

RESEAU FRANCAIS DE MECANOSYNTHESE

Lettre N°43

Octobre 1998

125 Groupes de Recherche
(dont 62 à l'étranger)

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

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Les différentes propositions reçues pour la conférence ISMANAM98 sont désormais en ligne sur le site web de la conférence :

<http://www.uow.edu.au/conferences/ismanam98>

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

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

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ANNONCE DE CONGRES ET / OU ECOLES CONGRESS AND SCHOOL ANNOUNCEMENTS

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All the details may be obtained by E-Mail to E. Gaffet

**The Reh binder Memorial International Conference
on Colloid Chemistry and Physical Chemical Mechanics**

Moscou (Russie) - 4 - 8 Octobre 1998

Contact : Prof. N.B. Uriev - Institute of Physical Chemistry - Leninsky Prospect 31 -
11795 Moscow - Russie

E-Mail : Reh binder98rehb.chem.msu.su ou <http://www.chem.msu.su>

Powder Metallurgy 98

Granada - Espagne - 18 - 22 Octobre 1998

Site Web : <http://www.epma.com/congress/>

Gorham / Intertech Conference on "Commercializing Nanotechnology 98 : Bridging New Materials to Market"

19 - 21 Octobre 1998 Nashville - TN USA

Contact : C.E. Spear - Intertech Corporation : Fax 207 - 781 - 2150

JA 98

Paris - 27 - 29 Octobre 1998

Symposium 1 : Phénomènes déterministes et aléatoires en science des matériaux

Symposium 2 : Matériaux poreux et mousses : élaboration, structures et propriétés

Symposium 3 : Les hydrures métalliques

Symposium 4 : Propriétés Thermophysiques et thermomécaniques des matériaux de la mesure à la simulation de procédés industriels

Symposium 5 : Analyse d'images et reconnaissance de formes en matériaux

Symposium 6 : Lois de comportement et calcul de structures

Org. SF2M - Contact : SFMM@wanadoo.fr

Fifth Internation Symposium on Quatum Confinement : Nanostructures

194th Meeting of the Electrochemical Society
1 - 6 Novembre 1998 - Boston - MA - USA
[http : //www.electrochem.org](http://www.electrochem.org)

Fine, Ultrafine and Nano Powders '98
Crowne Plaza Hotel - LaGuardia Airport - 8 - 10 Novembre 1998
Contact : E-Mail : Tombcc@aol.com

**Sixth Foresight Conference
on Molecular Nanotechnology**
Westin Hotel - Santa Clara - 12 15 Novembre 1998
California Satellite Conference to Nano'98
Contact : E-Mail : globus@nas.nasa.gov

Symposium on Advanced Technologies for Particle Production

AIChE Annual Meeting
15 - 20 November - Miami Beach - FL - USA
Technical Sessions and ChairPersons
1/ Particle Synthesis in Dispersions and Supercritical Fluids-R. Davis/MT Harris/D. Tomasko
2/ Sol - Gel Synthesis of Particles - A McCormick/PN Kumta/T. Okubo
3/ Chemical Kinetics during Particle Formation - J. Floess, K. Higashitani, S. E. Pratsinis
4/ In-Situ Diagnostics during Particle Formation-Ph. W. Morrison,R.M. Carangelo, D.T. Spicer
5/ Agglomerate Particle Dynamics - G. Fotou, SK Friedlander, Takahashi
6/ Computational Fluid Dynamics during Particle Formation and Growth - L. Collins, K. Kontomaris
7/ Aerosol Reactors - A.W. Weimer, M. Kamal Akhtar
8/ Particle Charging - T. Matsoukas
9/ Film synthesis by Particle Technologies - G. Grader, S. Bhandarkar
10/ Nanoparticles - M. Senna, TJ Mountziaris, H. Glicksmn
11/ Particulate deposits : Transport mechanisms, microstructure and properties : D. Rosner
12/ Posters on Advanced Technologies for Particles Production : G. Beaucage, H. Riemenschneider
Web Site : www.aiche.org

ISMANAM98

International Symposium on Metastable, Mechanically Alloyed and Nanocrystalline Materials
Wollongong (Sydney) - Australie - 7 - 12 Décembre 1998

International Advisory Committee :

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Contact : A. Calka E-Mail : Andrzej_Calka@uow.edu.au et

WebSite : <http://www.uow.edu.au/conferences/ismanam98>

**Satellite Symposium on Mechanochemistry / ISMANAM98
(Mechanochemical Synthesis and Mechanochemistry)**

Wollongong - Australie 7 /12 Decembre 1998

International Advisory Committee :

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E. Gaffet, E. Gutman, M. Senna, C. Suryanaryana, R. Schwarz

WebSite : <http://www.uow.edu.au/conferences/ismanam98>

Nanostructured Hybrid Materials

Symposium TMS Annual Meeting - San Diego CA - USA - 28 Février 4 Mars 1999
Contact : gmchow@anvil.nrl.navy.mil

4th International Workshop on Metastable Phases (IV IWOMP)

7 - 9 Avril 1999 - Bologne - Italie

Contact : Bonetti@df.unibo.it

12th International Conference on Wear of Materials

Atlanta - Georgie / USA - 25 - 29 Avril 1999

contact : Amy Richardson E-Mail A.Richardson@elsevier.co.uk

or web site : <http://www.elsevier.nl/locate/wom99>

**Nouveau
New**

JRFM'99

4èmes Journées du RFM
2 & 3 Juin 1999 - Dijon - France
Web Site :<http://www.bls.fr/amatech>

**Nanostructured Materials Symposium at the 5th IUMRS International Conference
on Advanced Materials
(IUMRS - ICAM'99)**

Beijing - Chine - 13 - 18 Juin 1999
Contact : Kelu@imr.ac.cn
WebSite - <http://www.chimeb.edu.cn>

PM2 Tec 98

1999 International Conference
on Powder Metallurgy and Particulate Materials
Vancouver - 20 / 24 Juin 1999
E-Mail : Info@mpif.org - Website: www.mpif.org

10th International Conference on Rapidly Quenched and Metastable Materials (RQ10)

Bangalore - Inde - 23 - 27 Août 1999
Website : <http://www.metalrg.iisc.ernet.in/rqten/>

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Annonces de Soutenance de Thèses
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**Transformations antiferromag - ferromag - paramagnétiques - verre de spin dans les alliages de Fe Rh
nanocristallisés par Broyage**

E. Navarro - Université de Complutense - Madrid - Espagne - 18 Mai 1998
Co directeurs : A. Hernando - A.R. Yavari

=====
**Modifications morphologiques et microstructurales du matériau actif des cathodes de batteries à l'ion lithium
induites par broyage et traitement thermique**

Ph. Perrot - Université de Poitiers - 6 Mai 1998
Co - Directeurs : E.L. Mathe, M. Grosbras

Jury : J. Mimault, H. Van Damme, A. Dager, M. Broussely, P. Goudeau, E.L. Mathe, M. Grosbras

=====
Effects of the mechanical milling on carbons : negative electrode materials of Li - ion batteries"

F. Salver Disma - Université de Picardie Jules Verne - 4 Février 98

Jury : Aymard L., Beguin F., Coulon M., Furdin G., Lassegues JC, Percheron Guegan A., Rouzaud JN, Tarascon JM.

=====
**"Elaboration et Caractérisations de Cermets Alumine - Métal à partir de poudres obtenues par
Mécanosynthèse"**

J.-L. Guichard - INPL - Nancy - 23 Janvier 1998

Jury : A. Simon, C. Carry, F. Thévenot, G. Le Caër, A. Mocellin

=====
**"Spinelles nanométriques à valence mixte et à fort taux de lacunes cationiques : Transfert électroniques dans
un ferrite de molybdène Fe_{2.47}Mo_{0.53}O₄, de la synthèse aux propriétés magnétiques dans le système fer -
vanadium Fe_{3-x}V_xO₄ (0²x<2).**

V. Nivoix - Université de Bourgogne - 17 Décembre 1997

Jury : M. Lenglet, H. Pascard, G. Bertrand, E. Gaffet, M. Guyot, M. Lallemand, A. Rousset, B. Gillot

=====
"The Preparation of Nitrides and Carbides by Mechanical Treatment - Phases and Structures"

G.M. Wang - School of Physics, University College, The University of New South Wales - Australian Defence Force
Academy - Canberra, ACT 2600 - Australia - 10/12/97

Supervisor - S.J. Campbell - **Co - Supervisors**: W.A. Kaczmarek and A. Calka

=====
**"Suivi par Diffraction X en Temps Réel de la Formation par Combustion des intermétalliques des systèmes Al -
Ni, Al - Ti, Al - Ni - Ti"**

J. F. Javel - Université de Nancy I - 3 Octobre 1997

Jury : J.F. Berar, F. Bernard, M. Bessiere, M. Dirand, J.C. Gachon, P. Galez, J.C. Jorda

=====
"Contribution à l'Etude de la Transformation - Tribologique Superficielle en Fretting"

E. Sauger - Ecole Centrale de Lyon - Génie des Matériaux - 26 Septembre 1997

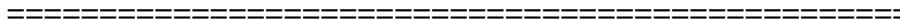
Jury : L. Mora - Ponsonnet, P. Blanchard, K. Dang Van, C. Esnouf, E. Gaffet, E. Rosset, A.B. Vannes, L. Vincent

Sites internet à découvrir

Site sur la cristallographie / Soft + Littérature

<http://www.lmcp.jussieu/sincris-top/logiciel>

N.B. : si vous connaissez d'autres sites en relation avec les thèmes développés par le RFM, faites nous les connaître



Post Doc Position Proposals

Belgique

The Department Metallurgy and Materials Engineering (MTM) of the K.U.Leuven (Belgium) has a research position available. Candidates are asked to contact the responsible staff member.

Area of research :

Metals and Alloys, Polymer Matrix Composites, Intelligent Processing of Materials, Surface Engineering and Tribology, Metal Forming and Mechanical Behaviour of Materials, Quality Control and Non-Destructive Testing of Materials, Ceramics, Thermodynamics, Corrosion, Nuclear Engineering

Description of research task

Tailor made powders by mechanical alloying of Fe and Cu based materials. Application field: specific composite materials, to be prepared by conventional PM consolidation techniques. Research activities: parametric study of MA, alloy design, microscopic

Staff member to be contacted

Prof. Dr. Ir. L. Froyen

Katholieke Universiteit Leuven - Dept. MTM

de Croylaan 2 - B-3001 Leuven (Belgium)

Tel. +32/16/22.09.31

Japon

Our group: Nanocomposite Group, Department of Composite Materials, National Institute of Materials and Chemical Research, Tsukuba, Ibaraki, Japan is now looking for post-doc researchers

The candidates would be integrated in the Nanocomposite Group of the Department of Composite Materials. The research interests of the group are mainly focused on nanocomposite preparation and its optical/chemical functionalities. Research projects currently under way aim to develop nanostructured and optically/chemically active thin films by sputtering, laser ablation and so on. For additional information about the Institute and group :

<http://www.nimc.go.jp/>

<http://www.aist.go.jp/NIMC/fcg/index.html>

Experience in the fields of materials science (ceramic or metal) is required.

There are two types of post-doc positions.

1. Long-term: from 6 months to 2 years

2. Short-term: from 1 to 3 months

If you or someone in your laboratory is interested in this fellowship, please contact as soon as possible to:

Dr. Naoto Koshizaki - Department of Composite Materials

National Institute of Materials and Chemical Research(NIMC) 1-1 Higashi, Tsukuba, Ibaraki 305-8565 JAPAN

Tel: +81-298-54-6335 - Fax: +81-298-54-6252 - E-mail: koshizaki@nimc.go.jp - <http://www.aist.go.jp/NIMC/fcg/index.html>

Bibliographie Récente

Livres ou "Special Issues"

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.

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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)

SPECIAL OFFER TO PARTICIPANTS OF ISMANAM-97. THIS UNIQUE BIBLIOGRAPHY IS AVAILABLE AT A REDUCED PRICE OF £50.00 (including postage and packing)

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, the entries will be mentioned in an alphabetical order according to the first-named author. Each reference provides the listing of all the authors (in the sequence they appear in the publication), full title of the publication, source (journal, book chapter, conference proceedings, patent, etc.), volume, year of publication, page numbers (starting and ending) and language of the document, if it is not in English. A reference, wherever available, to the entry number under which this publication was abstracted by Metals Abstracts (jointly published by the ASM International and Institute of Materials) will be given so that the reader can have access to the abstract before looking up the original article for full details. These items will be followed by letter symbols to indicate the topics and properties discussed in that publication. A comprehensive author index, subject index, and material index, is also provided at the end of the bibliographic entries. 234x156 mm, 440 pages, cased, Price to ISMANAM-97 delegates £50, ISBN: 1898326126

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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 assurée grâce au concours de M^{me} TAUZIN - FIN BiPSé)

N.B. : En cas de difficultés à vous procurer une copie des articles suivants,
n'hésitez pas à contacter E. Gaffet (CNRS / IPSé - Belfort)

[100] IN SITU FORMATION OF TiB₂ REINFORCED ALUMINIUM VIA MECHANICAL ALLOYING

Lu L. Lai MO. Niu XP. Ho HN. - Zeitschrift für Metallkunde. 89(8):567-572, 1998

This paper investigates the possibility of in-situ formation of TiB₂ ceramic particulates via the process of mechanical alloying of elemental Ti and B powders in a system diluted with Al. Elemental Al powder was mechanically milled together with Ti and B powders in a high energy planetary ball mill. 30 stainless steel balls with diameter of 15 mm were employed. The ball to powder weight ratio was 20:1. X-ray spectra revealed the evolution of structural changes of the alloyed powder particles. No evidence of formation of TiB₂ was manifested when the milling duration was shorter than 30 h. A TiB₂-like structure could be detected only after 40 hour of milling. Because of broadening and weakening of the X-ray diffraction (XRD) peaks corresponding to Al and TiB₂ structures as a result of refinement of crystalline size and the presence of large structural distortion, only very weak and vague TiB₂ XRD peaks could be detected. Clear evidence of formation of TiB₂ was, however, observed when the mechanically alloyed powder was annealed. Therefore, the results of the present investigation clearly indicate that formation of in-situ TiB₂ particulates within Al is possible although a longer milling duration is required. Mechanical testing shows that both the 0.2 % yield and the ultimate tensile stresses are increased with MA duration and sintering duration.

