

RESEAU FRANÇAIS DE MECANOSYNTHESE

Lettre N° 56

Novembre 1999

162(+2) Groupes de Recherche
(dont 90 (+2) à l'étranger / 34 Pays)

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3 Nouvelles Adhésions

J.M. Porto Lopez - INTEMA - Division Ceramicos - Mar Del Plata - Argentina
Alexander Patselov - High Pressure Physics Lab - Ekaterinburg - Russie
Vitaliy Pilyugin - High Pressure Physics Lab - Ekaterinburg - Russie

Le site web du RFM est :
<http://www.bls.fr/amatech>

Rubrique Pages Sciences et Techniques pour l'Ingénieur (Rubrique Sciences)
vous y trouverez les anciennes lettres du RFM (accessible par Adobe Acrobat)
les statuts du RFM ainsi que les annonces concernant les JRFM'99 et quelques éléments mis à jour régulièrement
concernant les derniers résultats dans ce domaine.

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Web Sites

- Pour ceux intéressés par le devenir du Rayonnement Synchrotron en France / Europe,
un site intéressant et particulièrement actif ces dernières semaines
- People interested by the Synchrotron Radiation Development in France / Europe

Website : <http://www.Lure.u-psud.fr/actus/avenir>

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JRFM'2000
5 èmes Journées du Réseau Français de Mécanosynthèse
Bordeaux-Pessac (CNRS-ICMCB)
Les 23 et 24 Mai 2000

Thématique 2000 : Mécanosynthèse : caractérisation Physico-Chimiques

Fiche d'Inscription : (à renvoyer à Jean-Louis BOBET le plus tôt possible, avant le 31.01.2000)

Nom : _____ Prénom : _____
Téléphone : _____ Fax : _____ E-mail : _____
Adresse de Facturation : _____

Pour les laboratoires propres ou associés au CNRS, indiquer le code et l'adresse complète de l'Unité.
Une facture sera établie directement à leur rencontre.

Pour les agent CNRS merci d'**indiquer votre indice majoré** :
(nécessaire pour obtenir un discount pour les dÈjeuners†!)

Frais d'inscription : (comprenant†: recueil des résumés, 2 déjeuners, 1 dîner)

Déjeuner du 23 mai oui / non

Banquet du 23 mai soir† oui / non (Prière d'entourer les réponses)

Déjeuner du 24 mai† oui / non

Tarifs

Etudiant(e)s en thèse : 100 FF Autres participant(e)s : 400 FF

Pour les fournisseurs et autres exposants, contacter directement E. Gaffet

Mode de paiement :

Dès réception du bulletin d'inscription dûment complété par vos soins (ou d'un bon de commande de votre organisme), une facture sera établie pour paiement : aucun règlement n'est à effectuer à l'inscription.

Rappel : le CNRS gérant ce colloque, il est fortement conseillé de privilégier le paiement par virement administratif du CNRS.

(-----)

Fiche d'inscription et résumés à retourner le plus tôt possible et avant le 31/01/2000 :

Jean-Louis BOBET (Journées RFM2000)

CNRS - Institut de Chimie de la Matière Condensée de Bordeaux

Av du Dr A. Schweitzer - 33608 Pessac Cedex

(Tel : 05.56.84.26.53 - fax : 05.56.84.24.80, E-mail : bobet@icmcb.u-bordeaux.fr)

Date limite d'Envoi des Résumés : 30 Avril 2000

Publications : Après sélection, les articles proposés pourront être publiés dans Journal of Metastable and Nanocrystalline Materials (J. Metastable & Nano. Mater. - Trans Tech Publ.)

Editors : A.R. Yavari, A. Inoue, R. Schulz, D. Morris

Int. Editorial Board, : J.H. Ahn, H. Bakker, M.D. Baro, R. Bormann,, A. Calka, G. Cocco, J. Eckert, E. Gaffet, S. Gialenella, A. Hernando,, A. Inoue, C. Koch, G. Le Caer, M. Magini, D. Morris, P.H. Shingu,, L. Schultz, R. Schulz

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IMPORTANT :

Les frais d'inscription ne comportent pas l'adhésion au RFM au titre de l'année 2000.

Pour participer aux JRFM2000, il est impératif d'envoyer votre cotisation de 100 FRF à l'ordre du RFM.

(Pour tout renseignements complémentaires sur cette adhésion contacter E. Gaffet).

Comité d'organisation

Jean-Louis Bobet

et toute l'équipe "Détermination structurale et matériaux magnétiques"

CNRS - ICMCB

(UPR 9048 CNRS - Université Bordeaux1)

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&

Eric Gaffet

Président du Réseau Français de Mécanosynthèse

Groupe "Nanomatériaux"

Université de Technologie de Belfort - Montbéliard -UTBM

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Tél : 03 84 58 31 02 - Fax : 03 84 58 30 27

E-mail : Eric.Gaffet@utbm.fr

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**ANNONCE DE CONGRES ET / OU ECOLES
CONGRESS AND SCHOOL ANNOUNCEMENTS**

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

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

J.A. 99 / SF2M

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

EURO PM99

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

MRS Fall Meeting 99

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

**PROCESSING AND CATALYTIC/CHEMICAL PROPERTIES
OF NANOSTRUCTURED MATERIALS**

January 16-21, 2000
Maui, Hawaii

Sintering 2000

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

Ultrafine Grained Materials

Strengthening, Fracture and Creep of Nanostructured Materials Symposia
12 - 16 Mars - TMS Annual Meeting - Nashville TN - USA
Contact : RSMIOSHRA@ucdavis.edu

**Nanomatériaux :
Vers les Applications Industrielles
Nanomaterials :
Towards Engineering Applications**

Colloque : France - Etats Unis - Canada
17-19 Mai 2000 - Montréal, Canada
Contacts : Champion@glvt-cnrs.fr et/ou Eric.Gaffet@utbm.fr

JRFM'2000

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

III European Conference on Fluidization

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

PM2 TEC2000

2000 International Conference on Powder Metallurgy & Particulate Materials
31 Mai - 3 Juin 2000 - New York - USA
Contact : MPIF

4th EUROMECH

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

ISMANAM 2000

International Symposium on Metastable Mechanically Alloyed and Nanocrystalline Materials
9 - 14 Juillet 2000 - St Catherines College - Oxford UK

E Mail :

ismanam2000@materials.ox.ac.uk

website : <http://www.materials.ox.ac.uk/Conferences.ISMANAM2000.html>

NOUVEAU

NCM8
**8th International Conference on the Structure of
Non - Crystalline Solid**

6 - 11 Aout 2000

Website : <http://www.sgt.org>

XIVth International Symposium on the Reactivity of Solids

Budapest, Hungary through 27-31 August 2000

<http://www.jate.u-szeged.hu/~isrs14>.

Solid State Chemistry 2000

Prague, Czech Republic, September 3 - 8,2000

and

3rd INCOME

International Conference on Mechanochemistry and Mechanical Alloying

Prague, Czech Republic, September 4 - 8,2000

Organised by the Institute of Inorganic Chemistry (UACH), Czech Republic

WebSite : <http://www.iic.cas.cz/INCOME.htm>

PM 2000

**Powder Metallurgy World
Congress & Exhibition**

12 - 16 Novembre 2000 Kyoto - Japon

Contact : Fax : 81 - 3 - 3423 - 1600

**The 1st International Conference
on Advanced Materials Processing,
Rotorua, New Zealand, 19-23 November 2000.**

Secretary, ICAMP 2000,

NOUVEAU

Department of Materials and Process Engineering The University
of Waikato

Private Bag 3105, Hamilton, New Zealand

Fax: 64-7-838 4835, e-mail: d.zhang@waikato.ac.nz

Or visit the conference web site:

<http://mape.waikato.ac.nz/conferences/amp.htm>

PM2 TEC2001

**2001 International Conference on Powder Metallurgy
& Particulate Materials**

13 - 17 May 2001 - New Orleans - USA

Contact : MPIF

**The 1st International Conference on Advanced Materials Processing,
Rotorua, New Zealand, 19-23 November 2000.**

Scope of the Conference:

In the last few decades of the 20th century, research in advanced materials processing has made a tremendous contribution to the development of novel, high performance and low cost materials. Without any doubt, in the new millennium, advanced materials processing will continue to be one of the most active research areas in materials science. To celebrate the historical achievement of scientists and engineers in this crucially important area, we are organizing the 1st International Conference on Advanced Materials Processing (ICAMP) in the very first year of the new millennium. Sponsored by the Institute of Materials, UK, this conference will provide a forum for international researchers to exchange their recent findings and views in the area of advanced materials processing. Internationally renowned experts in this area will be invited to give keynote lectures to the conference.

During the conference, a workshop will also be organized to discuss opportunities for international co-operation in research on advanced materials processing.

Key dates:

Preliminary Title/Expression of Interest: As soon as possible Second Circular: October 31, 1999

Abstract due by March 1, 2000

The conference will be held in Rotorua, New Zealand. Rotorua is in the central North Island, and holds many attractions, including the world famous thermally active reserves.

Areas of Interest include:

Casting, Electronic materials, Extrusion, Filament Winding, Fuel cells, Hot compaction
HIP, Joining, Mechanical Alloying, Moulding, Powder Metallurgy, pre-Preg Production
Pultrusion, Rapid Solidification, Recycling, Repair, Sintering Processes
Solid State Chemistry, Tape Casting, Thermoset Matrix Composites, Thin Film Deposition
Traditional Ceramic Processes

International Advisory committee:

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For further information or pre-registration please contact:

Dr Deliang Zhang
Secretary, ICAMP 2000,
Department of Materials and Process Engineering The University of Waikato
Private Bag 3105, Hamilton, New Zealand
Fax: 64-7-838 4835, e-mail: d.zhang@waikato.ac.nz

Or visit the conference web site:

<http://mape.waikato.ac.nz/conferences/amp.htm>

SOUTENANCE DE THESE

F.Charlot

Etude et compréhension des réactions auto-entretenuées activées mécaniquement. Elaboration du composé FeAl nanostructuré.

Déc. 1999 - Université de Technologie de Belfort - Montbéliard

Nanomatériaux : Elaboration et Transitions de Phases Hors Equilibre, UPR 806 CNRS, UTBM - Sévenans.

Matériaux à Grains Fins, LRSS UMR 5613 CNRS-Université de Bourgogne.

Jury (Provisoire) : G.LeCaer, F.Thévenot, F.Bernard, E.Gaffet, J.C.Gachon, M.Bessière, M.Gailhanou

L'objectif de ce travail est d'obtenir à partir d'un procédé de synthèse non conventionnel, le procédé MASHS, un matériau nanostructuré et dense. Ce procédé appliqué à la synthèse du composé FeAl, repose sur deux étapes : (i) une première étape dite d'activation mécanique (MA) qui est un prébroyage énergétique de courte durée conduisant à des poudres précurseurs nanostructurées. (ii) la seconde étape consiste à synthétiser des composés ayant une enthalpie de formation négative par une réaction SHS (Self-propagating High-temperature Synthesis. Une partie du travail a été, dans un premier temps, d'étudier l'influence des différents paramètres contrôlant ce procédé MASHS et, dans un second temps, de comprendre les chemins conduisant à ce matériau nanostructuré à partir d'un dispositif original autorisant le suivi in-situ et en temps réel des réactions de combustion en utilisant la diffraction des rayons X (rayonnement synchrotron) couplée à une thermographie infrarouge.

Il a été mis en évidence plusieurs modes d'activation mécanique, le mode de friction et le mode choc direct grâce au contrôle des conditions de broyage appliquées. Il a été clairement démontré que l'utilisation de poudres activées mécaniquement (friction ou mode direct) entraîne une diminution de la température d'amorçage d'au moins 100° C de la réaction de combustion ainsi qu'une vitesse de front de propagation multipliée par 4/5. Ces différentes modifications sont liées à l'existence de la nanostructure induite au cours du broyage. Une des autres conséquences importantes est l'obtention d'échantillons chimiquement homogènes. Les études in-situ en temps réel ont mis en valeur deux phénomènes, le premier est l'existence d'une phase liquide qui implique que la réaction est de type solide-liquide, le second est l'observation d'une phase transitoire, la forme allotropique gamma du fer.

Bien que le procédé MASHS permettent d'obtenir en une seule étape la phase désirée nanostructurée, un des points faibles reste le problème de la densification (densité relative comprise entre 70 et 80 %). Un procédé expérimental a été développé, en collaboration avec Z.A.Munir, permettant de synthétiser, de densifier et d'obtenir une nanostructure en une seule étape. Ces résultats permettent maintenant d'aller vers la compréhension de la granulo-dépendance des propriétés mécaniques.

The purpose of this work is to obtain a densified nanostructured materials with the application of a new process, the MASHS process. This process is applied to elaborate the FeAl compound. This ones involves mainly the combination of two steps; the first step, a mechanical activation, where pure elemental powders were co-milled to obtain precursor nanometric mixture, the second step, a Self-propagating High-temperature Synthesis (SHS) reaction, which uses the exothermicity reaction. A first step is to determine the parameters which control the different steps of MASHS reaction. The second step is to study the reactional mechanisms with an in-situ in real time study using X-ray diffraction (synchrotron beam) coupled with an infrared thermography. We have demonstrated that different kinds of mechanical activation exists (friction mode, direct shock mode), using the mechanically activated powders, the ignition temperature decreases (more than 100° C), the velocity of the combustion front increases (x 4/5) and the product are more homogeneous. The in-situ in a real time investigation shows the existence of a liquid phase, the reaction is solid-liquid, and an intermediate phase, the gamma iron. The MASHS process doesn't allow to obtain a nanodense material. A following work, with the collaboration of Z.A.Munir, allows to synthesize and densify in a one step a nanostructured materials.

O. Held

"Etude des réactions de combustions solide-solide ou solide liquide auto-entretenuées pour différents intermétalliques du système Al-Ni-Ti.

Elaboration de ces mêmes intermétalliques par broyage mécanique et étude de leur cinétique de cristallisation

Nancy, Faculté des Sciences, le 5/11/99.

Jury : J. L. Jorda, J. M. Moreau, P. Satre, J. C. Gachon, F. A. Kuhnast, F. Bernard, J. F. Bézar, M. Bessière.

