



**RESEAU FRANÇAIS DE
MECANOSYNTHESE**

Lettre N°86

Mai 2002

**187 (+1) Groupes de Recherche
(dont 113 (+1) à l'étranger / 34 Pays)**

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

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

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

⇒ vous y trouverez les anciennes lettres du RFM (accessible par Adobe Acrobat), les statuts du RFM ainsi que les annonces concernant les JRFM'2001 et quelques éléments mis à jour régulièrement concernant les derniers résultats dans ce domaine.

Congress and School Announcements

Nano 2002

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

NANO-7 / ECOSS-21

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

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

Workshops

Gordon Research Conference on Granular and Granular-Fluid Flow

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

ICSTR

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

RQ11

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

10th European Symposium on Comminution

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

8th ICCPS

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

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

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

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

ISMANAM-2002

International Symposium on
Metastable, Mechanically Alloyed and Nanocrystalline Materials
Seoul, Korea, 8-12 September, 2002.

Web site : <http://anu.andong.ac.kr/~ismanam/>



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

Matériaux 2002

Tours - France

21- 25 Octobre 2002

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

E_mail : materiaux@materiaux2002.net

Les JRFM'2002 seront intégrées dans le cadre du Congrès

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

Poudres et Matériaux Nanostructurés, du fondamental aux applications industrielles

Symposium 1 :

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



SOUTENANCES DE THESE

Sophie Soiron

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

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

Jury:

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

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

F. Dore

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

Université de Grenoble - 13 Novembre 2001

Jury :

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

Raphaël JANOT

Université de Nancy I – 24 octobre 2001

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

Jury :

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

Thierry Girot

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

Thèse INPL, 15 Octobre 2001

Jury :

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

Sébastien Lehnard

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

Université de Metz - 5 octobre 2001-08-23



Jury :

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

Nathalie Bouad

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

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

Montpellier, Université Montpellier II, 10 mai 2001

Jury :

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

Cooperative Research on Related Areas

France (12/04/2001)

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

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

Europe (6/03/2001)

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

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

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

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



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

Proposal from 4/04/2002

PhD CORUS Industrial CASE Studentship. Oxford University

Supported by EPSRC and CORUS, for postgraduate research on the structure, properties and processing of aluminium alloys or steel. The studentship will pay the holder a stipend of at least £11,000 per year, plus University and College fees.

Applicants should send a curriculum vitae and names of two academic referees to Kay Sims, Secretary to the Director of Graduate Studies, Department of Materials, University of Oxford, Parks Road, Oxford OX1 3PH, UK; tel: (01865) 273682; fax: (01865) 273783; e-mail: <mailto:kay.sims@materials.ox.ac.uk>kay.sims@materials.ox.ac.uk

Proposal from 11/03/2002

Announcing Ph.D. and postdoc positions:

A Ph.D. and a postdoc position is available in a joint project of the Fritz-Haber-Institut der Max-Planck-Gesellschaft (Matthias Scheffler, <http://www.fhi-berlin.mpg.de/th/th.html>) and The Pennsylvania State University (Henry C. Foley, <http://fenske.che.psu.edu/Faculty/Foley/index.html> and Kristen Fichthorn, <http://fenske.che.psu.edu/Faculty/Fichthorn/index.html>).

Theme:

The role of nano-porous carbon in dehydrogenation and oxidation catalysis

Project summary:

This is a highly interdisciplinary project involving, e.g. extensive density-functional theory calculations and Statistical Mechanics simulations (with DFT derived parameters).

The catalytic production of styrene is one of the most important processes in chemical industry (a key process for making most plastics). Recently it could be shown that the typically employed iron-oxide catalyst is in fact not the active material, but the true catalyst is formed during the induction period: The material that is actually doing the catalysis apparently is "nano porous carbon". This consists of strained and twisted graphite sheets that have a lot of defects (in particular five-fold rings). http://www.fhi-berlin.mpg.de/th/Slides/Scheffler_transparencies_pdf/npc-2002.pdf summarizes some aspects of our recent work.

The planned research may start with an analysis of the chemical reactivity of nanotubes of different diameter and of the different regions of nano porous carbon. At a later step it is planned to model the dynamics of the flow of steam + ethylbenzene at such carbon structures, and the process of ethylbenzene dehydrogenation.

Where:

The student/postdoc will spend some time in Berlin and some time in the US. Details will be decided along the progress of the work.

We are looking for computational physicists, chemists, or chemical engineers. Good background in electronic structure theory, thermodynamics, and statistical mechanics is important

Please send your application material to:

Matthias Scheffler

Fritz-Haber-Institut phone : ++49-30-8413 4711
der Max-Planck-Gesellschaft fax : ++49-30-8413 4701
Faradayweg 4-6 e-mail: scheffler@fhi-berlin.mpg.de
D-14 195 Berlin-Dahlem / Germany
WWW: <http://www.fhi-berlin.mpg.de/th/th.html>

Proposal from 8/03/2002

The Laboratory for Neutron Scattering (ETH Zurich & PSI Villigen) has an open position for a scientist to work in the field of powder neutron diffraction. The position is on a contractual basis and has a duration of 2-3 years with an option for prolongation. The starting date is November 1, 2002.

Research Scientists (Physicists, Chemists, Crystallographers) are invited to apply for this position

• Your tasks:

- Responsibility for the operation and further development of a powder neutron diffractometer at the spallation source SINQ at PSI Villigen.

- Co-operation with guest scientists in their experiments at SINQ.

- Performance of your own research projects.

• Your profile:

- You are a graduated research scientist (PhD) with some years' experience in the field of neutron scattering, particularly with neutron diffraction.

- You have some practical knowledge of computing and cryogenics.

- You are willing to work in a team and to communicate (establishing a professional relationship with guest scientists) as well as to work flexible hours.

• For further information

please contact Prof. Dr. A. Furrer, phone: +41-56-3102088, fax: +41-56-3102939, e-mail: albert.furrer@psi.ch

Please send applications with C.V., a list of publications and the names of two academic referees no later than by April 30, 2002, to: Prof. Dr. A. Furrer, Laboratory for Neutron Scattering, CH-5232 Villigen PSI, Switzerland.

Proposal from 28/01/2002

The Laboratory for Neutron Scattering (Paul Scherrer Institute and ETH Zuerich) announces three openings for research scientists (physicists, chemists, crystallographers) at the Swiss Spallation Neutron Source 'SINQ' (<http://sinq.web.psi.ch/>).



The posts represent excellent opportunities for postdoctoral scientists to develop their expertise, broaden experience and interact with scientists from many countries. We are looking for

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

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

(at your earliest convenience)

Reference Number: 3302A

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

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

(from 01/07/2001)

Reference Number: 3302B

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

(from 01/10/2002)

Reference Number: 3303A

Your tasks would be:

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

For further information (but not applications) please contact for

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

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

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

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

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

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

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



Périodiques

[59] OPTIMAL USE OF COLLOID BALL MILLS FOR WET MILLING [REVIEW] [GERMAN]

Geisler R. - Chemie Ingenieur Technik. 74(1-2):41-54, 2002

Stirred ball mills are being more and more integrated into the automatic processing system. To enhance the safety of the operation and increase the milling capacity, as well as to enable the prediction of the grinding results, it is necessary to understand the processes occurring in the milling chamber. Therefore, it is necessary to relate the optimal operating regime to dependence on the geometry, reactant, and operating parameters. The applicability and reliability of CoBall mills, as a typical representative of annual gap mills, can be determined more precisely by the modeling presented. At the same time, predictions can be made about the flow paths in the grinding gap and the motion of the grinding balls. For given geometries, the local temperature gradients for different inlet flows are discussed

[58] INFLUENCE OF MULLITE ADDITIONS ON THERMAL SHOCK RESISTANCE OF DENSE ALUMINA MATERIALS PART 1: PROCESSING STUDIES

Moreno R. Mezquita S. Baudin C. - British Ceramic Transactions. 100(6):241-245, 2001.

