RESEAU FRANÇAIS DE MECANOSYNTHESE

Lettre N°87

Juin 2002

188(+1) Groupes de Recherche

(dont 114(+1) à l'étranger / 34 Pays)

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1 Nnouvelle Adhésion

Dr. Jens-Uwe Wichmann Merck KgaA Pigments R&D - Germany

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IMPORTANT

Do Not Forget the Annual Subscription Fees
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Bulletin d'adhésion 2002 / Subcription Print

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

Eric GAFFET
CNRS UMR5060 « Métallurgies et Cultures »
Nanomatérials Research Group
Site de Sévenans (UTBM) - F90010 - Belfort Cedex - France

Nom/Name : .....Prénom / First Name : .....
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désire adhérer au Réseau Français de Mécanosynthèse / want to become a member of the French Mechanical Alloying Network
Chèque ci joint / Check enclosed in the amount of **20 Euros (20€)**
The check has to be to the order to : Reseau Francais de Mecanosynthèse

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Le site web du RFM est :
http://www.bls.fr/amatech
Rubrique Pages Sciences et Techniques pour l'Ingénieur (Rubrique Sciences)
⇒ vous y trouverez les anciennes lettres du RFM (accessible par Adobe Acrobat), les statuts du RFM ainsi que les annonces concernant les JRFM'2001 et quelques éléments mis a jour régulièrement concernant les derniers résultats dans ce domaine.

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

Press release (From Fritsch)

Planetary Ball Mills

Planetary mills are the effective laboratory mills for rapid batchwise ultra fine grinding down to colloidal fineness of hard to soft materials dry or in suspension. Mixing and perfect homogenising of emulsions and pastes as well for mechanical alloying.

QuickTime™ et un décompresseur
Photo - JPEG sont requis pour visualiser
cette image.

Typical fields of application:

Geology, Chemistry, Mineralogy, Ceramics Industry, Construction Industry, Metallurgy, Electrical Industry, Glass Industry, Nuclear Research, Soil Research, Biology, Pharmacology and Galenic Research, Medicine, Nuclear research, Technology, Spectroscopy, X-ray fluorescence.

Method of operation:

The Planetary ball mills work according to known and reliable planetary system. The sample is primarily crushed by the high-energy impact of grinding balls and in part by friction between the balls and the wall of the grinding bowl.

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

Features of performance:

- for brittle to hard solid samples
- grinding bowls in different sizes and materials
- feed quantity up to 1000 ml, max. feed particle size 10 mm
- simultaneous grinding of 1 to 8 samples in a single working process
- with RS232 interface for programming the grinding parameters, for validation, for instrument controlling, ...
- safe lock clamping system for the grinding bowls
- also available as hightec instrument with free selectable transmission ratios

5 different models with different feed quantities are available:

Vario-Planetary Mill “pulverisette 4”:
Max. feed particle size: <= 10 mm
Max. feed quantity: up to 2 x 225 ml
Final fineness: << 1 µm

Planetary Mill “pulverisette 5” (2 models):
Max. feed particle size: 10 mm
Max. feed quantity: Model I: up to 4 x 225 ml
Model II: up to 2 x 225 ml
Final fineness: < 1 µm

Planetary Mono Mill “pulverisette 6”:
Max. feed particle size: 10 mm
Max. feed quantity: up to 1 x 225 ml
Final fineness: < 1 µm

Planetary Micro Mill “pulverisette 7”:
Max. feed particle size: 5 mm
Max. feed quantity: up to 2 x 20 ml
Final fineness: < 1 µm

Please visit us
at MATÈRIAUX 2002 • 21. - 25. Octobre 2002
(http://www.materiaux2002.net/)
Vinci – Centre International de Congrès • Tours • France

contact: Fritsch GmbH
Manufacturers of Laboratory Instruments
Andrea Köhler - Industriestrasse 8 • D-55743 Idar-Oberstein
Phone 0 67 84/70-146 • Fax 0 67 84/70-11
E-Mail: koehler@fritsch.de • Internet: http://www.fritsch.de

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Congress and School Announcements

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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 ˆ 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
EIGTH 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

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ICAMP 2002
2nd International Conference on Advanced Materials Processing -
2nd to 4th December 2002 in Singapore.

You are invited to submit papers to the conference secretariat at
Denis Lam - ICAMP 2002 Secretariat
Integrated Meetings Specialist Pte Ltd
114 Middle Road - #05-02 Lee Kai House
Singapore 188971
E-mail: icamp@inmeet.com
Tel: (65) 6226 3069 - Fax: (65) 6226 3016

More detailed information can be found in ICAMP web site
http://www.eng.nus.edu.sg/PACentre/icamp

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Cooperative Research on Related Areas
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France (12/04/2001)

Le portail Internet "France Contact" a été lancé: ce portail s'adresse aux chercheurs etrangers 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 sejour 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

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

Proposal from 13/05/2002

POSTDOCTORAL RESEARCH FELLOW

Department of Materials and Process Engineering - School of Science and Technology - (Fixed term for 2 years)

We are seeking a postdoctoral research fellow for a 2-year fixed term contract, starting July 2002. The position involves research in processing, consolidating and developing titanium-based metallic and metal-ceramic composite powder materials, including titanium based metal-ceramic nanocomposites. The appointee will also assist the project leader in project planning and postgraduate supervision.

The candidate should either have a PhD degree in materials science and engineering, or expect to have completed a PhD by the time of appointment. Previous research experience and a strong publication track record in titanium-based metallic materials, mechanical alloying or powder metallurgy will be an advantage.

The position is funded by a New Economy Research Fund (NERF) grant from the Foundation for Research, Science and Technology.

The Department of Materials and Process Engineering hosts the Waikato Centre for Advanced Materials (WaiCAM) and the website for the department is http://mape.waikato.ac.nz

The salary range is NZ$46,564 - $52,666 per year.

Applications close on Friday, 7 June 2002.

Further information and an application form are available from; www.waikato.ac.nz/hrm/ or the Human Resource Management Division, The University of Waikato, Private Bag 3105, Hamilton, phone 64-7-8384003, fax 64-7-8560135, email; hrm@waikato.ac.nz.

The University is committed to providing equal opportunities for all.

Proposal from 30/04/2002

Queen's University - Department of Mechanical Engineering

Post Doctoral Research Position in - Nuclear Materials

The Department of Mechanical Engineering, Queen's University, invites applications for a post-doctoral research position to work in the NSERC Industrial Research Chair program in Nuclear Materials under the direction of the Chair-holder, Prof. Rick Holt. The chair program is co-funded by OPG Inc., COG Inc. and Nu-Tech Precision Metals Inc.

The purpose of the Chair is to further the fundamental understanding of nuclear materials and to develop the applied technology required by industry in this area. This includes radiation-induced deformation of nuclear materials (especially zirconium alloys), its relationship to manufacturing variables, crystallographic texture and microstructure, and other related topics.

Candidates must have completed a Ph.D. in metallurgical engineering, metallurgy, materials science or a related field.

The successful candidate will be an experienced electron-microscopist with expertise in TEM (specimen preparation techniques, bright field, dark field and weak beam imaging, EDX, CBED and SADP analysis). Expertise with SEM, especially orientation analysis using electron back scattering would be an asset.

The successful candidate will investigate the microstructure and microtexture of zirconium alloys using electron microscopy, and will assist with the supervision of post graduate students working in this area. Publication of the results is an expected outcome.