D. Cracco

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

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

A. Gentil - Sagot

**Amélioration de la tenue au fluage d'un alliage d'argent (AIC)
par introduction d'une dispersion d'oxydes.
Elaboration par Métallurgie des Poudres**

Ecole des Mines - Paris - 17 Juin 1999

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

Cooperative Research on Related Areas

COREE du SUD (6 May 1999)

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

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

We are very pleased to discuss for international cooperative research on above research topics with Member of Mecanosynthese Group
E-Mail : ShHong@Sorak.kaist.ac.kr

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

NOUVEAU

***** Postdoctoral Position Available for a Ph.D Physicist (13th October 1999)**

The High-Pressure (HP) group of the School of Physics & Astronomy at Tel Aviv University has a one year position available. This position could be extended to two years.

The HP group is known worldwide as one of the leading groups in experimental HP physics and is on the frontier of HP and diamond-anvil-cells based methodology. The main methods used are:

Mössbauer spectroscopy

X-ray diffraction with synchrotron radiation (ESRF, Grenoble) resistivity

Applicants must be between the final stage of dissertation and within three years of receipt of Ph.D diploma.

For more information, please contact:

Dr. Moshe P. Pasternak

School of Physics and Astronomy

Tel Aviv University

69978 Tel Aviv, ISRAEL

email: hh136@ccsg.tau.ac.il

***** Looking for Job Position (6th July 1999)**

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

For more information please contact: ph./fax +61-3-95433002 or email: Jonian.Nikolov@Minerals.CSIRO.au

****** Proposals**

FRANCE (2/07/99)

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

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

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

FRANCE (6/06/99)

P. Bracconi (Univ. Dijon) propose une position de Post Doc en Métallurgie des Poudres, de nationalité autre que française (Poste Fléché CNRS)

Contact : pbrac@u-bourgogne.fr

GRECE (11/06/99)

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

ISRAËL (14/5/99)

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

Denmark (22/02/99)

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

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

Québec (CANADA) (22/01/99)

Institut National de la Recherche Scientifique
Département Energie et Matériaux

POST-DOCTORAL POSITION IN Ni-MH BATTERY TECHNOLOGY

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

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

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

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

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

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

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

Bibliographie Récente

Livres ou "Special Issues"

"Non Equilibrium Processing of Materials"

R.W. Cahn - Elsevier Science - Volume 2 in the Pergamon Materials Series

A large number of technical papers have been published in reviews, monographs and conference proceedings, but have almost always been devoted to a single processing technique. This book, however, covers all the non equilibrium processing methods and their effects in a single volume.

web site : www.elsevier.nl/locate/isbn/0080426972

Bulk Amorphous Alloys : Preparation and Fundamental Characteristics

A. Inoue

Materials Science Foundation Vol. 4 - Trans Tech Publications : <http://www.ttp.net>

Interest in bulk amorphous alloys has increased rapidly throughout the world and these materials have now gained a position of great importance in basic science and engineering materials technology. Bulk amorphous alloys based upon the Zr - Al - Ni - Cu, Zr (Ti,Nb) - Al - Ni - Cu and Zr - Ti - Ni - Cu - Be systems have already achieved wide commercial success as components of various technical accessories ranging from sporting goods to optical instruments.

Here is a state of the art reviews on this new group of materials, covering all areas of interest, ranging from the synthesis of these special alloys and their fundamental properties, to their engineering characteristics and applications.

This work will therefore be of equal interest to those who wish to become fully acquainted with the subject, and to those who are already actively engaged in the field.

DISPERSION-STRENGTHENED ALUMINIUM PREPARED BY MECHANICAL ALLOYING

Michal Besterci, Institute of Materials Research, Slovak Academy of Sciences, Kosice

In the book, the author describes the theoretical and technological fundamentals of mechanical alloying the Al-C system. Special attention is given to material characteristics, the kinetics and mechanism of mechanical alloying, methods of mixture compaction and heat treatment of compacted parts. Models of dispersoid spatial arrangement, dispersoid evaluation and optimisation and experimental possibilities are discussed. The interpretation of the static and dynamic mechanical properties, especially strength and ductility properties at 20 °C, mechanical properties at elevated temperatures are discussed, with emphasis on the effect of interface, superplasticity, creep and creep-fatigue characteristics. Content

Introduction

1. Characteristics of dispersion-strengthened systems

2. Mechanical alloying (kinetics and mechanism of preparation of the Al-C system by mechanical alloying; compaction of powders and heat treatment of compacts;

3. Microstructure and quantitative evaluation of parameters of dispersion-strengthened materials (definition and properties of interparticle distance; experimental possibilities of determination of structural objects; models of heterogeneous structures and their evaluation; simulation of model structures; analysis of the spatial distribution of particles in the Al-Al4C3 material) 4. Static and dynamic mechanical properties (mechanical properties at elevated temperatures; mechanical properties at 20°C; effect of interface on the mechanical properties; superplastic properties of the system; thermal stability of the system; creep characteristics; creep-fatigue characteristics)

References

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

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

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

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

"Mechanical Alloying"

Auteurs : Li Lü & Man On Lai (National University of Singapore)

Kluwer Academic Publishers

Contents : Preface - Introduction to Mechanical Alloying - Experimental Set - Up - The Mechanical Alloying Process - Formation of New Materials - Characterization of Powders - Densification - Mechanical Properties - Mechanisms of Mechanical Alloying - Modeling of Mechanical Alloying - Index

"Surface-Controlled Nanoscale Materials for High-Added-Value Applications"

Editors: Kenneth E. Gonsalves, Marie-Isabelle Baraton, Rajiv Singh, Heinrich Hofmann, Jerry X. Chen, and Joseph A. Akkara.

Materials Research Society, Symposium Proceedings Volume 501, 1998

MRS, Warrendale, Pennsylvania, USA (website: <http://www.mrs.org/>)

"Nanomatériaux"

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

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

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

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

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

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

CHEMISTRY FOR SUSTAINABLE DEVELOPMENT Vol. 6, No. 2-3, MARCH-JUNE 1998

Proceedings of 2d International Conference on Mechanochemistry
(INCOME-2), which was held in Novosibirsk in 1997.

Contact : Prof. • N.Z. Lyakhov, Inst. Sol. State Chem.- Russian Acad Sci. - Kutaleladze, 18 - Novosibirsk - 630128 Russia - The Proceedings will be available by the price 80 USD.

Mechanochemistry of Materials Cambridge International Science Publishing

Emmanuel Gutman - Materials Eng. Dpt - Ben Gurion University - Beer Sheva - Israel

Considerable advances have been made in mechanochemistry in the last couple of decades. Training of experts in this field with a background in materials science, chemical and mechanical engineering, etc. requires study of the fundamentals of mechanochemistry. There is a need for a textbook in the general and compressed form which would cover many aspects and would be used as a basis for understanding the fundamental principles to control mechanochemical phenomena. This textbook is based on lectures given by Prof. Gutman in a graduate course in the mechanochemistry of materials at the Ben - Gurion University of the Negev. The book contains examples of experimental results to illustrate the mechanochemical phenomena and technologies.

BIBLIOGRAPHY ON MECHANICAL ALLOYING AND MILLING

Suryanarayana (Inst for Materials and Advanced Processes, University of Idaho, USA)

The present bibliography covers information on mechanical alloying and milling of materials starting from 1970 (when it was recognized that MA has become a commercial/viable material processing technique instead of just a grinding method) to 1996. All the available references will be presented in a chronological fashion. Under each year, (.....)

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Proceeding du Congrès "Mechanically Alloyed, Metastable and Nanocrystalline Materials"- Barcelone (1997)

Editor : M.D. Baro, S. Surinach - Materials Science Forum 269 - 272 (1998)

PERIODIQUES

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

[91] STRUCTURAL CHANGE BY MILLING IN A MIXTURE OF CUO AND CR2O3 AS A CATALYST FOR LOW-TEMPERATURE METHANOL SYNTHESIS VIA METHYLFORMATE

S Ohyama, H Kishida - SCIENCE AND TECHNOLOGY IN CATALYSIS 1998 (Series: STUDIES IN SURFACE SCIENCE AND CATALYSIS), 1999, Vol 121, pp 431-434

3RD TOKYO CONFERENCE ON ADVANCED CATALYTIC SCIENCE AND TECHNOLOGY; TOKYO, JAPAN. JULY 19-24, 1998

Low-temperature methanol synthesis via methyl formate was carried out in a liquid medium at 373 K and an initial pressure of 5 MPa using alkali alkoxide and a physical mixture of CuO and Cr₂O₃ as a catalyst. Methanol productivity was enhanced with prolonged milling of the mixture. Characterization of the mixtures by physical adsorption of N₂, X-ray diffraction (XRD), transmission electron microscopy with elemental analysis (TEM/EDX) and X-ray absorption fine structure (XAFS) suggested that long-term milling brought about intimate contact of fine grains of CuO and Cr₂O₃, and lattice disorder in the crystals, especially in CuO crystallites, both of which contribute to an increase in catalytic activity.

[90] ENHANCED SUPERPLASTICITY OF ULTRAFINE-GRAINED ALLOYS PROCESSED BY SEVERE PLASTIC DEFORMATION

RZ Valiev, RK Islamgaliev - TOWARDS INNOVATION IN SUPERPLASTICITY II (Series: MATERIALS SCIENCE FORUM), 1999, Vol 304-3, pp 39-46 - 2ND INTERNATIONAL CONFERENCE ON TOWARDS INNOVATION IN SUPERPLASTICITY; KOBE, JAPAN. SEPTEMBER 21-24, 1998

Nanocrystalline alloys - a boron doped Ni₃Al, intermetallic and Al alloy 1420 (Al-5.5%Mg-2.2%Li-0.12%Zr), as well as submicrocrystalline aluminium alloy 1420 were processed by two severe plastic deformation methods, namely, torsion straining under high imposed pressure and equal channel angular pressing (ECAP). The processed ultrafine-grained (UFG) alloys exhibit enhanced superplasticity at relatively low temperatures and/or high strain rates. From TEM/HREM results the features of superplastic behaviour in UFG alloys and the requirements to achieve enhanced superplasticity are discussed.

[89] NANO-GRAIN FORMATION IN A FULLY FERRITIC SUS 316L AUSTENITIC STAINLESS STEEL PRODUCED BY HS-PM PROCESS

H Fujiwara, K Ameyama - TOWARDS INNOVATION IN SUPERPLASTICITY II (Series: MATERIALS SCIENCE FORUM), 1999, Vol 304-3, pp 47-52 - 2ND INTERNATIONAL CONFERENCE ON TOWARDS INNOVATION IN SUPERPLASTICITY; KOBE, JAPAN. SEPTEMBER 21-24, 1998

In this paper, the high strain powder metallurgy (HS-PM) process was applied to an SUS 316L stainless steel powder, and the microstructural changes during the process are discussed. The HS-PM process is applied to an SUS 316L austenitic stainless steel. In the case of the SUS 316L stainless steel, room temperature recrystallization and recovery of an austenite phase take place because of the increased high angle grain boundary area and the existence of excess vacancies, which are stored during the milling process. Very fine ferrite grains are formed in the early stage of the milling. Although the amount of the alpha' martensite is not so large even after 98% cold rolling of the parent material, the extremely high density of defects introduced by the milling process increased free energy of the austenite phase, and thereby stabilized the alpha phase. In the late stage of milling, or in the case of higher energy milling, fully ferritic nano grain structure with an average grain size of approximately 20nm is formed. During heating, the fully ferritic powder showed complex microstructural changes. The powder compact sintered at 1173K for 3.6ks showed (alpha'+M₂C) microduplex structure with an average grain size of 250nm.

[88] GRAIN REFINING DURING HEAVY DEFORMATION IN FE-C ALLOYS WITH (ALPHA+THETA) TWO-PHASE STRUCTURE

H Hidaka, T Suzaki, Y Kimura, S Takaki - TOWARDS INNOVATION IN SUPERPLASTICITY II (Series: MATERIALS SCIENCE FORUM), 1999, Vol 304-3, pp 115-120 - 2ND INTERNATIONAL CONFERENCE ON TOWARDS INNOVATION IN SUPERPLASTICITY; KOBE, JAPAN. SEPTEMBER 21-24, 1998

Mechanical milling using high energy ball mill was applied to Fe-(0-3.8)mass%C alloy powders with (ferrite+cementite) two-phase structures to give an ultimate large strain to the powders. Relation between strength and microstructure was investigated in the mechanically milled Fe-C alloys, by means of S-ray diffractometry, transmission electron microscopy (TEM) and hardness testing. After milling for 360ks, the microstructure consists of nanocrystalline ferrite and grain boundary amorphous layer in the alloys with less than 2mass%C, while it is composed of nanocrystalline ferrite, nanocrystalline cementite and grain boundary amorphous layer in the alloys with more than 2mass%C. Below 1mass%C ferrite grains become smaller with increasing carbon content, and the grain size remains constant to be 10nm above 1mass%C. Hardness of the Fe-C alloy powders increases with grain refining, but it is independent of the volume fraction of undissolved cementite. TEM observation indicates that these alloy powders have equiaxed grains, which is characterized by the substructure having few dislocations within the grains. These findings suggest the outset of deformation in grain boundary amorphous layer, i.e., grain boundary sliding, in the nano-sized grain materials.

[87] TEMPERATURE DEPENDENCE OF TENSILE ELONGATION IN A MECHANICALLY MILLED, P/M AL-MG-CU ALLOY

T Hasegawa, K Okazaki - TOWARDS INNOVATION IN SUPERPLASTICITY II (Series: MATERIALS SCIENCE FORUM), 1999, Vol 304-3, pp 249-254 - 2ND INTERNATIONAL CONFERENCE ON TOWARDS INNOVATION IN SUPERPLASTICITY; KOBE, JAPAN. SEPTEMBER 21-24, 1998

A mechanically milled and thermo-mechanically treated Al-1.1Mg-1.2Cu (at%) alloy exhibits superplastic elongation at an intermediate temperature of 748K, neither at higher nor lower temperatures, and a large portion of total elongation is accounted for by the grain deformation. Computer simulation of stress-strain behavior from a viewpoint

of dislocation dynamics indicates that a combination of the largest re-mobilization probability of immobile dislocations, the highest mobile dislocation density and the modest immobilization rate of dislocations is responsible for the largest elongation at 748K.