[99] EFFECT OF MECHANICAL ALLOYING ON THE PROPERTIES OF POWDER METALLURGY COMPOSITES OF THE AL-SiC SYSTEM

Vishnyakov LR. Oniskova NP. Gribkov AN. Romashko IM. - POWDER METALLURGY AND METAL CERAMICS. 36(11-12):599-603, 1997

The process of mechanical alloying used in the powder metallurgy processing of Al-SiC materials was applied to the treatment of a mixture of aluminum and silicon carbide particles in an attritor. Optimal processing led to the formation of composite pellets containing SiC particles uniformly distributed in an aluminum alloy matrix. Subsequent compaction of these mechanically alloyed pellets produced material with ultimate strength 587 MPa, elastic modulus 10.5 GPa, and elongation 2.1%. Comparison of measured with calculated values of the elastic modulus indicated a high degree of matrix continuity in the structure of the mechanically alloyed composite material.

[98] SUPERSATURATED SOLID SOLUTIONS AND METASTABLE PHASES FORMATION THROUGH DIFFERENT STAGES OF MECHANICAL ALLOYING OF FeTi

Novakova AA. Agladze OV. Sveshnikov SV. Tarasov BP. - Nanostructured Materials. 10(3):365-374, 1998

Elemental equiatomic Fe-Ti powder mixture was mechanically alloyed in high energy ball mill. XRD, DTA and Mossbauer spectroscopy (at liquid nitrogen temperature) were utilized to monitor the kinetics as well as the accompanied structural and phase transformations through different stages of milling. Our experiments showed that formation of nanocrystalline FeTi compound proceeds via the formation of the supersaturated solid solutions beta-Ti(Fe) and alpha-Fe(Ti) at the interface. After 36 hours of milling, the main part of powder mixture transformed not only to FeTi but also to Fe₂Ti intermetallic compound. The transition of last part of supersaturated solid solutions beta-Ti(Fe) to those intermetallic phases was observed after annealing of this sample at 600 degrees C.

[97] A COMBINED STUDY OF NANOCRYSTALLINE ALUMINIUM BY X-RAY DIFFRACTION AND MECHANICAL SPECTROSCOPY

Bonetti E. Pasquini L. Sampaolesi E. - Nanostructured Materials. 10(3):437-448, 1998

Nanocrystalline aluminium was prepared by ball milling in different conditions. The milled powders were characterized by X-ray diffraction in order to determine accurately the crystal size and the internal strains. Mechanical spectroscopy measurements in the 300-700 K temperature range were performed with a torsion pendulum on consolidated nanocrystalline powders. The anelastic spectrum is characterized by a broad internal friction peak and an exponential background. These data are compared with those obtained on a coarse grained sample and correlated with the information derived from X-ray diffraction analysis.

[96] EFFECTS OF MILLING LIQUID ON THE REACTION-BONDED ALUMINUM OXIDE PROCESS

Watson MJ. Chan HM. Harmer MP. Caram HS. - JOURNAL OF THE AMERICAN CERAMIC SOCIETY. 81(8):2053-2060, 1998

The reaction-bonded aluminum oxide process begins with aluminum, Al₂O₃, and usually ZrO₂ powders that have been attrition-milled in an organic liquid. The attrition-milled powder is then compacted and heat-treated in air to produce polycrystalline, Al₂O₃-based ceramics. Safety considerations have made it desirable for the milling liquid to be changed from acetone to a less-flammable solvent. In this paper, mineral spirits, ethanol, and mineral spirits that contains 2 wt% stearic acid are presented as viable alternatives to acetone. The effects of changing the milling liquid on the reaction process and the properties of the final fired ceramic are investigated.

[95] GRINDING TIME FOR CONTROL OF THE SIZE FRACTION OF PRODUCTS IN THE ATTRITION MILLING

Park JK. Jeong Y. Yang JI. Jung MY. - Korean Journal of Chemical Engineering. 15(4):375-380, 1998

Grinding tests for garnet were carried out by using an attrition mill under wet processes. Effects of feed filling ratios and a chemical agent (sodium hexametaphosphate, SHP) were investigated on the grinding time of the garnet. The progeny particles obtained were screened into various particle size intervals, which were 100 mesh over, 100/400 mesh and 400 mesh under. In order to estimate the mass fraction of the particles in a given particle size interval, mathematical models were derived from the first-order reaction model, then compared to experimental data. It was observed that variation of the feed filling ratio did not show a significant effect on the mass fraction of the product. The chemical agent was, however, effective so that the mass fraction could be controlled by adjusting the content of SHP.

[94] SYNTHESIS OF NiAl-TiC NANOCOMPOSITE BY MECHANICAL ALLOYING ELEMENTAL POWDERS
Zhou LZ. Guo JT. Fan GJ. - MATERIALS SCIENCE AND ENGINEERING A-STRUCTURAL MATERIALS PROPERTIES MICROSTRUCTURE AND PROCESSING. 249(1-2):103-108, 1998

A NiAl-TiC nanocomposite has been synthesized by mechanical alloying from Ni, Al, Ti, and C powders. During milling, an abrupt reaction occurred, resulting in simultaneous formation of NiAl and TiC phases. It is suggested that two separate exothermic explosive reactions, i.e. $Ni + Al \rightarrow NiAl$ and $Ti + C \rightarrow TiC$, were involved. However, the reactions were incomplete with the existence of a small amount of elemental powders. Prolonged milling led to a gradual formation of NiAl and TiC as well as grain refinement. The final grain size for TiC was 3.5 times as large as that for NiAl. The formation mechanism of the NiAl-TiC nanocomposite during mechanical alloying was also discussed.

[93] MECHANICAL PROPERTIES AND FRACTURE BEHAVIOR OF SiCw REINFORCED Al-12Ti ALLOY PREPARED BY MECHANICAL ALLOYING TECHNIQUE

Jia DC. Zhou Y. - MATERIALS SCIENCE AND ENGINEERING A-STRUCTURAL MATERIALS PROPERTIES MICROSTRUCTURE AND PROCESSING. 252(1):44-52, 1998

Room and elevated temperature mechanical properties and thermal stability of SiC whisker (SiCw) reinforced Al-12Ti alloys fabricated by mechanical alloying were investigated. The results show that SiCw/Al-12Ti composites manifest higher room and elevated temperature strength and Young's modulus and specific strength and stiffness with increasing SiCw content, which can be attributed to the combined strengthening from Al₃Ti particles and SiC whiskers. The combined strengthening effect is especially good for lower SiCw content. The thermal stability of the composites at 550 degrees C worsens compared to that of the unreinforced MA Al-12Ti alloy. The in situ crack propagation observation under SEM shows that the fracture behavior of SiCw/Al-12Ti composites substantially differs from that of the MA Al-12Ti alloy, but changes slightly with increasing SiCw content. The strengthening mechanisms of the composites are also discussed.

[92] SYNTHESIS OF AN AMORPHOUS Zr-Al-Ni-Cu ALLOY WITH LARGE SUPERCOOLED LIQUID REGION BY COLD-ROLLING OF ELEMENTAL FOILS

Sagel A. Sieber H. Fecht HJ. Perepezko JH. - Acta Materialia. 46(12):4233-4241, 1998

Amorphous samples of a multicomponent Zr-Al-Ni-Cu alloy with a large supercooled liquid region of 98 K have been synthesized at ambient temperature from a layered array of individual elemental sheets by repeated low strain rate cold-rolling with intermediate folding. The alloying reactions during cold-rolling were monitored with electron microscopy, X-ray diffraction and thermal analysis. During the initial stage of processing a dissolution of solute into Zr along with a reduction in grain size to about 30 nm was observed and with further processing a fully amorphous sample was obtained after 120 deformation cycles. The characteristics of the amorphous phase developed by repeated cold deformation indicate that it is compositionally homogeneous, structurally more relaxed as compared to mechanically alloyed amorphous powders of similar composition and without the typical impurity levels obtained by mechanical alloying.

[91] SOLID SOLUBILITY OF CARBON IN COPPER DURING MECHANICAL ALLOYING

Saji S. Kadokura T. Anada H. Notoya K. Takano N. - Materials Transactions Jim. 39(7):778-781, 1998

Solubility of carbon in solid copper during mechanical alloying using an Attritor type of ball mill was investigated by means of X-ray diffraction, microscopy, electron probe microanalysis and chemical analysis. Carbon atoms less than about 28.5 atomic percent are soluble in the solid copper consisting of nano-crystals during mechanical alloying of copper-graphite mixed powders. The supersaturated solute carbon atoms take the interstitial positions in the fcc alpha-Cu solid solution and result in the lattice expansion of the alpha-Cu.

[90] DEFECT CHEMISTRY OF THE SHELL REGION OF WATER-MILLED BaTiO₃ POWDERS

Abicht HP. Voltzke D. Schneider R. Woltersdorf J. Lichtenberger O. - MATERIALS CHEMISTRY AND PHYSICS. 55(3):188-192, 1998

The sintering behaviour of BaTiO₃ powders and the resulting microstructure of the corresponding ceramics are controlled by the stoichiometry of the outer region of the grains of the starting powder. These grains exhibit a core-shell structure due to the water attack during powder milling. The hydrolysis results in the formation of lattice-hydroxyl groups. The existence of these processing-related hydroxyl defects is detected by H-1 magic-angle spinning nuclear magnetic resonance (H-1-MAS NMR) as well as differential thermal analysis/thermal gravimetry (DTA/TG) measurements and also verified by electron energy-loss spectroscopy (EELS) in combination with high-resolution transmission electron microscopy (HRTEM). The results are confirmed by detecting the near-edge structure of the O-K edge, the details of which can be interpreted by quantum-mechanical calculations of the electronic density of states in the conduction band.

[89] GRAIN GROWTH BEHAVIOR OF NANOCRYSTALLINE INCONEL 718 AND Ni POWDERS AND COATINGS
Jiang HG. Lau ML. Lavernia EJ. - Nanostructured Materials. 10(2):169-178, 1998

Nanocrystalline Inconel 718 and Ni powders were prepared using two approaches: methanol and cryogenic attritor milling. High velocity oxy-fuel (HVOF) spraying of milled Inconel 718 powders was then utilized to produce coatings with a nanocrystalline grain size. Isothermal heat treatments were carried out to study the thermal stability of the methanol milled and cryomilled powders, as well as the HVOF-derived coatings. All nanocrystalline Inconel 718 powders and coatings studied herein exhibited significant thermal stability against grain growth by maintaining a grain size around 100 nm following annealing at 1273 K for 60 min. In the case of the cryomilled nanocrystalline Ni powders, isothermal grain growth behavior was studied, from which the parameters required for the prediction of the microstructural evolution during a non-isothermal annealing were acquired. The theoretical simulation of grain growth behavior of nanocrystalline Ni during non-isothermal annealing conditions yields results that are in good agreement with the experimental results.

[88] ALLOY FORMATION IN THE Fe-Ni SYSTEM DURING COLD MECHANICAL ACTIVATION BY PRESSURE SHEAR

Mukoseev AG. Shabashov VA. Pilugin VP. Sagaradze VV. - Nanostructured Materials. 10(2):273-282, 1998
Binary alloys of the Fe-Ni system were used to study the regularities involved in the cold mechanical synthesis realized by pressure shear in Bridgman anvils. The alloy formation was found to depend on the crystal lattice symmetry: the solid solution is formed in mixtures of components having a similar symmetry of the crystal lattice but the alloy formation is retarded or does not occur altogether in mixtures of components having different symmetries of the crystal lattice. The kinetics of the alloy formation in mixtures of Fe-Ni solid solutions with a FCC lattice was determined. The phase transformations were found to affect the kinetics and the alloy formation mechanism in mixtures whose components are pressure- and strain-unstable.

[87] SINTERING OF NANOSTRUCTURED W-CU ALLOYS PREPARED BY MECHANICAL ALLOYING

Kim JC. Moon IH. - Nanostructured Materials. 10(2):283-290, 1998

Nanostructure Q (NS) powders with compositions corresponding to W-20wt%Cu and W-30wt%Cu were prepared by mechanical alloying. The microstructure and grain size of as-milled and annealed powders were analyzed by transmission electron microscopy. The compacted specimens were sintered at temperatures in the range 1000 degrees to 1300 degrees and then the microstructures of sintered parts were analyzed by scanning electron microscopy. Sintering of mechanically alloyed W-Cu alloys appears to be independent of Cu content, and may be explained in terms of recovery and grain growth in the mechanically alloyed powders as well as impurity activated sintering of W. After sintering, Cu pools are formed outside the mechanically alloyed powders. A relative sintered density of more than 95% is obtained by particle rearrangement during liquid-phase sintering, and the greatest homogeneity of W and Cu phases is achieved by sintering at 1200 degrees.

[86] EFFECT OF PARTICLE SIZE DISTRIBUTION AND MIXING HOMOGENEITY ON MICROSTRUCTURE AND STRENGTH OF ALUMINA/COPPER COMPOSITES

Oh ST. Sekino T. Niihara K. - Nanostructured Materials. 10(2):327-332, 1998

The powder mixtures of Al₂O₃/CuO with a variation in particle size distribution and mixing homogeneity were prepared using different milling processes. The mixing homogeneity was quantitatively determined by the coefficient of variation. Homogeneous microstructure and a high fracture strength of the hot-pressed Al₂O₃/Cu composites could be achieved using the powder mixture with narrow size distribution and high mixing homogeneity. The relationship between mixing homogeneity and properties was discussed based on the role of unevenly dispersed second phase for the matrix grain growth and for the local variation of mechanical characteristics.

[85] STRUCTURAL AND MAGNETIC PROPERTIES OF MECHANICALLY ALLOYED FE₈₁SI₉B₆NB₃CU₁ POWDERS

Gupta R. Enzo S. Frattini R. Hernando A. Marin P. Mulas G. Perin A. Principi G. - International Journal of Non-Equilibrium Processing. 10(3-4):283-296, 1998

Nanocrystalline powders of Fe₈₁Si₉B₆Nb₃Cu₁ composition have been synthesized by high-energy ball milling and characterized by means of X-ray and neutron diffraction, Mossbauer spectroscopy and magnetic tests. The diffraction and Mossbauer measurements point out the progressive formation of a bcc alpha-Fe(Si, Nb, Cu, B) disordered solid solution with average particle size in the nanometre range. In addition, for the specimens mechanically treated for prolonged times, the Mossbauer spectra show an ultrafine non-stoichiometric Fe_{3+x}Si_{1-x} crystalline phase. The coercive field of the powder after long milling time is higher than that of the partially crystallized metallic glass of similar composition. The annealing at 973 K in inert atmosphere of the samples mechanically alloyed for long times mainly produces the Fe₃Si crystalline phase and small amounts of borides and Fe-Nb aggregates.