Applicants should send their resumés to:

Prof. R.A. Holt
Materials Engineering Group
Department of Mechanical Engineering
Nicol Hall
Queen's University
Kingston ON K7L 3N6
holt@me.queensu.ca

Proposal from 24/04/2002

POST-DOCTORAL RESEARCHER
Nanoelectronics at the Quantum Edge
Postdoctoral Research Assistant in Scanning Probe Microscopy and Related Techniques
Grade RAIA / Salary: £17,626 - £26,491 / Job Ref: DJ02/023

Oxford and Cambridge Universities are working together with Hitachi Europe Ltd to produce radically new devices for future computing, in a project jointly funded by a Foresight Link Award from the Department of Trade and Industry and Hitachi

Europe Ltd. The project brings together research in physics, chemistry, materials science and electronics engineering to make prototype structures for advanced conventional computing and for the new field of quantum computing. See www.nanotech.org. Applications are invited for a postdoctoral position in scanning probe microscopy and related techniques, for atomic and electronic structural characterization of endohedral fullerenes and single walled carbon nanotubes. This is available until September 2004.
Candidates should have a good first degree and completed PhD or equivalent in a relevant physical science, a track record of innovative and effective experimental research using atomic resolution scanning probe microscopy, and evidence of quality publications in international peer reviewed journals and presentations at national and international seminars and conferences. They should have proven skills in maintaining a fundamental academic and research overview of the structural characterization of nanomaterials. Flexibility, excellent verbal and written communication skills in English, and the ability to work independently and in a team within an agreed time-scale are essential.
Before submitting an application, candidates should obtain further particulars available from The Deputy Administrator (Teaching), Department of Materials, University of Oxford, Parks Road, Oxford OX1 3PH (email: posts@materials.ox.ac.uk), or telephone 01865 273750 quoting reference: DJ02/023. The closing date for applications is 31 May 2002 and interviews are currently planned for the week beginning 17 June. Further information on the Department may be found on the web-site: http://www.materials.ox.ac.uk
See also http://www.ox.ac.uk/staff.html

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

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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 structuretheory, 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.
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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/rita1.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.

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Périodiques

[52] REACTIVE MILLING WITH THE SIMOLOYER (R): ENVIRONMENTALLY BENIGN QUANTITATIVE REACTIONS WITHOUT SOLVENTS AND WASTES

Kaupp G. Schmeyers J. Naimi-Jamal MR. Zoz H. Ren H. - Chemical Engineering Science. 57(5):763-765, 2002

The pilot set-up of a horizontal high energy ball mill (Simoloyer(18)) that has been successful in reactive milling, mechanical alloying, and formation of ductile metal-flakes is used for semi-continuous processing of organic solid-solid reactions which proceed rapidly and without wastes in the absence of solvents. 200 g batches were run in the exploratory investigation. Stoichiometric 1:1-mixtures of alpha-(D)-glucose and urea formed the 1:1-complex with quantitative yield after 5 min at a rotor frequency of 900 min(-1). Similarly, the condensation reaction of solid p-hydroxybenzaldehyde with solid p-aminobenzoic acid was complete after 15 min yielding the pure imine derivative in quantitative yield without waste producing work-up procedures. Further scale-up appears possible also with other solid-solid reactions.

[51] DRY FINE-MILLING AND CLASSIFICATION OF CERAMICS WITH PRACTICAL EXAMPLES OF APPLICATIONS

Maindok A. - Cfi, Ceramic Forum International/Berichte der Dkg (Deutsche Keramische Gesellschaft). 79(3):E22-+, 2002

[50] ATOMIC-LEVEL OBSERVATION OF DISCLINATION DIPOLES IN MECHANICALLY MILLED, NANOCRYSTALLINE FE

Murayama M. Howe JM. Hidaka H. Takaki S. - Science. 295(5564):2433-2435, 2002

Plastic deformation of materials occurs by the motion of defects known as dislocations and disclinations. High-resolution transmission electron microscopy was used to directly reveal the individual dislocations that constitute partial disclination dipoles in nanocrystalline, body-centered cubic iron that had undergone severe plastic deformation by mechanical milling, The mechanisms by which the formation and migration of such partial disclination dipoles during deformation allow crystalline solids to fragment and rotate at the nanometer level are described. Such rearrangements are important basic phenomena that occur during material deformation, and hence, they may be critical in the formation of nanocrystalline metals by mechanical milling and other deformation processes

[49] MOSSBAUER STUDY OF BALL-MILLED FE-GE ALLOYS

Cabrera AF. Sanchez FH. - Physical Review B. 6509(9):4202, 2002

In this work the mechanical alloying of Fe100-xGex was studied as a function of alloying composition (9 less than or equal toxless than or equal to40) processing time and annealing treatment. The alloys were prepared using a high-energy vibratory ball mill. For 9less than or equal toxless than or equal to27.5 a single phase A2 solid solution was formed while for xgreater than or equal to30 besides the solid solution an increasing amount of disordered B8(1) (NiAs type) Fe3Ge2 was found. Thermally induced A2-->D0(3) transition was investigated for 9less than or equal toxless than or equal to27.5. The detailed annealing temperature dependence of short- and long-range ordering into the D0(3) structure was studied for the case of Fe75Ge25 by means of Mossbauer-effect spectroscopy and x-ray diffraction. The solid solution orders gradually and homogenously into the D0(3) structure. To follow and quantify the evolution of short-range order a special fitting routine for Mossbauer spectra was used that takes into account local and nonlocal contributions to the hyperfine interactions experienced by a Fe-57 probe, and that assumes nonlinearity dependences on the number of neighbors of a given class, By this means the short-range D0(3) order parameter S was determined for as milled steady states as well as annealed states for 9less than or equal toxless than or equal to27.5. In the case of Fe75Ge25 the evolution of mechanical alloying with time was investigated. It was found that alloying proceeds in two stages. In the first one, iron rich (with A2 structure) and disordered Ge-rich regions coexist along with an almost equiatomic A(2) Fe-Ge solid solution, which was interpreted as an interphase physically located between the former two. The three phases collapse into just one bee solid solution with Sapproximate to0.3 for a critical time t(c), which was identified as the chemical mixing time reported by other authors. The second stage of mechanical alloying produces the complete homogenization of the solid solution. Plausible basic mechanisms of mechanical alloying of Fe-Ge mixtures are discussed on the basis of the results presented here

[48] FORMATION OF TIB2/TIN NANOCOMPOSITE POWDER BY HIGH ENERGY BALL MILLING AND SUBSEQUENT HEAT TREATMENT

Li JL. Hu K. Zhou Y. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 326(2):270-275, 2002

TiB2/TiN nanocomposite powder was fabricated by high energy ball milling and subsequent heat treatment. The microstructure development of the powder mixtures was monitored by X-ray diffraction, differential scanning calorimeter, scanning electron microscopy and transmission electron microscopy. It was found that TiN formed within 30 h of milling. After 40 h of milling, the resulting powder mixture was composed of a small amount of nanosized TiN particles, nanocrystalline Ti and amorphous phase. The nanocrystalline Ti and amorphous phase were transformed into TiN and TiB2 after subsequent heat treatment.

[47] THERMAL STUDIES AND MAGNETIC PROPERTIES OF MECHANICAL ALLOYED FE2B

Passamani EC. Tagarro JRB. Larica C. Fernandes AAR. - Journal of Physics-Condensed Matter. 14(8):1975-1983, 2002

In the present paper, a high energy milling process has been used to alloy Fe and B in the proportion 2:1. X-ray diffraction and Mossbauer spectroscopy were used to follow the solid state reaction among the alloy components and phase formation during thermal treatments of the final milled alloy, at distinct temperatures and environments. X-ray, DSC and Mossbauer data of the 310 h milled alloy show the presence of three phases: small fractions of alpha-Fe (n-Fe) and Fe2B (n-Fe2B) nanocrystalline phases and a large amount of amorphous Fe2B (a-Fe2B) phase (73%). Heat treatment of the milled alloy was done in a high vacuum furnace, followed by in situ Mossbauer measurements, or by annealing, the sample sealed in a quartz

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

tube. It is shown that segregation and crystallization effects thermally induced in the samples have been enhanced by the presence of oxygen in the residual atmosphere, being less effective in the case where the sample was continuously pumped during the annealing. These effects may have occurred due to boron oxidation. Also, the presence of a tet-Fe3B phase in the annealed samples has been observed. The high temperature Mossbauer spectra for the sample annealed at 823 K indicate that the n-Fe2B, n-Fe3B and n-Fe phases exhibit superparamagnetic behaviour, with estimated blocking, temperatures of 723 and 823 K, respectively. The amorphous a-Fe2B phase has a T-C value estimated at about 823 K that fits into the system reported for amorphous Fe/B ribbons.