[86] MECHANICAL ALLOYING

Suryanarayana C. - NON-EQUILIBRIUM PROCESSING OF MATERIALS. 2 PG. 49-85. 1999

[85] HYDRIDING PROPERTIES OF THE MgNi-H(D) SYSTEM WITH AMORPHOUS SINGLE PHASE - INVESTIGATION FROM A VIEWPOINT OF THE SHORT RANGE ORDERING [JAPANESE]

Orimo S. Ikeda K. Fujii H. Saruki S. Fukunaga T. - Journal of the Japan Institute of Metals. 63(8):959-964, 1999

The purpose of this work is, first, to get precise information about the thermal stability and the site-occupation of hydrogen (deuterium) in the MgNi-H(D) system with an amorphous single phase, and then to clarify the origin of its hydriding properties from a viewpoint of the short range ordering. The sample was prepared by a mechanical alloying (milling) method, and the structural and hydriding properties were characterized by X-ray and neutron diffraction measurements, and by electrochemical p-c(hydrogen pressure-composition) isotherm measurement. The following results were obtained; (1) The amorphous single phase was prepared by milling for 80 h, after the solid-phase reaction between elemental Ni and nanostructured Mg₂Ni. (2) The p-c isotherm clearly shows that there is a miscibility-gap (plateau), even in the amorphous phase. The hydrogen pressure of the miscibility-gap is located around 3×10^{-4} MPa at room temperature. (3) Hydrogen (deuterium) occupies the tetrahedral site composed of nearly [2Mg₂Ni] in a wide range of hydrogen compositions. (4) The origin of their notable hydriding properties is the short range ordering in the amorphous single phase similar to the CsCl-type cubic structure. The single miscibility-gap in the system is also of importance.

[84] EFFECT OF MECHANICAL GRINDING UNDER ARGON AND HYDROGEN ATMOSPHERES ON STRUCTURAL AND HYDRIDING PROPERTIES OF LaNi₅ [JAPANESE]

Munehiro S. Fujii K. Fujii H. Orimo S. - Journal of the Japan Institute of Metals. 63(8):970-976, 1999

Effect of mechanical grinding (MG) under argon and hydrogen atmospheres on structural and hydriding properties were investigated in detail. In MG-LaNi₅ under argon atmosphere, the grain size reaches similar to 20 nm for MG times of 60 min and almost remains unchanged for more longer MG times. The pressure-composition isotherm (P-C) in LaNi₅ at room temperature indicates an enhancement of hydrogen solubility, a lowering of plateau pressure and a narrowing of width of plateau by MG like FeTi but not Mg₂Ni. On the other hand, in reactive MG (RMG)-LaNi₅ under hydrogen atmosphere, a nanocrystallized LaNi₅H_{0.15} and amorphous phases coexist within 180 min of grinding time. For RMG times longer than 180 min, the nanostructured LaNi₅H_{0.15} phase disappears and the remaining amorphous-LaNi₅H_x dissociates into nano-Ni + LaNi_yH_z (y < 5). The P-C isotherm indicates no-plateau in the LaNi₅ produced by RMG longer than 60 min and the hydriding properties become worse and worse with increasing RMG times. From these results, we conclude that the hydriding properties could not be improved by structural modifications in the system containing metals with strong affinity for hydrogen like rare earth metals.

[83] THERMAL AND MECHANOCHEMICAL SELF-PROPAGATING DEGRADATION OF CHLORO-ORGANIC COMPOUNDS: THE CASE OF HEXACHLOROBENZENE OVER CALCIUM HYDRIDE

Cao G. Doppio S. Monagheddu M. Orru R. Sannia M. Cocco G. - Industrial & Engineering Chemistry Research. 38(9):3218-3224, 1999

We report on the highly exothermic solid-state reaction between hexachlorobenzene and calcium hydride. Once ignited by a thermal spike, the reaction displays a self-sustaining character in the CaH₂/C₆Cl₆ molar ratio of 3:18. The high temperatures reached, i.e., 2550-2900 K, ensure a complete breakdown of the aromatic molecule and of undesired chloro-organic congeners, with only inorganic halide salts being found among the end-products. Combustive-like reactions were also observed when reactant powders were subjected to intensive mechanical treatment by ball milling. The combustive range of mechanically driven processes falls within that found in the true self-sustaining regime even if the activation and the extinction of the reaction were ruled by completely different mechanisms. A neat correlation was worked out relating the temperatures at the combustion front to the total heat evolved in the reaction carried out in the mechanochemical mode. The same end-products were also found. The practical exploitation of a self-sustaining methodology for the disposal of hazardous organochlorine compounds seems feasible.

[82] TRANSFORMATIONS OF ORGANIC COMPOUNDS UNDER THE ACTION OF MECHANICAL STRESS [REVIEW] [RUSSIAN]

Dubinskaya AM. - Uspekhi Khimii. 68(8):708-724, 1999

Transformations of organic compounds (low-molecular-weight and polymeric) under the action of mechanical stress are considered. Two groups of processes are shown to occur. The first group involves disordering and amorphisation of crystal structure and conformational transformations as a result of rupture of intermolecular bonds. The second group includes mechanochemical reactions activated by deformation of valent bonds and angles under mechanical stress: the cleavage of valent bonds, oxidation and hydrolysis. Data on the organic mechanochemical synthesis of new compounds or molecular complexes are systematised and generalised. It is demonstrated that mechanical action provides the mass-transfer and contact of reacting species in these reactions. Proteins are shown to be especially sensitive to mechanical stress and undergo denaturation; enzymes undergo inactivation.

[81] PREPARATION OF SIMULATED INERT MATRIX FUEL WITH DIFFERENT POWDERS BY DRY MILLING METHOD

Lee YW. Kim HS. Kim SH. Joung CY. Na SH. Ledergerber G. Heimgartner P. Pouchon M. Burghartz M. - Journal of Nuclear Materials. 274(1-2):7-14, 1999

Simulated zirconium oxide-based inert matrix fuel (IMF) has been prepared using cerium oxide replacing plutonium oxide, following the conventional pelletising route, in which an improved two-stage attrition mill was employed. Different methods have been applied for the powder preparation. In addition to powder mixing, nitrate solutions have been co-precipitated to bulk gel and to microspheres by the internal gelation process. The calcined porous microspheres were crushed in the attrition mill with a few passes. This co-precipitated powder was compared with

three different mixtures of the four commercially available powders (ZrO₂, Y₂O₃, Er₂O₃ and CeO₂). Pellets of a standard size with relative densities higher than 90% TD and comparable grain and pore structures have been obtained. Based on these simulation tests both processes are found suitable for fabricating IMF pellets, which fulfil requirements of a commercial plutonium and uranium mixed oxide (MOX), where applicable. No intensive milling is required to form a solid solution in the sintering step for a (Zr,Y,Er,Ce)O_{2-x} pellet. For the fabrication of plutonium-containing IMF, a similar behaviour can be expected.

[80] STRUCTURE AND MAGNETIC PROPERTIES OF ND-FE-B POWDERS PREPARED BY HYDROGEN DECREPITATION

Kosobudskii ID. Sevost'yanov VP. Kuznetsov MV. - *Inorganic Materials*. 35(9):909-911, 1999

The magnetic and structural properties of Nd-Fe-B powders prepared via hydrogen decrepitation and the performance parameters of the magnets produced from these powders were studied. Hydrogen decrepitation, combined with vibration and disintegrator milling, can be useful in the production of permanent magnets since it reduces the particle size of the powder, improves the performance of the sintered magnets, and raises the effectiveness of the fabrication of permanent magnets.

[79] MECHANOCHEMICAL SYNTHESIS OF LEAD TITANATE

Zyryanov VV. - *Inorganic Materials*. 35(9):935-940, 1999

The structural properties of the PbTiO₃ materials with the perovskite structure prepared mechanochemically in a planetary mill were studied. The phase composition of the starting mixture was found to have a strong effect on the yield, composition, and structure of the resulting perovskite material. The experimental results lend support to conclusions of the theory of mechanochemical processes in powders: the threshold character of the formation of crystalline products, significant deviations from stoichiometry, high vacancy concentration, excess of the softer component in the reaction product, and the crucial effect of the difference in hardness on the yield of the perovskite phase. The large-sized ions Pb²⁺ are found to occupy B sites in the perovskite structure, which are typically occupied by small-sized cations. Annealing the lead titanate prepared by the mechanochemical process from minium and TiO₂ gives rise to the formation of a phase which is incommensurate at room temperature, with $a^* = 4 \sqrt{2} k_a$, and is likely to result from ordering of the small-sized cations Pb⁴⁺ in A sites of the perovskite structure.

[78] THE INFLUENCE OF TRIBOPHYSICAL ACTIVATION ON SOLID-STATE REACTION DURING THERMAL TREATMENT OF ZNO-CR2O3

Marinkovic BA. Zakula ZV. Sreckovic T. Tomasevic M. Ristic MM. - *Crystal Research & Technology*. 34(7):881-889, 1999

Tribophysical (mechanical) activation is used as a method for modification of physico-chemical properties of dispersed systems such as polycrystalline mixture of ZnO and Cr₂O₃ powders, obtained by conventional ceramic powder processing wet technique. Conventional mixtures were tribophysically activated by grinding in a vibro-mill with a continual regime in air. Conventional mixture and tribophysically activated mixtures, with activation times of 40 and 80 minutes, were thermally treated in non-isothermal and isothermal conditions. Solid-state reactions in non-isothermal conditions were examined by X-ray diffraction, differential thermal analysis and dilatometric measurements. The influences of tribophysical activation on solid-state reaction between ZnO and Cr₂O₃, during thermal treatment in isothermal conditions were investigated by X-ray diffraction line broadening theory. As a Fourier method for the pure physical line profile analysis a double-Voigt method is used. Mechanism of mass transport during solid-state reaction in ZnO-Cr₂O₃ system is proposed.

[77] MICRONIZATION OF PHARMACEUTICAL SUBSTANCES IN A SPIRAL JET MILL

Midoux N. Hosek P. Pailleres L. Authelin JR. - *Powder Technology*. 104(2):113-120, 1999

Many studies have been conducted to help understand the effects of the variables involved in jet milling. The first part of this work is an attempt to summarise the results published in the literature concerning horizontal and vertical jet mills. This focuses on the research of the optimal design of the mills and on the improvement of their performance. Several publications have been found, concerning mineral grinding. It seemed more interesting to present results on organic crystals' jet milling in the second part of this paper. The experiments concern three organic substances, and were run on three different spiral jet mills: Chrispro-Jetmill 50 and 100 and MicronMills 8 ". The results are presented in terms of specific energy consumption with an adaptation of the correlation proposed by Sarma. These representations show that, within the operating energy range and above a critical energy value, the creation of specific surface area corresponds to an increase of fine particles production.

[76] EFFECT OF MILLING ON THE THERMAL BEHAVIOUR OF POLY(ETHYLENE TEREPHTHALATE)

Font J. Muntasell J. Cesari E. - *Thermochimica Acta*. 333(2):169-172, 1999

The solid state amorphization of poly(ethylene terephthalate), applying mechanical milling, is described. Their effect on samples quenched or slowly cooled after the melt as well as in samples with different crystallinity degrees, attained from annealing treatments, is characterized using differential scanning calorimetry and X-ray diffraction at room temperature. In previously annealed poly(ethylene terephthalate) at several temperatures some differences appear in the amorphization after milling. However, effects other than the crystallinity degree have to be taken into account to explain them.

[75] NANO-SCALE METAL MULTILAYERS PRODUCED BY REPEATED PRESS-ROLLING

Shingu PH. Yasuna K. Ishihara KN. Otsuki A. Terauchi M. - *Radiation Effects & Defects in Solids*. 148(1-4):555-562, 1999

Multilayer structured alloys which have a layer thickness less than 10 nm have been produced by the application of repeated pressing and rolling to the initially macroscopically thick layered sample. The TEM observation of a Ag/Fe sample revealed a regular alternate lamination of Ag and Fe with the layer thickness in the range of 10 nm. The measured electrical resistivity showed a large dependence on the applied magnetic field which is characteristic to the composite structure of magnetic and non-magnetic metals in the thickness range of nanometers.

[74] THE MICROSTRUCTURE AND MAGNETIC PROPERTIES OF NANOCOMPOSITE FE-X(SIO2)(1-X) MATERIALS

Xiong CS. Yu KN. Xiong YH. - Nanostructured Materials. 11(4):477-486, 1999

Nanocomposite Fe-x(SiO₂)(1-x) samples were prepared using high energy ball milling. The microstructure and magnetic properties of these samples with different Fe contents and different ball milling time were studied using X-ray diffraction (XRD), transmission electron microscopy (TEM), Mossbauer spectroscopy, and magnetic balance over a wide temperature range. The results indicated that the microstructure and magnetic properties were closely related to the ball milling time and Fe contents. When the Fe content was less than 20 wt%, the sample after ball milling 80 hours demonstrated a very complex microstructure. The small alpha-Fe grains and Fe clusters were implanted in the SiO₂ matrix, and there were also nanometer-scale sandwich network-like structures. The nanocomposite Fe_x(SiO₂)_{1-x} samples also displayed the change of magnetic properties, which results from their unique nanostructure, the grain size effect, the strong interface interaction and the interface-osmosis effect at the Fe-SiO₂ boundaries.

[73] SHORT RANGE STRUCTURE OF AMORPHOUS SE100-XASX PREPARED BY MECHANICAL ALLOYING

Fukunaga T. Kajikawa S. - Journal of Non-Crystalline Solids. 250(Part 1):384-388, 1999

Amorphous Se_{100-x}As_x samples were synthesized by mechanical alloying. Short range structure for these samples during amorphization process was observed by neutron diffraction. When the amorphization proceeds, the coordination number of the nearest Se atoms around a Se atom decreased with increasing milling time and approached 1.91 +/- 0.02 atoms for pure Se. Moreover, the coordination number of As-As and Se-Se pairs decreased and that of the As-Se pair increased to be 2.63 +/- 0.03 atoms after 150 hours of milling of the Se(60)A(40) sample. These results definitely indicate that a number of dangling bonds exist in the amorphous Se_{100-x}As_x prepared by mechanical alloying, in comparison with those prepared by liquid quenching.

[72] LOCAL ORDER IN AMORPHOUS FE2ZR PREPARED BY MECHANICAL ALLOYING AND MECHANICAL GRINDING

Bionducci M. Navarra G. Bellissent R. Concas G. Congiu F. - Journal of Non-Crystalline Solids. 252(Part 2):605-610, 1999

Amorphous Fe₂Zr samples were prepared by mechanical grinding from the intermetallic crystalline phase and by mechanical alloying from elemental powders. The structural changes during milling were monitored by X-ray diffraction. Mossbauer absorption permitted us to follow the transition from the magnetically ordered phase of iron to the paramagnetic state of the final amorphous products. X-ray and neutron diffraction, coupled with Reverse Monte Carlo simulation, were used to extract the partial structure factors for the two samples. The results for the two alloys are compared and discussed.