[84] SYNTHESIS OF NANOCRYSTALLINE AL₆₀NI₄₀ ALLOY PRODUCED BY ROD MILLING

Kim HG. Myung WN. - International Journal of Non-Equilibrium Processing. 10(3-4):305-314, 1998.

We report on the synthesis of nanocrystalline Al₆₀Ni₄₀ powders by rod milling. X-ray diffraction, scanning electron microscopy, transmission electron microscopy, and a vibrating-sample magnetometer are utilized to characterize the as-milled powders. When the powders are milled for 200 h, the Al₃Ni₂ and b.c.c. AlNi phases appear. After 800 h, only the b.c.c. AlNi phase is detected on XRD patterns. The crystallite size and the saturation magnetization decrease drastically with increased time up to 200 h. At 800 h, the crystallite size and the saturation magnetization reach values of 5.7 nm and 1.28 emu/g respectively, suggesting the formation of a b.c.c. phase. Strain increases drastically with increased time up to 200 h and reaches a value of 7.07 x 10⁻⁴ above 200 h.

[83] EFFECT OF HEAT-ASSISTED GRINDING OF A CALCIUM HYDROXIDE GIBBSITE MIXTURE ON FORMATION OF HYDRATED CALCIUM ALUMINATE AND ITS HYDRATION BEHAVIOR

Kano J. Yamashita H. Saito F. - Powder Technology. 98(3):279-280, 1998

Heat-assisted grinding of a mixture of Al(OH)₃ and Ca(OH)₂ was conducted at different temperatures ranging from room temperature (about 293 K) to 773 K using a tumbling ball mill. The grinding of the mixture for up to 100 h at 373 K enabled us to synthesize hydrated calcium aluminate directly. This compound was not formed by grinding at room temperature nor at a temperature higher than 473 K. All the mixture ground for 100 h at any temperature was transformed into excess hydrated calcium aluminate by the addition of water but the unground mixture remained unchanged.

[82] MICROSTRUCTURAL STUDY OF A MECHANICALLY ALLOYED ODS SUPERALLOY

Mujahid M. Gater CA. Martin JW. - JOURNAL OF MATERIALS ENGINEERING AND PERFORMANCE.

7(4):524-532, 1998

Extruded bars of oxide-dispersion-strengthened (ODS) alloy MA-6000 have been annealed isothermally as well as in temperature gradients. The temperatures used for annealing produced secondary recrystallization in all the samples, although the final grain aspect ratio was different for each annealing process. Interrupted gradient anneal experiments showed a curved secondary recrystallization front, with the surface recrystallizing at a lower temperature than the interior. It is believed this is caused indirectly by the strain gradients arising during extrusion. Grain-orientation analysis of recrystallized material revealed that a <110> fiber texture is present. A progressive grain reorientation toward <110> has been measured behind the recrystallization front using microbeam electron diffraction. In addition,

changes in the distribution, size, and morphology of different types of precipitates and particles occurring during various stages of annealing have also been studied, Grain-boundary pinning by the stable oxide particles plays an important role in determining the grain growth behavior at high temperatures.

[81] MAGNETIC PROPERTIES OF AMORPHOUS YFE2 OBTAINED BY MILLING

Larica C. Passamani EC. Nunes E. Orlando MTD. Alves KMB. Baggiosaitovitch E. - Journal of Alloys & Compounds. 274(1-2):23-28, 1998

Fe-57 Mossbauer spectroscopy in zero and non-zero applied magnetic fields, a.c. magnetic susceptibility and d.c. magnetization measurements have been performed on amorphous YFe₂ (a-YFe₂) milled powders obtained from the intermetallic compound in the temperature range from 4.2 to 300 K. The Mossbauer spectra recorded under an external magnetic field indicate that a-YFe₂ powder does not display a pure ferromagnetic interaction and shows remanence effects at low temperatures. The peak of the a.c. magnetic susceptibility, which occurs at T(f) approximate to 70 K, has a weak frequency dependence behavior, characteristic of a spin-glass phase transition-type. This behavior and the Mossbauer results lead us to conclude the absence of fine particle superparamagnetism effects and that the a-YFe₂ obtained by milling presents a complex magnetic structure. These results are consistent with those reported for Y-Fe amorphous films of similar composition, which have been described as displaying asperomagnetic order. The 120 h milled powder shows the presence of segregated alpha-Fe clusters detected by Mossbauer spectroscopy. These clusters were no longer observed after the sample had been treated at 523 K for 6 h.

[80] ON THE POSSIBILITY OF METAL HYDRIDE FORMATION - PART I - THE SYNTHESIS OF MGNI3B2 BY MECHANICAL MILLING AND SINTERING

Gross KJ. Zuttel A. Schlapbach L. - Journal of Alloys & Compounds. 274(1-2):234-238, 1998

In this first part of an ongoing investigation, we present results on the crystal structure determination of MgNi₃B₂. We also describe the novel procedure that was used to synthesize this compound. Using powder X-ray diffraction, MgNi₃B₂ was indexed in the space group P6(2)22. A Rietveld refinement of the X-ray profile determined the structure to be of the CeCo₃B₂ type. This is a variation of the CaCu₅ (P6/mmm) structure of the classic hydride forming family of LaNi₅ compounds. MgNi₃B₂ also has an elemental composition which is characteristic of hydride-forming intermetallic compounds. That is, the combination of a rare or alkaline earth metal which has a high affinity for hydrogen (Mg), with a transition metal (Ni). However, gaseous hydrogen and electrochemical treatments of MgNi₃B₂ did not produce a hydride. This study led us to develop a very useful procedure for synthesizing compounds which are difficult to produce using more standard techniques. Because of the high melting point of B (2030 degrees C) and the low boiling point of Mg (1107 degrees C at 1 atm) it was not possible to prepare MgNi₃B₂ by melting. We were able to successfully produce this alloy by ball milling for 30 min and sintering for 10 min at 800 degrees C. The method is fast and simple, and should prove very useful in the synthesis of other similar intermetallic compounds.

[79] STRUCTURE AND MAGNETIC PROPERTIES OF BALL-MILLED IRON NITRIDE POWDERS

Paseka I. Bezdecka P. Klarikova A. Zaveta K. - Journal of Alloys & Compounds. 274(1-2):248-253, 1998

In the present work we studied the influence of ball milling of iron nitrides-alpha' martensite+gamma austenite in the NH₃ atmosphere on the content of nitrogen, the phase composition, and the magnetic moment of nitrides. Only a small increase in the nitrogen content (from 6.5 to 7.0 at.% N) was found after milling. XRD and Mossbauer spectroscopy analyses of milled samples showed that at first austenite was transformed into nanocrystalline alpha-Fe and amorphous gamma'-Fe₄N and FeN_x during milling. The decrease in the austenite content was gradually accompanied also by the change of martensite into the above mentioned compounds. The structure of the samples milled for the longest time (8 h) was so destroyed that these samples were not able to be transformed into alpha''-Fe₁₆N₂ by subsequent annealing. The magnetic moment of milled samples firstly slightly increased and then decreased with increasing milling time.

[78] THE EFFECT OF HEAT TREATMENT ON THE ELECTRODE CHARACTERISTICS OF THE BALL-MILLED ZR-CR-NI

Jung CB. Lee KS. - Journal of Alloys & Compounds. 274(1-2):254-259, 1998

Electrodes of Zr-Cr-Ni alloys have been prepared by the ball milling process, starting from the are-melted Zr(Cr_{0.5}Ni_{0.5})₂ alloy and from the mixtures of elemental Zr, Cr and Ni powders. The nanocrystalline C14 structure has been obtained by either ball milling the are-melted alloy or heat-treating the ball-milled Zr-Cr-Ni powders. The plateau pressure for hydrogenation of nanocrystalline alloys was lower than that of conventionally produced alloys. The heat-treated Zr-Cr-Ni powders showed a similar discharge capacity as the are-melted alloy, but the activation was much easier. The structure and phase transformation occurring during the ball milling and heat-treating were analyzed and discussed.

[77] MECHANICALLY INDUCED REDUCTION OF ILMENITE (FeTiO3) AND RUTILE (TiO2) BY MAGNESIUM

Welham NJ - Journal of Alloys & Compounds. 274(1-2):260-265, 1998

Ilmenite (FeTiO₃) and rutile (TiO₂) have been ball milled with various ratios of metallic magnesium for 100 h in a laboratory mill. The reduction of both oxides was evident directly after milling: with substantial formation of MgO. Ilmenite also formed a mixed cation titanate (Mg, Fe)TiO₃ and elemental iron, no reduced phases were observed far rutile. There was no difference between the products obtained by thermal reaction and milling, although energetic reactions observed in milled powder by DTA were not seen in the milled sample and there was no obvious crystallisation of a reduced titanium phase. Solubilisation of titanium from milled powders was found to increase with magnesium content and was >90% for a starting ratio of 1: greater than or equal to 1 ilmenite: Mg. The unleached material was composed of incompletely reduced phases such as TiO₂, MTiO₃ and Ti₃O₅.

[76] COMPOSITE HYDRIDE ELECTRODE MATERIALS

Yang QM. Ciureanu M. Ryan DH. Stromolsen JO. - Journal of Alloys & Compounds. 274(1-2):266-273, 1998

A new class of electrode materials - composite hydride materials - is proposed for anodes in hydride-based rechargeable batteries. The composites were synthesized by mechanical mixing of two components: a major component having good hydrogen storage properties and a minor component used as surface activator. The major

component was selected among nonconventional or conventional hydride electrode materials; alloys and intermetallic compounds of the AB(2) or AB(5) type. The minor component was amorphous Mg_xNi_{1-x} ($x=0.4, 0.5$) or a 1:1 mixture of Mg_xNi_{1-x} and $LmM(5)$ (where Lm is a La-rich mischmetal and $M-5$ is $Ni_3.6Co_0.8Mn_0.3Al_0.3$). The role of the minor component was to improve the kinetics of electrochemical charging and to eliminate the initial activation of the major component. Experimental results have shown that the electrodes manufactured from composite materials have the following advantages: (1) elimination of the initial activation; (2) a significant improvement of electrode kinetics, resulting in better rate capabilities and higher charging efficiency; (3) a drop of the charging overpotential, resulting in better energy efficiency of the battery; (4) better cycling behavior; (5) in most cases, a considerable increase of the discharge capacity with respect to that of the major component.

[75] FORMATION OF MODIFIED TBCU7 AND TH2ZN17 TYPE STRUCTURES DURING ANNEALING OF MECHANICAL-ALLOYED SM-Fe POWDERS

Teresiak A. Kubis M. Mattern N. Wolf M. Muller KH. - Journal of Alloys & Compounds. 274(1-2):284-293, 1998
Compounds with the nominal composition near Sm_2Fe_{17} were prepared by mechanical alloying starting from the elemental powders and subsequent annealing at temperatures, T-A, between 600 degrees C and 900 degrees C. For crystal structure investigations of the non-equilibrium phases formed at various temperatures, XRD methods with following Rietveld analysis were applied. For T-A between 600 degrees C and 750 degrees C a modified $TbCu_7$ -type structure of space group $P6/mmm$ was found, in which the $Fe(2c)$ site is replaced by the partially (1/3) and randomly occupied $Fe(6l)$ site. Its approximate composition is $SmFe_{8.8-9.0}$. For T-A between 800 degrees C to 900 degrees C a disordered modified Th_2Zn_{17} structure (space group $R(3) \overline{6}m$) was found that is formed by introducing additional randomly occupied $Fe(6c)$ and $Sm(3a)$ positions, respectively. The degree of order of Sm atoms and Fe -dumbbells along the c -direction increases with increasing T-A. A decrease in the Fe concentration in the cell is observed for increasing T-A. The completely ordered stoichiometric Th_2Zn_{17} -type structure could not be reached by annealing samples prepared from the ball-milled elemental powders.

[74] SYNTHESIS AND ELECTROCHEMICAL PROPERTIES OF HIGH-ENERGY BALL-MILLED LAVES PHASE (ZR,TI)(V,MN,CR), ALLOYS WITH NICKEL POWDER

Jurczyk M. Rajewski W. Majchrzycki W. Wojcik G. - Journal of Alloys & Compounds. 274(1-2):299-302, 1998
The relations between the structural and electrochemical properties of two nanocrystalline alloys, including: ZrV_2/Ni , which has the cubic $C15$ type structure and $Zr_0.5Ti_0.5V_0.8Mn_0.8Cr_0.4/Ni$, which has the hexagonal $C14$ type structure, have been investigated. We have found that electrochemical activities of ZrV_2 and $Zr_0.5Ti_0.5V_0.8Mn_0.8Cr_0.4$ can be stimulated by high-energy ball-milling (HEBM) of the precursor alloy powders with Ni powder. XRD analysis indicate that there is a high degree of nickel dispersion in HEBM alloy particles. This improves the electrochemical properties of the studied powders. In nanocrystalline $Zr_0.5Ti_0.5V_0.8Mn_0.8Cr_0.4/Ni$ material a discharge capacity of 214 mA h g^{-1} has been measured.

[73] MECHANOCHEMICAL REDUCTION OF $FeTiO_3$ BY Si

Welham NJ. - Journal of Alloys & Compounds. 274(1-2):303-307, 1998
Mechanically induced chemical reactions between $FeTiO_3$ and silicon have been examined using X-ray diffraction and thermal processing. Reduction of $FeTiO_3$ by Si during milling was observed with the formation of elemental iron and TiO_2 or metal silicides, depending on the Si content. An amorphous phase was also present and was probably composed of silicon and oxygen. The reaction occurred in two stages, activation followed by reaction, the length of the activation stage was affected by the milling intensity with a higher intensity giving a shorter activation time. This is attributed to the necessity to attain a critical level of internal energy (in the form of defects, grain boundaries, lattice strain etc.) prior to the onset of reaction.