Homogeneous, fine grained (approximate to 0.5-2 μm), and dense (greater than or equal to 97% of theoretical density) alumina-mullite composites have been prepared by a colloidal filtration route. In order to improve the uniformity of the composites, the rheological behaviour of aqueous suspensions of alumina, mullite, and alumina mullite mixtures was studied, focusing on the optimum concentration of deflocculant, the mixing procedure, and milling time. The sintered materials were characterised (scanning electron microscopy, dynamic Young modulus, Vickers indentation hardness, and toughness) and compared with an alumina material with similar density and microstructure. Small mullite additions (5-15 vol.-%) allow desirable values of hardness and toughness of alumina to be maintained while reducing the Young modulus below that of alumina, so that it may be expected that the thermal shock behaviour will be improved

[57] SYNTHESIS OF BATIO₃ BY MECHANOCHEMISTRY [SPANISH]

Rodriguez-Paez JE. Diaz F. Raigoza CFV. - Boletín de la Sociedad Española de Cerámica y Vidrio. 41(1):177-181, 2002

The barium titanate, BaTiO₃, is one of the electronic ceramics with more history of technological application in the industry of the ceramic capacitors. The actual technological demands, and the high quality required for the electronic devices, have driven to new alternatives of prosecution of these materials and to optimize the traditional techniques of raw materials synthesis it prevails. An alternative synthesis is to use the mechanical energy to modify the physicochemical properties of the dispersed system and to favor the obtaining of powders of BaTiO₃. In this work the results are indicated obtained when using mechanical activation, physicochemistry, in the obtaining of the BaTiO₃. It has been analyzed the effect of the time of mill and the nature of the precursor of the titanium on the transformation of phases in the samples during the thermal treatment to that are subjected. X-ray diffraction, DRX, was used to study the phases evolution and infrared spectroscopy; FTIR, to determine the functional groups present in the samples

[56] COMPOSITIONAL DEPENDENCE OF CRYSTALLIZATION BEHAVIOR OF MECHANICALLY ALLOYED AMORPHOUS FE-NI-ZR-B ALLOYS

Liu YJ. Chang ITH. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 325(1-2):25-30, 2002

The effect of the Ni/Fe ratio on the glass transition, the crystallization behavior and glass forming ability of mechanically alloyed amorphous Fe-Ni-Zr-B alloys has been investigated. Differential scanning calorimetry was used to obtain the glass transition, crystallization temperatures, and the melting points of the amorphous alloys. The glass forming ability is discussed with relation to the Ni/Fe ratio. The crystallization products have been studied using a combination of X-ray diffractometry and transmission electron microscopy. Finally, a metastable phase diagram for crystallization is established

[55] DENSE NANOCRYSTALLINE TiB₂-TiC COMPOSITES FORMED BY FIELD ACTIVATION FROM HIGH-ENERGY BALL MILLED REACTANTS

Lee JW. Munir ZA. Ohyanagi M. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 325(1-2):221-227, 2002

The formation of dense nanometric TiB₂-TiC composites from ball milled elemental reactants was investigated. Elemental powders (Ti, C and B) were milled to (a) produce nanometric powders without product formation or (b) to effect a reaction during milling to produce nanostructured TiB₂ and TiC. The products of these two mechanical activations were reacted/consolidated or only consolidated under the influence of a high current and a uniaxial pressure. Dense (up to 98.6%) nanocomposites were formed. The effect of prior milling on the crystallite sizes of the composite components was studied. The crystallite sizes of TiB₂ and TiC in the dense composite formed from powders milled for 10 h were 71.4 and 62.5 nm, respectively. The microhardness of this composite was 20.6 GPa.

[54] SMALL-ANGLE X-RAY SCATTERING STUDY OF NANOCRYSTALLINE FE₂CU_{1-y} ALLOYS PRODUCED BY BALL MILLING

van Raap MBF. Socolovsky LM. Sanchez FH. Torriani IL. - Journal of Physics-Condensed Matter. 14(4):857-864, 2002

Small-angle x-ray scattering measurements on ball-milled Fe₂Cu_{1-y} giant-magneto-resistive alloys (0.141 less than or equal to y less than or equal to 0.45) have been performed using synchrotron radiation. For samples with different nominal compositions, prepared under exactly the same conditions, the scattered intensity recorded as a function of the wavevector varied systematically. For this reason, the strong scattering signal was attributed mainly to composition fluctuations in the crystalline grains. The system was treated as a two-phase model consisting of Fe-rich regions in a homogeneous Cu matrix, with composition-dependent relative volume fractions. Real-space analysis of the scattering was performed in terms of a



volumetric distribution of spherical particles. Results indicate that the main contribution corresponds to 2 nm scattering objects of well-defined density contrast. The invariant Q was calculated to assess the variations in electron-density contrast as a function of the Fe content in the samples. Calculations based on these results allowed the determination of 0.35 at.% iron concentration in the iron-rich phase for all the nominal compositions studied

[53] MAGNETIC PROPERTIES OF ND-Fe-CO(CU)-AL-B AMORPHOUS ALLOYS PREPARED BY NONEQUILIBRIUM TECHNIQUES

G. Eckert J. Roth S. Loser W. Ram S. Schultz L. - Journal of Applied Physics. 91(6):3764-3768, 2002

The amorphous alloys Nd₄₀Fe₄₀Co₅Al₈B₇, Nd₅₇Fe₂₀Co₅Al₁₀B₈, and Nd₅₇Fe₂₀Cu₅Al₁₀B₈ were prepared by copper mold casting, melt spinning, and mechanical alloying. Despite their similar x-ray diffraction patterns, samples display different magnetic and thermal behavior correlated with the method of preparation. The fully amorphous melt-spun ribbons exhibit relatively soft magnetic properties with coercivities approximate to 40 kA/m at room temperature and a Curie temperature (T_C) approximate to 474 K. Apparently only the mold-cast cylinders of 3 mm diameter show hard magnetic behavior with a coercivity in the range of 258-270 kA/m (depending on composition) and have approximately the same T_C as that of the melt-spun ribbons. An additional magnetic transition at 585 K due to the presence of Nd₂Fe₁₄B phase in the case of Nd₄₀Fe₄₀Co₅Al₈B₇ cast rod has been observed. Heat treatment above crystallization temperature in as-cast Nd₅₇Fe₂₀Co₅Al₁₀B₈ and Nd₅₇Fe₂₀Cu₅Al₁₀B₈ samples destroys the hard magnetic properties. In contrast, mechanically alloyed amorphous samples are soft magnetic with maximum coercivity up to 11 kA/m but show an entirely different T_C approximate to 680-740 K, which is rather characteristic of an Fe solid solution. The magnetic properties are discussed in terms of different local atomic environment and cluster sizes in amorphous samples prepared by different methods.

[52] MAGNETIC PROPERTIES OF IRON-CONTAINING PARTICLES IN A QUARTZ MATRIX AFTER MECHANOCHEMICAL PROCESSING

Mofa NN. Ketegenov TA. Ryabikin YA. Chervyakova OV. Ksandopulo GI. - Inorganic Materials. 38(2):132-136, 2002

[51] MECHANOCHEMICAL SYNTHESIS OF ZrO₂-CeO₂ SOLID SOLUTIONS

Morozova LV. Lapshin AE. Panova TI. Glushkova VB. - Inorganic Materials. 38(2):153-158, 2002

ZrO₂-CeO₂ solid solutions (12 and 13 Mol % CeO₂) were prepared by mechanochemical synthesis in different media. The mechanical activation medium was shown to have a significant effect on the phase composition and particle size of tile resultant powders. The conditions for sintering the powders were optimized

[50] MECHANICAL ACTIVATION OF DIOPSIDE IN CO₂

Kalinkin AM. Politov AA. Boldyrev VV. Kalinkina EV. Makarov VN. Kalinnikov VT. - Inorganic Materials. 38(2):163-167, 2002

The mechanical activation (MA) of diopside in a controlled CO₂ atmosphere was studied using different grinding facilities. The results demonstrate that diopside may sorb CO₂ by two mechanisms, depending on the nature of the MA process. If grinding is not accompanied by structure breakdown, the sorption process is similar to that reported for metal oxides, and the adsorbate consists of undistorted CO₃²⁻ groups. If the MA process leads to diopside amorphization, CO₂ is sorbed in the form of distorted carbonate groups, and the IR spectrum contains a split absorption band (1433 and 1522 cm⁻¹) similar to the CO₃²⁻ band in the spectra of carbonate-containing silicate glasses. After ball milling in an AL-1000 mechanical activator in CO₂ for 580 min, diopside contains up to 15 wt % CO₂. Subsequent heating ensures partial or complete removal of the carbonate. Acid treatment of diopside after MA in CO₂ leads to decomposition of the carbonate and almost complete leaching of the Ca and Mg cations.