[46] LOCAL STRUCTURAL ORDERS IN NANOSTRUCTURED AL2O3 PREPARED BY HIGH-ENERGY BALL MILLING

Scholz G. Stosser R. Klein J. Silly G. Buzare JY. Laligant Y. Ziemer B - Journal of Physics-Condensed Matter. 14(8):2101-2117, 2002

Nanostructured Al2O3 powders were prepared by high-energy ball milling of corundum. Both the solid state nuclear magnetic resonance spectra of the Al3+ ions and the solid state electron paramagnetic resonance spectra of incorporated Fe3+ ions are governed by noticeable spectral changes dependent on the duration of the mechanical treatment. The quadrupolar parameters of the Al-27 nuclei and the zero-field splitting parameters of the Fe3+ ions as well as their statistical distributions were determined as functions of the milling time. Structural changes of the Al2O3 matrix were also followed by powder x-ray diffraction and transmission electron microscopy measurements. Direct relations between the structural disorder as obtained by x-ray data and the spin Hamiltonian parameters of both ions could be established. These results suggest that the milled powders consist of nanocrystalline grains embedded in amorphous grain boundaries even for the longest milling time. The grains can be described in terms of ordered AlO6-octahedra as in the starting crystalline material exhibiting a slight rhombic distortion. The,ram boundaries look like random arrangements of these octahedral units. The specific behaviour of the environment of the Fe3+ paramagnetic probe points out that such a point defect acts as an activation centre of the amorphization process.

[45] PREPARATION OF SILVER ION CONDUCTING AMORPHOUS MATERIALS IN THE SYSTEM AG2S-SIS2 BY MECHANICAL MILLING PROCESSES

Peng HF. Machida N. Shigematsu T. - Journal of Materials Chemistry. 12(4):1094-1098, 2002.

Silver ion conducting amorphous materials in the system Ag2S-SiS2 were synthesized over the composition range from 0 to 60 mol% Ag2S by use of a high-energy ball-milling process. The electrical and electrochemical properties of the obtained samples were evaluated by using ac impedance spectra, dc polarization measurements, and cyclic voltammetry. These measurements suggested that the silver-ion conductivities of the samples increased with an increase in the Ag2S content and that the activation energy for conduction decreased with an increase in the Ag2S content. The 60Ag(2)S.40SiS(2) (mol%) sample showed its highest ion conductivity, 6.9 x 10(-2) S m(-1), at 298 K in the system Ag2S-SiS2. This sample also possessed a wide electrochemical window over 2.0 V vs. Ag/Ag+.

[44] THE SONOCHEMICAL PREPARATION OF TUNGSTEN OXIDE NANOPARTICLES

Koltypin Y. Nikitenko SI. Gedanken A. - Journal of Materials Chemistry. 12(4):1107-1110, 2002.

Amorphous tungsten oxide has been prepared by ultrasound irradiation of a solution of tungsten hexacarbonyl W(CO)(6) in diphenylmethane (DPhM) in the presence of an Ar (80%)-O-2 (20%) gaseous mixture at 90 degreesC. Heating this amorphous powder at 550 degreesC under Ar yields snowflake-like dendritic particles consisting of a mixture of monoclinic and orthorhombic WO2 crystals. Annealing of the as-prepared product in Ar at 1000 degreesC causes the formation of a WO2-WO3 mixture containing nanorods (around 50 nm in diameter) and packs of these nanorods. Heating the product in air for 3 hours leads to triclinic WO3 crystal formation, with a basic size of 50-70 nm. The prepared oxides have been characterized by elemental analysis, X-ray powder diffraction measurements, FTIR spectroscopy, differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), transmission electron microscopy (TEM), scanning electron microscopy (SEM), and energy dispersive X-ray analysis (EDX).

[43] CHARACTERIZATION OF MECHANICALLY ATTRITED SI/SIOX NANOPARTICLES AND THEIR SELF-ASSEMBLED COMPOSITE FILMS

Phely-Bobin T. Chattopadhyay D. Papadimitrakopoulos F. - Chemistry of Materials. 14(3):1030-1036, 2002

The surface chemistry and structure of nanosized silicon, as obtained by high-energy ball milling and its subsequent dispersion in ethanol, via sonication-assisted oxidation, is presently described. These Si/SiOx suspensions that spontaneously assemble on glass surfaces were investigated as a function of pH in a variety of oxidizing agents and atmospheres. Spectroscopic characterization indicates that at pH 5 or below and in the absence of H2O, the Si-OEt groups present on the surface of these nanoparticles get protonated, providing the driving force to assemble on less positively charged glass substrates. X-ray photoelectron spectroscopy concurs with a core/shell (Si/SiOx) nanoparticle structure, with an average Si-atom ratio of 45/55 and x in the order of 1.5. In addition, Raman investigation suggests that the Si core in these nanoparticles (45% of the total Si-atom content) is composed of crystalline (34%) and amorphous (11%) parts.

[42] ELECTROCHEMICAL PROPERTIES OF RUTHENIUM-BASED NANOCRYSTALLINE MATERIALS AS ELECTRODES FOR SUPERCAPACITORS

Soudan P. Gaudet J. Guay D. Belanger D. Schulz R. - Chemistry of Materials. 14(3):1210-1215, 2002

Nanocrystalline TixFeyRuzOn materials were prepared by mechanical alloying using high-energy ball milling. The electrochemical properties of the materials were investigated in 1 M NaOH and 1 M H2SO4 aqueous solutions, using a composite electrode technology. The tested materials fall in two categories. On one hand, when the O/Ti ratio is larger than 1, Ru atoms are found in an hexagonal phase. Upon cycling in H2SO4 or NaOH, these materials exhibit a significant

increase of their capacitance from similar to5 to similar to50 F/g. This is due to the progressive growth of a ruthenium oxide layer at the surface. On the other hand, when the O/Ti ratio is smaller than 1, Ru atoms are found in a cubic phase (CsCl), along with Ti and Fe atoms. In that case, the growth of a stable oxide phase at the surface of the material occurs only when it is cycled in basic electrolyte. The maximum attainable capacitance is also close to 50-60 F/g. The individual crystallites of as-milled nanocrystalline materials suffer from a strong tendency to agglomerate together. For example, it is shown that the electrochemically active surface area of nanocrystalline RuO2 is only increased by a factor of 2 when the crystallite size is decreased from 600 to 15 nm, which amounts to a 40-fold increase of the specific surface area. Thus, higher surface area materials were obtained by performing an additional milling step with Al, which is followed by a subsequent leaching of Al with a NaOH solution. With that procedure, the best performances were obtained with leached Ti2FeRuO2, with a maximum capacitance of 110 F/g.

[41] SOLVENT-FREE MECHANOCHEMICAL SYNTHESIS OF PHOSPHONIUM SALTS

Balema VP. Wiench JW. Pruski M. Pecharsky VK. - Chemical Communications. (7):724-725, 2002.

Phosphonium salts have been prepared during high- energy ball- milling of triphenylphosphine with solid organic bromides; the reactions occur at ambient conditions without a solvent; in the case of 2- bromo- 2- phenylacetophenone the reaction in a solution usually produces a mixture containing both the C- phosphorylated and O- phosphorylated compounds, while the solvent- free mechanically induced transformation results in the thermodynamically favorable C-phosphorylated product; the occurrence of the observed transformations during mechanical processing of solid reactants is confirmed by the solid-state P-31 NMR spectroscopy and X- ray powder diffraction.