[72] PREPARATION OF Mg_2Ni BASE ALLOY BY THE COMBINATION OF MECHANICAL ALLOYING AND HEAT TREATMENT AT LOW TEMPERATURE

Aymard L. Ichitsubo M. Uchida K. Sekreta E. Ikazaki F. - Journal of Alloys & Compounds. 274(1-2):309, 1998

[71] KINETICS OF GALLIUM FILMS CONFINED AT GRAIN BOUNDARIES

Konrad H. Weissmuller J. Birringer R. Karmonik C. Gleiter H. - Physical Review B-Condensed Matter. 58(4):2142-2149, 1998

We report on a combined study, by in situ x-ray diffraction at temperatures between 293 and 80 K, differential scanning calorimetry, and coherent quasielastic neutron scattering (QENS), of thin Ga films confined at grain boundaries in Al_2O_3-Ga nanocomposites that are produced by high-energy ball milling. We find that a molar fraction of Ga of up to 0.18 does not undergo phase change in the temperature interval investigated, which contains the bulk melting temperature $T-M(bulk)$, and conclude that this Ga is located in confined layers at grain boundaries, with an estimated thickness of six atomic monolayers. In samples with a higher overall molar fraction, the Ga in excess of molar fraction 0.18 melts and crystallizes reversibly when cycled between 100 and 320 K. For such samples, QENS at 320 K, that is, 17 K above $T-M(bulk)$, indicates three distinct diffusion coefficients D for Ga : one below the resolution limit, $D-1 < 1 \times 10^{-6} \text{ cm}^2/\text{s}$, one agreeing with the bulk liquid value, $D-2 = 3.4 \times 10^{-5} \text{ cm}^2/\text{s}$, and a faster coefficient $D-3 = 2.4 \times 10^{-4} \text{ cm}^2/\text{s}$. We argue that the smaller and medium diffusion coefficients, $D-1$ and $D-2$, represent, respectively, solidlike layers near the Al_2O_3-Ga interface and Liquid Ga in the core of interfacial layers and in liquid droplets. The fast diffusion coefficient ($D-3$) may be due to diffusion on free surfaces.

[70] THERMOELECTRIC POWER OF DOPED BETA- $FeSi_2$ MATERIALS PREPARED BY MECHANOCHEMICAL SYNTHESIS

Mamylov SG. Belyaev EY. Lomovskii OI. - Inorganic Materials. 34(7):683-685, 1998
 β - $FeSi_2$ doped with various amounts of cobalt (n-type) and aluminum (p-type) was prepared by mechanochemical synthesis. Depending on synthesis conditions, the 400-K thermoelectric power of the resultant materials varies from 2 to 800 $\mu V/K$.

[69] HIGH-ENERGY BALL MILLING OF BA-HEXAFERRITE/Fe MAGNETIC COMPOSITE

Bercoff PG. Bertorello HR. - Journal of Magnetism & Magnetic Materials. 187(2):169-176, 1998

A Ba-hexaferrite/Fe composite is synthesized using a high-energy ball mill. Changes in the crystalline structure and magnetic properties are studied with the aid of X-ray diffraction and magnetization measurements. The product after an 80 h mill is magnetite (Fe₃O₄) and barium carbonate (BaCO₃). After heating up to 1300 K in air and Ar atmospheres it is found that in the former case the product is phase M (BaFe₁₂O₁₉) and hematite (alpha-Fe₂O₃) with improved magnetic properties while in the second, BaFe₁₂O₁₉ and Fe₃O₄ are obtained. This is a magnetic composite material having both dipolar and exchange couplings. The coercivity observed is explained in terms of the modified Brown's equation for inverse domain nucleation fields.

[68] GIANT MAGNETORESISTANCE IN MECHANICAL ALLOYED LA_{0.7}SR_{0.06}CA_{0.24}MNO₃ PEROVSKITE

Jin ZQ. Tang W. Zhang JR. Du YW. - Journal of Magnetism & Magnetic Materials. 187(2):237-241, 1998
Nanocrystalline ferromagnetic La_{0.7}Sr_{0.06}Ca_{0.24}MnO₃ perovskites have been successfully synthesized by mechanical alloying. The effect of grain size on magnetoresistance (MR) has been investigated. The results show that after milling for 100 h the as-made samples exhibit a complete amorphous phase, which crystallizes into a nanocrystalline perovskite-like compound when annealed above 920 K. With increasing annealing temperatures the grain size and the metal-semiconductor transition temperature increases, and the MR effect at 77 K decreases. The peak temperature T_p in MR behavior is found to be less affected by the grain size. All the annealed samples exhibit a larger MR at 77 K rather than around the temperature T_p.

[67] SYNTHESIS OF FULL-DENSITY NANOCRYSTALLINE TITANIUM NITRIDE COMPACTS BY PLASMA-ACTIVATED SINTERING OF MECHANICALLY REACTED POWDER

Eleskandarany MS. Omori M. Konno TJ. Sumiyama K. Hirai T. Suzuki K. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 29(7):1973-1981, 1998
Nearly equiatomic nanocrystalline titanium nitride (Ti₅₆N₄₄) powder with an average grain size of 5 nm has been synthesized by ball milling elemental Ti powder under nitrogen gas flow at room temperature. During the first stage of reactive ball milling (RBM) (time < 3.6 ks), the metallic Ti powder tends to agglomerate to form powder particles with a larger diameter. At the second stage (3.6 to 22.0 ks), the agglomerated particles of Ti fragment to form smaller particles. These smaller particles that have new or fresh surfaces begin to react with the milling atmosphere (nitrogen) during the third stage of milling (22 to 86 ks) to form TiN powder coexisting with unreacted Ti powder. Toward the end of milling (86 to 173 ks), a single phase of nanocrystalline TiN (NaCl structure) is obtained. The powder of this end-product has a spherical-like morphology with an average particle size of about 0.4 μm diameter. A sintering procedure using plasma activation has been employed to consolidate the powder particles at several stages of the RBM. The as-milled and as-consolidated powders have been characterized as a function of the RBM time by means of X-ray diffraction (XRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM), optical metallography, and chemical analyses. Density measurements of the consolidated samples show that after 86 to 173 ks of the RBM time, the compacted samples are essentially fully dense (above 96 pct of the theoretical density for TiN). The results also show that the consolidated TiN compacts still maintain their unique nanocrystalline properties with an average grain size of about 65 nm. The hardness and some mechanical properties of the consolidated TiN compacts have been determined as a function of the RBM time.

[66] CHARACTERIZATION OF ALUMINOSILICATE (MULLITE) PRECURSORS PREPARED BY A MECHANOCHEMICAL PROCESS

Temuujin J. Okada K. Mackenzie KJD. - Journal of Materials Research. 13(8):2184-2189, 1998
Aluminosilicate precursors were prepared by mechanochemical treatment of gibbsite-silica gel mixtures. The effect of grinding on their structure and thermal behavior has been examined by Al-27 and Si-29 MAS NMR, x-ray diffraction (XRD), differential thermal analysis-thermogravimetry (DTA-TG), and Fourier transform infrared (FTIR). After 8 h grinding, the hydrated alumina was completely changed to an amorphous phase which showed a new exothermic DTA peak at about 980 degrees C due to the formation of gamma-Al₂O₃ or spinel phase. This behavior was related to changes in the Al and Si environments, as deduced from the MAS NMR spectra. With increased grinding time, some 4-coordinated Al appears, together with an Al resonance at about 30 ppm. Simultaneously, a new ²⁹Si resonance appears at about -90 ppm, indicating a greater degree of homogeneity in the ground samples. Mullite crystallizes at 1200 degrees C from samples ground for 8-20 h, its XRD intensity increasing with increased milling times, in agreement with the NMR, DTA, and FTIR data. Changes in the Al and Si environments during heat treatment, as reflected by the NMR spectra, are also reported.

[65] CYCLE LIFE IMPROVEMENT OF LIMN₂O₄ CATHODE IN RECHARGEABLE LITHIUM BATTERIES

Liu ZL. Yu AS. Lee JY. - Journal of Power Sources. 74(2):228-233, 1998
Spinel LiMn₂O₄ and LiCo_{0.1}Mn_{1.9}O₄ were synthesized by solid state reactions and were used as the positive electrode in liquid cells with Li as the negative electrode. Through extensive ball milling of the spinel with carbon black during the preparation of the cathodic mix, significant decrease in contact resistance between the oxide phase and carbon particles was achieved. The spinel LiCo_{0.1}Mn_{1.9}O₄ cathode so prepared showed good cyclability and rate capability.

[64] LITHIUM INSERTION IN CARBON-SILICON COMPOSITE MATERIALS PRODUCED BY MECHANICAL MILLING

Wang CS. Wu GT. Zhang XB. Qi ZF. Li WZ. - Journal of the Electrochemical Society. 145(8):2751-2758, 1998
Carbons containing nanosize silicon particles were prepared by mechanical milling of graphite and silicon mixtures with different atomic ratios. The microstructure, morphology, and electrochemical performance of ballmilled C₁-chiSi_{chi} (chi = 0, 0.1, 0.2, 0.25) were analyzed by X-ray diffraction, Raman, high-resolution and transmission electron microscopy, and electrochemical methods. After ballmilling, the crystal size of graphite increased but the size of silicon decreased with increasing content of silicon. Ballmilled C₁-chiSi_{chi} materials reacted reversibly with lithium, and the reversible specific capacity increased from 437 mAh/g in the ballmilled pure graphite to 1039 mAh/g in ballmilled C_{0.8}Si_{0.2} materials. The excess capacity due to the Li extraction from silicon appeared at a potential about 0.4 V vs. Li metal. After 20 charge/discharge cycles the reversible capacity of C_{0.8}Si_{0.2} was 794 mAh/g. This

behavior is a result of nanosize silicon particles decreasing the crumbling rate during Li insertion and extraction. These materials appear to be promising candidates for negative electrodes in lithium-ion rechargeable batteries.

[63] STUDIES ON GAMMA SILVER IODIDE

Patnaik JRG. Sunandana CS. - Journal of Physics & Chemistry of Solids. 59(6-7):1059-1069, 1998
gamma-AgI with an Ag+-disordered zincblende structure has been synthesized by several methods including a wet route, based on LiI and a mechano-chemical reaction involving Ag powder and iodine crystals, and thoroughly characterized by XRD ($a = 0.6507$ nm) and density (5.96 g mL⁻¹) measurements, gamma-AgI is found to be thermodynamically stable at room temperature and pressure, up to 413 K and 588 MPa. beta-AgI is formed beyond pressures of 883 MPa. AC/DC electrical conduction in gamma-AgI is promoted by the migration of cationic Frenkel defects (Ag+ interstitials) that are already present below 250 K and by the creation of more such defects above 250 K but below 420 K. The activation energy for Ag+ ion migration is 0.39 eV. Electronic conductivity through I-V characteristics, of gamma-AgI shows the existence of thermally generated electrons and their migration and establishes gamma-AgI as an n-type semiconductor, like beta-AgI. The activation energy for electron transport is 0.42 eV. Weaker excitonic effects (relative to beta-AgI) are observed in gamma-AgI from optical absorption measurements.

[62] FORMATION AND SURFACE STRUCTURE OF TI-ZN-DOUBLE OXIDES AND OF ZN FERRITE

Steinike U. Druska P. Wallis B. Uecker DC. Sepelak V. - Chemical Papers-Chemicke Zvesti. 52(3):147-151, 1998
In powder mixtures of ZnO and TiO₂ the compounds Zn₂TiO₄, ZnTiO₃, and Zn₂Ti₃O₈ can be formed as a result of solid state reactions. The formation of the double oxides is essentially determined by the microstructure of the powder mixture. Furthermore, the type of the double oxide depends on the TiO₂ modification. There exist structural similarities between Zn₂TiO₄ (spinel) and TiO₂ (anatase) as well as between ZnTiO₃ and TiO₂ (rutile). Compound Zn₂Ti₃O₈ is formed only on the basis of the Zn₂TiO₄ phase. The Zn²⁺ ions on the surface of Zn₂Ti₃O₈ occupy only tetrahedral sites and not octahedral ones, like it was derived for the crystal structure of the bulk. The formation of zinc ferrite, ZnFe₂O₄ at room temperature is possible by means of mechanical activation in a high-energy ball-milling process of a ZnO-Fe₂O₃ mixture (mechanosynthesis). The surface structure of mechanosynthesized zinc ferrite corresponds to the inverse spinel structure type. The structure of inverse spinel type is also created by means of mechanical activation of zinc ferrite of the normal spinel type.

[61] EFFECTIVE MAGNETIC ANISOTROPY OF NANOCRYSTALLINE ND-FE-TI-N HARD MAGNETIC ALLOYS

Jin ZQ. Tang W. Zhang JR. Qin HX. Du YW. - European Physical Journal B. 3(1):41-44, 1998
The intermetallic compound Nd-Fe-Ti-N has been successfully synthesized by a mechanical alloying process. The structure and magnetic properties of the sample have been studied using X-ray diffraction and magnetic measurements. It is found that Nd₉Fe₈₃Ti₈ alloy exhibits a nanocrystalline ThMn₁₂-type tetragonal structure with lattice parameters of $a = 0.8723$ nm and $c = 0.4896$ nm. The saturation magnetization M-S and effective magnetic anisotropy K-eff of the compound have been determined by investigating magnetization processes. The calculated results based on the law of approach to magnetic saturation have been successfully used to determine the constant K-eff. The difference between observed and calculated values in magnetization is lower than 3%. Of all terms in the law of approach to saturation, it is the 1/H² term, which is attributed more to non-compensated anisotropy energy, that has the prevailing effect for the compound. The absorption of nitrogen is found to increase unit cell volume, M-S and K-eff.

[60] THE RELATIONSHIP BETWEEN STRUCTURE AND MAGNETIC PROPERTIES IN NANOSTRUCTURED FePd FERROMAGNETS

Okumura H. Soffa WA. Klemmer TJ. Barnard JA. - IEEE Transactions on Magnetics. 34(4 Part 1):1015-1017, 1998
FePd L1(0), ferromagnets with ultra-fine microstructures (submicron/nanoscale) have been studied. The powders and thin films with nanoscale microstructure were produced using high energy ball, milling and vacuum sputtering, respectively. These ultra-fine grained materials were characterized using TEM, SEM, and X-ray diffraction. The line broadening method was used to estimate the crystallite size and lattice strain of the ball-milled powders with the aid of TEM observation. The magnetic properties of the epoxy-bonded powders, isostatically pressed pellets and the thin films were measured using a vibrating sample magnetometer (VSM). The enhanced coercivities exhibited by these ferromagnets compared to bulk materials are discussed in terms of the mechanism of magnetization reversal operating in these fine-particle aggregates and thin films in the grain size range of $D < D_c$ but well above the superparamagnetic regime. Micromagnetic analysis is applied to discuss the mechanism of coercivity, and a "pinning" model is proposed in these so-called "interaction" domain ferromagnets.