[49] STRUCTURE AND MAGNETIC PROPERTIES OF NANOSTRUCTURED PrCo₇-XTi_x(x=0-0.4) PREPARED BY MECHANICAL MILLING AND SUBSEQUENT ANNEALING

Zhang J. Shen BG. Zhang SY. Wang YQ. Duan XF. - Applied Physics Letters. 80(8):1418-1420, 2002

The Pr(Co,Ti)₇ phase alloy, which can not be prepared directly by arc melting, has been synthesized by mechanical milling and subsequent annealing. A coercivity of 7.3 kOe, remanence ratio of 0.69 and maximum energy product of 10.6 MGOe have been achieved for the PrCo_{6.7}Ti_{0.3} powder milled for 3 h and annealed at 800 degreesC for 3 min. The high coercivity obtained indicates that the addition of Ti causes the anisotropy of PrCo₇ phase to change from planar to uniaxial. Transmission electron microscopy reveals that a uniform Pr(Co,Ti)₇ microstructure with an average grain size of about 20 nm is developed in the powders. The electron energy-loss spectrum acquired from one grain proves that the Ti enters the Pr(Co,Ti)₇ grain. The high coercivity obtained apparently arises from the high uniaxial anisotropy of Pr(Co,Ti)₇ phase and the uniform nanoscale microstructure developed by mechanical milling and subsequent annealing.

[48] CRYSTALLOGRAPHIC FEATURES OF MECHANICALLY MILLED AND ALLOYED NANOSIZED CRYSTALLINE AND AMORPHOUS MATERIALS

Tonejc A. - Acta Chimica Slovenica. 49(1):1-28, 2002

High energy mechanical ball milling, which includes mechanical alloying (MA) and mechanical grinding (MG), has been extensively used in the last 20 years as a non-equilibrium processing method analogous to the previous interest in very rapid solidification cooling (rates from 10⁶ to 10¹⁰ K/s) for synthesizing, at room temperature, all kinds of materials. Both mechanical alloying of dissimilar powders and mechanical grinding of single composition powder have resulted in a wide variety of entirely new metastable alloys including amorphous alloys, nanocrystalline materials, extended solid solutions, alloys from elements with a widely different melting point, all sorts of compounds and composites and otherwise difficult to process novel materials, and even those alloys which are immiscible by conventional processing methods. The crystallite size of MA and MG materials decreases rapidly with milling time to reach a saturation value, generally in the range from 1



to 30 nm, attributing the name nano-sized materials to MA and MG end products. During the milling process the interaction between milling balls and powder particles can be characterized by processes like cold-welding, plastic deformation and further fragmentation of the particles. A high defect structure of the lattice, the immense magnification of the boundary surface and high diffusion rate leads to low activation energies for the transformation of the structure. The current state of the mechanism of the metastable phase formation during milling systems which exhibit a negative heat of mixing of the components will be given from the thermodynamic and kinetic viewpoints. On the other hand, although phase formation in systems with positive heats of mixing is far from being understood, a possible explanation will also be given. Various application areas where the MA technology could be used will be illustrated as well.

[47] ON FRICTION LAYER FORMATION IN POLYMER MATRIX COMPOSITE MATERIALS FOR BRAKE APPLICATIONS

Filip P. Weiss Z. Rafaja D. - *Wear*. 252(3-4):189-198, 2002

Due to the complexity of friction phenomena in polymer matrix composites, the friction mechanisms have not been fully understood. This paper concentrates on the characterization of friction layer formation and correlation of friction layer properties to the performance of a recently developed family of polymer matrix composites. It was demonstrated that character of the friction layer determines the friction performance of the investigated composite material. Structure and chemical composition of the friction layer generated on the friction surface significantly differs from the bulk. Mechano-chemical interaction occurring in the friction process is compared to a "non-friction" situation where an "equivalent" apparent temperature and compressive loading, respectively were applied to the same material. No simple relationship exists between composition of the friction layer and bulk material formulation. Phase stability and kinetics of interactions for "friction" and "equivalent non-friction" loading conditions significantly differ.

[46] A MICRO-CONTACT AND WEAR MODEL FOR CHEMICAL-MECHANICAL POLISHING OF SILICON WAFERS

Zhao YW. Chang L. - *Wear*. 252(3-4):220-226, 2002

A micro-contact and wear model for chemical-mechanical polishing (CMP) of silicon wafers is presented in this paper. The model is developed on the basis of elastic-plastic micro-contact mechanics and abrasive wear theory. The synergetic effects of mechanical and chemical actions are formulated into the model. A close-form equation of material removal rate from the wafer surface is derived relating to the material, geometric, chemical and operating parameters in a CMP process. The model is evaluated by comparing the theoretical removal rates with those experimentally determined. Good agreement is obtained for both chemically active and inactive polishing processes. The model reveals some insights into the micro-contact and wear mechanisms of the CMP process. It suggests that the removal rate is sensitive to the particle concentration in the slurry, more sensitive to the applied load and operating speed and most sensitive to the surface hardness and slurry particle size. The model may be used to study the effects of different materials, geometry, slurry chemistry and operating conditions on CMP processes.

[45] MECHANICAL MILLING OF FULLERENE WITH CARBIDE FORMING ELEMENTS

Liu ZG. Tsuchiya K. Umemoto M. - *Journal of Materials Science*. 37(6):1229-1235, 2002

Mechanical alloying of Ti, V, Cr, Mo and W with fullerene (C-60(C-70)) and graphite reveals that fullerene is more reactive than graphite. The formation heat of carbide is the driving force for reaction in the mechanical alloying process. Higher heat of formation results in the direct formation of carbide in Ti-C systems, and the formation of carbide in V-C systems during the subsequent heating of milled powder. In the systems with lower carbide heat of formation, a mixture of metal with carbon is obtained by ball milling. No carbide was obtained even after heating the milled powders up to 973 K. Small amount of fullerene remained when milled with Mo and W for 10 hours. (C) 2002 Kluwer Academic Publishers

[44] SYNTHESIS AND CHARACTERISATION OF DUAL-PHASE Y-TZP AND RuO₂ NANOPOWDERS: DENSE ELECTRODE PRECURSORS

van Zyl WE. Winnubst L. Raming TP. Schmuhl R. Verweij H. - *Journal of Materials Chemistry*. 12(3):708-713, 2002.

The synthesis and characterisation of nanopowders in the dual-phase system tetragonal-Y₂O₃-doped ZrO₂ (Y-TZP) and RuO₂ are described. Five powders were prepared from a co-precipitation (CP) method with stoichiometric variation in the RuO₂ content (5-46 mol%) and two powders were prepared from solid-phase mechanical mixing of the above oxides prepared separately. In the CP method, an aqueous chlorometal solution containing appropriate precursor ions was co-precipitated in a concentrated aqueous ammonia (pH similar to 14) solution. Following filtration, washing and drying (100 degreesC), the CP synthesis route yielded black coloured amorphous powders. Crystalline dual-phase powders were obtained after calcination in stagnant air at 600 degreesC for 2 h. The average Y-TZP and RuO₂ crystallite sizes were, respectively, 10 and 20 nm, i.e. a nano/nano powder. The powders were characterised by X-ray diffraction (XRD), transmission electron microscopy (TEM), backscatter Raman spectroscopy (ZrO₂ phase determination) and quantitative X-ray fluorescence (XRF). Time-dependent calcination experiments for samples of similar composition revealed that under the synthesis conditions employed, a composite was formed where a fraction of the crystalline RuO₂ phase was initially dissolved in the ZrO₂ phase and which gradually transforms to a more stable, distinctly dual-phase system upon prolonged (greater than or equal to 20 h) calcination at 600 degreesC

[43] CU-MGO SAMPLES PREPARED BY MECHANOCHEMISTRY FOR CATALYTIC APPLICATION

Varga M. Molnar A. Mulas G. Mohai M. Bertoti I. Cocco G. - *Journal of Catalysis*. 206(1):71-81, 2002

High-energy ball milling was applied to prepare powder samples by treating copper or copper oxides with magnesium or magnesium oxide. The five samples thus prepared were characterized by physical methods. According to X-ray diffraction the mechanical treatment resulted in the formation of nanostructured powders. Carbon and oxygen impurities and Mg



(mainly as oxide) were detected by X-ray photoelectron spectroscopy of the as-milled samples. Four samples, namely, nanocomposite materials prepared by the self-sustaining reaction of copper oxides and magnesium applied in stoichiometric amounts [(Cu₂O)Mg and (CuO)Mg] and samples produced by mechanical treatment of copper oxides and magnesium oxide [(Cu₂O)(7)(MgO)(93) and (CuO)(13)(MgO)(87)] also had copper on the surface. The latter specimens and 3% Cu + MgO were unique in their characteristics to have, after a short hydrogen treatment or after catalytic application, an unusually large concentration of Cu-0 measured by the N₂O titration method. Temperature-programmed reduction showed, in each case, the existence of reducible copper species on the surface. The quantity of reducible copper was quite low for (Cu₂O)Mg, (CuO)Mg, and 3% Cu + MgO, whereas almost the total amount of copper of (Cu₂O)(7)(MgO)(93) and (CuO)(13)(MgO)(87) was available for reduction. Two types of copper species have been detected: bulk CuO with a reduction temperature of about 260degreesC [characteristic of (Cu₀)Mg and also found in (Cu₂O)Mg] and species strongly interacting with the support with a reduction temperature of about 350degreesC [3% Cu + MgO, (Cu₂O)(7)(MgO)(93), (CuO)(13)(MgO)(87), and also detected in (Cu₂O)Mg]. The 3% Cu + MgO, exhibited the highest basicity measured by the decomposition of 2-methyl-3-butyn-2-ol and the dimerization of acetone. The samples showed high activity in the dehydrogenation of 2-propanol, whereas their activity in the one-step synthesis of methyl isobutyl ketone from acetone was moderate. Methyl isobutyl ketone (MIBK) was formed in low selectivity over the samples prepared by a self-sustaining reaction and 3% Cu + MgO, whereas very high MIBK selectivities were observed over the mixed oxides. These features were correlated with the relative concentration of active sites capable of hydrogenating the carbon-oxygen and carbon-carbon double bond.