[71] AMORPHOUS PHASE FORMATION DURING COLD ROLLING OF AL-SM AND ZR-CU-NI-AL MULTILAYER STRUCTURES

Sieber H. Wilde G. Sagel A. Perepezko JH. - Journal of Non-Crystalline Solids. 252(Part 2):616-620, 1999

The early stages of the mechanically activated solid state amorphization reaction (SSAR) were investigated in multilayered samples prepared by cold rolling of elemental foils of Al₉₂Sm₈ and the bulk glass forming alloy Zr₆₈Cu₁₈Ni₉Al₅. The microstructural development in the cold rolled samples during the mechanical alloying process was measured by X-ray diffraction, scanning, and transmission electron microscopy at different deformation levels to compare the amorphization reaction for these different glass forming systems. The mechanical alloying process first yields the formation of a multilayered structure with layer thicknesses below 100 nm. During continued rolling, the elemental grain size decreases (to approximately 50 nm for Zr₆₈Cu₁₈Ni₉Al₅ and 20-30 nm for Al₉₂Sm₈) and the formation of amorphous phases starts in localized regions of the samples. In the final stage of the rolling process the Zr₆₈Cu₁₈Ni₉Al₅ sample transforms completely to the amorphous state.

[70] THE CRYSTALLIZATION PROCESS OF AMORPHOUS AL80FE20 ALLOY POWDERS PREPARED BY BALL MILLING

Zhou F. Luck R. Scheffer M. Lang D. Lu R. - Journal of Non-Crystalline Solids. 252(Part 2):704-708, 1999

The amorphous-to-crystalline transition during thermal annealing of amorphous Al₈₀Fe₂₀ alloy powders prepared by ball milling was investigated by means of magnetothermal analysis and X-ray diffraction. Following structural relaxation and stress relief processes, a metastable phase precipitates during primary crystallization, and a mixture of intermetallic compound (theta-Al₁₃Fe₄) and fcc Al is developed further. On heating at constant rate annealing the first and succeeding reactions overlap to some extent in temperature. The apparent activation energy for primary crystallization amounts to 1.99 +/- 0.10 eV by using the Kissinger method. In addition, the structure of the metastable phase, which has not been identified yet, is briefly discussed.

[69] STRUCTURAL AND MAGNETIC STUDY OF CRYSTALLINE FE80NI20 ALLOYS WITH NANOMETER-SIZED GRAINS

Pekala M. Oleszak D. Jartych E. Zurawicz JK. - Journal of Non-Crystalline Solids. 252(Part 2):757-761, 1999

Samples of the Fe₈₀Ni₂₀ alloys were synthesized in the low energy conventional horizontal and high energy planetary ball mills. Both in the low and high energy processes, the solid bcc Fe(Ni) solutions with a similar structure are formed. The lattice constants are slightly larger than for the pure iron. Thermal stability of alloys was measured upto 1100 K. The magnetization and ferromagnetic Curie temperatures confirm a ferromagnetic ordering. The magnetic moment equal to 2.07 mu(B) for the longest milling periods proves that the electronic structure of the crystalline alloy with nanometer-sized grains does not differ from that of polycrystalline samples. The distributions of the hyperfine magnetic fields revealed that an iron atom has one to two Ni atoms in the nearest neighborhood.

[68] HYDROGENATION PROPERTIES OF COMPLEX ALKALI METAL HYDRIDES FABRICATED BY MECHANO-CHEMICAL SYNTHESIS

Zaluski L. Zaluska A. Strom-Olsen JO. - Journal of Alloys & Compounds. 290(1-2):71-78, 1999

Mechano-chemical synthesis was used to produce complex alkali metal hydrides. Various complex hydrides have been synthesized, for example Li₃AlH₆, Na₃AlH₆, (Li-Na)(3)AlH₆, (Li-Na-B)(3)AlH₆. Complex alkali metal hydrides fabricated by mechano-chemical processes exhibit very good reactivity in hydriding/dehydriding reactions. They have excellent cyclability and cover a wide range of operational temperatures and pressures.

[67] KINETICS AND FORMATION MECHANISM OF AMORPHOUS FE52NB48 ALLOY POWDER FABRICATED

BY MECHANICAL ALLOYING

El-Eskandarany MS. Bahgat AA. Gomaa NS. Eissa NA. - Journal of Alloys & Compounds. 290(1-2):181-190, 1999
A single phase amorphous Fe₇₀Nb₃₀ alloy has been synthesized through a solid state interdiffusion of pure polycrystalline Fe and Nb powders at room temperature, using a high-energy ball-milling technique. The mechanisms of metallic glass formation and competing crystallization processes in the mechanically deformed composite powders have been investigated by means of X-ray diffraction, Mossbauer spectroscopy, differential thermal analysis, scanning electron microscopy and transmission electron microscopy. The numerous intimate layered composite particles of the diffusion couples that formed during the first and intermediate stages of milling time (0-56 ks), are intermixed to form amorphous phase(s) upon heating to about 625 K by so-called thermally assisted solid state amorphization, TASSA. The amorphization heat of formation for binary system via the TASSA, Delta H-a, was measured directly as a function of the milling time. Comparable with the TASSA, homogeneous amorphous alloys were fabricated directly without heating the composite multilayered particles upon milling these particles for longer milling time (86 ks-144 ks). The amorphization reaction here is attributed to the mechanical driven solid state amorphization. This single amorphous phase transforms into an order phase (mu phase) upon heating at 1088 K (crystallization temperature, T-x) with enthalpy change of crystallization, Delta H-x, of -8.3 kJ mol⁻¹.

[66] CALORIMETRIC AND STRUCTURAL ANALYSIS OF THE NEW PHASE AL₃₃NI₁₆ZR₅₁ PRODUCED BY DIRECT SYNTHESIS AND MECHANICAL ALLOYING

Held O. Braganti JP. Kuhnast FA. - Journal of Alloys & Compounds. 290(1-2):197-202, 1999

A new phase has been synthesized in the ternary phase diagram Al-Ni-Zr: its nominal composition is Al₃₃Ni₁₆Zr₅₁. For the Al₃₃Ni₁₆Zr₅₁ compound obtained by mixing the three components in suitable proportions, a study has been carried out by direct synthesis (calorimetry) and mechanical alloying in our laboratory. With the first method we know directly the enthalpy of formation of this alloy. For the amorphous alloys prepared by mechanical alloying we can determine the crystallisation enthalpy with the differential scanning calorimetry (DSC) method. So it is possible to determine a fundamental piece of information: the amorphous alloy formation enthalpy.

[65] FORMATION OF CELSIAN FROM MECHANICALLY ACTIVATED BaCO₃-Al₂O₃-SiO₂ MIXTURES

Boskovic S. Kosanovic D. Bahloul-Hourlier D. Thomas P. Kiss SJ. - Journal of Alloys & Compounds. 290(1-2):230-235, 1999

A ternary mixture BaCO₃-Al₂O₃-SiO₂ was mechanically activated for different lengths of time. Chemical composition of the mixture corresponded to BaAl₂Si₂O₈-BAS As a function of activation time, reaction course was followed in the temperature range 750-1200 degrees C. Reaction of celsian formation was followed using thermogravimetry as well as conventional and high-temperature X-ray diffractometry. The obtained data show that reaction rate increases with prolonged activation time, under the same conditions of thermal treatment. Formation of hexacelsian via a series of solid state reactions involving Ba-silicates, was favoured with increasing activation time. Direct formation of monoclinic celsian was retarded, however, with prolonged activation,

[64] MECHANICALLY ALLOYED MMNI(5)-TYPE MATERIALS FOR METAL HYDRIDE ELECTRODES

Jurczyk M. Rajewski W. Majchrzycki W. Wojcik G. - Journal of Alloys & Compounds. 290(1-2):262-266, 1999

Nanocrystalline MmNi(5)-type alloys have been prepared by mechanical alloying (MA) followed by annealing. The structural and electrochemical properties of these materials have been investigated at room temperature. The amorphous phase forms directly from the starting mixture of the elements, without other phase formation. Heating the MA powders at 1070 K for 1 h resulted in the formation of the hexagonal CaCu₅-type structure. For nanocrystalline MmNi(4.2)Al(0.8) material discharge capacities of 207 mAh g⁻¹ were obtained.

[63] INFLUENCE OF PROCESS CONTROL AGENT ON INTERDIFFUSION BETWEEN AL AND MG DURING MECHANICAL ALLOYING

Lu L. Zhang YF. - Journal of Alloys & Compounds. 290(1-2):279-283, 1999

The influence of a process control agent (PCA) on the reaction rate during mechanical alloying has been studied. Mechanical alloying of Al-Mg powder mixtures with different PCAs was carried out using a planetary ball mill. X-ray diffraction spectra showed that a small amount of PCA might lead to fast interdiffusion and reaction between Al and Mg although the crystalline sizes were in general the same for the powders when using different amounts of PCA. The fast interdiffusion and advanced reaction are closely associated with the diffusion distance between the powder particles due to the presence of PCA and to the actual weight ratio of ball to powder.

[62] SOME OBSERVATIONS ON MECHANICAL ACTIVATION OF THE CeO₂-ZrO₂-SiO₂ SYSTEM

Kiss SJ. Zec S. Boskovic S. - Journal of Alloys & Compounds. 290(1-2):L1-L2, 1999

Mechanical activation of CeO₂-ZrO₂-SiO₂ mixtures was performed in a high energetic vibratory mill with WC lining and grinding media. It was shown that in air atmosphere the reduction of Ce⁴⁺ into Ce³⁺ took place at 1400 degrees C. The Ce³⁺ ion is stable in air in the form of Ce-2(WO₄)₃ contrary to the Ce³⁺ ion in Ce-silicates.

[61] LATTICE PARAMETER CHANGES ON DISORDERING INTERMETALLICS

Cahn RW. - Intermetallics. 7(10):1089-1094, 1999

Most intermetallic compounds have long-range atomic (chemical) order. This can sometimes be destroyed, in some instances by quenching from a high temperature, in others by mechanical milling or irradiation. The destruction of the superlattice is normally accompanied by an increase in lattice parameter. This paper addresses the question whether this lattice parameter increase for binary intermetallic compounds is better correlated with the ordering energy or with the radius mismatch of the constituent atoms; the available evidence is surveyed. That evidence suggests that the ordering energy is the more significant variable, and this can be interpreted in terms of bond shortening associated with strong "unlike" bonds when an intermetallic acquires long-range order.

[60] EFFECT OF CARBON CONTENT ON MECHANOREDUCTION OF COPPER OXIDE

Liu L. Yuan SL. Dong YD. Cui K. - Chinese Physics Letters. 16(8):591-593, 1999.

The reduction of CuO with different amount of carbon (CuO:C = 2:1, 2:1.5, and 2:2, in molar ratio) induced by ball milling is examined by x-ray diffraction and transmission electron microscopy. It is found that reduction process is closely related to the carbon content. CuO can be completely converted into Cu if an excessive C is used (i.e., C = 1.5

or 2.0 mol). However, the reduction of CuO follows a two-step process, $\text{CuO} \rightarrow \text{Cu} \rightarrow \text{Cu}_2\text{O}$, if the mixture only contains a nominal C content (i.e., C = 1 mol). A tentative model in terms of solid state reaction at the interfaces is proposed to explain the effect of carbon content on the mechanoreduction of CuO.

[59] HIGH-RESOLUTION TRANSMISSION ELECTRON MICROSCOPY (HRTEM): IMAGE PROCESSING ANALYSIS OF DEFECTS AND GRAIN BOUNDARIES IN NANOCRYSTALLINE MATERIALS

Tonejc A. - Acta Chimica Slovenica. 46(3):435-461, 1999

A brief overview of the application of the high resolution transmission electron microscopy (HRTEM) method is given: the principle of HRTEM image formation in an electron microscope; different modes of image formation; the role of contrast transfer function (CTF) in HRTEM image formation; definition and improvement of electron microscope resolution. As an example, the image processing of HRTEM micrographs of mechanically alloyed ZrO₂-10mol% Y₂O₃ powders was performed in order to obtain information about alloying and transformation at the atomic level. In this work the results obtained by applying the CRISP program to analyse the HRTEM photographs of mechanically alloyed nanocrystalline materials are presented. The investigation is focused on the following regions: a) the grain boundary region; b) the region of stacking faults; c) the region of overlapping layers of zirconia and yttria. Fourier filtering revealed at the atomic level one possible sequence of alloying that occurred at the grain boundary, on the stacking faults and in the overlapping layers. Performing the Fourier filtering with different filtering mask, it was possible to isolate the separate planes introduced into the correct order of particular family of m-ZrO₂ lattice. The introduced planes belonging to m-ZrO₂ lattice. Since they were identified in corresponding FT, from which the filtering was performed, it was possible to give an interpretation about the transformation due to the mechanical alloying observed in the particular HRTEM image.

[58] CORRELATION OF SIZE REDUCTION RATE OF INORGANIC MATERIALS WITH IMPACT ENERGY OF BALLS IN PLANETARY BALL MILLING

Kano J. Mio H. Saito F. - Journal of Chemical Engineering of Japan. 32(4):445-448, 1999

Room temperature grinding of four kinds of powder materials chosen based on their grindability was conducted using a planetary ball mill, in which the powder is charged in different amounts besides media balls. The median diameter of the ground materials was monitored to experimentally determine the size reduction rate. The size reduction rate of each material is proportional to specific impact energy of the balls simulated by the PEM (or called DEM). The gradient of the straight lines for each material in the prescribed relation is correlated to a net work index, which is calculated by substituting the impact energy of balls into the term of energy in Bond's equation. The relation between gradient and net work index is also expressed by a straight line in normal coordinates. Therefore, the size reduction rate can be estimated by introducing a specific impact energy simulated by the PEM.