[40] MICROSTRUCTURE AND MECHANICAL PROPERTIES OF MOSI2-X (X = AL, B, NB) ALLOYS FABRICATED BY MA-PDS PROCESS

Shan AD. Fang W. Hashimoto H. Park YH. - Materials Transactions. 43(1):5-10, 2002

In order to improve the mechanical proper-ties Of MoSi2 alloys, alloys with Al, B or Nb addition were prepared by an advanced consolidation process that combined mechanical alloying with pulse discharge sintering (MA-PDS). Their microstructure and mechanical properties were investigated. The microstructure of MoSi2 alloys fabricated by the MA-PDS process was much finer than that of the sample sintered from the commercial MoSi2 powders. Alloys made from powders milled in Ar gas had fewer silica or alumina phases as compared to their counterparts sintered from powders milled in air, presumably because of the suppression of the oxidation process during milling in At gas. Both Vickers hardness and tensile tests indicated that the alloys fabricated by the MA-PDS process showed better performance than the sample sintered from commercial MoSi2 powders because of the finer grain sizes. The Al-added alloy sintered from the powders milled in air gave the best mechanical properties due to the suppression Of SiO2 and formation of fine Al2O3 particles

[39] SYNTHESIS AND THERMOELECTRIC PERFORMANCE OF (BI0.25SB0.75)(2)TE-3 POLYCRYSTALS DISPERSED WITH AG AND BN

Liu XD. Park YH. - Materials Transactions. 43(1):30-35, 2002

Mechanical allowing followed by pulse-discharge sintering (NIA-PDS) is used to fabricate bulk (Bi0.25Sb0.75)(2)Te-3 polycrystals dispersed with metal Ag, ceramic BN or both. During sintering, the dispersed second phase particles, especially BN, effectively hinder the growth of the (Bi. Sb)(2) Te-3 matrix phase, giving rise to a refined microstructure in compacts. Property measurements demonstrate that the metal Ag is effective in raising, electrical conductivity. while the ceramic BN is effective in decreasing thermal conductivity through reinforced phonon scattering. B adding Ag alone, we have identified a large potential for further improving the room-temperature figure of merit of the (Bi0.25Sb0.75)(2)Te-3 mother allot. The maximum figure of merit appears at 0.02mass% Ag with value in the order of 3.4 x 10(-3) K-1, which is 18% higher than that of the mother alloy. This result is of practical significance. Because of the deterioration of Seebeck coefficient, addition of BN either alone or in combination with Ag does not produce a favorable figure of merit

[38] LITHIUM STORAGE PROPERTIES OF BALL MILLED NI-57 MASS% SN ALLOY

Ahn JH. Kim YJ. Wang GX. Lindsay M. Liu HK. Dou SX. - Materials Transactions. 43(1):63-66, 2002

Ni-57 mass%Sn alloy has been examined as a new anode material for Li-ion batteries. Li ions can reversibly intercalate and de-intercalate in this alloy. Ball milled nanocrystalline and subsequently annealed microcrystalline Ni-57 mass%Sn alloy showed very high initial discharge capacity. The cell capacity decayed rapidly after the first discharge. The capacity of the ball milled Ni-57 mass%Sn alloy faded continuously on cycling, while the annealed alloy exhibited good cyclic properties. Therefore, Ni-57 mass%Sn alloy is an attractive intercalation host for Li.

[37] SYNTHESIS OF FERRITE NANOPARTICLES BY MECHANOCHEMICAL PROCESSING USING A BALL MILL

Todaka Y. Nakamura M. Hattori S. Tsuchiya K. Umemoto M. - Journal of the Japan Institute of Metals. 66(1):34-39, 2002

Ferrites with the spinel structure have a wide field of technological applications. In the present study, various ferrite (Fe, Co and Ni-Zn) nanoparticles were synthesized by mechanochemical reaction in aqueous solution of various chlorides (FeCl3, CoCl2 and NiCl2/ZnCl2) and NaOH in a horizontal ball mill. Structures, morphologies, compositions and magnetic properties of the synthesized nanoparticles were investigated using X-ray diffraction (XRD), analytical high-resolution transmission electron microscope (HRTEM) and vibrating sample magnetometer (VSM). It was revealed that the size and morphology of ferrite nanoparticles can be controlled by milling conditions, such as, milling time and pH value (R value) of starting solution. The particle sizes of Fe3O4/-Fe2O3 milled for 72 h and 120 h at R = 1 were 30 nm and 20 nm in diameter, respectively. Meanwhile the particles milled for 72 h at R = 0.5 had a particle size of 100 nm and a coercivity of 10.8 kA/m. The particle sizes of CoFe2O4 milled for 72 h at R = I and Ni0.5Zn0.5Fe2O4 milled for 96 h at R = 1 were about 30 nm. The

magnetization values of CoFe2O4 and Ni0.5Zn0.5Fe2O4 were about 55 muWb (.) m/kg, with coercivity values of 43 kA/m and 5.3 kA/m, respectively

[36] ESTIMATION OF MECHANOCHEMICAL DECHLORINATION RATE OF POLY(VINYL CHLORIDE)

Mio H. Saeki S. Kano J. Saito F. - Environmental Science & Technology. 36(6):1344-1348, 2002

Poly(vinyl chloride) (PVC) was ground in air with CaO in the presence of quartz powder as a grinding aid by a small-scale planetary ball mill to investigate the relation of the dechlorination rate of PVC with the impact energy of the balls calculated from a computer simulation based on the Discrete Element Method under various conditions. Mechanochemical dechlorination proceeds as the grinding progresses and is improved with an increase in both the mill speed and the amount of balls introduced into the mill. The same trend can be seen in the relation between the specific normal impact energy of the balls and the rotational speed. The relationship between the observed dechlorination rate and the computed normal impact energy of the balls is linear, with a correlation coefficient of 0.965. This relationship can be used to estimate the dechlorination rate of PVC in a large-scale planetary ball mill

[35] PEROVSKITE-TYPE OXIDES SYNTHESIZED BY REACTIVE GRINDING PART 11: CATALYTIC PROPERTIES OF LACO((1-X))FEXO3 IN VOC OXIDATION

Szabo V. Bassir M. Van Neste A. Kaliaguine S. - Applied Catalysis B-Environmental. 37(2):175-180, 2002

The catalytic properties in n-hexane total oxidation of perovskite type mixed oxides, LaCo(1-x)FexO3 prepared by a new technique designated as reactive grinding, are investigated. All catalysts were found significantly more active than a reference sample prepared by conventional synthesis using amorphous citrate complexes. The activity per unit surface area was found to depend on grinding conditions and calcination temperature. These enhanced activities are associated with both the high surface area and high defect density reached by the reactive grinding synthesis method. Cobalt rich perovskites were shown more active per unit surface area than iron perovskites calcined at the same temperature

[34] FORMATION MECHANISM OF NANOCRYSTALLINE FERRITE DURING BALL MILLING PURE FE [CHINESE]

Yin J. Zhou CR. Wang JQ. Hu ZQ. Umemoto M. Liu ZG. Tsuchiya K. - Chin Shu Hsueh Pao: Acta Metallurgica Sinica. 38(2):113-118, 2002

Nanocrystalline ferrite formation during ball milling pure Fe (0.004%C) has been studied through morphology observation and microhardness measurements. It was found that layer-like structure consisting of nano ferrite firstly formed at the outer surface area of the powder particle at the early stage of ball milling. The microstructures between nano-ferrite and work-hardened regions are different and the boundaries between them are quite clear under SEM and TEM. The hardness of nano-ferrite region is obviously higher than that of the work-hardened region. Further milling led to the particle refinement and formation of nano-ferrite. It has been suggested that the nano crystalline ferrite formed through a transition from dislocation cell wall created by work hardening during ball milling to grain boundary, which is regarded to contribute to the hardness gap between the work hardened structure and the nanocrystalline ferrite

[33] ENHANCEMENT OF ELECTROCHEMICAL CHARACTERISTICS OF MG2NI/MMNI(5-X)(COALMN)(X) BY FORMING NANO-PHASE [CHINESE]

Wang ZM. Peng CH. Ouyang LZ. Li ZX. Zhu M. - Chin Shu Hsueh Pao: Acta Metallurgica Sinica. 38(2):189-192,

Hydrogen storage composite was prepared by high energy ball milling of the mixture powder Of Mg2Ni and MnNi5-x(CoAlMn)x alloys and subsequently wrapping with electroless plating. XRD and SEM analysis show that Mg2Ni/MmNi5-x(CoAlMn)x nano-phase composite has a morphology of particles composed of aggregate of constituent phases. The electrode properties Of Mg2Ni alloy, MmNi5-x(CoAlMn)x alloy and nano-phase composite with different grain sizes were tested by the simulated battery test. It was found that the discharge capacity Of Mg2Ni alloy was obviously enhanced with increasing milling time, but vice verse for MmNi5-x(CoAlMn)x alloy. As to the nanophase composite, its discharge capacity was not simply the linear sum of the capacity of components constituted the composites. An enhancement effect on capacity has been observed in the nano-phase composite when the size of the phase inside composite is refined to less than 100 nm.