[42] BALL-MILLED CARBON AND HYDROGEN STORAGE

Awasthi K. Kamalakaran R. Singh AK. Srivastava ON. - International Journal of Hydrogen Energy. 27(4):425-432, 2002
We report the formation of carbon in different nanoparticle forms obtained by ball-milling of graphitic carbon. Ball-milling of graphite was carried out in Szegvari attritor at room temperature for varied times i.e. 24, 48 and 100 h in hexane medium. The characterization of ball-milled graphitic carbon (BMC) samples was done by X-ray diffractometry, scanning electron microscopy and transmission electron microscopy, The self-coagulated carbon agglomerates were obtained for the case of 24 and 100 h BMC samples. The fort-nation of coiled nanotubes and nanofibres was observed in the BMC sample. The BMC samples with and without nickel (Ni) catalyst were subjected to hydrogenation cycling in a Sievert's type apparatus fabricated in our laboratory. It has been found that BMC sample can adsorb hydrogen. The hydrogen adsorption capacity has been found to be similar to 0.6 wt%.

[41] THE ELECTROCHEMICAL HYDRIDING PROPERTIES OF MG-NI-ZR AMORPHOUS ALLOY

Goo NH. Lee KS. - International Journal of Hydrogen Energy. 27(4):433-438, 2002
The amorphous alloys of Mg-Ni-Zr were synthesized by mechanical alloying process, in which the effects of Zr addition were studied. A small shift of the broadening amorphous peak due to Zr additions was observed. The equilibrium hydriding pressure increased continuously with hydrogen contents in the amorphous alloys, while there was an obvious plateau pressure of 10(-4) MPa in the crystalline Mg₂Ni phase. The Zr addition lowered the equilibrium hydriding pressure and increased the hydrogen solubility from 1.4 wt% for Mg-Ni to 2.2 wt% for Mg-Ni-Zr. Accompanied by amorphization, the hydrogen discharge capacity increased largely, and reached 580 mA h/g for Mg-Ni-Zr alloy. The chronopotentiometric experiments were performed to investigate the hydrogen diffusion in the alloy powder in the high over-potential range. The diffusivity of hydrogen in the Zr-contained alloy was lower than the Mg-Ni binary amorphous alloy or in Mg₂Ni nanocrystalline alloy.

[40] MECHANICALLY ACTIVE ATHERMALIZATION OF A FORWARD LOOKING INFRARED SYSTEM

Bayar M. Farsakoglu OF. - Infrared Physics & Technology. 43(2):91-99, 2002
This study discusses the mechanically active athermalization of a forward looking infrared (FLIR) system, Athermalization is the principle of stabilizing the optical performance with respect to temperature. First optical design for a FUR system that makes thermal imaging at 8-12 mum band is done using a commercial design and analysis program. Then the optical elements are transferred to a 3D solid modeling program where the housing for the system is designed using the optomechanical engineering principles. Subsequently, all the system with lenses, electronic cards and housing are transferred to a finite element analysis (FEA) software. Initial conditions and obtained results are verified by a laboratory study. After making thermal and structural analysis by FEA software; the temperature distribution, expansions and contractions in the system are determined. These data are used to optimize the system and as a result to ensure the system work at different environmental temperatures.

[39] COMBUSTION OF QUARTZ-CONTAINING OXIDE SYSTEMS MODIFIED BY ORGANIC COMPOUNDS UNDER MECHANOCHEMICAL TREATMENT

Ksandopulo GI. Mofa NN. Ketegenov TA. Chervyakova OV. Tyumentseva OA.- Combustion Explosion & Shock Waves. 38(1):54-59,

The paper deals with the combustion of a SiO₂-Al stoichiometric mixture after mechanochemical treatment in activator mills of two types: mills using predominantly shear loading and those using shear-dynamic compression. It is shown that treatment in different loading regimes, leading to different energy states of the material, and the use of modifying organic additives change significantly the ignition and combustion temperature of the mixture. Modification of the surface of the quartz particle by mechanochemical treatment in the presence of butanol or polystyrene and aluminum activates the combustion process, thus ensuring greater completeness of the oxidation-reduction reaction.

[38] PREPARATION AND CHARACTERIZATION OF ENERGETIC AL-MG MECHANICAL ALLOY POWDERS

Shoshin YL. Mudryy RS. Dreizin EL. - Combustion & Flame. 128(3):259-269, 2002



Metals such as Al and Mg have high combustion enthalpies and they are widely used as additives in energetic materials for propellants, explosives, and pyrotechnics. However, long ignition delays and slow combustion kinetics limit their current applications. An approach suggested in this work is to design new metal-based materials in which pre-determined phase changes will occur and trigger ignition at a desired temperature and also accelerate the rate of heat release during combustion. As a first step, metastable solid solutions of Mg in Al (10-50% of Mg) have been produced by mechanical alloying. The ignition temperatures of the produced alloys in air were determined using digital imaging and three-color pyrometry of the electrically heated filaments coated with different alloy powders. Combustion of mechanical alloys in air was studied using a laminar, premixed flame aerosol burner. The ignition temperatures were around 1,000 K, much lower than the pure aluminum ignition temperature of about 2,300 K. The steady flames of mechanical alloy powders were produced at lower equivalence ratios and had higher propagation velocities than similar pure aluminum powder flames. Phase compositions of the combustion product, were determined using X-ray diffraction. In addition to Al₂O₃ and MgO, significant amounts of Al₂MgO₄ were found in experiments.

[37] MECHANICAL MILLING OF FULLERENE WITH CARBIDE FORMING ELEMENTS

Liu ZG. Tsuchiya K. Umemoto M. - Journal of Materials Science. 37(6):1229-1235, 2002

Mechanical alloying of Ti, V, Cr, Mo and W with fullerene (C-60(C-70)) and graphite reveals that fullerene is more reactive than graphite. The formation heat of carbide is the driving force for reaction in the mechanical alloying process. Higher heat of formation results in the direct formation of carbide in Ti-C systems, and the formation of carbide in V-C systems during the subsequent heating of milled powder. In the systems with lower carbide heat of formation, a mixture of metal with carbon is obtained by ball milling. No carbide was obtained even after heating the milled powders up to 973 K. Small amount of fullerene remained when milled with Mo and W for 10 hours

[36] SYNTHESIS AND CHARACTERISATION OF DUAL-PHASE Y-TZP AND RuO₂ NANOPOWDERS: DENSE ELECTRODE PRECURSORS

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[34] A NEW HIGH-TEMPERATURE, OXIDATION-RESISTANT TI-BASED MATERIAL

Gao W. Li Z. Zhang D. - Oxidation of Metals. 57(1-2):99-114, 2002



Ti₃Al(O)-Al₂O₃ composites were fabricated in situ using a mechanical-alloying and reaction-sintering technique. Their oxidation behaviors were studied in air at 700-900degreesC. The oxidation rates were much lower than those of Ti₃Al. The behaviors of isothermal and cyclic oxidation were very similar. The oxide scales that formed exhibited excellent spallation resistance under all testing conditions. No scale cracking or spallation could be observed, even along the edges or corners of the samples, implying that growth and thermal stresses generated during heating and cooling periods had been effectively released. The mechanisms of the decrease in oxidation rate and the improvement on spallation resistance are discussed based on microstructure studies. This composite has advantages of light-weight, simple fabrication from inexpensive materials, and superior high-temperature oxidation resistance; it may provide opportunities to be used in some high-temperature structural applications