[59] MAGNETOTRANSPORT AND MAGNETISM IN GRANULAR EU-S-CO AND EU-S-AG NANOCOMPOSITES PREPARED BY MECHANICAL ALLOYING

Oconnor CE. Feng L. Seip CT. Tang JK. - IEEE Transactions on Magnetics. 34(4 Part 1):1123-1125, 1998
Magnetic and magnetotransport properties have been studied in nanocomposites (EuS)-Co and (EuS)-Ag prepared by mechanical alloying. For (EuS)₍₇₀₎Co₋₃₀, the large negative magnetoresistance (similar to -50%) found at 20 K, just above the Curie temperature (T_c) of EuS, arises from the suppression of spin fluctuation in EuS. Below T_c, the positive magnetoresistance (similar to 15%), which is uncharacteristic of ferromagnetic semiconductors, is probably due to defect-induced magnetic fluctuation. On the other hand, (EuS)₍₇₀₎Ag₋₃₀, where nonmagnetic silver replaces cobalt, exhibits negative magnetoresistance below T_c, which is possibly related to the spin dependent scattering commonly seen in GMR materials. While (EuS)₍₇₀₎Co₋₃₀ shows semiconducting conductivity, (EuS)₍₇₀₎Ag₋₃₀ is metallic. Reentrant spin glass behavior has been observed in EuS-Ag system, where the rise of antiferromagnetic coupling is due to the defects and disorder caused by mechanical milling.

[58] MAGNETIC PROPERTIES OF BALL MILLED FE-40AL AT.PERCENT ALLOYS

Amils X. Nogues J. Surinach S. Baro MD. Munoz JS. - IEEE Transactions on Magnetics. 34(4 Part 1):1129-1131, 1998
A direct correlation between the lattice parameter and the saturation magnetization, during the disordering (ball

milling) and posterior reordering (annealing) processes, has been found in Fe-40Al at.% compounds. These results indicate that the paramagnetic-ferromagnetic-paramagnetic transitions induced by ball milling and subsequent annealing could be related to the changes in volume, and not only to nearest neighbors effects as is commonly assumed. Moreover, these alloys have been found to become spin glass at low temperatures, independently of their structural state (ordered or disordered).

[57] REACTION SYNTHESIS OF TITANIUM SILICIDES VIA SELF-PROPAGATING REACTION KINETICS

Yen BK. Aizawa T. Kihara J. - Journal of the American Ceramic Society. 81(7):1953-1956, 1998

Self-propagating exothermic reactions in the titanium-silicon system induced by mechanical milling, shock loading, and thermal ignition of elemental powder mixtures have been investigated. After an induction period of 3 h, the 5Ti + 3Si powder mixture abruptly reacted during milling to form single-phase Ti₅Si₃ via a mechanically induced self-propagating reaction (MSR). Moreover, the formation of porous Ti₅Si₃ solid indicated that the melting of powder particles had occurred during the mechanical alloying process. The Ti + Si powder mixture also reacted via the MSR mode, but the end-product was multiphase. Shock and combustion synthesis (thermal-explosion mode) experiments essentially produced the same result.

[56] ZIRCON SYNTHESIS VIA SINTERING OF MILLED SiO₂ AND ZrO₂

Spearing DR. Huang JY. - Journal of the American Ceramic Society. 81(7):1964-1966, 1998

The formation of zircon (ZrSiO₄) via sintering of milled SiO₂ and ZrO₂ powders was studied, and the effects of slurry vs dry milling, sintering time, and particle size on zircon yield were examined. It was found that very high zircon yields could be obtained via slurry milling, cold pressing, and sintering of the oxide precursors. The controlling factor in determining zircon yield was found to be the particle size of the SiO₂ and ZrO₂ powders. Zircon yield as a function of sintering time was examined, and found to be similar to previous studies in which sol-gel precursors seeded with zircon were used. SEM studies reveal a homogeneous product with particle sizes on the order of 1-5 μm. It was found that complete reaction to zircon can be achieved from a once-through milling, pressing, and sintering process of SiO₂-ZrO₂ powders.

[55] MECHANOCHEMICAL SYNTHESIS OF FE-S MATERIALS

Jiang JZ. Larsen RK. Lin R. Morup S. Chorkendorff I. Nielsen K. Hansen K. West K. - Journal of Solid State Chemistry. 138(1):114-125, 1998

Powder mixtures of metallic iron and sulfur have been milled in a high-energy planetary ball mill and the formation of iron sulfides has been studied by x-ray diffraction, Mossbauer spectroscopy, and scanning electron microscopy. For Fe:S ratios of 1:1 and 1:2 the final products are FeS with the modified NiAs structure and FeS₂ (pyrite), respectively. No other iron sulfides were formed for any of the Fe:S ratios studied. The FeS phase has been tested as an electrode material in lithium batteries.

[54] ROLE OF WATER IN THE MECHANOCHEMICAL REACTIONS OF MGO-SiO₂ SYSTEMS

Temuujin J. Okada K. Mackenzie KJD. - Journal of Solid State Chemistry. 138(1):169-177, 1998

The products of mechanochemical activation of mixtures in the magnesia-silica system ground in a planetary pot mill for up to 20 h. were found to depend on the water content of the siliceous starting materials. Mixtures containing silica gel produced no new products detectable by XRD and Si-29 MAS NMR, but their thermal reaction to form forsterite at 890 degrees C was slightly enhanced by grinding. In contrast, mixtures containing more highly hydrated silicic acid produce a layer-lattice magnesium silicate with an 8.4-Angstrom basal spacing and a Si-29 MAS NMR spectrum similar to that of the product obtained when the same mixture is subjected to hydrothermal reaction conditions. A possible explanation in terms of a mechanochemically induced hydrothermal reaction mechanism is discussed.

[53] SPREADING OF MOO₃ ON GAMMA-AL₂O₃ INDUCED BY MECHANICAL ACTIVATION

Mestl G. Knozinger H. - Langmuir. 14(14):3964-3966, 1998

[52] MECHANOMAKING OF HIGH SPEED STEEL AISI M2 - POWDER CONSOLIDATION

Matteazzi P. Wolf F. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 248(1-2):19-34, 1998

AISI M2 powders were produced from the elements by mechanomaking, a room temperature process driven by high energy ball milling. The as-mechanomade powders with relative densities 0.5, were consolidated by cold and then hot isostatic pressing. Fully dense solids with carbide sizes around 20 nm are obtained after Hot Isostatic Pressing (HIP) at 950 degrees C, while the matrix is constituted of a very fine array of crystallites below 10 nm. An as-quenched hardness of 69 HRC was obtained and a Hall-Petch behavior was observed for Vickers hardness. No evidence of residual austenite was found in either as-HIPed or quenched samples.

[51] MECHANICAL ALLOYING OF BISB SEMICONDUCTING ALLOYS

Martinlopez R. Lenoir B. Devaux X. Dauscher A. Scherrer H. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 248(1-2):147-152, 1998

Polycrystalline Bi_{100-x}Sb_x (x = 7, 10, 12, 15, 22) semiconducting alloys were synthesized by mechanical alloying in order to achieve performant homogeneous thermoelectric materials. The influence of the milling parameters on the final homogeneity and morphology of the powders, such as ball-to-powder weight ratios (BPR = 10:1, 40:1, 92:1) and balls sizes (diameters of 30 and 20 mm), was investigated. Powder samples were characterized by X-ray diffraction, differential scanning calorimetry, scanning electron microscopy and electron microprobe analysis. The results show that the samples obtained with the 10:1 BPR and four balls of 30 mm in diameter present the best homogeneity whatever the alloy composition.

[50] APPLICATION OF MOSSBAUER SPECTROSCOPY IN THE CHARACTERIZATION OF NANOSTRUCTURED MATERIALS

Rawers J. Cook D. Kim T. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 248(1-2):212-220, 1998

Understanding of nanostructured materials is often limited by experimental characterization methods that measure only bulk properties. For example, numerous studies have characterized nanostructured materials using X-ray diffraction for phases present, average grain size, internal strain, etc. In this study, Mossbauer analysis is used to characterize the local atomic site characterization, distribution, and concentrations of attrition milled nanostructure powder. Interatomic analysis provided insight into the mechanical alloying process and the resulting nanostructure not previously reported. Iron powder, blends of iron with 2 wt% aluminum powder, and prealloyed iron-aluminum powder were processed with both argon and nitrogen gas as the processing environments. Mechanical processing resulted in micrometer-size particles with essentially defect-free nanograin interiors. Mechanical alloying iron powder with aluminum resulted in the aluminum being restricted to the grain boundary region. Mechanical processing iron powder in a nitrogen gas environment resulted in nitrogen being either on the grain boundary or in the outer layer of the grain boundary distorting the local b.c.c.-Fe lattice into a b.c.t.-Fe lattice.

[49] FORMATION OF MICRONISED WC FROM SCHEELITE (CAWO4)

Welham NJ. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 248(1-2):230-237, 1998

A stoichiometric mixture of graphite and scheelite have been mechanically milled together for 40 h. The resultant powder, and an unmilled powder, have been examined by thermal analysis, isothermal annealing and X-ray diffraction to determine the effect of milling on the reduction of scheelite to tungsten carbide. The milled powder underwent a rapid reduction reaction at 800 degrees C, 300 degrees C lower than the unmilled powder. The reduction sequence was shown to be multistage with Ca_3WO_6 , W_2C , and W all formed as intermediate phases prior to WC . The milled powder showed complete reduction to WC in 1 h at 1000 degrees C, whereas the unmilled powder was incompletely reduced, even after 1 h at 1200 degrees C. Unoptimised acid leaching of the reduced powder showed that separation of the final phases was readily achievable, leaving highly porous WC particles which were composed of grains around 200 nm in size.

[48] MILLING CHARACTERISTICS OF EXTRUDED EUTECTOID ZN-AL ALLOY

Hirata VML. Munoz MS. Hernandez JCR. Zhu YH. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 247(1-2):8-14, 1998

Mechanical milling-induced phase transformations, microstructural changes and preferred crystal orientation of the extruded eutectoid Zn-Al-based alloy are reported. Decomposition of a metastable ϵ phase and a four-phase transformation: $\alpha + \epsilon \rightarrow \text{T}' + \epsilon$, were greatly accelerated by mechanical milling. The mechanism of the milling-induced microstructural change from a lamellar structure to a fine grain structure was found to be different from that which occurred under tensile and creep deformations in the extruded eutectoid Zn-Al-based alloy. A preferred crystal orientation was observed as well, in the alloy during milling, which was supported by a rolling-induced preferred orientation in the same alloy.

[47] STRUCTURAL CONFIGURATION AND MAGNETIC EFFECTS IN AS-MILLED AND ANNEALED NANOCRYSTALLINE IRON

Delbianco L. Hernando A. Navarro E. Bonetti E. Pasquini L. - Journal de Physique IV. 8(P2):107-110, 1998
Mechanical spectroscopy, room temperature Mossbauer spectroscopy and magnetisation measurements have been employed to get insight into the structural relaxation process of the grain boundaries in 10 nm-grain sized iron powders produced by ball milling. In particular, by performing the Mossbauer analysis on milled powders previously subjected to proper thermal treatments in the 570-670 K range, the structural evolution of the grain boundaries has been followed through the changes in the hyperfine magnetic field distribution associated to the interfacial region. The results are consistent with a decrease of the magnetic moment of the atoms at the interface, following the relaxation process.

[46] FORMATION OF MULLITE FROM MECHANOCHEMICALLY ACTIVATED OXIDES AND HYDROXIDES

Temuujin J. Okada K. Mackenzie KJD. - Journal of the European Ceramic Society. 18(7):831-835, 1998.
The formation of mullite from different precursors prepared by a mechanochemical method were investigated. The structural changes caused by grinding various oxide/hydroxide mixtures were characterized by the Si-29 and Al-27 MAS-NMR, FTIR, DTA and XRD. Thermal treatment of precursors prepared by mechanochemical activation from gibbsite and silica gel led to the crystallization of mullite at approximate to 1200 degrees C via a spinel-phase, while precursors prepared from $\alpha\text{-Al}_2\text{O}_3$ and silica gel or gibbsite and fused silica formed mullite at about 1400 degrees C by solid state reaction between $\alpha\text{-Al}_2\text{O}_3$ and cristobalite. MAS-NMR spectroscopy shows that the crystallization of mullite at lower temperatures is associated with good homogeneity, as judged by the amount of Si present in the aluminosilicate resonances.

[45] SURFACE ANALYSIS OF AN AMORPHOUS MGNI ALLOY PREPARED BY MECHANICAL ALLOYING FOR USE IN NICKEL METAL HYDRIDE BATTERIES

Zhang SG. Yorimitsu K. Nohara S. Morikawa T. Inoue H. Iwakura C. - Journal of Alloys & Compounds. 270(1-2):123-126, 1998

An amorphous MgNi alloy prepared by mechanical alloying has been investigated by means of X-ray photoelectron spectroscopy (XPS) and X-ray induced Auger electron spectroscopy (XAES) in combination with an Ar^+ sputtering technology. At the top surface (ca. 30 Angstrom), Mg was dominant and existed in oxide forms while Ni mainly existed in the metallic state. Underneath the top surface, there was a layer enriched with metallic Ni which penetrates to more than 150 Angstrom. Such a distribution of Mg and Ni at the surface layer tightly correlates to the unique hydriding and dehydriding properties of the amorphous MgNi alloy.

[44] HYDRIDING AND ELECTROCHEMICAL CHARACTERISTICS OF A HOMOGENEOUS AMORPHOUS $\text{Mg}_2\text{Ni-Ni}$ COMPOSITE

Iwakura C. Inoue H. Zhang SG. Nohara S. - Journal of Alloys & Compounds. 270(1-2):142-144, 1998
Hydriding and electrochemical characteristics of a homogeneous amorphous $\text{Mg}_2\text{Ni-Ni}$ composite prepared by ball-milling of Mg_2Ni alloy with Ni (70 wt.% vs. Mg_2Ni) were investigated. It was found that the $\text{Mg}_2\text{Ni-Ni}$ composite

absorbed hydrogen to the amount of 4.0 wt.% vs. Mg₂Ni [2.4 wt.% vs. (Mg₂Ni+70 wt.% Ni)] at a high rate under a hydrogen pressure of 3 MPa at 30 degrees C. Furthermore, this alloy exhibited an extremely high discharge capacity of similar to 1082 mAh g(Mg₂Ni)(-1) [636 mAh g(Mg₂Ni +70 wt.% Ni)(-1)] at 30 degrees C, which exceeded the theoretically calculated value of 999 mAh g(Mg₂Ni)(-1) on the basis of Mg₂NiH₄ for crystalline Mg₂Ni.