[57] THE USE OF A PRECISELY CONTROLLED METHANE-HYDROGEN GAS MEDIUM WHEN REGENERATING TUNGSTEN-BASE HARD ALLOY WASTE

Bondarenko VP. Pavlotskaya EG. - International Journal of Hydrogen Energy. 24(9):853-859, 1999

The kinetics and the mechanism of reduction and carburization processes of products of WC-Co hard alloy waste oxidation in methane-hydrogen gas medium of a precision composition have been studied. The use of a gas medium whose composition is close to the equilibrium with respect to carbon at a given temperature is shown to allow obtaining regenerated mixtures of stoichiometric content of combined carbon and almost with no impurities of free carbon. It has been found that variation of process parameters (the carburization temperature, the rate of milling the regenerated mixture, the conditions of sintering alloys) allows controlling the structure and physico-mechanical properties of regenerated alloys over a wide range. A new ecologically acceptable and highly efficient process has been devised and optimized for the regeneration of hard alloy waste employing a combined process of reduction-carburization of oxidation products in the methane-hydrogen gas medium. It has been shown that the alloys regenerated with this technology meet the requirements of State Standards on commercial alloys as to their composition, structure and physico-mechanical properties, and are highly competitive with them as far as their performance characteristics is concerned.

[56] EFFECT OF NICKEL ALLOYING BY USING BALL MILLING ON THE HYDROGEN ABSORPTION PROPERTIES OF TiFe

Bououdina M. Fruchart D. Jacquet S. Pontonnier L. Soubeyroux JL. - International Journal of Hydrogen Energy. 24(9):885-890, 1999

After ball-milling, TiFe under a pure argon atmosphere, with a small amount of pure nickel, can readily absorb large amounts of hydrogen without any activation process. The H/M system reaches equilibrium after 8 h under 10 bars of hydrogen pressure (H/M = 0.62). Even after 10 days exposure to the air, the same sample reacts with hydrogen, but with slower kinetics. The impact of the ball-milling time on the hydrogen absorption kinetics and the maximum hydrogen content is discussed.

[55] INFLUENCE OF ATTRITION MILLING ON NANO-GRAIN BOUNDARIES

Rawers J. Cook D. - Nanostructured Materials. 11(3):331-342, 1999

Nanostructured materials have a relatively large proportion of their atoms associated with the grain boundary and the method used to develop the nano-grains has a strong influence on the resulting grain boundary structure. In this study; attrition milling iron powders and blends of iron powders produced micron-size particles composed of nano-size grains. Mechanical cold-working powder resulted in dislocation generation; multiplication; and congealing that produced grain refinement. As the grain size approached nano-dimensions; dislocations were no longer sustained within the grain and once generated rapidly diffuse to the grain boundary. Dislocations on the grain boundary strained the local lattice structure which; as the grain size decreased; became the entire grain. Mechanical alloying of substitutional aluminium atoms into iron powder resulted in the aluminium atoms substituting for iron atoms in the grain boundary cells and providing a grain boundary structure similar to that of the iron powder processed in an on. Attrition milling iron powder in nitrogen gas resulted in nitrogen atoms being adsorbed onto the particle surface. Continued mechanical milling infused the nitrogen atoms into interstitial lattice sites on the grain boundary which also contributed to expanding and straining the local lattice.

[54] SYNTHESIS AND MICROSTRUCTUREAL CHARACTERIZATION OF NANOSTRUCTURED GAMMA-NI-Fe POWDER

Qin XY. Lee JS. Nam JG. Kim BS. - *Nanostructured Materials*. 11(3):383-397, 1999

The microstructures and their relation to synthesis conditions of nanostructured gamma-Ni-Fe alloys, synthesized by a mechano-chemical process, were investigated by using various techniques which include EDS, infrared and optical emission spectroscopy, XRD, TEM, BET and Laser particle analyzer as well as field emission SEM. The results indicate that nanostructured gamma-Ni-xFe alloys with x approximate to 32, 46, 55, and 64wt% were successfully synthesized. The impurity contents in these gamma-Ni-Fe alloys were very low. Typically, the microstructures of nano gamma-Ni-46Fe and their variations with synthesis conditions were focused on. It was documented that gamma-Ni-46Fe alloy with a minimum average grain size of 20nm and minimum average particle size similar to 70nm can be obtained under specific synthesis conditions. Micro-pore analysis combined with BET and XRD experimental results indicated that the particles usually consisting of less than one hundred grains were actually small (sub-micrometer) polycrystals with no micro-pores within them. Experiments also showed that there were large hard-agglomerates which had an average size of similar to 100nm in the nano gamma-Ni-46Fe powders obtained at temperatures below 800 degrees C. Above this temperature both the grain growth and the sintering process were significant. Moreover, it was revealed that upon annealing at temperatures below 800 degrees C, the grains of nano gamma-Ni-46Fe have an elongated shape with their long axis being in $\langle 111 \rangle$ direction and with an aspect ratio of 1.37-1.25. Annealing at temperatures above 800 degrees C caused their grains to change shape gradually to a disk-like form. In addition, the lattice of the nano gamma-Ni-46Fe was found to be in a shrunken state with an average grain size of $d < 50\text{nm}$, contradicting results of mechanical alloying by Hays et al. (14).

[53] MECHANOCHEMICAL REACTION AND RELATIONSHIP TO TRIBOLOGICAL RESPONSE OF SILICON NITRIDE IN N-ALCOHOL

Hibi Y. Enomoto Y. - *Wear*. 231(2):185-194, 1999

Milling tests and sliding tests were conducted to see how mechanochemical reactions influenced the friction and wear characteristics of Si₃N₄ in normal-chain, (n)-alcohols. Si₃N₄ powder was milled with n-alcohol to obtain mechanochemical reaction products. Silicon alkoxide and long-chain hydrocarbons might be produced in the mechanochemical reaction between Si₃N₄ and n-alcohols. Silicon alkoxide were hydrolyzed to form silica gel to determine the yield by post-treatment. Silica gel was the main product in lower alcohol and hydrocarbons were the main products in higher alcohol. In the sliding tests, Si₃N₄ was slid against the same material in CH₃OH and in C₉H₁₉OH. Hydrodynamic lubrication, where the friction coefficient was < 0.01 , occurred in CH₃OH for sliding speed exceeding 1600 mm/s. Friction coefficient of Si₃N₄ in C₉H₁₉OH was 0.05-0.11. Wear of Si₃N₄ in CH₃OH was much higher than that in C₉H₁₉OH. Precursors of silica gel, reduced friction of Si₃N₄ in alcohol; hydrocarbons did not act as a lubricant, however. The specific wear rate of Si₃N₄ ball in the sliding test correlated very well with the yield of silica gel produced in the milling test. Our results suggest that friction and wear of Si₃N₄ are influenced by the reactivity of the mechanochemical reaction producing silicon alkoxide.

[52] HEAT TREATMENT OF SINTERED POROUS STEELS

Grevnov LM. - *Powder Metallurgy & Metal Ceramics*. 37(11-12):583-592, 1998

Heat-treatment features are considered for steels prepared from mechanical powder mixtures The rise in the temperature of the critical point Ac-1 and the fall in the point Ac-3 as the homogeneity of steel increases is described by a linear dependence on the coefficient of concentration variation. A chemically inhomogeneous low-alloy powder steel has the kinetics of the pearlite-austenite transformation limited by the diffusion growth of nuclei. In more homogeneous steels, the limiting stage is nucleation. Decrease in stability of the supercooled austenite and narrowing of a temperature difference between the pearlite and intermediate regions occur with reduction in homogeneity of a high-alloy powder steel. The porosity and chemical inhomogeneity activate the decomposition of martensite on low-temperature annealing and reduce the temperatures of the four transformations on tempering.

[51] USE OF POWDERS OF THE TUNGSTEN-RHENIUM ALLOYS FOR THE PREPARATION OF IMPREGNATED CATHODE SKELETONS - I. DENSIFICATION AND FORMATION OF POROUS STRUCTURE IN SINTERING OF POWDERS OF TUNGSTEN-RHENIUM ALLOYS

Get'man OI. Panichkina VV. Rakitin SP. Radchenko OG. Skorokhod VV. - *Powder Metallurgy & Metal Ceramics*. 37(11-12):618-625, 1998

The effect of phase composition, fineness, and morphology of W-Re powders with different rhenium content on compaction and formation of porous structure has been studied Fine particle powders with a specific surface of 5-8 m²/g, mechanical mixtures of tungsten and rhenium powders, and W-Re alloy powders were used. Local nonuniform compaction is observed during sintering of powder mixtures and alloy powders. It was established that alloy powders and intermetallic powders have lower sintering activity.

[50] SYNTHESIS OF TiC AND Ti/TiC COMPOSITES BY REACTION MILLING FOLLOWED BY HOT CONSOLIDATION OF Ti WITH GRAPHITE OR HEPTANE [JAPANESE]

Yasukawa A. Nagumo M. - *Journal of the Japan Institute of Metals*. 63(7):822-827, 1999

Synthesis of TiC and Ti/TiC composite has been conducted by mechanical alloying and following Spark-Plasma-Sintering starting from elemental Ti powder with graphite or heptane. From graphite, formation of TiC takes place rapidly after a definite period of milling while it proceeds gradually from heptane. The apparent compositional range of Ti_{1-x}C_x is extended to a C/Ti ratio as low as 30/70 when graphite is used, while a mixture of Ti and TiC is obtained at a C/Ti ratio of 32/68 with heptane. Sintering can proceed at substantially lower temperatures compared with the conventional process, the sintering temperature decreasing with lower C/Ti ratios. The lattice parameter of TiC and the density of the compacts decrease with lower C/Ti ratios, but are larger than previously reported ones through conventional methods. The mechanism which causes the differences between graphite and heptane is discussed with respect to the reaction process during milling.

[49] EFFECT OF VANADIUM ADDITION ON LATTICE PARAMETER AND DISTORTION OF Li(2)-(ALX)(3)Ti PHASE [JAPANESE]

Hashimoto K. Yamamoto Y. Kimura T. Moriya H. Nakamura M. - Journal of the Japan Institute of Metals. 63(7):874-881, 1999

Composition, lattice parameter and lattice distortion of six kinds of quaternary Lit-type $(AlX)_3Ti(V)$ phases with ternary element X(Mn, Cr, Fe, Ni, Cu or Ag) have been examined by electron micro-probe analysis and X-ray diffraction analysis. Quaternary element, V from 1 to 12 mol% was added to the six kinds of L1(2)-type $(AlX)_3Ti(V)$ phases. Measured lattice parameters of Lit-type $(AlX)_3Ti(V)$ phases exhibited good linear correlation with calculated lattice parameter from atomic sizes and occupation sites of constitutional atoms in the L1(2)-type $(AlX)_3Ti(V)$ phases except for the $(AlNi)_3Ti(V)$ and $(AlCu)_3Ti(V)$ systems. A large amount of residual strain induced by milling of the specimen into powder was found in the Lit-type $(AlMn)_3Ti(8 \text{ mol\% V})$. A large amount of lattice distortion was found in the powder specimen of the Lit-type $(AlFe)_3Ti(8 \text{ mol\% V})$ phase which was well annealed at 1223 K for 40 min. It was suggested that the Lit-type $(AlX)_3Ti(V)$ phase may have poor ductility at ambient temperature if their powder specimen exhibit a large amount of lattice distortion after well annealing.

[48] X-RAY DIFFRACTION PEAK FROM MINOR PHASE IN MECHANICALLY ALLOYED AL-TI [JAPANESE]

Kim HS. Jeong HG. Hanada S. Kum DW. - Journal of the Japan Institute of Metals. 63(7):938-943, 1999

Refining of Ti powder particles and their dissolution into the Al matrix during mechanical alloying (MA) were investigated by using X-ray diffraction (XRD) and transmission electron microscopy (TEM) as functions of alloy composition, milling time and ban to powder ratio (BPR). It is found that Ti particles less than 20 nm are observed in a dark field image of mechanically alloyed Al-10 mass%Ti whose XRD pattern exhibits no Ti peak. The observed change of lattice constant of Al indicates that about 1 mass%Ti can be solved in Al after MA for a long time, independent of alloy composition, milling time and BPR, suggesting that most of Ti particles are retained in the Al matrix. It is concluded that the disappearance of XRD peaks in mechanically alloyed Al-10 mass%Ti is not simply attributable to the dissolution of the Ti into Al, but is associated mainly with extreme refining and/or heavy straining of Ti particles.

[47] LOW-TEMPERATURE METHODS FOR SYNTHESIS OF ALUMINOMAGNESIUM SPINEL AND EFFECT OF YTTRIUM OXIDE ON ITS SINTERING

Morozova LV. Panova TI. Lapshin AE. - Russian Journal of Applied Chemistry. 72(4):567-571, 1999

Low-temperature methods for synthesis of aluminomagnesium spinel were studied: sol-gel method, joint crystallization of magnesium and aluminum nitrate solutions with their subsequent decomposition, and mechanochemical activation of hydrated magnesium and aluminum nitrates. X-ray phase analysis was used to compare the effectiveness of the methods used with respect to $MgAl_2O_4$ formation.

[46] PHASE DEVELOPMENT IN TITANIUM BY MECHANICAL ALLOYING UNDER HYDROGEN ATMOSPHERE

Cheng ZH. MacKay GR. Small DA. Dunlap RA. - Journal of Physics D-Applied Physics. 32(15):1934-1937, 1999

Titanium hydride was successfully synthesized at room temperature by ball milling. X-ray diffraction patterns demonstrated that a transformation of elemental Ti powders into TiH_2 could be induced at room temperature by ball milling under a hydrogen atmosphere. Almost no hydrogen absorption was observed during the initial few minutes of the milling process on the basis of x-ray diffraction patterns and hydrogen pressure in the reservoir system, although the grain size of Ti decreased significantly. A non-stoichiometric titanium hydride with an approximate composition of $TiH_{1.9}$ was first formed after about 7 min of milling and existed together with unhydrided Ti flakes. The stoichiometric hydride phase TiH_2 was developed after further milling at room temperature. Due to the trapping of hydrogen by numerous defects and grain boundaries, the dilute solution $Ti(H)$ does not exist in the nanograin milled metal powders.

[45] FORMATION, CHARACTERIZATION, AND PROPERTIES OF A NEW BORON-CARBON-NITROGEN CRYSTAL

Yao B. Liu L. Su WH. -Journal of Applied Physics. 86(5):2464-2467, 1999

Amorphous boron-carbon-nitrogen (BCN) powders were produced by mechanically milling a mixture of hexagonal boron nitride (h-BN) and graphite powders for 120 h in an argon atmosphere. By annealing the amorphous BCN powders isothermally under 4 GPa at temperatures above 880 K, a new B-C-N crystal with a tetragonal structure was obtained. Its lattice constants decrease with the increasing annealing temperature and are $a=1.685 \text{ nm}$ and $c=0.537 \text{ nm}$, respectively at an annealing temperature of 1170 K. The conductivity measurement suggests the new B-C-N phase is a semiconductor with a very small band gap and the band gap changes depending on the temperature in the temperatures between room temperature and 840 K.