[33] SIZE CONTROL OF SPHERICAL LEUCITE CRYSTALS [JAPANESE]

Hashimoto S. Yamaguchi A. - Nippon Seramikkusu Kyokai Gakujutsu Ronbunshi-Journal of the Ceramic Society of Japan. 110(1):27-31, 2002

Spherical leucite crystals with 100 μm in diameter were obtained when a starting sample consisting of K₂SO₄:Al₂(SO₄)₃:SiO₂ (amorphous silica, 10 μm) = 5.9:1.0:1.1 (molar ratio) was heated at 1000degreesC for 3 h and then treated with hot HCl. The effect of SiO₂ polymorphs and their starting particle size on the size of the resulting spherical leucite crystals was investigated. In addition, the effect of preheating and grinding the starting sample on the size of leucite crystals was studied. The size of the spherical leucite crystals decreased with decreasing the size of SiO₂ particles. When quartz particles with 3.2 to 52.8 μm in diameter were used, the diameter of the spherical leucite crystals was in the range 19.0 to 55.2 μm. When tridymite particles with diameter 3.0 to 72.0 μm were used, the size of spherical leucite crystals was between 12.4 and 135.8 μm. When the starting sample with amorphous SiO₂ was preheated to 800degreesC, cooled to room temperature rapidly, and ground to 4.5 μm, the size of spherical leucite crystals reached on average size of 2.6 μm

Author e-mail Address kayjune@hanmail.net

[32] EXAFS STUDY FOR MECHANICALLY ALLOYED CO50C50

Yang DS. Kim I. Yoo YG. Yu SC. - Journal of the Physical Society of Japan. 71(2):487-490, 2002

Co₅₀C₅₀ alloys were fabricated by mechanical alloying process from the mixed powder of pure cobalt and carbon. The variations of the local structure and magnetic properties were examined by EXAFS analysis and the VSM magnetometer. It is shown that the alloying was progressed as the milling time increases. The significant changes of the magnetization and the structure has been occurred in 12h milling. EXAFS spectrum and its Fourier transform confirms that the final product is in the amorphous state. The EXAFS spectrum of the alloy milled for 24 h was fitted to the conventional EXAFS equation. The interatomic distances obtained from the fitting were 1.94 +/- 0.02 Angstrom for Co-C pair and 2.52 +/- 0.02 Angstrom for Co-Co pair, respectively. The magnetization was almost saturated in 24 h milling.

[31] CERAMIC PIGMENTS OBTAINED BY SOL-GEL TECHNIQUES AND BY MECHANOCHEMICAL INSERTION OF COLOR CENTERS IN AL₂O₃ HOST MATRIX

Ricceri R. Ardizzone S. Baldi G. Matteazzi P. - Journal of the European Ceramic Society. 22(5):629-637, 2002

Nanocrystalline pigments for ceramic glazes have been obtained by mechanochemical insertion of Fe and Ti in an alpha-Al₂O₃ host matrix (Fe and Ti oxides were reduced by high energy milling with metallic Al with subsequent formation of additional Al₂O₃ by oxidation of Al(0)). The powders have been characterized by X-ray diffraction, laser granulometry and by surface area and porosity measurements. Sol-gel techniques have also been used in order to obtain pigments with pure Al₂O₃ and Cr or Fe/Ti-doped Al₂O₃-Pigments have been prepared using Al(III)-alkoxides or Al(III)-inorganic salts, obtaining products with different characteristics in the two cases. Pigments have been studied by X-ray diffraction, surface area and porosity measurements and by the analysis of electrochemical reactivity in aqueous suspensions. Coloring properties of pigments obtained by mechanomaking and by sol-gel techniques have been tested in ceramic glazes; their properties have also been analyzed by insertion in ceramic bodies for glazed gres (porcelain) technology.

[30] ELECTROCHEMICAL CYCLING BEHAVIOR OF LICOO₂ CATHODE PREPARED BY MECHANICAL ALLOYING OF HYDROXIDES

Jeong WT. Lee KS. - Journal of Power Sources. 104(2):195-200, 2002

Well-ordered high-temperature LiCoO₂ (HT-LiCoO₂) is synthesized by mechanical alloying (MA) of LiOH.H₂O and Co(OH)₂ powders and subsequent firing. Its electrochemical properties are investigated. The maximum discharge capacity of a sample mechanically alloyed and fired at 600 degreesC for 2 h is 152 mAh g⁻¹ at the C/40 rate, which is comparable to that obtained from a sample made by conventional solid state reactions. The cycleability is inferior, however, due to a relatively low crystallinity. When the firing temperature is increased to 850 degreesC, the first discharge capacity of 142 mAh g⁻¹ at the C/5 rate is increased by more than 10%, and retains 93% of its maximum value after 30 cycles. These cycling properties are about the same, or slightly higher, than those synthesized by firing a sample mixture of the same starting materials at 600 degreesC for 8 h and then at 850 degreesC for 24 h. Consequently, given the lower firing temperature and/or reduced reaction time, MA could prove a promising synthetic process for cathode materials used in rechargeable lithium batteries.

[29] EFFECTS OF F-TREATMENT ON DEGRADATION OF MG₂Ni ELECTRODE FABRICATED BY MECHANICAL ALLOYING

Kim JS. Lee CR. Choi JW. Kang SG. - Journal of Power Sources. 104(2):201-207, 2002

The effects of surface fluorination on the electrochemical charge-discharge properties of a Mg₂Ni electrode, prepared by mechanical alloying in Ni-MH batteries are investigated. After 20 h milling, Mg and Ni powder form nanocrystalline



Mg₂Ni. The discharge capacity of this alloy increases greatly on the initial cycle but, due to the formation of a Mg(OH)₂ passive layer, displays rapid degradation in alkaline solution within 10 cycles. In a 6 M KOH+x M KF electrolyte (x = 0.5, 1, and 2), a continuous and stable fluorinated layer is formed and the durability of the Mg₂Ni electrode increases markedly and a high rate discharge capability is obtained (90-100 mAh/g). Addition of 2 M KF leads to the highest durability of all the electrodes tested. The improvement is due to a thin MgF₂-fluorinated layer, which reduces the charge-transfer resistance and protects the Mg₂Ni electrode from forming a Mg(OH)₂ layer.

[28] THERMAL STABILITY OF MECHANICALLY ALLOYED NITRIDE/AMORPHOUS Zr₆₅Al_{7.5}Cu_{17.5}Ni₁₀ ALLOY COMPOSITES

Zhou CR. Xu J. - Journal of Non-Crystalline Solids. 297(2-3):131-142, 2002

Elemental powder mixtures with a composition of Zr₆₅Al_{7.5}Cu_{17.5}Ni₁₀ together with 10 vol.% AlN, Si₃N₄ and TiN particles were mechanically alloyed to form nitride/amorphous alloy matrix composites. The microstructure of ball-milled products was characterized using X-ray diffraction and transmission electron microscopy. Glass transition and crystallization kinetics of the nitride-containing composites were investigated with differential scanning calorimetry. In the final products of ball milling, initial particles of crystallite nitride were refined to a nanoscale and homogeneously dispersed in the amorphous Zr-based alloy matrix to form the composites. The nitride-containing composites exhibit a larger supercooled liquid region AT, compared with the amorphous Zr-based alloy without nitrides. The isothermal crystallization kinetics of the mechanically alloyed amorphous Zr₆₅Al_{7.5}Cu_{17.5}Ni₁₀ alloy with or without nitride does not follow the conventional Johnson-Mehl-Avrami model.