[32] MECHANOCHEMICAL SYNTHESIS OF A NOVEL C-60 DIMER CONNECTED BY A SILICON BRIDGE AND A SINGLE BOND

Fujiwara K. Komatsu K. - Organic Letters. 4(6):1039-1041, 2002

A novel C-50 dimer connected by a silicon bridge and a single bond was synthesized by the mechanochemical solid-state reaction employing a high-speed vibration milling technique and fully characterized by the H-1 and C-13 NMR, APCI mass, and UV-vis spectroscopy. The presence of the electronic interaction between the two C-60 cages was demonstrated by the electrochemical method

[31] SEQUENTIAL COMBINATION OF CONSTITUENT OXIDES IN THE SYNTHESIS OF PB(FE1/2NB1/2)O-3 BY MECHANICAL ACTIVATION

Gao XS. Xue JM. Wang J. Yu T. Shen ZX. - Journal of the American Ceramic Society. 85(3):565-572, 2002

Pb(Fe1/2Nb1/2)O-3 (PFN) has been successfully synthesized via a novel mechanical activation of mixed oxides and columbite precursor consisting of lead oxide and FeNbO4. A nanocrystalline perovskite phase 5-15 nm in crystallite size was formed after 30 h of mechanical activation at room temperature for both types of starting materials. However, the nanocrystalline PFN phase derived from the mixed oxides of PbO, Fe2O3, and Nb2O5 is unstable, and develops pyrochlore phases when calcined at 500degrees-900degreesC, while no pyrochlore phase is observed for the material derived from the columbite precursor consisting of PbO and FeNbO5. Different sintering behavior and dielectric properties were also observed between the two types of PFN. These differences are accounted for by the compositional inhomogeneity in the material derived from the mixed oxides, as was revealed by Raman spectroscopic studies. This suggests that mechanical activation is

analogous to thermal activation, where the phase development is strongly dependent on the sequence of combining the constituent oxides

[30] DEPENDENCE OF SILICON CARBIDE PRODUCT MORPHOLOGY ON THE DEGREE OF MECHANICAL ACTIVATION

Shaw LL. Yang ZG. Ren RM. - Journal of the American Ceramic Society. 85(3):709-711, 2002

Mechanical activation before carbothermic reduction can substantially enhance the formation of SiC from SiO2 and carbon mixtures. However, the morphology (e.g., particles or whiskers) of SiC formed from mechanically activated SiO2 and carbon mixtures is dependent of the degree of mechanical activation and the condition of the subsequent carbothermic reduction. These phenomena are investigated and rationalized based on the increased reactivity of the reactants and SiC formation mechanisms.

[29] TRANSPARENT PLZT8/65/35 CERAMICS FROM CONSTITUENT OXIDES MECHANICALLY MODIFIED BY HIGH-ENERGY BALL MILLING

Kong LB. Ma J. Zhu W. Tan OK. - Journal of Materials Science Letters. 21(3):197-199, 2002

[28] DILATOMETRIC ANALYSIS ON THE SINTERING BEHAVIOR OF NANOCRYSTALLINE W-CU PREPARED BY MECHANICAL ALLOYING

Ryu SS. Kim YD. Moon IH. - Journal of Alloys & Compounds. 335(1-2):233-240, 2002

Unlike conventional W-Cu powder, the sintering of nanocrystalline W-Cu powder was significantly enhanced at solid phase sintering temperature. In the present study, in order to clarify this enhanced sintering behavior of nanocrystalline W-Cu powder prepared by mechanical alloying, the sintering behavior during heating stage was analyzed by dilatometry with various heating rates. The sintering of the nanocrystalline W-Cu powder was characterized by the densification of two stages, having two peaks in shrinkage rate curves. The temperature at which the first peak appeared was well below the Cu melting point and significantly dependent on the heating rate. On the basis of dilatometric data and microstructural observation, the possible mechanism for the enhanced sintering of the nanocrystalline W-Cu powder in the solid phase was attributable to the coupling effect of the sintering occurring inside as-milled powder and the sintering between powder particles.

[27] STRUCTURAL AND ELECTROCHEMICAL CHARACTERIZATION OF FLUORINATED AB(2)-TYPE LAVES PHASE ALLOYS OBTAINED BY DIFFERENT PULVERIZATION METHODS

Higuchi E. Li ZP. Suda S. Nohara S. Inoue H. Iwakura C. - Journal of Alloys & Compounds. 335(1-2):241-245, 2002

In order to enhance the effect of fluorination treatment (F-treatment) on charge-discharge characteristics of AB(2)-type Laves phase alloys, three pulverization methods were applied for the F-treated alloys; e.g. hydriding-dehydriding, dry ball-milling and wet ball-milling. As a result, it was found that the F-treated alloy powder obtained by the wet ball-milling had smaller particle size and larger specific surface area than those obtained by the other two methods. The F-treated alloy electrode prepared from the alloy powder obtained by the wet ball-milling showed good initial activation, high-rate discharge ability and relatively high discharge capacity. These results indicate that the wet ball-milling is a very effective pulverization method in enhancing the effect of the F-treatment

[26] THE RELATIONSHIP BETWEEN THE HIGH-RATE DISCHARGEABILITY AND THE DIFFUSION COEFFICIENT AND EXCHANGE CURRENT FOR TI0.5NI0.25AL0.25 METAL HYDRIDE ALLOYS

Xu YH. Chen CP. Wang XL. Wang QD. - Journal of Alloys & Compounds. 335(1-2):262-265, 2002

Commonly, the high-rate dischargeability (HRD) of metal hydride electrodes is related to its dynamic behavior. i.e. the exchange current and diffusion coefficient are two main factors influencing HRD. The effect of ball milling with or without Ni powders on the discharge capability of Ti0.5Ni0.25Al0.25 metal hydride electrodes at large currents was investigated and the dependence of HRD on the apparent diffusion coefficient and apparent exchange current was discussed. We considered that the nature of the improvement of HRD is to increase the electro-catalytic activity and to accelerate the diffusion rate of hydrogen in alloy.

[25] EFFECTS OF MODIFIED FLUORINATION TREATMENT ON STRUCTURAL AND ELECTROCHEMICAL CHARACTERISTICS OF AB(2)-TYPE LAVES PHASE ALLOY

Higuchi E. Hidaka K. Li ZP. Suda S. Nohara S. Inoue H. Iwakura C. - Journal of Alloys & Compounds. 335(1-2):277-280, 2002

Effects of a modified fluorination treatment (F-treatment), ball-milling in. a fluoride solution, on structural and electrochemical characteristics of Zr0.9Ti0.1Mn0.6V0.2Co0.1Ni1.1 alloy were investigated. As a result, it was found that the modified F-treatment improved charge-discharge characteristics such as the initial activation property, discharge capacity and high-rate dischargeability of the alloy. It was also found that the modified F-treatment could effectively deposit Ni species on the alloy surface, compared with the conventional F-treatment. These results suggest that the combination of F-treatment and ball-milling is very effective in forming a Ni-rich surface layer with catalytic activity, leading to improved charge-discharge characteristics.

[24] PHASE FORMATION AND THERMAL STABILITY OF (ZR1-XTIX)O-2 SOLID SOLUTION VIA A HIGH-ENERGY BALL MILLING PROCESS

Kong LB. Ma J. Zhu W. Tan OK. - Journal of Alloys & Compounds. 335(1-2):290-296, 2002

Solid solutions of (Zr1-xTix)O-2 (x=0.44, 0.48, 0.52 and 0.60) with a srilankite structure were produced directly from ZrO2 and TiO2 oxide powders via a high-energy ball milling process in air at room temperature. (Zr1-xTix)O-2 phase was formed as the samples were milled for 8 h. After milling for 20 h, a single phase of (Zr1-xTix)O-2 could be obtained. The effects of annealing on the phase formation and stability of the solid solution (Zr1-xTix)O-2 were investigated in the oxide mixtures milled for different time durations

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

[23] PEROVSKITE-TYPE OXIDES SYNTHESIZED BY REACTIVE GRINDING PART 11: CATALYTIC PROPERTIES OF LACO((1-X))FEXO3 IN VOC OXIDATION

Szabo V. Bassir M. Van Neste A. Kaliaguine S. - Applied Catalysis B-Environmental. 37(2):175-180, 2002

The catalytic properties in n-hexane total oxidation of perovskite type mixed oxides, LaCo(1-x)FexO3 prepared by a new technique designated as reactive grinding, are investigated. All catalysts were found significantly more active than a reference sample prepared by conventional synthesis using amorphous citrate complexes. The activity per unit surface area was found to depend on grinding conditions and calcination temperature. These enhanced activities are associated with both the high surface area and high defect density reached by the reactive grinding synthesis method. Cobalt rich perovskites were shown more active per unit surface area than iron perovskites calcined at the same temperature.