[43] STRUCTURAL AND HYDRIDING PROPERTIES OF AMORPHOUS MGNI WITH INTERSTITIALLY DISSOLVED CARBON

Funaki K. Orimo S. Fujii H. Sumida H. - Journal of Alloys & Compounds. 270(1-2):160-163, 1998
MgNiC_x with an amorphous structure were synthesized up to x=1.31 by mechanical alloying of amorphous (a-)MgNi and graphite as starting materials. The core-level X-ray photoelectron spectroscopy revealed that the original bonding states with metallic character of a-MgNi remained even in the sample with high carbon content although the graphite dissolved as interstitial carbon and it mainly bounded with Mg in a-MgNi. The crystallization temperature of MgNiC_x increased with increasing carbon content up to x=0.85, which was accompanied by a drastic change in the crystallization processes. Upon hydrogenation of MgNiC_x, the atomic ratio of hydrogen plus carbon to metal (H+C)/M remained a constant value of ≈0.9 through the whole system, indicating that carbon atoms dissolved into the sites that otherwise would have been occupied by hydrogen atoms.

[42] A STUDY OF IRON NITROGENATION BY HOT ISOSTATIC PRESSING

Chartier P. Eymery JP. Dedieu E. Zouggar M. Beaufort MF. Grosbras M. - Journal of Alloys & Compounds. 270(1-2):168-174, 1998

In this study a hot isostatic pressing (HIP) furnace under high temperature and high nitrogen pressure is used to perform nitrogen diffusion in mechanically processed iron powders. After hipping, the gamma'-Fe₄N phase is formed while unreacted b.c.c. iron also remains in the powder. The amount of gamma'-Fe₄N is estimated by calculating the relative X-ray diffraction peak intensity. The latter amount increases with increasing milling time because of new surface formation and defect creation. The nitrogen diffusion into grains occurs for a minimum value of the HIP pressure, then the alloying of nitrogen with iron appears for a minimum temperature. Mossbauer and EXAFS analyses indicate that the iron nitride is mainly located at the surface of the powder grains.

[41] MECHANOCHEMICAL SYNTHESIS OF NONSTOICHIOMETRIC TANTALUM DISILICIDE

Golubkova GV. Belyaev EY. Lomovsky OI. - Journal of Alloys & Compounds. 270(1-2):224-227, 1998
Tantalum disilicide synthesis was developed by means of mechanochemical activation under intensive mechanical treatment. The concentration interval from Ta₇₅Si₂₅ to Ta₁₈Si₈₂ (at. %) was under investigation. Activator-mills of planetary type were used for mechanical treatment with balls accelerated under 600 ms⁻². The increase of elementary cell parameter takes place during the initial period of mechanical activation up to 12 min. The intensity of Ta lines diminishes, and reflexes of the TaSi₂ phase appears during treatment. The metastable structures on the base of TaSi₂ were produced containing up to 15 at. % of silicon and 5 at. % of tantalum excess. The homogeneity region of the TaSi₂ phase can be extended from Ta_{38.3}Si_{61.7} to Ta_{18.3}Si_{81.7}. Tantalum disilicide materials with tantalum excess have better oxidation resistance.

[40] MECHANOCHEMICAL REACTION BETWEEN ILMENITE (FeTiO₃) AND ALUMINIUM

Welham NJ. - Journal of Alloys & Compounds. 270(1-2):228-236, 1998
A natural ilmenite (FeTiO₃) and aluminium powder have been mechanically milled together for 100 h in a laboratory ball mill. The as-milled powder and an unmilled powder of identical composition were annealed at up to 1200 degrees C and examined by X-ray diffraction and differential thermal analysis (DTA). The unmilled sample showed aluminium melted prior to an exothermic reaction starting at -850 degrees C. The milled powder showed no thermal activity, other than a reversible phase transition at 1067±4 degrees C, indicating that reaction occurred within the mill. The products of both powders were the same, TiAl₃, Fe₄Al₃, and Al₂O₃, although in the milled powder these phases were nanocrystalline until annealing caused crystallite growth. The thermal reaction seemed to occur in two stages, formation of TiAl₃, Al₂O₃ and elemental iron followed by a slower, diffusion controlled reaction between the elemental iron and residual aluminium to form Fe₄Al₁₃. The reaction during milling was attributed to increased intermixing between the ilmenite and aluminium causing a change in the rate determining step from solid-state diffusion to another, unknown, controlling mechanism.

[39] MATHEMATICAL MODELLING OF THE MECHANICAL ALLOYING KINETICS

Pabi SK. Das D. Mahapatra TK. Manna I. - Acta Materialia. 46(10):3501-3510, 1998
A rigorous mathematical model, based on the modified iso-concentration contour migration method, has been developed to predict the kinetics of diffusive intermixing in a binary miscible system in the course of mechanical alloying (MA). The present model considers the variation of diffusion coefficient with composition, and interface shift due to both interdiffusion and mechanical deformation. Comparison of the kinetics predicted by the present model with the relevant experimental data from Cu-Ni and Cu-Zn shows that the effective mass transport operative in MPI attains a rare intermediate between that for volume or grain boundary diffusion. An effective temperature for diffusion (T-eff) has been proposed to simulate the observed alloying kinetics. T-eff is a function of composition but is not related to the adiabatic temperature rise at the point of ball-powder collision. Finally, the ratio of T-eff to the corresponding liquidus temperature for all the composition studied lies between 0.4 and 0.5.

[38] MECHANOCHEMICAL PREPARATION OF ALUMINOSILICATE PRECURSORS FROM GIBBSITE SILICIC ACID MIXTURES

Temuujin J. Jadambaa TS. Okada K. Mackenzie KJD. - Materials Letters. 36(1-4):48-51, 1998
The influence of mechanochemical treatment on the homogeneity of gibbsite-silicic acid mixtures was investigated by Si-29 and Al-27 MAS NMR, DTA-TG and XRD. Increased homogeneity of mullite precursors prepared by grinding mixtures of the appropriate stoichiometry for 20 h is indicated by the appearance of metakaolinite-like 4-coordinated Al resonances in the Al-27 NMR spectra and new resonances at -93 and -101 ppm in the 29Si spectra. The influence of hydration water on the grinding reactions is also discussed.

[37] SYNTHESIS OF 7075 AL/SIC PARTICULATE COMPOSITE POWDERS BY MECHANICAL ALLOYING

Pankar R. Singh P. - Materials Letters. 36(1-4):201-205, 1998

Mechanical alloying as a viable processing route for the manufacture of 7075(Al-5.6Zn-2.5Mg-1.6Cu-0.23Cr)-SiC particulate (SiCp) composite is confirmed. XRD and STEM studies indicated that the alloying takes place through the supersaturated solid solutions to the formation of the precipitate.

[36] PREPARATION AND THERMAL STABILITY OF CERAMIC/METALLIC GLASS COMPOSITES PREPARED BY MECHANICAL ALLOYING

Lu IR. Moelle C. Sagel A. Wunderlich RK. Perepezko JH. Fecht HJ. - Materials Letters. 35(5-6):297-302, 1998
We present the formation of ceramic/metallic glass composite powders by mechanical alloying of an elemental powder mixture with the composition Zr₆₅Al_{7.5}Ni₁₀Cu_{17.5} together with SiC particles. The effect of the added ceramic particles on glass formation and thermal stability was investigated with structural and thermal analysis. During the milling process an amorphous matrix with a homogeneous dispersion of SiC particles develops. This composite material reveals a distinct glass transition followed by crystallization. The onset temperatures for these transformations are found to be increased by about 10 K in comparison to the amorphous Zr-based alloy without SiC. This observation may be explained by small compositional changes in the amorphous matrix during the milling process. No indication for SiC dispersoids acting as heterogeneous nucleation sites was found. The kinetics of primary crystallization was found to be delayed. The microhardness of the ceramic/metallic glass composites was increased by about 25% compared to the value found for the single amorphous powder. As such, mechanical alloying represents a useful method for the preparation of ceramic/metallic glass composite powders combining the mechanical properties of the amorphous state and the ceramic particle dispersions.

[35] MAGNETIC PROPERTIES OF SM-(FE,CO)-N MAGNETS CONTAINING SMALL ADDITIONS OF GA

Januszewski D. Leonowicz M. - Materials Letters. 35(5-6):364-369, 1998

Addition of small amounts of Ga (up to 1 at.%) to mechanically alloyed Sm-(Fe,Co)-N magnets was investigated. The coercivity initially increased (up to 0.25 at.%) and then decreased for higher Ga contents. The remanence adversely followed the coercivity being lowest for 0.25 at.% Ga. The addition of Ga up to 1 at.% did not change the phase constitution of the alloys. A small decrease of the Curie temperature and increase of the decomposition temperature of the Sm-2(Fe,Co,Ga)(17)N-x phase were observed. The change of the magnetic parameters is considered to be caused by the change of lattice constants and interstices size which affect the amount of nitrogen dissolved in the Sm-2(Fe,Co,Ga)(17) lattice.

[34] FORMATION OF CUBIC C-BN BY CRYSTALLIZATION OF NANO-AMORPHOUS SOLID AT ATMOSPHERE

Yao B. Liu L. Su WH. - Journal of Materials Research. 13(7):1753-1756, 1998

An amorphous carbon-boron nitride (C-BN) solid was prepared by ball milling the mixture of graphite and hexagonal BN powders for a period of 120 h. After annealing the amorphous C-BN solid for 1 h at atmosphere in the temperature range from 800 to 900 K and then quenching it to room temperature, a small amount of cubic C-BN solid solutions with diamond-like structure, which belong to a high energy phase and can only be synthesized previously under high pressure and temperature (30 GPa, 2000 K), were observed in the annealed amorphous C-BN solid. The lattice constant of the cubic C-BN solid solution was 0.3587 nm, and its grain size was in the range of 10 to 50 nm.

[33] MAGNETIC PROPERTIES AND STRUCTURE OF NANOCRYSTALLINE FE70AL30 ALLOY PREPARED BY MECHANOSYNTHESIS

Jartych E. Zurawicz JK. Oleszak D. Pekala M. Sarzynski J. Budzynski M. - Journal of Magnetism & Magnetic Materials. 186(3):299-305, 1998

Nanocrystalline powder samples of Fe₇₀Al₃₀ alloy were prepared by the mechanical alloying method. X-ray diffraction, Mossbauer and magnetic studies indicated that the ferromagnetic solid solution with BCC structure was formed with an increase in milling time. The magnetic ordering temperature T_c of the nanocrystalline mechanically alloyed Fe₇₀Al₃₀ was larger than that of the corresponding alloy prepared by rapid quenching, while the average magnetic moment per Fe atom in the investigated milling alloy was smaller than in cold-worked Fe₇₀Al₃₀ alloy. The high value of the hyperfine magnetic field of about 29 T measured in the Fe₇₀Al₃₀ alloy was explained in the frame of a local environment model, where five or more Fe atoms in the nearest neighbourhood of Fe-57 constitute the most probable configuration.

[32] THE EFFECT OF NOX AND CO ON THE RATE OF TRANSITION METAL OXIDE CATALYZED CARBON BLACK OXIDATION - AN EXPLORATORY STUDY

Mul G. Zhu WD. Kapteijn F. Moulijn JA. - Applied Catalysis B-Environmental. 17(3):205-220, 1998

The catalytic activity of Cr₂O₃, Co₃O₄, and CuD for the oxidation of carbon black in various gas compositions is reported. In 10% O₂ in Ar, the carbon black oxidation activity is improved considerably by intensifying the contact between the transition metal oxide (TMO) and carbon black by ball-milling (creation of 'tight contact') or impregnation, and also by increasing the surface area of the non-supported metal oxides. The effect of CO addition is strongly dependent on the catalyst: CO promotes the CuO catalyzed carbon black oxidation, it hardly affects the Cr₂O₃ activity, and inhibits the Co₃O₄ activity. Addition of NO_x always enhances the catalytic carbon black oxidation rate. This is attributed to bifunctional catalysis: NO₂ reacts with carbon black, yielding NO, CO acid CO₂ and NO is reoxidized to NO₂ over the TMO. NO improves the efficiency of the TMO by transportation of activated oxygen. In tight contact conditions the NO₂/carbon black reaction might also be catalyzed. Catalytic filter configurations for the practical use of the NO₂/carbon reaction over the investigated catalysts in diesel soot abatement are discussed.

[31] SUPPORTED OXIDES - PREPARATION, CHARACTERIZATION AND CATALYTIC ACTIVITY OF CROX/ZRO2, MOOX/ZRO2 AND VOX/ZRO2

Indovina V. - Catalysis Today. 41(1-3):95-109, 1998

In the paper, we review the preparation and the characterization of CrO_x/ZrO₂, MoO_x/ZrO₂ and VO_x/ZrO₂. Catalysts were prepared by various methods (equilibrium adsorption, impregnation or mechanical mixing) and characterized by XRD, XPS, ESR and FTIR techniques. In the paper, we also review the catalytic activity of the ZrO₂

supported systems for propene hydrogenation (CrOx/ZrO₂ and MoOx/ZrO₂), propane or isobutane dehydrogenation (CrOx/ZrO₂), abatement of NO with H₂, propane, or propene (CrOx/ZrO₂), abatement of NO with NH₃ (VOx/ZrO₂). In selected cases, in order to assess the influence of the support on the catalytic activity, we compare the activity of the MeOx/ZrO₂ samples with that of the same MeOx, on other supports.

[30] MECHANICAL AMORPHIZATION OF MULLITE AND THERMAL RECRYSTALLIZATION

Schmucker M. Schneider H. Mackenzie KJD. - Journal of Non-Crystalline Solids. 226(1-2):99-104, 1998
Commercial fused-mullite was ball-milled up to 240 h and its structural changes were monitored by XRD, TEM and solid-state Al-27 MAS NMR. Milling for 240 h reduces the mullite XRD reflections to about 10% of their original intensity. Broadening of the XRD lines occurs, suggesting drastic crystal size reduction, but TEM indicates that the loss of XRD intensity is due to amorphization rather than to the effect of particle size or lattice strain. MAS NMR spectra show that the amorphous material retains the tetrahedral and octahedral Al environments of the original mullite. However, the Al NMR signal near 40 ppm in mullite which is associated with the tetrahedral tricluster Al* sites is gradually replaced by a resonance at about 30 ppm. This NMR signal also appears in alumina-rich gels and glasses acid has been explained either by the presence of fivefold coordinated aluminum or by distorted triclustered AlO₄ tetrahedra. Thermal treatment at > 700 degrees C causes gradual recrystallization of the non-crystalline material to mullite with the simultaneous replacement of the 30 ppm Al-27 NMR resonance by the Al* resonance at about 40 ppm. Complete recrystallization of the ground material leads to extremely small mullite grains indicating higher nucleation densities than in crystallized mullite gels and glasses. The low activation energy of recrystallization in mechanically amorphized mullite and the low crystallization temperature suggest that ball-mill-produced mullite 'glass' has a structural short-range-order similar to that of crystalline mullite.