[27] MOLECULAR ALLOYS FORMED BY SOLID-STATE VITRIFICATION

Nagahama M. Suge H. - Journal of Molecular Liquids. 95(3):261-284, 2002

A new example of solid-state vitrification for trehalose from its crystalline sample is reported. DSC experiments on vitrified trehalose clarified the heat effects associated with glass transition, crystallization, and fusion, respectively. Increases in the glass transition temperature T_g and the enthalpy of crystallization with the milling time that have commonly occurred in solid-state vitrification of molecular crystals was observed also in trehalose. Mechanical milling on several mixtures of trehalose and sucrose, both being geometrical isomers of disaccharides C₁₂H₂₂O₁₁, produced a series of molecular alloys with T_g value varying monotonously with the composition. Observation of only a single T_g in each solid indicated that the two component molecules mixed on a molecular level to exhibit a sole process for structural relaxation. Formation of other molecular alloys of trehalose with tri-O-methyl- α -cyclodextrin and glucose is also discussed. The former was totally miscible over the whole composition range, while the latter was partially miscible

[26] PHASE FORMATION LEAD ZIRCONATE TITANATE VIA A HIGH-ENERGY BALL MILLING PROCESS

Kong LB. Ma J. Zhu W. Tan OK. - Journal of Materials Science Letters. 21(1):25-27, 2002

[25] INFLUENCE OF MECHANICAL ACTIVATION OF PRECURSORS ON THE STRUCTURE AND PROPERTIES OF DONOR DOPED Ba_{0.95}Pb_{0.05}TiO₃ CERAMICS

Briancin J. Medvecky L. - Journal of Materials Science Letters. 21(1):55-59, 2002

[24] AMORPHOUS Ni₃₀Ta₇₀ POWDERS PRODUCED BY MECHANICAL ALLOYING

Lee CH. Fukunaga T. - Journal of Materials Science Letters. 21(2):141-143, 2002

[23] GRAIN SIZE EFFECT ON THE NEEL TEMPERATURE AND MAGNETIC PROPERTIES OF NANOCRYSTALLINE NiFe₂O₄ SPINEL

Chinnasamy CN. Narayanasamy A. Ponpandian N. Joseyphus RJ. Jeyadevan B. Tohji K. Chattopadhyay K. - Journal of Magnetism & Magnetic Materials. 238(2-3):281-287, 2002

Nanocrystalline NiFe₂O₄ spinel ferrites with various grain sizes have been synthesized by ball milling the bulk NiFe₂O₄. The average grain sizes were estimated from the X-ray line broadening of the (3 1 1) reflection. The Neel temperatures of NiFe₂O₄ for various grain sizes were determined by magneto thermogravimetric method. The magnetic behaviour has been explained by combining the effects of changes in cation distribution on milling and finite size scaling. The shift in B-H loops has been correlated to the surface spin effects. The high coercivities observed here may be due to high anisotropies of the milled samples. The Hopkinson peak observed just below the Neel temperature has been explained by the mathematical formalism given by the Stoner Wohlfarth model.

[22] PECULIARITIES OF DIFFUSION CONTROLLED PHASE FORMATION IN THE Al-Co SYSTEM

Bokstein B. Klimov M. - Zeitschrift fur Metallkunde. 92(10):1186-1188, 2001

By using differential scanning microcalorimetry, X-ray phase analysis, electron microscopy and secondary ion-mass spectrometry, the phase formation was studied in Al-Co powder, prepared by mechanical alloying, and also in Al-Co thin films of Al₁₃Co₄ composition. It was shown that solid-phase reaction between Al and Co starts from Al₉Co₂ formation. The heat of Al₉Co₂ formation was estimated, approximately the same in both types of materials. In mechanically alloyed powders the Al₉Co₂ phase formed at lower temperatures than in thin films. The growth of the Al₉Co₂ phase is a two-stage process. It is supposed that the first stage is controlled by Co diffusion in Al, and the second stage is controlled by Co diffusion through the intermetallic phase Al₉Co₂.

[21] INITIAL STAGE OF LIQUID PHASE SINTERING: LIQUID REACTION AND PARTICLE GROWTH IN W-Ni-Fe-(Co)

Antonsson T. Ekbom L. - Powder Metallurgy. 44(4):325-332, 2001.

The initial stage of liquid phase sintering, involving liquid reaction and particle growth, has been investigated under microgravity in experiments using tungsten heavy alloys for short periods of time (8-14 seconds). The influence of different factors, such as alloy composition, plastic deformation, and non-equilibrium conditions, have been evaluated. During the



liquid phase sintering of tungsten heavy alloys at about 1470degreesC, the liquid matrix penetrates the tungsten particle agglomerates. A fraction of the tungsten particles goes into solution in the liquid phase and the original tungsten powder size will initially be reduced. At the same time, the agglomerates of tungsten particles are effectively separated. In a second stage, larger particles grow in equilibrium with the matrix while pure tungsten particles are dissolved into the matrix. When equilibrium is reached, the tungsten particles start to grow in the liquid Ni-Fe-W matrix phase in accordance with the Ostwald ripening process. A theoretical treatment of the particle solution and growth during these stages is proposed. The addition of iron and cobalt to the W-Ni system reduces the rate of penetration and growth. Non-equilibrium conditions during the formation of a liquid phase have a marked effect on the tungsten particle separation. Milling of the tungsten powder increases the initial growth of tungsten particles

[20] MICROSTRUCTURAL AND FRACTURE CHARACTERIZATION OF NB-CR-TI MECHANICALLY ALLOYED MATERIALS

Davidson DL. Chan KS. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 33(2):401-416, 2002

Three materials containing Nb, Cr, and Ti were fabricated by consolidating powders made by mechanical alloying. The Nb/Ti ratio was maintained at about 1.3 and Cr was increased to form the intermetallic Cr₂Nb. X-ray diffraction, metallography, and transmission electron microscopy were used to thoroughly characterize the microstructure and substructure of the materials. Fatigue and fracture toughness properties were also evaluated at ambient temperature. The alloyed powders contained only small amounts of intermetallic, but during the consolidation heat treatment, two of the materials precipitated large volume fractions of Cr₂Nb. In the third material, Cr₂Nb was precipitated by heat treatment, although this was not expected from the composition based on the Nb-Cr-Ti phase diagram. Maximum fracture toughness of the composites was approximate to Mpa rootm. The low fracture toughness was attributed to the high plastic constraint of matrix deformation by the Cr₂Nb and compositional change in the matrix

[19] MICROSTRUCTURE FORMATIONS IN COPPER-SILICON CARBIDE COMPOSITES DURING MECHANICAL ALLOYING IN A PLANETARY ACTIVATOR

Kudashov DV. Aksenov AA. Klemm V. Martin U. Oettel H. Portnoy VK. Zolotarevskii VS. - Materialwissenschaft und Werkstofftechnik. 31(12):1048-1055, 2000

In the present paper the structure formation process of the powder metallurgical produced copper composite materials was studied. The volume part of the reinforcing SiC particles was varied from 5 to 25 wt.-%. It was discovered that while milling in a planetary activator first of all a "puff- pastry" structure appeared. There are important differences between this structure formation process and other known processes of milling. The homogeneous distribution of SiC particles was obtained after 60-100 minutes of treatment in "Gefest11-3" planetary activator. Phase composition of the powder and composite samples at the interface SiC/Cu (particles/matrix) was analysed after consolidation of the powder mixture and after the high temperature annealing. It was still determined that not only pure copper powder can be as a starting material for Cu-composites production used, but also the wastes of copper mechanical treatment, for instance, copper shaving.

[18] THE MECHANICAL ALLOYING OF TITANIUM ALUMINIDES

Lu L. Lai MO. Froes FH. - JOM-Journal of the Minerals Metals & Materials Society. 54(2):62-64, 2002

This article reviews the mechanical alloying of titanium aluminides carried out in the past decades. Research has proven that mechanical alloying is an efficient means to synthesize nanostructured and non-equilibrium titanium aluminides. Although fine-grained structures have been successfully produced, effort is still needed to reduce contamination and to consolidate nanostructural powders

[17] A NEW EXPERIMENTAL SETUP FOR THE TIME RESOLVED X-RAY DIFFRACTION STUDY OF SELF-PROPAGATING HIGH-TEMPERATURE SYNTHESIS

Vrel D. Girodon-Boulandet N. Paris S. Mazue JF. Couqueberg E. Gailhanou M. Thiaudiere D. Gaffet E. Bernard F. - Review of Scientific Instruments. 73(2 Part 1):422-428, 2002

A new experimental setup for time resolved x-ray diffraction is described. Designed for the LURE H10 beamline and its 4 (+2) circles goniometer, it allows simultaneous recordings of x-ray patterns with a rate of 30 patterns per second, a maximum 2theta range of 120degrees, infrared thermography at the same rate, and thermocouples readings at a frequency of up to 3x10(4) Hz. Preliminary results obtained using this setup are presented, showing how it is possible to analyze a solid-solid or solid-liquid reaction. As an example, an in situ study of phase transformation and temperature evolution during the self-sustaining synthesis of an FeAl intermetallic compound starting from a mechanically activated mixture is investigated. The versatility of the setup was proved and could even be enhanced by the design of new sample holders, thus expanding its area of use at low cost.