[22] STUDY ON NITRIDING REACTIONS OF TA-N-2 BY MECHANICAL ACTIVATION [CHINESE]

Liu L. - Acta Physica Sinica. 51(3):603-608, 2002

The nitriding reactions between tantalum and molecular nitrogen activated by mechanical milling are investigated by X-ray diffraction and transmission electron microscopy. It is found that the high-energy milling at ambient temperature can activate Ta/ N-2 reaction and causes the formation of three different structures, including tetragonal Ta2N, hexagonal Ta3N5 and an amorphous phase. The chemisorption of nitrogen onto the clean tantalum surfaces created by milling is the dominated processes governing Ta/ N-2 reaction. The binding energy of Ta-N band much higher than that of N-N band provides the thermodynamic driven force for N-2 decomposition, while the energy input due to introduction of high density of grain-boundaries and lattice defects offers the activation energy for the transition from Ta-N bands to Ta-nitrides. The amorphous phase formed in the later stage of milling is caused by iron intake due to the contamination from milling tool. The results demonstrate that the amorphous phase obtained consists of three elements of Ta, Fe and N.

[21] MECHANICAL ALLOYING IN IMMISCIBLE ALLOY SYSTEMS [REVIEW]

Authors Fang F. Zhu M. - Progress in Natural Science. 12(3):170-174, 2002

In recent years, mechanical alloying (MA) of immiscible alloy systems characterized by positive heat of mixing has been extensively investigated. The present article reviews the latest progress in MA of immiscible alloy systems including the mechanisms of non-equilibrium phase transformation and metastable phase formation of the MA-driven supersaturated solid solutions, amorphous phases and nanophase composites as well as their mechanical and physical properties related to those metastable phases

[20] INFLUENCE OF LONG MILLING TIME ON THE STRUCTURE OF FINEMET TYPE POWDERS

Sovak P. Fechova E. Kollar P. Kavecansky V. Hornak P. - Physica Status Solidi A-Applied Research. 189(3):747-751, 2002

The aim of this work was to investigate the crystallographic structure, size distribution and chemical composition of FINEMET type powders after long milling (20 h up to 1800 h). Three different powder samples have been prepared and investigated: the first one by milling of amorphous ribbon with a chemical composition of Fe73.5Cu1Nb3Si13.5B9, the second by milling of the same ribbon but in a partially nanocrystalline state, and the third was prepared from powders of the pure elements of which FINEMET consists. Experimental results confirm that particle size distribution, crystallographic structure and chemical composition of the powders depend on all the investigated parameters: milling time and the character of the initial material before milling.

[19] VARIATION OF MAGNETISM AND STRUCTURE ON MECHANICALLY ALLOYED CO-C POWDER

Yoo YG. Kim KS. Yu SC. Yang DS. - Physica Status Solidi A-Applied Research. 189(3):769-773, 2002

The variation of magnetic properties during mechanical alloying was studied in Co100-xCx (x = 20, 50, 80) granular alloys. The structural evolution during mechanical alloying was complemented by X-ray diffractometry. With increasing processing time, X-ray diffraction peaks from Co and C phases became weaker and finally disappeared. The grain size was estimated to be about 20 nm from the broadening of the XRD peaks. The saturation of magnetization at a steady state value was another evidence for the formation of a saturated solid solution in the mechanically alloyed sample. During mechanical alloying, the exchange interaction among the Co atoms becomes weak? er due to inter-diffusion of Co and C atoms. Both the Curie temperature, Tc, and the spin wave stiffness constant, D, which were estimated from the temperature dependence of magnetization, decrease with increasing C content. This tendency indicates the isolation of Co clusters from the C matrix.

[18] MAGNETIC AND STRUCTURAL PROPERTIES OF MECHANICALLY ALLOYED FE,MN-0.70,AL-0.30 (X=0.40 AND 0.45) ALLOYS

Rico MM. Medina G. Alcazar GAP. Munoz JS. Surinach S. Baro MD. - - Physica Status Solidi A-Applied Research. 189(3):811-816, 2002

Alloys of the system FexMn0.70-xAl0.30, x = 0.40 and 0.45, prepared by mechanical alloying during 24, 48, 72 and 84 hours, were studied by X-ray diffraction, Mossbauer spectroscopy and ac magnetic susceptibility. The alloys with x = 0.45 have a bcc structure for all the milling times whereas those with x = 0.40 are bee for 24 and 48 hours and bcc fee for 72 and 84 hours. Mossbauer spectroscopy at room temperature shows that the alloys with x = 0.40 are paramagnetic for all the milling times and their spectra were fitted with a doublet (bcc) for times of 24 and 48 hours and with a singlet (fcc) + a doublet in the other cases. For the x = 0.40 samples, by ac magnetic susceptibility measurements a reentrant spin-glass to ferromagnetic transition (RSG-F) was detected at low temperatures and a ferromagnetic to paramagnetic (F-P) transition at higher temperatures, whereas for the samples with x = 0.45 only a superparamagnetic-like blocking process to ferromagnetic transition (SPM-F) was detected. Moreover, for both compositions, the ferromagnetic phase increases its stability with the milling time due to the increasing disorder induced by milling. The presence of the RSG phase is a consequence of the

disorder and the competitive interactions, and presence of the SPM-like blocking process is due to the disorder and the increase of Fe content

[17] MOSSBAUER STUDY OF MECHANICALLY ALLOYED FE57CR31CO12

Bentayeb FZ. Alleg S. Bouzabata B. Greneche JM. - Physica Status Solidi A-Applied Research. 189(3):841-844, 2002

Nanostructured powders of Fe57Cr31Co12 were prepared by mechanical alloying from elemental Fe, Cr and Co powders, using a planetary ball mill type Fritsch Pulverisette 7. The powders were characterized by X-ray diffraction and Fe-57 Mossbauer spectrometry. A detailed analysis of the diffraction patterns reveals a decrease of the crystalline grain size as a function of milling time. For the first hours of milling, Mossbauer spectra are composed of a magnetic contribution and a single line. The paramagnetic component, whose relative area increases with milling time, can be attributed to paramagnetic Cr-rich clusters. The results are compared to those obtained on the same alloy prepared by conventional melting technique.

[16] MAGNETIC PROPERTIES OF THE FINEMET TYPE POWDER PREPARED BY LONG TIME MILLING

Kovac J. Petrovic P. Fechova E. Fuzer J. Kollar P. - Physica Status Solidi A-Applied Research. 189(3):859-863, 2002

We have investigated the influence of milling on the magnetic properties of powder FINEMET material as a function of long milling time (up to 3500 h). The results show that magnetic properties significantly depend on milling time. The coercivity of the sample increases with the increase of milling time up to 1700 h almost linearly while further milling leads to saturation at the value of 25 kA/m (at the milling time of 3500 h). The evolution of Mossbauer spectra of samples as a function of milling time shows the decreasing amount of ferromagnetic phases of alpha-Fe grains seen as a sextet. With the increase of milling time a doublet of small superparamagnetic particles accumulates and after 2500 h of milling it is a dominant feature of spectra

[15] EARLY STAGES OF MECHANICAL CRYSTALLIZATION OF AMORPHOUS FE86ZR7B6CU1 SOFT MAGNETIC MATERIAL: THE ROLE OF SHEAR BANDS

Friedrich J. Herr U. Samwer K. - Physica Status Solidi A-Applied Research. 189(3):897-902, 2002

Nanocrystalline soft magnetic materials can have superior properties such as low coercivity combined with a high saturation magnetization. Whereas these materials are usually made by thermal crystallization of amorphous precursors, mechanical crystallization using ball milling provides an alternative route for the generation of nanocrystalline materials. The focus of this study is the development of the magnetic properties and the microstructure at the early stages of the transformation of initially amorphous Fe86Zr7B6Cu1. Controlled deformation of amorphous starting material by cold rolling is performed to clarify the origin of the coercivity maximum. Optical and magneto-optical investigations reveal the influence of the microstructure on the coercivity. The shear band density obtained from the magneto-optical observations is directly correlated with the increase in coercivity.