[29] POSSIBILITY OF MECHANICALLY STIMULATED TRANSMUTATION OF CARBON NUCLEI IN ULTRADISPERSE DEUTERIUM-CONTAINING MEDIA

Lipson AG. Kuznetsov VA. Ivanova TS. Saunin EI. Ushakov SI. - Technical Physics. 42(6):676-682, 1997
The variation of the concentration of C-14 in graphite subjected to vibratory milling in the presence of heavy (or light) water is investigated using methods of radiocarbon beta-spectral analysis. It is discovered that the C+D₂O system exhibits a statistically significant increase in the beta counting rate in comparison to the C+H₂O system milled under similar conditions. A quantitative model of the transmutation of the C-13 isotope into the C-14 isotope involving an interaction of multiphonon excitations with deuterons in the graphite crystal lattice in the presence of background thermal neutrons is proposed.

[28] TRANSFORMATION OF CADMIUM FERRICYANIDE BY HEATING, MILLING AND SONICATION

Reguera E. Balmaseda J. Quintana G. Gomez A. Fernandezbertran J. - Polyhedron. 17(13-14):2353-2361, 1998.
On heating, milling and sonication, cadmium ferricyanide decomposes and reduces, to cadmium ferrocyanide. Two compositions were studied, pure cadmium ferricyanide and mixed potassium-cadmium ferricyanide. The role of reducing agent is played by the CN- anions liberated during the decomposition process. The solid decomposition product is composed by several phases of cadmium ferrocyanide, an Fe³⁺ phase and also potassium ferrocyanide in the case of the mixed complex salt; The decomposition process was monitored using DTA, XRD, IR and Mossbauer techniques. No occurrence of linkage isomerization in cadmium ferricyanide was detected.

[27] SYNTHESIS AND ELECTROCHEMICAL PERFORMANCES OF TRIBASIC AND TETRABASIC LEAD SULFATES PREPARED BY REACTIVE GRINDING

Grugeondewaele S. Laruelle S. Joliveauvallat F. Torcheux L. Delahayevidal A. - Journal of Power Sources. 72(2):126-131, 1998

Tribasic lead sulfate (3BS) and tetrabasic lead sulfate (4BS), used as precursors of the positive active material in the lead/acid batteries, were prepared by a new method: reactive grinding. The effects of various experimental parameters (stoichiometry, hygrometry of the starting compounds, duration of mechanical treatment) upon the nature and morphological features of the resulting phase were investigated. Among them, hygrometry turned out to be the most critical one. With water in excess, only 3BS was produced while dry reagents led to 4BS. In both cases, samples with a small particle size and high reactivity were obtained. In order to evaluate the influence of the morphology upon the electrochemical performances of such grinding produced samples, the capacity was measured and compared with that of traditional 3BS and 4BS samples.

[26] EFFECT OF BALL-MILLING ON ELECTROCHEMICAL AND PHYSICO-CHEMICAL CHARACTERISTICS OF CRYSTALLINE MG₂Ni ALLOY

Inoue H. Ueda T. Nohara S. Fujita N. Iwakura C. - Electrochimica Acta. 43(14-15):2215-2219, 1998.
Two-step surface modifications for crystalline Mg₂Ni alloy were tried to improve the charge-discharge characteristics at 30 degrees C. The surface modification of the alloy by ball-milling led to the improvement of the discharge capacity, probably due to the increase in surface area and the decrease in diffusion length for the desorption of absorbed hydrogen as the result of the production of nanocrystallites. The subsequent surface modification with graphite led to a further increase in discharge capacity due to the improvement of electrocatalytic activity.

[25] PREPARATION AND ELECTROCHEMICAL CHARACTERIZATION OF MG₂Ni ALLOYS WITH DIFFERENT CRYSTALLINITIES

Inoue H. Hazui S. Nohara S. Iwakura C. - Electrochimica Acta. 43(14-15):2221-2224, 1998.
Mg₂Ni alloys with different crystallinities were prepared by mechanical alloying (MA), followed by a subsequent heat treatment and their charge-discharge characteristics in (6 M KOH + 1 M LiOH) aqueous solution were measured at 30 and 70 degrees C. At both temperatures, charge-discharge characteristics of the Mg₂Ni prepared by MA came close to those of the Mg₂Ni alloy prepared by induction melting (IM), with an increase in heat-treating period at 400 degrees C. The presented approach was believed to be very effective for the preparation of hydrogen storage alloys whose constituent metals have large differences in melting points.

[24] HOT ISOSTATICALLY PRESSED SIC-ALN POWDER MIXTURES - EFFECT OF MILLING ON SOLID-SOLUTION FORMATION AND RELATED PROPERTIES

Li JF. Kawasaki A. Watanabe R. - Journal of the American Ceramic Society. 81(6):1445-1452, 1998
Powder mixtures with nominal compositions of 75 mol% SiC and 25 mol% AlN as well as 50 mol% SiC and 50 mol% AlN were prepared by ball milling for 1-300 h and subsequently sintered to full density by hot isostatic pressing (HIPing) at 1850 degrees C under a pressure of 200 MPa. Microstructures and properties of the HIP-sintered SiC-AlN powder mixtures were investigated with special attention to the effect of ball milling on the formation of SiC-AlN solid solutions. Extensive ball milling facilitated effectively the formation of solid solutions by improvement in mixing homogeneity and pulverization of the SiC and AlN powders. Thermal conductivity of the HIP-sintered SiC-AlN powder mixtures decreased remarkably with ball-milling time, mainly because of the enhanced formation of solid solutions. Mechanical properties-such as strength, fracture toughness, and microhardness-were dependent on the microstructural homogeneity and the grain refinement resulting from the formation of solid solutions.

[23] AN X-RAY DIFFRACTION AND MOSSBAUER SPECTROSCOPY STUDY OF THE REACTION BETWEEN HEMATITE AND ALUMINUM ACTIVATED BY BALL MILLING

Concas G. Corrias A. Manca E. Marongiu G. Paschina G. Spano G. - Zeitschrift fur Naturforschung Section A-A Journal of Physical Sciences. 53(5):239-244, 1998

The reaction between hematite and aluminum in presence of alumina as diluent activated by Ball Milling powder mixtures in different energetic conditions has been investigated. To this purpose, the powders at different milling times have been characterized by X-ray Diffraction and Mossbauer Spectroscopy. A self-sustained combustion reaction was observed when the strongest energetic conditions of milling were adopted. The intermediate products of the reaction also depend on the energetic conditions: the formation of hercynite is favoured by the use of strong energetic conditions while the formation of an Fe-Al alloy was observed when a low energy per single hit is transferred to the powders.

[22] OPTICAL BEHAVIOR OF ORGANIC PIGMENTS IN AQUEOUS DISPERSIONS AND ITS APPLICATION

Fu JM. Li Y. Guo JL. - Journal of Colloid & Interface Science. 202(2):450-455, 1998

The intensity of a beam of light on passing through a dilute pigment dispersion is attenuated as a result of absorption and scattering of light by the pigment particles. It was found that the particle size distribution of an organic pigment not only controls the light scattering but also affects the light absorption. As the milling time increases, the absorption coefficient increases while the scattering coefficient decreases. The peak-to-valley ratio obtained from the extinction spectra of the pigment dispersions can be used to determine the dispersion degree of organic pigments, and this method was confirmed by particle size measurements.

[21] MECHANICAL ACTIVATION OF THE FORMATION OF AN ALUMINA-TITANIUM TRIALUMINIDE COMPOSITE

Welham NJ. - Intermetallics. 6(5):363-368, 1998

The fabrication of a powder composed of alumina and titanium trialuminide TiAl₃ from TiO₂ and aluminium powder is reported in this paper. The powders were ball-milled together under vacuum for 100h in a laboratory scale mill with no reaction evident directly after milling. Differential thermal analysis showed that the milled powder underwent an exothermic reaction below the melting point of aluminium. An unmilled powder underwent a slow reaction until an exothermic reaction occurred at 1000 degrees C. The milled powder showed complete reaction to Al₂O₃ and TiAl₃ after heating to 600 degrees C whereas the unmilled powder reaction was incomplete after 1 h at 1200 degrees C. Analysis of the energetics of the reactions suggested that the milled powder would undergo self-propagation, whereas the unmilled powder would not.

[20] MECHANICAL ACTIVATION OF IRON AND VANADIUM OXIDES IN ORDER TO OBTAIN NANOMETRIC VANADIUM FERRITE SIMILAR TO A SOFT CHEMISTRY COMPOUND [French]

Nivoix V. Gaffet E. Bernard F. Gillot B. - Comptes Rendus de l'Academie des Sciences Serie II Fascicule C-Chimie. 1(3):183-189, 1998

Co-milling of iron and vanadium oxides allows us to obtain an intimate oxide mixture at a nanoscale, similar to a coprecipitate elaborated by soft chemistry. Reduction of such a mixture in the same temperature and oxygen partial pressure conditions (500 degrees C and 10⁻²⁵ Pa) as the soft chemistry produces leads to a nanometric vanadium ferrite with only a spinel phase. Homogeneity of grain size and chemical composition is reached if the initial oxides have similar grain sizes.

[19] HYPERFINE INTERACTIONS IN NANOCRYSTALLINE FE-AL ALLOYS

Jartych E. Zurawicz JK. Oleszak D. Pekala M. - Journal of Physics-Condensed Matter. 10(22):4929-4936, 1998
Nanocrystalline powder samples of Fe-30 at% Al, Fe-40 at% Al and Fe-50 at% Al alloys were prepared by the mechanical alloying method. X-ray diffraction studies indicated that the solid solution with bcc structure was formed with increasing milling time for all investigated compositions. The magnetic ordering temperature T_c of the nanocrystalline mechanically synthesized alloys was larger than that of the corresponding alloys on a micrometric scale. The magnetization curves as well as the Mossbauer spectra revealed that the Fe-Al alloys formed during the low energy ball milling process contained different magnetic phases.

[18] SPECTROSCOPIC AND DIFFRACTOMETRIC STUDY OF THE V2O5/TIO2 SYSTEM PREPARED VIA MECHANOCHEMICAL ACTIVATION

Dallacqua L. Baricco M. Berti F. Lietti L. Giamello E. - Journal of Materials Chemistry. 8(6):1441-1446, 1998
Materials consisting of a vanadium oxide (VO_x) phase dispersed on a TiO₂ matrix and obtained by mechanochemical activation of the V₂O₅ and TiO₂, are compared with materials obtained by high temperature solid state reaction between the same two oxides. As a consequence of grinding, a non-negligible formation of isolated V-IV is evidenced by EPR, whose nature is typically vanadylic (VO₂⁺). Diffuse reflectance UV-VIS spectroscopy indicates that the reduction of the dispersed phase also induces the formation of more reduced vanadium species like V-III. Various distinct VO₂⁺ species are stabilized on the surface by thermal treatments, and their coordinative environment has been tested by adsorption of NH₃ and H₂O. The phase changes brought about in the TiO₂ matrix by the grinding process as well as the presence of crystalline V₂O₅ have been monitored by XRD.

[17] CHARACTERIZATION OF NANOCRYSTALLINE TANTALUM NITRIDE FORMED BY SOLID-GAS REACTION DURING MECHANICAL ALLOYING

Qin Y. Liu L. Chen L. - Journal of Alloys & Compounds. 269(1-2):238-240, 1998

Tantalum nitride formed by solid-gas reaction of tantalum with molecular nitrogen driven by mechanical alloying has been examined by X-ray diffraction and transmission electron microscopy. The experimental results reveal that Ta₂N with nanometric structure with grain size of 5-20 nm is produced by milling tantalum powders in nitrogen atmosphere. Trace Ta₃N₅ is also detected by electron diffraction pattern. The formation of tantalum nitrides is believed to be associated with the chemisorption of nitrogen onto clean metal surfaces created by milling. The defects generated by mechanical alloying may have also an effect on solid-gas reaction.

[16] MECHANICALLY MILLED MG COMPOSITES FOR HYDROGEN STORAGE - THE RELATIONSHIP BETWEEN MORPHOLOGY AND KINETICS

Gross KJ. Chartouni D. Leroy E. Zuttel A. Schlapbach L. - Journal of Alloys & Compounds. 269(1-2):259-270, 1998

Magnesium based alloys are potentially the best materials for gaseous hydrogen storage due to their high capacity per weight. Unfortunately, their practical use is limited by poor hydrogen absorption and desorption kinetics. This problem can be overcome by mechanically milling Mg alloys with other phases to catalyze the hydriding and dissociation reactions. We have investigated composites formed by mechanically milling La₂Mg₁₇ with LaNi₅. The hydrogen absorption and desorption rates of these composites were maximized by the addition of 40 wt. % LaNi₅. The kinetics for this composition proved far superior to those of the base La₂Mg₁₇ component. It absorbed 95% of its full hydrogen capacity (3.7 wt. % H₂) in 27 a at 250 degrees C and desorbed the same quantity of hydrogen in 4 min. Under the same conditions pure La₂Mg₁₇ took 32 min to absorb and 3 h to desorb 95% of its full hydrogen capacity (5.0 wt. % H₂). Understanding the mechanisms behind the improved kinetics of these composites is critical for the development of better hydrogen storage materials. It is known that, after a few hydrogen absorption-desorption cycles at high temperatures (300 degrees C), the composites are transformed into a very fine powder matrix (grain size similar to 1 μm) of La, Mg and Mg₂Ni. The enhanced properties of this new composite are due to changes in its microscopic morphology and catalytic interactions between the three new component phases. SEM, Microprobe and XPS depth profiling have been used to investigate the complex morphology of the composite particles. 'Full kinetics' and 'incremental kinetics' measurements were performed to compare the absorption and desorption rates of the main hydriding phases. These measurements demonstrate the relationships which exist between the component phases and their contribution to the excellent overall kinetics of these composite materials.