[44] MAGNETIC HYSTERESIS PROPERTIES AND ROTATIONAL HYSTERESIS LOSSES OF SYNTHETIC STRESS-CONTROLLED TITANOMAGNETITE ($Fe_{2.4}Ti_{0.6}O_4$) PARTICLES - I. MAGNETIC HYSTERESIS PROPERTIES

Keller R. Schmidbauer E. - Geophysical Journal International. 138(2):319-333, 1999

Hysteresis parameters as a function of temperature were measured for milled and etched synthetic 2.4, 12.5 and 165 μm titanomagnetite (TM60) particles. The results show characteristics that appear to be dictated by microstresses, generated by a high density of dislocations and other lattice defects, that were introduced in the spinel lattice by the milling procedure. The following features were found. (1) 2.4 μm particles are single domain (SD) with a saturation remanent magnetization ratio σ_{rs}/σ_s (20 degrees C) approximate to 0.5 and coercive force H_c (20 degrees C) approximate to 75 kA m^{-1} ; the latter value can only be interpreted in terms of stress-induced anisotropy because shape and magnetocrystalline anisotropies are too low by far. The nearly temperature-independent coercivity ratio H_{cr}/H_c approximate to 1.5 up to similar to 200 degrees C is higher than expected for Stoner-Wohlfarth SD particles with uniaxial anisotropy; this behaviour is attributed to irregularly directed microstresses as a consequence of lattice defects. By contrast, a Henkel plot shows almost the behaviour expected for Stoner-Wohlfarth particles. Due to fracturing processes during milling, partial low-temperature oxidation has taken place, resulting in an enhanced Curie temperature T_c approximate to 230-260 degrees C, as extrapolated from data of saturation magnetization. (2) 12.5 μm particles ($T_c = 205 \text{ degrees C}$) show typical pseudo-single-domain (PSD) behaviour, with σ_{rs}/σ_s (20 degrees C) approximate to 0.18, H_c (20 degrees C) approximate to 11 kA m^{-1} , both

values being much higher than for multidomain (MD) particles, and H-cr/H-c(20 degrees C) approximate to 1.9. Magnetization processes appear to be dictated largely by reversible and irreversible domain rotations; that is, domain wall displacements play a minor role because of strong pinning forces for domain walls at lattice defects. As the temperature rises, a comparatively weak decrease in $\sigma(\text{rs})/\sigma(\text{s})$ takes place, whence it follows that the PSD character persists up to the Curie point. H-c falls with temperature steadily to a minimum of H-c(160 degrees C) approximate to 4 kA m(-1); a further increase towards 200 degrees C may be associated with an inhomogeneous magnetic behaviour throughout a particle because of the formation of some sort of SD regions, related in part to the microcrystalline particle structure; furthermore, thermal fluctuations may play a role. (3) 165 μm particles ($T_c = 200$ degrees C) exhibit MD behaviour with $\sigma(\text{rs})/\sigma(\text{s})(20$ degrees C) approximate to 0.025, H-c(20 degrees C) approximate to 1 kA m(-1) and H-cr/H-c(20 degrees C) approximate to 8. While H-c shows a smooth variation with temperature up to 160 degrees C, H-cr/H-c falls with temperature to a minimum H-cr/H-c(160 degrees C) approximate to 2 and an increase occurs again to H-cr/H-c(200 degrees C) approximate to 4.5.

[43] MAGNETIC HYSTERESIS PROPERTIES AND ROTATIONAL HYSTERESIS LOSSES OF SYNTHETIC STRESS-CONTROLLED TITANOMAGNETITE (Fe_{2.4}Ti_{0.6}O₄) PARTICLES - II. ROTATIONAL HYSTERESIS LOSSES

Keller R. Schmidbauer E. -Geophysical Journal International. 138(2):334-342, 1999

Rotational hysteresis losses W-RH of a ferro- or ferrimagnet (determined by torque measurements) give information on anisotropies related to irreversible magnetization processes. We measured W-RH as a function of magnetic field H for milled and etched synthetic titanomagnetite Fe_{2.4}Ti_{0.6}O₄ particles of 2.4, 12.5 and 165 μm grain size, which are assumed to show stress-controlled magnetic properties, between room temperature and the Curie point. For uniaxial single-domain (SD) particles, typical parameters that can be determined are in the low-field range a critical field for the onset of W-RH, a peak W-RH and a further typical field in the high-field region where W-RH tends to zero all these parameters are associated with the anisotropy constant. For 2.4 μm particles, assumed to be SD, the large experimental W-RH at all fields in the whole temperature range is ascribed predominantly to the action of high internal microstresses arising from a large density of dislocations and other lattice defects introduced by the milling process. The reduced magnitude of W-RH for pseudo-single-domain 12.5 μm particles and the still smaller W-RH for multidomain (MD) 165 μm particles is thought to be in the first place the result of much lower defect densities in conjunction with irreversible domain rotations. For each particle size, a rise of some W-RH parameters is observed for temperatures > 120 degrees C, which coincides for MD 165 μm particles with an increase of magnetic hysteresis parameters. The non-zero W-RH at the maximum applied held of H = 1200 kA m(-1) approximate to 15 kOe for all particles in the whole temperature region analysed is attributed to some kind of exchange anisotropy, caused by centres or areas in a particle related to a very high anisotropy, due probably to Fe²⁺ in the neighbourhood of lattice defects.

[42] MECHANICAL ALLOYING - A NOVEL METHOD FOR SYNTHESIS AND PROCESSING OF MATERIALS

Oleszak D. -Acta Physica Polonica A. 96(1):101-112, 1999

High energy ball milling (mechanical alloying) has recently become a popular research topic. As the non-equilibrium processing method, this technique may be used for the synthesis of many materials, including metastable ones. In many cases these materials exhibit the structure and properties quite different from materials prepared by conventional methods. In this paper some examples of metastable structures synthesized by mechanical alloying are described. The results of mechanical alloying in the Fe-Al system are presented as well

[41] THERMAL EVOLUTION OF BALL MILLED NANOCRYSTALLINE IRON

Bonetti E. Del Bianco L. Pasquini L. Sampaolesi E. - Nanostructured Materials. 12(5-8 Part B Special Issue SI):685-688, 1999

Mechanical spectroscopy and X-ray diffraction measurements were employed to investigate the thermally induced structural evolution of nanocrystalline iron, prepared by mechanical attrition through different ball milling equipment in different structural states. The evolution of the elastic modulus, grain size and microstrain as a function of temperature and after selected isothermal treatments indicates that the rearrangement of structural configuration at the interfaces may stabilise the nanostructure against grain growth.

[40] NONEQUILIBRIUM PHASE TRANSITION OF Nd₃(Fe,Ti)₂₉ COMPOUND DURING MECHANICAL MILLING

Tang SL. Jin ZQ. Zhang SY. Du YW. - Nanostructured Materials. 12(5-8 Part B Special Issue SI):705-708, 1999

A nonequilibrium phase transition has been found during mechanical milling of monoclinic Nd₃(Fe,Ti)₂₉ compound. The transition process and the mechanically milled products have been investigated by X-ray diffraction and AC initial susceptibility. The results show that mechanical milling results in the transformation of Nd₃(Fe,Ti)₂₉ compound into a nanostructure during the early stage of milling, and then generates it to decompose to the hexagonal TbCu₇-type Nd(Fe,Ti)₇ disorder phase and alpha-Fe(Ti), and finally transforms the material to a mixture of the amorphous phase and alpha-Fe(Ti). The structure of mechanically milled Nd(Fe,Ti)₇ phase is similar to but not exactly identical with that of annealed Nd(Fe,Ti)₇ phase. The magnetic-ordering transition of the former is broader than that of the later. The broadening of the magnetic-ordering transition can be attributed to the effect of the nanostructure, and the inhomogeneity of the concentration of Nd(Fe,Ti)₇ phase.

[39] MAGNETIC STRUCTURE EVOLUTION BN MECHANICALLY MILLED NANOSTRUCTURED ZNF₂O₄ PARTICLES

Jiang JZ. Wynn P. Morup S. Okada T. Berry FJ. - Nanostructured Materials. 12(5-8 Part B Special Issue SI):737-740, 1999

Nanostructured partially-inverted ZnFe₂O₄ particles have been prepared from bulk ZnFe₂O₄ by high-energy ball milling in an open container The grain size reduction, cation site distributions, and the evolution of magnetic structures have been studied by x-ray diffraction with Rietveld structure refinements, transmission electron microscopy, and Mossbauer spectroscopy. It is found that a change of magnetic structure from an antiferromagnetic to a ferrimagnetic (or ferromagnetic) structure occurs in the milled samples This change is correlated with the

redistribution of the cations, Zn and Fe, in the A and B sublattices.

[38] MICROSTRUCTURE AND HARDNESS OF A NANOSTRUCTURED FE-40AL AT% ALLOY

Amils X. Nogues J. Surinach S. Munoz JS. Lutterotti L. Baro MD. - Nanostructured Materials. 12(5-8 Part B Special Issue SI):801-806, 1999

Microstructural changes induced by ball milling under different conditions and posterior annealing in Fe-40Al at% B2 alloys have been studied. High speed milling results in more disordered samples and milling media contamination, which affects the thermal reordering. The microhardness is closely correlated to the concentration of antisite defects of the samples.

[37] MAGNETOELASTICITY AND INTERNAL STRAINS IN NANOCRYSTALLINE NICKEL

Bonetti E. Campari EG. Pasquini L. Sampaolesi E. Scipione G. - Nanostructured Materials. 12(5-8 Part B Special Issue SI):817-820, 1999

The magnetic field dependence of the dynamic elastic modulus (Delta E effect) was studied in ball-milled nanocrystalline Nickel, as a function of the milling times and annealing treatments. The analysis of the crystallite size and internal strains has been performed as well by Fourier analysis of the X-ray spectra. A correlation between Delta E and the lattice strain calculated by X-ray analysis is observed, suggesting that the magnetic anisotropy is mainly determined by the internal strains.

[36] COMPRESSIBILITY OF NANOSTRUCTURED FE-CU MATERIALS PREPARED BY MECHANICAL MILLING

Jiang JZ. Olsen JS. Gerward L. Morup S. - Nanostructured Materials. 12(5-8 Part B Special Issue SI):847-850, 1999

The compressibility of nanostructured Fe-Cu materials prepared by mechanical milling has been investigated by in-situ high-pressure x-ray diffraction using synchrotron radiation. It is found that the bulk modulus of both fcc-Cu and bcc-Fe phases decreases with decreasing grain sizes. The unstable ferromagnetic fcc-FeCu solid solution prepared by mechanical alloying has a bulk modulus of about 85 GPa, which is much smaller than the corresponding values for bulk fcc-Cu and bcc-Fe.

[35] ROOM-TEMPERATURE AGEING EFFECTS ON THE MAGNETIC PROPERTIES OF MECHANICALLY MILLED SMCOS

Leslie-Pelecky DL. Kirkpatrick EM. Schalek RL. - Nanostructured Materials. 12(5-8 Part B Special Issue SI):887-890, 1999

Mechanical milling of ferromagnetic SmCo5 produces dramatic increases in coercivity or short (15 minutes to 2 hours) milling times. The milled nanostructured material is highly metastable and undergoes room-temperature recovery during the first two weeks after fabrication. Although changes in the coercivity with ageing time are significant the diffracting crystallite size determined from x-ray diffraction remains constant. We conclude that disorder is a primary determinant of the coercivity of mechanically milled alloys.

[34] MAGNETIC PROPERTIES AND STRUCTURE OF NANOCRYSTALLINE FE-AL AND FE-NI ALLOYS

artych E. Zurawicz JK. Oleszak D. Pekala M. - Nanostructured Materials. 12(5-8 Part B Special Issue SI):927-930, 1999

Magnetic and structural properties of nanocrystalline Fe-Al and Fe-Ni alloys were investigated. Alloys were prepared by the mechanical alloying process. The standard X-ray diffraction, Mossbauer and magnetic studies were carried out on the powder samples. The structural investigations proved that in the low-energy mechanical alloying process of Fe and Al the disordered bcc solid solution was formed up to 50 at.% Al. Alloys were amorphous for Al content between 50 and 90 at.%. The final products of milling exhibited interesting magnetic properties different as compared with microcrystalline alloys. In the case of Fe-Ni alloys the bcc solid solution was formed during high-energy ball milling for the composition with 20 at.% Ni, while above 35 at.% Ni the fcc solid solutions were obtained. All mechanically synthesized Fe-Ni alloys were ferromagnetic and no Invar effect was observed for 35 at.% Ni.

[33] MAGNETIC PROPERTIES OF MECHANICALLY ALLOYED NANOCRYSTALLINE Ni3Fe

Chinnasamy CN. Narayanasamy A. Chattopadhyay K. Ponpandian N. - Nanostructured Materials. 12(5-8 Part B Special Issue SI):951-954, 1999

Disordered nanocrystalline Ni3Fe alloy was prepared by mechanical alloying of elemental powders. X-ray diffractograms show the formation of Ni3Fe single phase. The chemical composition and morphology of the powder have been obtained by using EDAX and SEM analysis respectively. While the saturation magnetisation decreases with milling time, the coercivity increases. The width of the hyperfine field distributions obtained from Mossbauer studies shows that the alloy is highly disordered. Atomic ordering is found to take place at a faster rate compared to that in the bulk alloy.

[32] PRODUCTION OF MAGNETICALLY SOFT MATERIALS BY MECHANICAL MILLING?

Arrott A. - Nanostructured Materials. 12(5-8 Part B Special Issue SI):1135-1140, 1999

It may be possible to produce a useful magnetically soft material for industrial applications such as high-temperature motors by means of mechanical milling. The complexity of the micromagnetics involved makes it difficult to come to a theoretical conclusions concerning the potential for creating soft magnetic materials with high saturation magnetization and low hysteresis loss from materials with substantial crystalline anisotropy by using the exchange interaction to couple many grains with different anisotropy axes. This should be a tractable experimental problem starting with supermendur (49Fe49Co2V) which has very small anisotropy and then working with less and less Co which increases the saturation magnetization, the magnetic anisotropy and the electrical resistivity while lowering the magnetostriction and the cost. It is argued that this is an interesting problem.

