 1130 to 1070 degreesC, with a resultant improvement in the coercivity due to less grain growth and better grain isolation. The addition of dysprosium hydride to a Nd₁₄Fe₇₉B₇ alloy by blending resulted in a further enhancement of the coercivity, a 2 at% addition yielding an approximate doubling in the value of intrinsic coercivity. Examination of the microstructure of dysprosium-hydride blended magnets showed the dysprosium to be concentrated in the outer regions of the matrix grains, with the centres being essentially dysprosium-free. Hydrogen has also been employed in improving the milling characteristics of the high melting point elements niobium and vanadium. Fine powders of niobium and vanadium hydride were prepared and were successfully blended into magnets with an even distribution throughout the microstructure. These studies have shown that powder blending of metal hydrides is an effective way of both promoting liquid-phase sintering in low rare earth composition magnets and of modifying the grain boundary phases and hence the magnetic properties.

[5] STUDY OF MG-M (M = CO, NI AND FE) MIXTURE ELABORATED BY REACTIVE MECHANICAL ALLOYING: HYDROGEN SORPTION PROPERTIES

Bobet JL. Akiba E. Darriet B. - International Journal of Hydrogen Energy. 26(5):493-501, 2001

Reactive Mechanical Alloying (MA in H₂) Of magnesium powder allows to obtain partial conversion of Mg to MgH₂. The ratio MgH₂/Mg can be improved by increasing the milling time or by adding a 3d-element (with the same initial particle size). X-ray diffraction was applied successfully to determine the quantity of MgH₂ formed. The RMA allows to suppress the activation procedure generally requested for magnesium. The hydriding is determined to be a two step process: first nucleation and then diffusion. A direct relationship exists between the nucleation duration and the specific surface. It is demonstrated that RMA for short time (2 h) is an effective way to strongly improve the hydrogen storage properties of both magnesium and (Mg + 10 wt% M) mixtures

[4] THE EFFECT OF HYDROGEN INCORPORATION IN THE NANOCRYSTALLINE IRON PARTICLES ON THEIR MAGNETIC PROPERTIES

Authors Novakova AA. Kiseleva TY. Agladze OV. Perov NS. Tarasov BP. - International Journal of Hydrogen Energy. 26(5):503-505, 2001

The effect of hydrogen on the structure and magnetic properties of nanocrystalline iron powders was investigated by XRD, Mossbauer spectroscopy and magnetic measurements. The nanocrystalline Fe powders were produced by two methods: iron hydroxide reduction in hot hydrogen flow and high-energy ball milling of carboxylic iron in hydrogen atmosphere during 8 h. The formation of nanocrystalline state with mean grain size 13 nm was obtained in both cases. Hydrogen incorporation in the grain boundaries was observed in the nanocrystalline ball-milled iron. Formation of oxy-hydroxide amorphous surface layer was observed in "reduced"

[3] PARTICLE FRACTURE AND PLASTIC DEFORMATION IN VANADIUM PENTOXIDE POWDERS INDUCED BY HIGH ENERGY VIBRATIONAL BALL-MILL

Chatterjee P. Sen Gupta SP. Sen S. - Bulletin of Materials Science. 24(2):173-180, 2001

An X-ray powder profile analysis in vanadium pentoxide powder milled in a high energy vibrational ball-mill for different lengths of time (0-250 h), is presented, The strain and size induced broadening of the Bragg reflection for two different crystallographic directions ([001] and [100]) was determined by Warren-Averbach analysis using a pattern-decomposition method assuming a Pseudo-Voigt function, The deformation process caused a decrease in the crystallite size and a saturation of crystallite size of similar to 10 nm was reached after severe milling. The initial stages of milling indicated a propensity of size-broadening due to Fracture DF the powder particles caused by repeated ball-to-powder impact whereas with increasing milling time microstrain broadening was predominant. WA analysis indicated significant plastic strain along with spatial confinement of the internal strain fields in the crystallite interfaces, Significant strain anisotropy was noticed in the different crystallographic directions. A near-isotropy in the crystallite size value was noticed for materials milled for 200 h and beyond, The column-length distribution function obtained from the size Fourier coefficients progressively narrowed down with the milling time



[2] ROOM TEMPERATURE ACID EXTRACTION OF CO FROM LiCo_{0.2}Ni_{0.8}O₂ SCRAP BY A MECHANOCHEMICAL TREATMENT

Zhang QW. Lu JF. Saito F. Nagata C. Ito Y. - Advanced Powder Technology. 11(3):353-359, 2000.