[14] THE TEMPERATURE AND MILLING-TIME DEPENDENCE OF COERCIVITY IN BALL-MILLED ND2FE14B-GD

Thong AW. Cashion JD. - Physica Status Solidi A-Applied Research. 189(3):1035-1037, 2002

Exchange coupled 20 vol% Nd2Fe14B-80 vol% Gd magnetic powders were synthesised by ball milling. Magnetometer measurements at 5 and 300 K showed that coercivity increased with milling time. The temperature dependence of the coercivity of a 30 h milled sample showed a gradual decrease from 5 K until approximately 270 K, after which the coercivity increased markedly to a peak before decreasing again.

[13] AMORPHOUS-TO-CRYSTALLINE TRANSFORMATION INDUCED BY THERMAL ANNEALING OF A METASTABLE AL90FE10 COMPOSITE

Zhou F. Luck R. Lu K. Lavernia EJ. Ruhle M. - Philosophical Magazine A-Physics of Condensed Matter Defects & Mechanical Properties. 82(5):1003-1015,

Crystallization from the amorphous state to metastable crystalline Al6Fe was induced by thermal annealing of mechanically alloyed Al90Fe10 composite powders (where the composition is given in atomic per cent), which consisted of fcc Al nanocrystals in an amorphous matrix. The microstructural evolution and the transformation kinetics were characterized in detail. A thermodynamic analysis of the transformation was given, showing the nature of the polymorphous crystallization in the amorphous composite. The influence of the pre-existing crystals on crystallization was not morphologically distinctive. The observed linear kinetics law behaviour in combination with an Avrami exponent value of 2.7 +/- 0.1 suggest that the growth of a fixed nuclei population at a constant rate in three dimensions governs crystallization. The activation energy for crystallization was found to be close to the activation energy for the diffusion of Fe atoms in Al

[12] MECHANOCHEMICAL SYNTHESIS AND CATALYTIC PROPERTIES OF THE CALCIUM FERRITE CA2FE2O5

Isupova LA. Tsybulya SV. Kryukova GN. Budneva AA. Paukshtis EA. Litvak GS. Ivanov VP. Kolomiichuk VN. Pavlyukhin YT. Sadykov VA. - Kinetics & Catalysis. 43(1):122-128, 2002

The formation of the real structure of calcium ferrite prepared by the calcination of a mechanochemically activated hydroxide mixture at 600-1100degreesC was studied by X-ray diffraction analysis, electron microscopy, thermal analysis, Moessbauer spectroscopy, IR spectroscopy, small-angle X-ray scattering, and secondary-ion mass spectrometry. It was found that low-temperature calcium ferrite is an anion-modified oxide, in which the ordering of oxygen vacancies was incomplete. Regions with a disordered structure were detected on the surface of crystallites. As the calcination temperature was increased, the brownmillerite crystal structure was improved and the intercrystalline boundaries were formed and then annealed. At the surface, these processes were accompanied by a change in the predominant form of adsorbed NO from nitrosyl to dinitrosyl species. An increase in the specific catalytic activity of samples with calcination temperature can be associated with the perfection of the brownmillerite structure and with a change in the state of adsorption centers

[11] PHYSICOCHEMICAL AND CATALYTIC PROPERTIES OF LA1-XCAXFEO3-0.5X PEROVSKITES PREPARED USING MECHANOCHEMICAL ACTIVATION

Isupova LA. Tsybulya SV. Kryukova GN. Alikina GM. Boldyreva NN. Vlasov AA. Snegurenko OI. Ivanov VP. Kolomiichuk VN. Sadykov VA. - Kinetics & Catalysis. 43(1):129-138, 2002

The phase composition of La1-xCaxFeO3-0.x5 perovskites synthesized from preactivated oxides was studied by powder X-ray diffraction analysis and differential dissolution. The system does not form a continuous series of homogeneous solid solutions. No intermediate samples from this series are monophasic. It was found that the synthesis under nonequilibrium conditions (mechanical activation + calcination at 900degreesC for 4 h) resulted in nonequilibrium microheterogeneous solid solutions with degrees of calcium substitution for lanthanum of no higher than 0.5. A longer calcination (for 16 h) or an increase in the calcination temperature of solutions up to 1100 degreesC decreased the calcium content of the samples down to x similar to 0.2 because of the formation of a brownmillerite phase. The catalytic activity of the test samples in the oxidation of CO changed nonmonotonically with x, and it was maximum at x = 0.5-0.6, which correlates with the maximum density of interphase boundaries in these samples.

[10] SOLID STATE RADICAL RECOMBINATION AND CHARGE TRANSFER ACROSS THE BOUNDARY BETWEEN INDOMETHACIN AND SILICA UNDER MECHANICAL STRESS

Watanabe T. Hasegawa S. Wakiyama N. Usui F. Kusai A. Isobe T. Senna M. - Journal of Solid State Chemistry. 164(1):27-33, 2002

A composite of indomethacin (IM) and SiO2 was prepared in a solid state by cogrinding. Dehydration was observed as a consequence of a chemical reaction between silanol groups of SiO2 and carboxyl groups of IM. Several specific radical species found on separately ground SiO2 disappeared preferentially after cogrinding as a result of their recombination. These chemical interactions under mechanical stress bring about a charge transfer, as confirmed by X-ray photoelectron spectroscopy, leading to Si-O-C bridging bond formation at the IM/SiO2 interface

[9] MODELING HIGH-ENERGY BALL MILLING IN THE ALUMINA-YTTRIA SYSTEM

Alkebro J. Begin-Colin S. Mocellin A. Warren R. - Journal of Solid State Chemistry. 164(1):88-97, 2002

Experimental results from high-energy ball milling of alumina-yttria powder mixtures have been analyzed with models collected from the literature. Depending on the milling conditions, either there is formation of an intermediate phase in the alumina-yttria system (yttrium aluminum perovskite, YAP), or the sample becomes mostly amorphous. Variations due to milling tool material can be accounted for by local models based on the Hertzian theory of elastic bodies, but the effects of changing mills are poorly accounted for by published models. Therefore, the concept of an impact frequency distribution over the energy spectrum is introduced as a tool for studying the characteristics of the mills. The pressure on the powder trapped between two colliding bodies has been found to be the factor deciding the outcome of the process. The threshold behavior of the system yields an amorphous structure for low pressures, and formation of YAP when impact pressures exceed the threshold value.