[13] DEHYDRIDING PROPERTIES OF TERNARY MG₂NI_{1-x}ZRX HYDRIDES SYNTHESIZED BY BALL MILLING AND ANNEALING

Zhang YS. Yang HB. Yuan HT. Yang ED. Zhou ZX. Song DY. - J. All. & Compounds. 269(1-2):278-283, 1998

The ternary hydrogen-storage alloys Mg₂Ni_{1-x}Zrx (0 < x less than or equal to 0.3) have been successfully synthesized by ball milling followed by annealing. Studies of the synthesis conditions of the ternary alloys and their dehydriding properties have been carried out. The results show that the ternary Mg₂Ni_{1-x}Zrx (0 < x less than or equal to 0.3) alloys have the same hexagonal crystal structure as that of Mg₂Ni. Compared with a Mg₂Ni alloy, they have a larger specific surface (similar to 1.20 m² g⁻¹), more promising dehydriding kinetics, lower enthalpy of formation of hydrides than that of Mg₂NiH₄, and lower decomposition temperatures in an open system. An optimum desorption storage capacity of about 3.3 wt.% is observed.

[12] MAGNETIC PROPERTIES OF NANOCOMPOSITE ND₂(FE,CO,M)(14)B/ALPHA-FE-BONDED MAGNETS

Jakubowicz J. Jurczyk M. - Journal of Alloys & Compounds. 269(1-2):284-287, 1998

Nanocomposite Nd_{12.6}Fe_{69.8-x}CO_{11.6}MxBb/alpha-Fe (M=Al-Cr, V, Cr, Ni, Zr or Mo) powders, with a volume fraction of magnetically soft alpha-Fe phase of 10 and 37.5%, have been prepared by a high-energy ball-milling (HEBM) and annealing. Enhanced remanent magnetic polarizations up to 1.15 T were obtained. It has been found that addition of a small amount of Al-Cr, Cr, Zr or Mo to Nd_{12.6}Fe_{69.8-x}Co_{11.6}MxB₆/alpha-Fe can improve the intrinsic coercivity and the hysteresis squareness of the Nd₂(Fe,Co)(14)B/alpha-Fe-based nanocomposite materials. The improvements may be related to refined grain sizes realized by these additives. Bonded magnets of J(r) and H-J(c) ranging from 0.81 T and 840 kA m⁻¹ to 1.10 T and 760 kA m⁻¹ have been produced from Nd_{12.6}Fe_{67.8}Co_{11.6}Cr₂B₆/alpha-Fe, with a 10 and 37.5% volume fraction of soft alpha-Fe phase, respectively.

[11] NB- AND CR-AL₂O₃ COMPOSITES WITH INTERPENETRATING NETWORKS

Garcia DE. Schicker S. Janssen R. Claussen N. - Journal of the European Ceramic Society. 18(6):601-605, 1998.

Cr-Al₂O₃ and Nb-Al₂O₃ composites containing 50 vol% metal have been fabricated by pressureless sintering of compacts of attrition milled powder mixtures. Successful fabrication of high-strength and high toughness composites requires fine and homogeneous powders. Strength and fracture roughness of the composites increase with increasing milling time. Short milling times do not lead to the required particle fineness and powder homogeneity. For a composite containing 50 vol% Nb, strengths of up to 690 MPa with corresponding fracture toughness of 6.6 +/- 0.4 MPa m^{1/2} and hardness of 11.2 GPa (H-V20) have been obtained, whereas strengths of 592 MPa, fracture roughness of 6.6 +/- 0.3 MPa m^{1/2} and hardness of 9.3 GPa have been obtained for Cr-Al₂O₃ composites.

[10] AMBIENT TEMPERATURE FORMATION OF AN ALUMINA TITANIUM CARBIDE METAL CERAMIC

Willis PE. Welham NJ. Kerr A. - Journal of the European Ceramic Society. 18(6):701-708, 1998

The fabrication of a sub-micron sized powder composed of alumina, titanium carbide and iron in a single low temperature stage is reported in this paper. The starting materials were the mineral ilmenite (FeTiO₃), graphite and aluminium powder. A similar composite without iron was also produced using rutile (TiO₂) as the starting material. The powders were ball milled together for 100 h in a laboratory scale mill and subjected to annealing at up to 1200 degrees C. X-ray diffraction showed that the phases formed during the milling step were nanocrystalline and underwent crystallite growth on annealing. Differential thermal analysis indicated that the reaction was complete within the mill with no evidence for residual elemental aluminium. Mixtures of the same composition showed only a

slight reaction when heated to 1200 degrees C.

[9] MICROSTRUCTURAL AND KINETIC ASPECTS OF THE TRANSFORMATIONS INDUCED IN A FEAL ALLOY BY BALL-MILLING AND THERMAL TREATMENTS

Gialanella S. Amils X. Baro MD. Delcroix P. Lecaer G. Lutterotti L. Surinach S. - Acta Materialia. 46(9):3305-3316, 1998

A Fe-40Al (at.%) alloy powder having a B2 ordered structure was milled in a high energy planetary ball-mill. The microstructural evolution of the alloy was followed by analysing powder specimens milled for different times by X-ray diffraction, Mossbauer spectroscopy and magnetisation measurements. Grain refinement and chemical disordering were the main transformations resulting from milling. A complete destruction of the long-range order under the adopted conditions of milling was not achieved. From diffraction analyses it was possible to see how the concentrations of such defects as dislocations, planar faults, antiphase domain boundaries, etc., were modified by the heavy deformations involved with milling. Starting from the specimens milled for the longest time, considered in this study, isothermal annealing experiments were carried out to monitor the reverse transformations. The annealing temperatures were selected on the basis of differential scanning calorimetric and thermogravimetric magnetic measurements, which revealed that several transformations occur when treating the deformed powders. Recovery and reordering take place at temperatures ranging from 100 up to 250 degrees C. A complete reordering is possible only at higher temperatures, i.e. 700 degrees C, when recrystallisation is fully accomplished.

[8] ELASTIC STRESS IN COMPOSITE FETI HYDROGEN STORAGE MATERIALS

Tessier P. Schulz R. Stromolsen JO. - Journal of Materials Research. 13(6):1538-1547, 1998

A simple model of the elastic stress in a composite hydrogen absorbing material is developed to account for the hydrogen storage properties of nanocrystalline FeTi with a network of intergranular phase having a wide storage site energy distribution. The model accounts for the equilibrium properties of nanocrystalline FeTi hydrogen absorbers made by ball-milling such as the narrowing of the miscibility gap and changes in plateau pressure. A second model is proposed for disconnected inclusions of the second phase. The effect of chemical disorder is also briefly examined.

[7] MECHANICALLY INDUCED REACTION BETWEEN ALKALINE EARTH METAL OXIDES AND TiO2

Welham NJ. - Journal of Materials Research. 13(6):1607-1613, 1998

This paper outlines the formation of alkaline earth metal titanates, of the general formula $MTiO_3$, directly from the metal oxides and rutile by mechanical activation in a laboratory ball mill at room temperature. X-ray diffraction analysis showed that the reaction was essentially complete within 100 h for all metals except magnesium. The titanates formed all had a Scherrer crystallite size of 11-12 nm and a lattice strain of 0.5-0.6%, neither of which were affected by extended milling. Annealing studies confirmed that the titanate was formed during milling and showed that grain growth could be achieved at temperatures below that generally used for their formation. Mixed cation titanates could also be formed by milling, but tended to be barium rich until annealed.

[6] SANS INVESTIGATION OF SINTERING PROCESS BASED ON NANO-CRYSTALLINE POWDER

Grosz T. Bartha L. Borbely S. Harmat P. Rosta L. - Physica B. 241:350-351, 1997

At the Budapest Research Reactor, we plan to initiate the systematic SANS investigation of nano-crystalline-based sintered composite materials. The results of preliminary SANS experiments carried out on pure nanosized WC powder samples reveals different particle sizes in the initial materials due to the different milling technologies.

[5] X-RAY DIFFRACTION STUDY OF NANOCRYSTALLINE PHASES FORMATION IN METALLIC SYSTEMS

M Baricco, P Rizzi, S Enzo - EPDIC 5, PTS 1 AND 2 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 278-2, pp 559-564 - 71ST ANNUAL MEETING OF THE AMERICAN ASSOC FOR THORACIC SURGERY; WASHINGTON, D.C.. MAY 6-8, 1991 - R Delhez, EJ Mittemeijer TRANSTEC PUBLICATIONS LTD, BRANDRAIN 6, CH-8707 ZURICH-UETIKON, SWITZERLAND.

The synthesis and structural characterization of some nanocrystalline alloys is reported in this paper. Fe-based nanocrystals, with interesting soft magnetic properties, have been obtained by crystallization of amorphous alloys. After thermal treatment of amorphous $Fe_{73.5}Cu_{1}Nb_{3}Si_{13.5}B_9$ and $Fe_{90}Zr_{7}B_3$, the nanocrystalline bcc phase, with a grain size of the order of 15 nm, is embedded in the amorphous matrix. Its formation is due to the low growth rate of the crystal phase, which is related to the presence of atomic species with low diffusivity. Single fcc solid solutions has been obtained by ball milling Cu and Co pure elements, which show, in equilibrium conditions, a wide immiscibility gap in solid state. The lattice parameter values, which have been followed for $Cu_{70}Co_{30}$ and $Cu_{50}Co_{50}$ as a function of milling time, indicate the occurrence of the alloying reaction. The miscibility gap can be overcome because of the creation of a large number of defects and interfaces, which rise the free energy of the system

[4] MOSSBAUER STUDY OF FE-POWDER MECHANICALLY ALLOYED BY N AND C

VM Nadutov, JC Rawers - EPDIC 5, PTS 1 AND 2 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 278-2, pp 565-570 eds R Delhez, EJ Mittemeijer - TRANSTEC PUBLICATIONS LTD, BRANDRAIN 6, CH-8707 ZURICH-UETIKON, SWITZERLAND.

Mossbauer spectroscopy was used to characterize the change in the local iron environment with the addition of nitrogen or carbon in Fe-powder as a function of mechanically processing time in gases atmospheres. The analysis of the Mossbauer spectra did show the solution of interstitials in mechanically processed Fe-powder. The distribution of interstitial elements was found to be inhomogeneous. The distortion of iron crystal lattice both at the grain boundary and interior of nano-grains was found on the basis of lines width analysis. The results of Mossbauer measurements confirmed the X-ray diffraction observations data concerning the formation of the solid solution of nitrogen and carbon in Fe-powder.

[3] NANOCRYSTALLINE STRUCTURE OF THE METASTABLE BALL-MILLED INVERSE SPINEL-FERRITES

V Sepelak, A Buchal, K Tkacova, KD Becker - EPDIC 5, PTS 1 AND 2 (Series: MATERIALS SCIENCE FORUM), 1998, Vol 278-2, pp 862-867 - Eds R Delhez, EJ Mittemeijer - TRANSTEC PUBLICATIONS LTD, BRANDRAIN 6, CH-8707 ZURICH-UETIKON, SWITZERLAND

Changes in the structure of nickel ferrite ($NiFe_2O_4$) and magnesium ferrite ($MgFe_2O_4$) caused by the high-energy

ball-milling have been investigated by X-ray diffraction, Mossbauer spectroscopy electron microscopy and the particle size analysis. Attention is concentrated on the explanation of Hn origin of the mechanically induced changes in magnetic properties of the nanoscale spinel ferrites. The range of thermal stability of the mechanically induced metastable states is determined by studying the response of the metastable mechanically treated ferrites to changes in temperature.

[2] MORPHOLOGICAL CHARACTERIZATION OF MECHANICALLY ALLOYED PET/VECTRA BLENDS BY X-RAY MICROSCOPY

AP Smith, C Bai, H Ade, RJ Spontak, CM Balik, CC Koch, CK Saw - CONFERENCE PROCEEDINGS AT ANTEC '98: PLASTICS ON MY MIND, VOLS I - 3 (Series: SOCIETY OF PLASTICS ENGINEERS TECHNICAL PAPERS), 1998, Vol 44, pp 1474-1478 - SYMP AT THE 22ND ANNUAL CONVENTION OF THE INTERNATIONAL METALLOGRAPHIC SOC : COMPUTER-AIDED MATERIALS CHARACTERIZATION; CHARLOTTE, NORTH CAROLINA. JULY 23-24, 1989 - SOC PLASTICS ENGINEERS, 14 FAIRFIELD DR, BROOKFIELD CENTER, CT 06805.

High-energy mechanical milling constitutes a novel means by which to produce intimate mixtures of immiscible polymers. In the present work, this technique has been employed to blend poly(ethylene terephthalate) (PET) and a thermotropic copolyester (Vectra). Morphological characterization by conventional transmission electron microscopy (TEM) is confounded due to the absence of a preferential stain to enhance phase contrast. Based on Near Edge X-ray Absorption Fine Structure spectroscopy, x-ray microscopy is sensitive to the chemistry and orientation of functional groups and can be used to discriminate between PET and Vectra. X-ray micrographs reveal that PET-rich blends exhibit Vectra-rich domains ranging from 0.10 to 20 μ m in diameter. X-ray microscopy is also utilized here to determine the molecular orientation within the Vectra-rich domains.

[1] MICROSTRUCTURAL CHARACTERIZATION OF A POLYMER/METAL MECHANICAL ALLOY

WJD Shaw, HJ Danilkewich - ROLL OF CHARACTERIZATION IN UNDERSTANDING ENVIRONMENTAL DEGRADATION OF MATERIALS (Series: MICROSTRUCTURAL SCIENCE), 1998, Vol 25, pp 297-302 - 8TH INTERNATIONAL CONF ON COMPUTATIONAL METHODS IN WATER RESOURCES; VENICE, ITALY. JUNE 11-15, 1990 - MCSC ASM INTERNATIONAL, 9503 KINSMAN RD, MATERIALS PARK, OH 44073.

A mechanical alloy was synthesised using polystyrene and tin as a first attempt in looking at a polymer metal combination. The mechanical alloying technique is known to work for metal, intermetallics as well as more recently for polymeric materials. The work conducted here has shown a large dependency upon the gaseous environment surrounding the materials being processed. If argon is used, considerably better material properties are obtained. Analysis strongly suggests that the tin bonds itself to the benzene ring in the polystyrene during the processing technique. The alloy produced has shown that it is quite sensitive to environment or processing conditions as reflected in it's microstructure and overall behaviour. The characterization of this material has shown that organometallic interactions take place primarily during the consolidation process and that the technique holds great promise for the fabrication of various combinations of alloys between polymers and metals.

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