[16] MICROSTRUCTURAL AND FRACTURE CHARACTERIZATION OF NB-CR-TI MECHANICALLY ALLOYED MATERIALS

Davidson DL. Chan KS. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 33(2):401-416, 2002

Three materials containing Nb, Cr, and Ti were fabricated by consolidating powders made by mechanical alloying. The Nb/Ti ratio was maintained at about 1.3 and Cr was increased to form the intermetallic Cr₂Nb. X-ray diffraction, metallography, and transmission electron microscopy were used to thoroughly characterize the microstructure and substructure of the materials. Fatigue and fracture toughness properties were also evaluated at ambient temperature. The alloyed powders contained only small amounts of intermetallic, but during the consolidation heat treatment, two of the



materials precipitated large volume fractions of Cr₂Nb. In the third material, Cr₂Nb was precipitated by heat treatment, although this was not expected from the composition based on the Nb-Cr-Ti phase diagram. Maximum fracture toughness of the composites was approximate to Mpa rootm. The low fracture toughness was attributed to the high plastic constraint of matrix deformation by the Cr₂Nb and compositional change in the matrix

[15] ELECTROCHEMICAL LI-INSERTION PROCESSES INTO CARBONS PRODUCED BY MILLING GRAPHITIC POWDERS: THE IMPACT OF THE CARBONS' SURFACE CHEMISTRY

Aurbach D. Markovsky B. Nimberger A. Levi E. Gofer Y. - Journal of the Electrochemical Society. 149(2):A152-A161, Carbon powders were prepared by milling graphite particles in different atmospheres including air, highly pure argon, nitrogen, and CO₂. Part of the samples was further exposed to air after milling. The carbonaceous materials thus obtained were tested as Li-insertion anodes in nonaqueous Li salt solutions. It was found that the atmosphere in which the active mass was prepared had a pronounced impact on the specific surface area of the particles obtained, probably due to the effect of surface groups on the carbons developed in the milling process. It was also found that the atmosphere to which the powders were exposed had a remarkable impact on the electrochemical behavior of the carbon electrodes in Li-insertion processes. The milling of the graphite produces highly reactive carbon surface sites, which react readily with active gases such as oxygen and CO₂. The surface groups thus formed influence the surface chemistry developed on these electrodes in solutions. Since the electrochemical behavior of Li-C insertion electrodes depends strongly on the nature of the passivation films developed on these electrodes at low potentials, the milling atmosphere strongly influences their electrochemical behavior. The electrochemical processes were studied by chronopotentiometry, cyclic voltammetry, and impedance spectroscopy, the electrodes' structure and morphology were explored by X-ray diffraction and scanning electron microscopy, and the carbons' surface chemistry was studied by Fourier transform infrared and X-ray photoelectron spectroscopy.

[14] DISPERSION AND GRINDING OF OXIDE POWDERS INTO AN AQUEOUS SLURRY

Houivet D. El Fallah J. Haussonne JM. - Journal of the American Ceramic Society. 85(2):321-328, 2002

The present paper deals with a comprehensive analysis of the dispersion behavior of oxide powders to determine accurate conditions for mixing and grinding dense slurries without the use of steric dispersing agents. The experimental support of the work was the synthesis of (Zr,Sn)TiO₄ microwave dielectric ceramics by the solid-state reaction of raw materials mixed and ground by attrition milling. Zeta potential measurements were performed on the raw materials versus pH to determine the optimum pH of the slurry, allowing a good dispersion of all the species: the absolute value of the zeta potential of every powder >20 mV, with all potentials having the same sign. During the grinding process, as the surface of the materials increases due to the breakup of the grains, surface reaction occurs with the dispersion liquid, and pH must be continuously adjusted to be maintained at an adequate level. We have correlated these characterizations of the optimal processing conditions with the rheological behavior of the slurries, thus providing an analysis of the flocculated or deflocculated state. When applied to synthesizing (Zr,Sn)TiO₄ microwave dielectric ceramics, these conclusions made it possible to produce reproducible resonators with a k = 37 dielectric constant and characterized by a quality factor, Q x F > 60 000 GHz measured at 3 GHz, the highest value reported for this composition.

[13] ATOMIC DISORDER-ORDER PHASE TRANSFORMATION IN NANOCRYSTALLINE FE-AL

Sarkar S. Bansal C. - Journal of Alloys & Compounds. 334:135-142, 2002

The atomic disorder to order (A₂→DO₃) phase transformation was studied in nanocrystalline Fe-Al alloys prepared by mechanical alloying near Fe₃Al stoichiometry. Important differences in the microstructure evolution as well as the kinetics of the transformation were observed as compared to the corresponding coarse grained alloys prepared by splat quenching. These were understood in terms of the small grain sizes and the presence of grain boundary regions in the nanophase alloy systems.

[12] DEGRADATION KINETICS OF DISCHARGE CAPACITY FOR AMORPHOUS MG-NI ELECTRODE

Mu D. Hatano Y. Abe T. Watanabe K. - Journal of Alloys & Compounds. 334:232-237, 2002

Amorphous MgNi alloy was prepared by ball-milling of Mg₂Ni and Ni powders. Current density dependence of the discharge capacity was studied in the range from 5 to 250 mA/g for the MgNi electrode by charge-discharge cycle tests in 6 M KOH electrolyte using a conventional two-electrode system. The amount of hydrogen used for the discharge decreased with cycles. The extent of capacity degradation at a given cycle increased with increasing discharge current density. The capacity degradation curves could be expressed by $\phi(t) = \phi(\text{proportional to}) + (\phi(0) - \phi(\text{infinity})) \exp[-kt]$, where $\phi(t)$ is the discharge capacity at time t, that is the period of time in which the electrode was immersed in the electrolyte solution, $\phi(\text{infinity})$ the ideal discharge capacity expected for the virgin electrode, the final steady discharge capacity after a number of cycles, and k the apparent rate constant for the degradation. The rate constant increased linearly with the current density. According to diffusion analyses of discharge curves under different current densities, the hydrogen diffusion in the bulk is not the rate-determining step for the measured discharge rate, but surface processes play dominant roles in affecting the MgNi electrode properties

[11] MG-FETI_{1,2} (AMORPHOUS) COMPOSITE FOR HYDROGEN STORAGE

Wang P. Wang AM. Ding BZ. Hu ZQ. - Journal of Alloys & Compounds. 334:243-248, 2002

Amorphous FeTi_{1,2} was prepared from the powder mixture of elemental Fe and Ti by ball milling. The catalytic reaction ball milling method was adopted to prepare Mg-FeTi_{1,2} (amorphous) composite. The composite possesses rapid H-absorption rate, high H-capacity, low working temperature, as well as superior oxidation-resistance. During catalytic reaction ball milling and hydriding/dehydriding cycles, the phase stability of amorphous FeTi_{1,2} was examined. The favorable



hydrogenation performance is mainly attributed to the combined effects of the catalytic efficiency of amorphous FeTi_{1,2} and the nanostructure of Mg.

[10] FORMATION OF TiB₂/TiN/Ti (C_xN_{1-x}) NANOCOMPOSITE POWDER VIA HIGH-ENERGY BALL MILLING AND SUBSEQUENT HEAT TREATMENT

Li JL. Li F. Hu K. Zhou Y. - Journal of Alloys & Compounds. 334:253-260, 2002

TiB₂/TiN/Ti (C_xN_{1-x}) nanocomposite powder was fabricated by high-energy ball milling and subsequent heat treatment. The microstructure development of powder mixtures was monitored by X-ray diffraction and transmission electron microscopy. It was found that TiN and TiC formed within 10 h of milling. After 30 h of milling, the resulting powder mixtures were mainly composed of nanocrystalline Ti, TiN, TiC and TiB₂. The as-milled powder was transformed into Ti (C_xN_{1-x}), TiN and TiB₂ after subsequent heat was thus obtained. treatment at 1300degreesC. During annealing, TiC reacted with TiN, and Ti (C_xN_{1-x}) was thus obtained.

[9] CATALYTIC EFFECT OF GE ON HYDROGEN DESORPTION FROM MgH₂

Gennari FC. Castro FJ. Urretavizcaya G. Meyer G. - Journal of Alloys & Compounds. 334:277-284, 2002

We studied the influence of Ge on hydrogen desorption from MgH₂ produced by mechanical alloying at room temperature under a hydrogen atmosphere. The structural and morphological properties, and the desorption kinetics of the products were examined by X-ray diffraction, differential scanning calorimetry, thermal desorption spectroscopy and scanning electron microscopy. The mechanical milling of Mg-Ge mixtures under hydrogen leads to the formation of Mg₂Ge and MgH₂. The presence of Ge decreases the hydride decomposition temperature in a range from 50 to 150degreesC, depending on the Ge amount. On the contrary, Mg₂Ge does not show any effect on hydrogen desorption

[8] MODIFICATION OF THE MAGNETIC PROPERTIES OF SmCo₅ PARTICLES DEPENDING ON THE GRINDING ATMOSPHERE

Kahn ML. Bobet JL. Weill F. Chevalier B. - Journal of Alloys & Compounds. 334:285-292, 2002

Particles of SmCo₅ were synthesized and ground mechanically under two different gaseous atmospheres (Ar or H-2). The influence of the gaseous atmosphere on the crystallinity, the morphology of the particles and their magnetic properties has been studied. The major differences in terms of crystallinity and morphology are reached after 20 min of grinding. The sample ground under H-2 is still well crystallized whereas the homologous sample ground under Ar is almost amorphous. For longer grinding times, both the crystallinity and morphology are very close whatever the gaseous atmosphere. The sample is amorphous and consists of aggregates. However, for the sample ground under H-2 during 2 h, formation of SmH₂+6 and Co is observed. Values of the coercivity and saturation magnetization are discussed in regard to these characteristics.