[31] LOCAL STRUCTURE OF TI-SI AMORPHOUS ALLOYS

Fukunaga T. - Journal of Physics & Chemistry of Solids. 60(8-9):1479-1481, 1999

TixSi100-x (x = 55, 60) amorphous alloys were synthesized by a mechanical alloying method. The S-CC(Q) defined by Bhatia and Thornton was directly derived by neutron diffraction for Ti-45.3 at.% Si amorphous alloy. Provided that the S-CC(Q) is equal to that for Ti60Si40 amorphous alloy, other partial structure factors S-NN(Q) and S-NC(Q) were deduced by eliminating S-CC(Q) from S(Q)s by neutron and X-ray diffractions. The Faber-Ziman type partial structure factors S-TiTi(Q), S-TiSi(Q) and S-SiSi(Q) were calculated from the S-CC(Q), S-NN(Q) and S-NC(Q).

[30] NEUTRON SCATTERING STUDY OF AMORPHOUS TRI-O-METHYL-BETA-CYCLODEXTRIN

Tsukushi I. Kanaya T. Yamamuro O. Matsuo T. Kaji K. - Journal of Physics & Chemistry of Solids. 60(8-9):1545-1547, 1999

Low energy excitations in crystalline and amorphous solids of Tri-O-methyl-beta-cyclodextrin (TMCD) were studied by neutron scattering and calorimetric techniques. Amorphous solids of TMCD were prepared by two different ways: mechanical milling and liquid-quenching amorphization. Both the amorphous solids showed a boson peak at around 2 meV and there was no significant difference between them. It was noted that crystalline TMCD also showed a boson peak. This anomalous boson peak in the crystalline state was confirmed by low temperature heat capacity measurements.

[29] FE_{61.6}NI_{15.4}CU_{1NB2P14B6} AMORPHOUS PREPARED BY MECHANICAL ALLOYING

Li F. Wu BY. Ji YL. Wang GQ. Zhao JW. Zhang SY. - Journal of Materials Science Letters. 18(13):1021-1023, 1999

[28] MECHANICAL MILLING OF THE INTERMETALLIC COMPOUND ALFE

Meyer M. Mendoza-Zelis L. Sanchez FH. Clavaguera-Mora MT. Clavaguera N. - Physical Review B-Condensed Matter. 60(5):3206-3212, 1999

The intermetallic compound AlFe in its ordered B2 phase has been ground under Ar in a ball-milling device. The system evolution under mechanical work has been examined as a function of time of milling by x-ray diffraction, transmission Mossbauer spectroscopy, and differential scanning calorimetry. Short and long range disorder was observed. The evolution of the long range order parameter with milling time is analyzed up to the completion of the solid state reaction when a steady state value is reached. The short range order evolution is consistent with a nonstatistical order distribution throughout the sample but mainly distributed in two regions with different degrees of order each. Kinetic analysis of the reordering and recrystallization has been performed under continuous heating and isothermal calorimetric regimes. Modeling of these processes is presented as well as the estimated values of both the enthalpy and the activation energy of the reordering process. Results are compared with preceding information on related systems.

[27] ELECTROCHEMISTRY OF NANOCRYSTALLINE COPPER

Heim U. Schwitzgebel G. - Nanostructured Materials. 12(1-4 Part A Special Issue SI):19-22, 1999

Nanocrystalline copper (Cu-nc), prepared by inert gas condensation, pulsed electrodeposition and by ball-milling, was characterized by XRD (mean grain diameter D; mean micro strain epsilon). Under fetal exclusion of O₂, the electrode potentials E-nc of these materials as well as E-mc of microcrystalline copper could be measured in acid solutions of CuSO₄. The differences Delta E-ex = E-nc-E-mc which range up to 16.8 mV for the sample with the lowest diameter, D = 8 nm, are inversely proportional to D according to a modified Gibbs-Thomson equation, when the deformation energy is taken into account. Thus, it can be concluded that Cu_n forms a metastable equilibrium with Cu ions and that the electrode potentials of Cu-nc are quasi reversible, within the time of observation. The intrinsic grain growth of Cu-nc (outside the solution) is much slower than the apparent growth in solution. Corrosion on Cu-nc could best be detected by electrochemical impedance spectroscopy: Channels and pores in the surface give rise to an unusual diffusion type.

[26] MECHANOCHEMICAL SYNTHESIS OF METAL SULPHIDE NANOPARTICLES

Tsuzuki T. McCormick PG. - Nanostructured Materials. 12(1-4 Part A Special Issue SI):75-78, 1999

The synthesis of ZnS, CdS and Ce₂S₃ nanoparticles by mechanochemical reaction has been reviewed. During mechanical milling, solid-state displacement reactions between the respective metal chloride and alkali sulphide or alkaline earth sulphide were induced in a steady-state manner, leading to the formation of metal sulphide nanoparticles. A simple washing process to remove the chloride by-product yielded separated particles of ZnS, CdS and Ce₂S₃ of similar to 8 nm, similar to 4 nm and similar to 20 nm, respectively. The resulting particles and crystallite sizes were dependent on the milling conditions, starting materials and the presence of a diluent. Structural change with decreasing particle size was observed for CdS and Ce₂S₃.

[25] HIGH STRENGTH AL-ALLOYS WITH NANO-QUASICRYSTALLINE PHASE AS MAIN COMPONENT

Schurack F. Eckert J. Schultz L. - Nanostructured Materials. 12(1-4 Part A Special Issue SI):107-110, 1999

The formation of quasicrystals by ball milling as well as by rapid quenching in the systems AlMnCe and AlMnFe was investigated. Melt spinning resulted in a nanoscale mixed structure of up to 60 vol.% quasicrystalline particles embedded in a fee Al-matrix revealing a strong influence of the alloy composition on the solidification behaviour. Milling leads to the formation of an amorphous phase, however, there was no significant effect for Ce addition on phase formation. While the use of process-control agent or different milling atmospheres mainly affects the powder morphology, the milling intensity and, hence, the temperature during milling was found to be the most important parameter concerning phase evolution. The nature of observed metastable phase transitions was clarified by DSC measurements combined with x-ray diffraction.

[24] PROPERTIES OF MG-Y-CU GLASSES WITH NANOCRYSTALLINE PARTICLES

Schlorke N. Weiss B. Eckert J. Schultz L. - Nanostructured Materials. 12(1-4 Part A Special Issue SI):127-130, 1999

Nanocrystals embedded in an amorphous Mg-Y-Cu phase can lead to an increase in fracture stress compared to the homogeneous amorphous alloy. Therefore, mechanical alloying of elemental Mg, Y and Cu powders with Si-, Ce-, Y₂O₃- and MgO-additives was used to prepare amorphous/nanocrystalline phase mixtures. Despite the existence of nanocrystalline particles in the amorphous matrix, all samples exhibit a temperature difference Delta T-x (= T-x - T-g) between the crystallization temperature T-x and the glass transition temperature T-g of about 50 - 60 K and a decrease of viscosity above T-g. The additives enhance the thermal stability of the amorphous Mg-Y-Cu phase. Bulk amorphous/nanocrystalline samples can be prepared via consolidation and subsequent hot working in the viscous state above T-g. Finally, first results on the mechanical properties of the samples are presented.

[23] FORMATION OF IRON-NICKEL NANOCRYSTALLINE ALLOY BY MECHANICAL ALLOYING

Tcherdyntsev VV. Kaloshkin SD. Tomilin IA. Shelekhov EV. Baldokhin YV. - Nanostructured Materials. 12(1-4

Part A Special Issue SI):139-142, 1999

Fe.,Ni, alloys were prepared by mechanical alloying of elemental powders in high-energy planetary ball mill in a wide concentration range of components (10 less than or equal to x less than or equal to 90). The structure was studied by X-ray diffractometry. It is shown that concentration ranges of single-phase solid solution of MA samples are markedly wider than that of thermodynamically stable alloys. Character of X-ray peaks indicates that there is a high density of crystalline lattice defects including stacking faults. Block sizes, calculated without considering stacking faults presence, was found to be 8 - 15 nm, and calculated with considering of stacking faults 30 - 80 nm. The results were discussed on the basis of thermodynamic model of MA.

[22] SYNTHESIS AND CHARACTERIZATION OF NANOCRYSTALLINE ZINC FERRITE

Jiang JS. Yang XL. Gao L. Guo JK. Jiang JZ. - Nanostructured Materials. 12(1-4 Part A Special Issue SI):143-146, 1999

Nanocrystalline zinc ferrite powders with a partially inverted spinel structure were synthesized by high-energy ball milling in a closed container at ambient temperature from a mixture of alpha-Fe₂O₃ and ZnO crystalline powders in equimolar ratio. From low-temperature and in-field Mossbauer measurements it is revealed that ZnFe₂O₄ particles prepared are in superparamagnetic state at ambient temperature. A doublet with an average quadrupole splitting of 0.8 mm/s is observed for the as-milled sample at 295 K, which is much larger than that for bulk ZnFe₂O₄ prepared by traditional ceramic method and that for ultrafine ZnFe₂O₄ particles prepared by the co-precipitation method. This indicates larger structural defects in the nanometer-sized ZnFe₂O₄ particles prepared by high-energy ball milling.

[21] HIGH ENERGY BALL MILLING OF CO89B11 POWDERS

Jachimowicz M. Fadeeva VI. Matyja H. - Nanostructured Materials. 12(1-4 Part A Special Issue SI):159-162, 1999

Mechanical alloying was performed on elemental cobalt and baron powders with atomic ratio Co₈₉B₁₁. X-ray diffraction analysis (XRD) and differential scanning calorimetry were used to characterize the structure of the milled samples before and after the heating. The high-energy ball milling was found to influence the resulting phases. The starting powder consisted of the two modifications of cobalt: hcp (hexagonal close packed) and fee (face centered cubic). Depending on the milling time, single hcp, single fee, amorphous phases or their mixture were formed. After milling for 20 h, one can observe that the fcc phase transforms into hcp. Further milling caused the amorphization of the alloy. The fee phase formed from the amorphous matrix only after 80 h of milling and then transformed partially into the hexagonal phase after further milling. The average crystallite size in the final sample obtained after 150 h of ball milling was about 6 nm.

[20] INFLUENCE OF THE MECHANICAL ALLOYING PARAMETERS ON CRYSTALLITE SIZE OF TI-AL POWDERS

Szewczak E. Wyrzykowski JW. - Nanostructured Materials. 12(1-4 Part A Special Issue SI):171-174, 1999

Nanocrystalline Ti_xAl_{100-x} (x=75, 50, 40,35 at.%) alloys were prepared by mechanical alloying. The influence of milling parameters (the milling equipment milling time and energy) on the crystallite size of the alloys obtained as well as on the thermal stability of the crystallite size was examined. The powders were characterized by X-ray diffraction, scanning differential calorimetry and transmission electron microscopy. Attention was also paid to the influence of the phase transitions during heating of milled powders upon the crystallite size of the product.

[19] FORMATION AND MAGNETIC PROPERTIES OF NANOSIZED SM₂CO₁₇ MAGNETIC PARTICLES VIA MECHANOCHEMICAL/THERMAL PROCESSING

Liu W. McCormick PG. - Nanostructured Materials. 12(1-4 Part A Special Issue SI):187-190, 1999

Nano-sized Sm₂Co₁₇ magnetic particles have been prepared by mechanical milling and subsequent heat treatment of a mixture of Sm₂O₃, CoO and Ca with a suitable amount of CaO as diluent. The mechanical milling produced a nanostructured composite consisting of a nanocrystalline CaO supersaturated with Ca, CoO and Sm₂O₃, Co nanoparticles and a small amount of CoO. Heat treatment of as-milled powders at temperatures above 300 degrees C resulted in the formation of ultrafine Sm₂Co₁₇ particles of 10-250 nm in size embedded in a CaO matrix. The particle size of Sm₂Co₁₇ grew larger and its distribution became wider with increasing heat treatment temperature. A high coercivity of 14.2 kOe was measured on the ultrafine Sm₂Co₁₇ particles embedded in the CaO matrix in the sample heat treated at 800 degrees C.

[18] KINETICS OF FORMATION OF NANOCRYSTALLINE TiO₂II BY HIGH ENERGY BALL-MILLING OF ANATASE TiO₂

Begin-Colin S. Girot T. Mocellin A. Le Caer G. - Nanostructured Materials. 12(1-4 Part A Special Issue SI):195-198, 1999

Ball-milling is a way of inducing phase transformations, chemical reactions and changes in the reactivity of solids. The high pressure and high temperature phase of TiO₂ : TiO₂ II is formed transiently during room-temperature grinding of anatase TiO₂. Rutile is the only phase present after prolonged ball milling. The influences of physical and of chemical processing conditions on the kinetics of phase transformations are considered.

[17] PHYSICO-CHEMICAL CHARACTERIZATION OF NANOCRYSTALLINE (FE, NI) ALLOYS

St Werkmeister K. Rullang F. Koch M. Heyer M. Schwitzgebel G. - Nanostructured Materials. 12(1-4 Part A Special Issue SI):229-232, 1999

Nanocrystalline (Fe, Ni) alloys were prepared using pulsed electrodeposition (PE) and mechanical alloying (MA). When compared with thermally alloyed metals the phase separation into fee and bcc is more pronounced, but incomplete mixing cannot be avoided especially in the MA samples. The specific interfacial enthalpies determined by solution calorimetry (SC) are systematically higher than those from differential scanning calorimetry (DSC). Electromotive force (EMF) measurements with solid electrolyte cells showed that the metastability of fcc alloys (e.g. invar) causes large shifts in the electrode potential. Corrosion processes' of nanocrystalline (Fe, Ni) were observed and studied by atomic force microscopy (AFM), by DSC and especially by electrochemical impedance spectroscopy (EIS).

[16] MECHANICAL MILLING OF FE₃O₄/SiO₂: FORMATION OF AN AMORPHOUS FE(II)-SI-O-CONTAINING PHASE

Koch CB. Jiang JZ. Morup S. - Nanostructured Materials. 12(1-4 Part A Special Issue SI):233-236, 1999

The product of ball milling of magnetite and amorphous silica (40 mole% Fe₃O₄ in SiO₂) for an extended period of time (800 h) in a closed vial, has been investigated by Mossbauer spectroscopy, X-ray diffraction and infrared spectroscopy. It is found that the milling induces extensive reduction of Fe(III). The material constitutes a mixture of ultrafine Fe-rich spinel particles (magnetite/maghemite) and amorphous Fe(II)-containing silicate with a magnetic transition temperature of approximately 25 K. The amorphous phase has a rather high Fe content and is distinctly different from the initial amorphous silica.