[8] REACTIVITY AND STRUCTURAL PROPERTIES OF A MECHANOCHEMICALLY TREATED AG2O-V2O5 SYSTEM IN RELATION TO AGVO3 POLYMORPHS

Kittaka S. Nishida S. Iwashita T. Ohtani T. - Journal of Solid State Chemistry. 164(1):144-149, 2002

Formation and chemical properties of amorphous AgVO3, which was prepared by mechanochemical treatment of an Ag2O-V2O5 mixture, and crystaffine AgVO3 were studied in relation to AgVO3 polymorphs. A ball-milled sample of the mixture was assigned as a highly deformed beta-AgVO3 rather than the low density phase alpha-AgVO3. Crystalline alpha-AgVO3 and beta-AgVO3 were converted into deformed beta-AgVO3 by ball milling, which produced a clear change. delta-AgVO3 is resistant to mechanical treatment and its structure was not markedly affected. The dissolved chemical species from the ball-milled sample precipitates to form alpha-AgVO3 without a seeding crystal, but other polymorphs deposit if they are present; i.e., beta-AgVO3 and delta-AgNO3 grow on the seeding crystal

[7] MECHANICAL ACTIVATION OF STRUCTURAL AND CHEMICAL TRANSFORMATIONS IN A ZR-C-H SYSTEM IN TWO STAGES

Borchers C. Leonov AV. Morozova OS. - Journal of Physical Chemistry B. 106(8):1843-1848, 2002

The effect of mechanical activation on the structural and chemical transformations in the zirconium-carbon-hydrogen system was studied using X-ray powder diffraction, transmission electron microscopy and kinetic techniques. A zirconium-graphite mixture was subjected to high-energy impact milling in a permanent hydrogen flow. A two-stage process was observed: first, the formation of tetragonal epsilon-ZrH1.8-2 accompanied by carbon amorphization, and second the decomposition of epsilon-ZrH1.8-2 synthesized at the first stage to cubic delta-ZrH1.6 + gamma-ZrH + alpha-Zr. In the first stage, the size of the hydride particles decreased drastically to only a few nanometers dispersed in larger particles of amorphous carbon, while in the second stage the newly formed Zr particles compacted again to gain the size of microns, while the carbon partially crystallized. While the first stage is governed by the fast reaction kinetics of hydrogen with zirconium, the second stage is governed by the greater affinity of zirconium to carbon as compared to hydrogen

[6] SONOCHEMICAL SYNTHESIS OF MESOPOROUS IRON OXIDE AND ACCOUNTS OF ITS MAGNETIC AND CATALYTIC PROPERTIES

Srivastava DN. Perkas N. Gedanken A. Felner I. - Journal of Physical Chemistry B. 106(8):1878-1883, 2002

Synthesis of mesoporous iron oxides has been reported using a sonochemical technique. Iron(III) ethoxide was used as an inorganic precursor and CTAB as an organic structure directing agent. After sonication the surfactant was removed by calcination and solvent extraction methods. FTIR spectra demonstrated the removal of the surfactant from the pores of the

mesoporous iron oxide. The calcinated material was characterized by XRD, TEM, TGA, and BET surface area measurements. The surface area after solvent extraction is found to be 274 m(2)/g. The magnetic and catalytic properties of the materials have been studied. The as-prepared amorphous Fe2O3 has shown a paramagnetic behavior, while after calcination at 350 degreesC it changes to gamma-Fe2O3 with good magnetic properties. The catalytic activity of mesoporous iron oxide was studied in the reaction of cyclohexane oxidation under mild conditions. The mesoporous Fe2O3 catalyst has shown high conversion of cyclohexane into cyclohexanone and cyclohexanol, with a high selectivity

[5] SIMULTANEOUS SYNTHESIS AND CONSOLIDATION OF NANOSTRUCTURED MOSI2

Gras C. Bernard F. Charlot F. Gaffet E. Munir ZA. - Journal of Materials Research. 17(3):542-549, 2002

A new process combining electric field activation and the imposition of pressure from mechanically activated powder mixtures is demonstrated as a means to simultaneously synthesize and density nano-MoSi2 in one step. Nanophase reactants (Mo + 2Si) produced by mechanical activation are reacted by field activation with the simultaneous application of a uniaxial pressure. Mo + 2Si powders were comilled in a specially designed planetary mill to obtain nanometric reactants but to avoid formation of any product phases. These were then subjected to high alternating currents (1600 A) and pressures of 106 MPa. Under these conditions, a reaction is initiated and completed within a short period of time (3-6 min). The relative density of the product ranged from 82 to 93%. The crystallite size of the MOSi2 compound was determined by x-ray diffraction line-broadening analysis using the Langford method. The size ranged from 58 to 75 nm

[4] ICOSAHEDRAL PHASE FORMATION DOMAIN IN AL-CU-FE SYSTEM BY MECHANICAL ALLOYING

Barua P. Murty BS. Mathur BK. Srinivas V. - Journal of Materials Research. 17(3):653-659, 2002

A systematic composition dependence study on icosahedral phase (i-phase) formation in the Al-Cu-Fe system has been carried out. Structural evolution during mechanical alloying and on subsequent heat treatment has been investigated by x-ray diffraction and transmission electron microscopy. The i-phase is observed to evolve from the reaction between the Al2Cu and P phases. The influence of the Cu to Fe ratio (RC,,Fe), Al to transition metal ratio (R-AITM), and the electron to atom ratio (e/a) on the volume fraction of the i-phase has been studied. The analysis of the present results and those published earlier indicates that quasicrystal-forming ability correlates better with the R-AITM and e/a ratios. The volume fraction of the i-phase is maximum when the R-AITM similar to 2.3 and e/a ratio similar to 2.0. Formation of the i-phase in Al65Cu25Fe10 by mechanical alloying is reported for the first time.

[3] GRINDING ASSISTANCE IN THE TRANSFORMATION OF GIBBSITE TO CORUNDUM

Sanchez RMT. Boix A. Mercader RC. - Journal of Materials Research. 17(3):712-717, 2002

After gibbsite was milled for 5 min in a Cr-steel oscillating mill, corundum was obtained by heating the powder for 3 h at 800 degreesC. We found that iron contamination, produced by the milling process, is essential to attain the transformation at this low temperature and is located at the surface of the gibbsite particles, The knowledge of the oxidation state and location of the contaminant elements, necessary to control the solid-state reactions and/or phase transformations induced by the milling, was realized in this work by a characterization performed by chemical analysis, x-ray photoclectron spectroscopy, Mossbauer spectroscopy, and isoelectric point determination. The iron contamination amounted to about 3% (as Fe2O3) for the sample milled for 60 min. That the iron contamination that occurred mainly on the gibbsite amorphous surface was concluded after a comparison of the isoelectric point determination of the milled samples with that of a mechanical mixture of gibbsite and hematite. X-ray diffraction studies showed that gibbsite looses its crystallinity after the first 5 min of milling.

[2] MICROSTRUCTURE AND MAGNETIC PROPERTIES OF UNCOUPLED SM2CO17-CU NANOCOMPOSITES

Zhang JX. Bessais L. Djega-Mariadassou C. Leroy E. Percheron-Guegan A. Champion Y. - Applied Physics Letters. 80(11):1960-1962, 2002

Nanocomposite Sm2Co17-Cu particles have been fabricated by low energy comilling of mechanically alloyed Sm2Co17 particles with Cu nanoparticles. The x-ray diffraction analyses show that the diffraction crystallite size (DCS) of Sm2Co17 decreases with increasing comilling time. Scanning and transmission electron microscopy observation demonstrates that the particle size is in the range of the DCS and the Sm2Co17 particles are separated by Cu particles. The coercivity as well as the remanence ratio decreases with increasing milling time due to the grain size reduction and grain separation. The nanocomposite Sm2Co17-Cu exhibits suitable magnetic and microstructure properties for high-density recording

[1] ULTRA FAST SYNTHESIS OF METASTABLE TETRAGONAL ZIRCONIA BY MEANS OF MECHANOCHEMICAL ACTIVATION

Kuznetsov PN. Kuznetsova LI. Zhyzhaev AM. Pashkov GL. Boldyrev VV. - Applied Catalysis A-General. 227(1-2):299-307, 2002

Ultra fast solid-phase synthesis of nanostructured metastable zirconia of tetragonal modification from the amorphous hydroxide with no cation and anion promoters and from the zirconia of monoclinic modification was performed at the ambient temperature by mechanical stimulation of the reactions of dehydration, crystallisation and solid-phase transition in a high-energy centrifugal planetary ball mill. The process occurred by stages as the mechanical loading increased and crystallisation of metastable form from dry powder of precursors was accomplished in 15 min. Short-term mechanical activation of the amorphous zirconium hydroxide allowed also subsequent thermal crystallisation into the metastable zirconia to occur at low temperature (320degreesC). The generation of nano-sized zirconia is suggested to be one of the factors favouring the formation of the metastable form.

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

Correspondants du Réseau Français de Mécanosynthèse
188 Laboratoires ou Groupes de Recherche (34 Pays)
Bureau : E. Gaffet (Président), G. Le Caër (Secrétaire Général), A.R. Yavari (Trésorier)

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