[7] THE MECHANICALLY ACTIVATED COMBUSTION REACTION IN THE Fe-Si SYSTEM: IN SITU TIME-RESOLVED SYNCHROTRON INVESTIGATIONS

Gras C. Bernsten N. Bernard F. Gaffet E. - Intermetallics. 10(3):271-282, 2002

Mechanical high-energy ball milling of Fe + 2Si elemental powder mixtures was used to activate self Sustaining combustion reaction in the case of iron disilicide synthesis. The reaction path as well as the influence of the microstructural parameters on phase transformation have been investigated in detail. Time-resolved X-ray diffraction (TRXRD) using the fast recording kinetics offered by the synchrotron radiation was coupled to an infrared camera in order to study the internal structure of the combustion wave. The crystallite size and the amount of mechanically induced phases play an important role during the combustion; the reaction path and the end product composition mainly depend on the degree of mechanical activation (i.e. shock power and ball milling duration). beta-FeSi₂ is formed during a slow diffusion process in the post-combustion zone. The polyinterfaces created at a nanometric scale during the mechanical activation stage are responsible for this peculiar behaviour

[6] A COMPARATIVE STUDY ON THE MAGNETIC PROPERTIES OF ARC-MELTED AND BALL-MILLED Fe_{0.9}-XMn_{0.1}Al_x

Restrepo J. Alcazar GAP. Gonzalez JM. - Hyperfine Interactions. 134(1-4):27-35, 2001.

Samples of nominal composition Fe_{0.9}-xMn_{0.1}Al_x (0.1 less than or equal to x less than or equal to 0.5) were prepared both by mechanical alloying and arc-melting. In order to elucidate the effect of the synthesis method upon the magnetic properties of this system, we have carried out a comparative study involving the use of different experimental techniques (Mossbauer. X-ray diffraction, vibrating sample magnetometry and magnetic susceptibility). Results revealed that independently of the employed method and milling time, the samples exhibit ferromagnetism below similar to 34 at.% Al. Above this concentration, the preparation method became a determinant factor upon the magnetic properties of the system. The differences are attributed, in the case of the mechanically alloyed samples, to Fe contamination arising from jars material. The results of our study are summarized in a magnetic phase diagram including ferromagnetic, paramagnetic, pure spin glass and reentrant spin glass regions

[5] HIGH-ENERGY BALL MILLING OF SOME INTERMETALLICS

Principi G. - Hyperfine Interactions. 134(1-4):53-67, 2001.

High-energy ball milling of metallic powders has been used in recent years for the synthesis of alloys through reactions mainly occurring in solid state. The diffusive phenomena accompanying and promoting the reactions of formation are related to the microstructure acquired by the powders as a consequence of the intense mechanical deformations. The process induces a remarkable comminution of powder particles, inside of which domains of nanometric size are formed and compositional variations often occur. Several analytical techniques are suitable for following the structural evolution of the powders during



milling. Among them, Mossbauer spectroscopy is suitable for obtaining detailed local information on the atomic arrangement of the treated materials, if one of the constituents is a Mossbauer isotope, and for detecting little changes occurring at an atomic scale. For these reasons Mossbauer spectroscopy is more sensitive than other analytical techniques especially in the early stages of the process. Some recent results are presented regarding in particular the Fe-Cu, Fe-Al, Fe-Al-Cu, NiAl(Fe) and Fe-Mn systems

[4] MECHANOCHEMICAL REACTIONS IN Fe₂O₃-M (M : AL, TI)

Cuadrado-Laborde C. Damonte LC. Mendoza-Zelis L. - *Hyperfine Interactions*. 134(1-4):131-140, 2001.

The production of metal-ceramic nanodispersion by mechanical milling of powders through the displacement reaction $Fe_2O_3 + M \rightarrow Fe + M\text{-oxide}$ (with M: Al, Ti) was studied. The reaction progress with milling time was followed by recording the temperature and pressure during the process. The samples were characterized by X-ray diffraction and Mossbauer spectroscopy at the intermediate and final stages. In both cases self-sustained reactions were observed with different activation times. The results confirm that mechanical work at room temperature yields the reduction of hematite by Ti and Al. The final oxides were identified as Ti₂O₃ and Al₂O₃, respectively. The dependence of the intermediate and final stages on the milling conditions and the starting composition will be discussed

[3] MAGNETIC PROPERTIES OF THE MECHANICALLY ALLOYED (Fe_{0.85}Mn_{0.15})(0.3)Cu-0.7 SYSTEM

Restrepo J. Morales AL. Gonzalez JM. Alcazar GAP. Medina G. Barrero CA. Tobon J. Perez G. Arnache O. Betancur JD. Giraldo MA. - *Hyperfine Interactions*. 134(1-4):199-206, 2001.

Samples of nominal composition (Fe_{0.85}Mn_{0.15})(0.3)Cu-0.7 were prepared by mechanical milling starting from pure element powders. In order to elucidate the effect of the alloying time upon the magnetic properties of the system, milling times ranging from 1 hour up to 72 hours were considered. The phase distribution present on the as-milled materials was identified from the analysis of X-ray diffraction data, The room temperature magnetic properties of the samples were studied by means of Fe-57 Mossbauer spectroscopy and vibrating sample magnetometry, whereas their low temperature magnetic behavior was characterized through magnetic susceptibility measurements. The results evidenced a strong dependence of the magnetic properties on the milling time and, concretely, the occurrence of a superparamagnetic behavior in the long-time-milled samples for which an extended solid solution was obtained. This fact is attributable both to the obtained crystallite sizes, which resulted to be of the order of a few nanometers, and to a milling-driven increase of the lattice parameter

[2] FESiB AMORPHOUS ALLOY PREPARED BY MECHANO-SYNTHESIS

Marcatoma JQ. Rodriguez VAP. Baggio-Saitovitch EM. - *Hyperfine Interactions*. 134(1-4):207-212, 2001.

In this work we show that mechano-synthesis is a good method to prepare the FeSiB amorphous alloy starting from Fe, Si, B pure elements with relative atomic composition of 75, 15 and 10%, respectively. We have used a high-energy ball-milling system keeping the material under an Ar atmosphere. The evolution of the microstructure inside the grains is followed by X-ray diffraction and Mossbauer spectroscopy at room temperature. The dependence of crystalline size, lattice parameter and hyperfine parameters on milling time is discussed. Our results show that the milled samples obtained after 10 h of milling contained a bcc structure in the amorphous matrix. After 19 h of milling the sample became fully amorphous

[1] EFFECTS OF PRESSURE, TEMPERATURE AND GRAIN SIZE ON SYNTHESIS AND STRUCTURES OF Fe-N ALLOYS [CHINESE]

Liu L. Yao B. Zhao XD. Guo XY. Ning FL. Su WH. - *Chemical Journal of Chinese Universities-Chinese*. 23(1):6-9, 2002
Effects of pressure, temperature and grain size on the solid state reaction between alpha-Fe and amorphous BN and crystal structures of the resultant of the reaction were studied by using mechanical milling and high pressure techniques at the temperatures ranging from 580 to 930 K under pressures between 1×10^{-3} Pa and 4 GPa. It was found that the pressures and refinement of grain size can greatly accelerate the solid state reaction process. There is a critical grain size for a-Fe, below which the reaction begins to occur. In the present experiment, this grain size was measured to be about 8 nm. The crystal structures of the resultants change with the variation of pressure and temperature. A new Fe-N alloy with orthorhombic structure was produced firstly at 2 GPa and 800 K. Single phase epsilon-Fe_xN alloy can be obtained in the temperature range of 690 to 800 K at the pressures of 3 to 4 GPa. However, the resultant is not any Fe-N alloy but Fe₃B alloy when the pressure is 4 GPa and the temperature is above 930 K



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