[15] ZNO-FE NANOCOMPOSITES VIA BALL MILLING AND ANNEALING

Takacs L. Mossion A. Lazar K. Varga LK. Pardavi-Horvath M. Bakhshai A. - Nanostructured Materials. 12(1-4 Part A Special Issue SI):245-248, 1999

Metallic iron can be formed by reacting Fe₃O₄ with Zn in a ball mill. A mixed Fe-Zn oxide forms first, followed by decomposition into Fe and ZnO upon further milling. A large amount of Zn remain dissolved in the bcc phase and a fraction of the Fe retains an oxidized state. Decomposition can also be achieved by annealing the intermediate oxide. This process is investigated by thermomagnetic measurements, XRD, and Mossbauer spectroscopy. It is shown that the intermediate phase decomposes at relatively low (300-400 degrees C) annealing temperatures. The separation of Fe and Zn between the metal and oxide phases of the end product is more complete than found in samples prepared by milling alone.

[14] PARTICLE BEHAVIOR OF NANOCRYSTALLINE 316-STAINLESS STEEL DURING HIGH VELOCITY OXY-FUEL THERMAL SPRAY

Lau ML. Gupta VV. Lavernia EJ. - Nanostructured Materials. 12(1-4 Part A Special Issue SI):319-322, 1999

The present paper investigates the particle behavior of nanocrystalline 316-stainless steel powders during high velocity oxy-fuel (HVOF) spray. The feedstock powders were synthesized by mechanical milling to produce flake-shaped agglomerates with an average grain size of less than 50 nm. The powders were then introduced into the HVOF spray to produce a nanocrystalline coating. A mathematical model is developed to study the variation of different agglomerate sizes resulting from the mechanical milling process on the particle behavior during thermal spraying. A generalized Newtonian equation describing the momentum transfer was used, which incorporates a geometric ratio to account for the size and morphological variations of the micron-sized agglomerates with nano-grained structure. Particle velocity from the numerical calculations indicated that the velocity profile depends strongly on the thickness of the particles, which is a function of milling time and milling media.

[13] NANOPHASE COMPOSITES IN EASY GLASS FORMING SYSTEMS

Eckert J. Seidel M. Xing LQ. Borner I. Weiss B. - Nanostructured Materials. 12(1-4 Part A Special Issue SI):439-442, 1999

Nanophase composites based on Zr- and Mg-base easy glass forming alloys were prepared by partial crystallization of melt-quenched amorphous alloys or by mechanical alloying of elemental powder mixtures blended with oxide particles. The amorphous phases exhibit a wide supercooled liquid region before crystallization, even in the presence of second phase particles. This allows an easy consolidation of powders or shaping of bulk samples due to viscous flow. Room temperature mechanical properties will be presented and discussed with respect to the potential of the nanoscale microstructure for mechanical strengthening.

[12] NANOPARTICLES IN AN AMORPHOUS ZR55AL10CU30NI5 MATRIX - THE FORMATION OF COMPOSITES BY MECHANICAL ALLOYING

Kubler A. Eckert J. Schultz L. - Nanostructured Materials. 12(1-4 Part A Special Issue SI):443-446, 1999

Mechanical alloying of pure elemental powders blended with W, Y₂O₃ and SiO₂ particles was used to synthesize nanoscale composites consisting of an amorphous Zr₅₅Al₁₀Cu₃₀Ni₅ matrix with a homogeneous dispersion of nanoparticles. The powders are characterized by x-ray diffraction, calorimetric measurements, thermal mechanical analysis and testing the microhardness. The amorphous matrix exhibits an extended supercooled liquid region before crystallization. The supercooled liquid region is not significantly reduced due to the addition of nanocrystalline particles whereas the viscosity of the composites increases with increasing volume fraction of nanoscaled particles within the amorphous matrix.

[11] NANOCOMPOSITES PRODUCED BY MECHANICAL ALLOYING OF THE AL50FE25TI25 POWDERS MIXTURE

Krasnowski M. Fadeeva VI. Matyja H. - Nanostructured Materials. 12(1-4 Part A Special Issue SI):455-458, 1999

Nanocomposite powders were produced by high energy ball milling of the Al₅₀-Fe₂₅-Ti₂₅ (at.%) powder mixture added with ethanol (C₂H₅OH) and ammonium carbonate ((NH₄)₂CO₃). The phase transformations occurring in the material during the process were monitored by X-ray diffractometry and the structures of the milled samples were examined using transmission electron microscopy. The nanocrystalline composite powder, consisting of complex (Ti,Al)C carbide distributed in the Fe(Al) matrix was obtained as the final product of the milling with participation of ethanol whereas the process performed with an addition of ammonium carbonate led to the formation of (Ti,Al)N-Fe(Al) nanocomposite.

[10] MECHANO-CHEMICAL SYNTHESIS OF NANOSIZED STAINLESS STEEL POWDER

Nam JG. Lee JS. - Nanostructured Materials. 12(1-4 Part A Special Issue SI):475-478, 1999

The present investigation has attempted to fabricate nanosized stainless steel powder of Fe-18Cr-8Ni(wt%) by mechano-chemical synthesis. The synthesis process was conducted by hydrogen reduction of a high energy ball milled mixture of Fe₂O₃-NiO-Cr(NO₃)₃·9H₂O. In-situ alloying of Ni-Fe-Cr nanophase simultaneously occurring during reduction process is presumably responsible for the formation of nanophase stainless steel powders. The powder characteristics were examined by X-ray diffractometry and SEM observation. The kinetic phenomena in association with the oxide reduction and the alloying process were investigated by thermogravimetry, hygrometry and X-ray diffractometry, and discussed in relation to powder characteristics.

[9] STRUCTURE, PHASE COMPOSITION AND MAGNETIC CHARACTERISTICS OF THE NANOCRYSTALLINE IRON OBTAINED BY MECHANICAL MILLING IN HEPTANE

Yelsukov EP. Lomayeva SF. Konygin GN. Dorofeev GA. Povstugar VI. Mikhailova SS. Zagainov AV. Kadikova

AH. - Nanostructured Materials. 12(1-4 Part A Special Issue SI):483-486, 1999

The paper states that in the absence of carbon in the particle bulk the formation of the nanocrystalline state in alpha-Fe by mechanical milling is not accompanied by the change in structural and magnetic parameters, with the exception of the increase of the coercivity. In case the source of carbon is present all the changes in magnetic characteristics of nanocrystalline alpha-Fe, caused by milling time increase, may be explained by carbon segregation in boundary zones with the formation of supersaturated solid solutions and Fe₃C.

[8] STRUCTURAL AND MAGNETIC INVESTIGATION OF MECHANICALLY ALLOYED FE₇₀CO_{3.5}Cu₁Nb₃B₉Si_{13.5} POWDERS

Chiriac H. Moga AE. Urse M. Necula F. - Nanostructured Materials. 12(1-4 Part A Special Issue SI):495-498, 1999
Results concerning the preparation of nanocrystalline Fe₇₀Co_{3.5}Cu₁Nb₃B₉Si_{13.5} powders by high energy ball milling, in argon atmosphere, and their magnetic properties are presented. From X-ray diffraction patterns it follows that the powders with the above mentioned composition, obtained after 250 hours of milling, are nanocrystalline, with the crystallite size of about 8.6 nm. An improvement in the magnetic properties of the mechanically alloyed powders, by controlled heat treatment, can be noticed. A comparison between the magnetic properties of the Fe₇₀Co_{3.5}Cu₁Nb₃B₉Si_{13.5} and Fe_{73.5}Cu₁Nb₃B₉Si_{13.5} (FINEMET) mechanically alloyed powders is also reported.

[7] A METHOD TO PRODUCE TWO-PHASE NANOCOMPOSITES IN SOLID STATE

He L. Allard LF. Ma E. - Nanostructured Materials. 12(1-4 Part A Special Issue SI):543-546, 1999

We demonstrate that full-density, bulk, two-phase nanocomposites can be produced starting from a powder mixture of two coarse-grained individual phases in equilibrium. Instead of going through liquid or vapor phase precursors, mixing is achieved through mechanical milling, a highly nonequilibrium driven process imposing external forcing. The precursor created is a supersaturated solid solution with nanocrystalline grain sizes. Upon subsequent hot consolidation to bulk samples, phase separation occurs on the scale of the nano-grains, forming two-phase nanocomposites in situ. Transmission electron microscopy of the Cu₆₀Fe₄₀ sample presented in this paper confirm that, after full density processing using the sinter forging consolidation scheme, nanophase grains are maintained and a homogeneous two-phase microstructure is obtained.

[6] A STUDY OF AL-MO ALLOYS SYNTHETIZED BY MECHANICAL TREATMENT AND ANNEALED IN-SITU.

Enzo S. Frattini R. Canton P. Mulas G. Radaelli P. - Nanostructured Materials. 12(1-4 Part A Special Issue SI):547-550, 1999

Al₇₅Mo₂₅ alloys were synthesised by ball milling of pure elements. The decrease of fcc aluminium peaks from the neutron diffraction patterns as a function of mechanical treatment time suggests the formation of an extended Mo(Al) solid solution, even though no significant change of the bcc lattice parameter is observed. Further evidence of the existence of a metastable phase is supplied a posteriori by in situ annealing of the processed metal powders at temperatures below 750 degrees C. The main product is the intermetallic Al₈Mo₃ compound.

[5] PHASE TRANSFORMATIONS IN NANOCRYSTALLINE MECHANICALLY ALLOYED NI-MO POWDERS

Oleszak D. Portnoy VK. Matyja H. - Nanostructured Materials. 12(5-8 Part B Special Issue SI):621-624, 1999

The mechanical alloying process of Ni-20 at.% Mo powder mixture led to the formation of fcc Ni(Mo) solid solution, characterized by lattice parameter of 0.3612 nm, crystallite size 30 nm and lattice strain 0.8%. The synthesis in argon of Ni-43 at.% Mo alloy, both under high and low energy conditions, resulted in the formation of two-phase structure. amorphous + nanocrystalline Mo. The presence of oxygen during milling in air and with addition of ethanol, reduced the structure refinement and formation of Ni(Mo) solid solution. Moreover, the amorphous structure was not observed in this case.

[4] NANOCRYSTALLINE BCC SOLID SOLUTIONS OF AL-FE-V SYSTEM PREPARED BY MECHANICAL ALLOYING

Fadeeva VI. Portnoy VK. Baldokhin YV. Kotchetov GA. Matyja H. - Nanostructured Materials. 12(5-8 Part B Special Issue SI):625-628, 1999

Solid state reactions in Al₅₀Fe₅₀, Al₅₀Fe₄₅V₅ and Al₅₀Fe₂₅V₂₅ powder mixtures by mechanical alloying (MA) and following annealing were studied. MA was performed in the high energy planetary ball mill in an argon atmosphere. X-ray diffraction (XRD) and Mossbauer spectroscopy (MS) methods were used. It was established that the partially ordered solid solution of B2 type has been formed in the Al₅₀Fe₅₀ alloy and the BCC (A2) phase has been formed in the V containing alloys. According to MS one can conclude that the short range order of B2 type in the mechanically alloyed Al₅₀Fe₄₅V₅ and Al₅₀Fe₂₅V₂₅ alloys was presented too. MA phases were characterised by a crystallite size of 10 nm. Annealing at 750 degrees C for 2h of mechanically alloyed Al₅₀Fe₅₀ and Al₅₀Fe₄₅V₅ resulted in the formation of completely ordered B2 phases. After annealing of the Al₅₀Fe₂₅V₂₅ alloy the A2 structure transformed in two phases - with B2 and D8(a) structure.

[3] CHARACTERIZATION OF FERRITES SYNTHESIZED BY MECHANICAL ALLOYING AND SOFT CHEMISTRY

Millot N. Colin SB. Perriat P. Le Caer G. Welter R. Malaman B. - Nanostructured Materials. 12(5-8 Part B Special Issue SI):641-644, 1999

For the purpose of comparing nanostructured materials prepared by different synthesis methods, nanocrystalline Fe-based spinels were synthesized using two different routes: soft chemistry and high-energy ball milling. The as-prepared powders were characterized notably by thermogravimetric analyses and Fe-57 Mossbauer spectrometry.

[2] EXAFS STUDY ON NANOCRYSTALLINE FE₄₀CO₁₀Cu₅₀ ALLOY PROCESSED BY MECHANICAL ALLOYING

Yang DS. Yoo YG. Yu SC. - Nanostructured Materials. 12(5-8 Part B Special Issue SI):665-668, 1999

Metastable Fe₄₀Co₁₀Cu₅₀ alloys were fabricated by mechanical alloying process from the mixed powder. The structural changes during mechanical alloying process and post heat treatment were examined by Fe, Co, and Cu K-edge EXAFS measurements. The EXAFS analysis verified that Fe and Co atoms diffused to the Cu matrix so that the metastable Fe-Co-Cu with fcc structure was formed by the mechanical alloying. Also we found that the metastable Fe-Co-Cu alloy was decomposed into Fe-Co alloy and Cu by annealing process.

[1] NEUTRON DIFFRACTION STUDIES OF CERIA-ZIRCONIA CATALYSTS PREPARED BY HIGH-ENERGY MECHANICAL MILLING

Enzo S. Frattini R. Delogu F. Primavera A. Trovarelli A. - Nanostructured Materials. 12(5-8 Part B Special Issue SI):673-676, 1999

The structural transformation occurring to a mixture of cubic CeO₂ and monoclinic ZrO₂ by High-energy Mechanical Milling (MM) has been followed by neutron diffraction. Rietveld analysis indicates that 9 hours of milling produce a tetragonal solid solution for Zr-rich composition and a nanocrystalline cubic solid solution for Ce-rich composition.

JRFM'2000

5èmes Journées du Réseau Français de Mécanosynthèse
23 et 24 Mai 2000 - Bordeaux, ICMCB

Thématique retenue

Caractérisations physico-chimiques des matériaux élaborés par mécanosynthèse

Conférences Invitées

- **Vers la Modélisation de la Mécanosynthèse**
Prof. Jacques FOCT, Université de LILLE

- **Elaboration et caractérisation de nanotubes élaborés par mécanosynthèse**
Dr Laurent CHAFFRON, CEA saclay-CEREM-SRMP/DTM, Gif sur Yvette

- **La Mécanosynthèse au Japon**
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