Room temperature extraction of valuable substances from LiCo_{0.2}Ni_{0.8}O₂ scrap containing polyvinylidene fluoride (PVDF) has been carried out using 1 N HNO₃ solution after mechanochemical (MC) treatment by a planetary mill with and without Al₂O₃ powder. Crystalline LiCo_{0.2}Ni_{0.8}O₂ in the scrap was pulverized and became amorphous by MC treatment for 60 and 240 min, respectively, with and without Al₂O₃ powder. This shows that the addition of Al₂O₃ is very effective for MC treatment. Accordingly, Co as well as Ni and Li were extracted at a high yield of more than 90% from the amorphous scrap sample. About 1% of fluorine in the PVDF was dissolved in the filtrate when the Al₂O₃ powder was added to the scrap during the MC treatment, while no fluorine was detected in the filtrate obtained from the ground scrap sample without Al₂O₃ powder

[1] EFFECTS OF MILLING RAW MATERIALS AND SLURRY CONCENTRATION ON THE SYNTHESIS OF MAGNESIUM TITANATE

Hamada K. Yamamoto SI. Senna M. - Advanced Powder Technology. 11(3):361-371, 2000.

Wet milling by a multi-ring media mill was carried out for aqueous slurries of Mg(OH)₂ and TiO₂ powder mixtures in the concentration range from 6.9 to 50 wt%. Rings used as grinding media were made of partially stabilized zirconia or polyoxymethylene. Products were characterized by thermogravimetry-differential thermal analysis. X-ray diffractometry and EPMA. ZrO₂ contamination in the product was determined by X-ray fluorescence analysis. The temperature of endothermic peaks of dehydration remained almost unchanged regardless of slurry concentration but decreased with increasing compressive force exerted by grinding rings. The homogeneity of the product is correlated with the temperature of exothermic peaks above 1100 K due to the crystallization of MgTiO₃, but not with the endothermic peaks around 650 K. The increase of the slurry concentration increases the homogeneity of the product, facilitating the formation of single-phase MgTiO₃ on subsequent heating. The increase of the compressive force of grinding rings brings about not only the homogeneity but also the phase purity of MgTiO₃



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COMMUNIQUE DE PRESSE : Un rhéomètre pour l'évaluation des propriétés d'écoulement de la poudre

Ce rhéomètre, breveté en Grande-Bretagne, permet de résoudre bon nombre des problèmes associés au traitement de la poudre humide et sèche dans les domaines de la recherche, du développement et de la production.

Grâce à un principe des plus novateurs, le **rhéomètre à poudre FT3 de Freeman Technology** évalue les propriétés d'écoulement des poudres et des semi-solides. De fait, la fluidité se mesure à la quantité d'énergie requise pour induire un état d'écoulement dynamique. Ainsi, avec le FT3, les mesurages peuvent être répétés quasiment à l'infini et les procédures d'essai et d'analyse sont automatisées, d'où un gain de temps précieux et l'absence de toute intervention humaine.

Le traitement des poudres se révèle souvent être une opération ardue, en raison des nombreux facteurs qui influent sur les propriétés d'écoulement, comme la vitesse de coulée, la compaction, la ségrégation, l'attrition, l'adhésivité et la fluidification. Or, le rhéomètre à poudre FT3 est capable de classer les poudres en mesurant la dépendance de leur fluidité vis-à-vis de chacun de ces facteurs.

Le mesurage de la fluidité est généralement compliqué par les variations observées en termes de tassement. La solution apportée par le FT3 à ce problème est un procédé de conditionnement, qui produit une densité de tassement uniforme et reproductible préalablement aux essais de fluidité. Il est ainsi possible de comparer, de façon parfaitement fiable, des mesures prises à différents moments et en différents lieux.

Outre les essais programmés, des routines d'homogénéisation entièrement programmables peuvent être conçues en vue d'homogénéiser des poudres sèches ou des mélanges poudre-liquide, voire des matières plus complexes encore, telles la farine et l'eau. Grâce à ces routines, des programmes complexes peuvent être définis de bout en bout et, le cas échéant, répétés à volonté. Une analyse énergétique complète du programme d'homogénéisation est également possible.

Les applications du FT3 sont diverses, depuis les études de formulation en recherche et développement (R&D) jusqu'à l'évaluation des effets d'attrition dans un procédé de fabrication donné, en passant par la définition de critères de fluidité aux fins du contrôle qualité (CQ). Et ces applications présentent de grands avantages puisqu'elles permettent, entre autres, de réduire les délais d'élaboration des produits nouveaux, de limiter le nombre des arrêts de production et d'améliorer le contrôle de la qualité des matières premières comme des produits finis.



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