



**RESEAU FRANÇAIS DE  
MECANOSYNTHESE**

**Lettre N°98**

**Mai 2003**

**189 Groupes de Recherche  
(dont 115 à l'étranger / 34 Pays)**

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**Bulletin d'adhésion 2003 / Subscription Print**

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Eric GAFFET

CNRS UMR5060 « Métallurgies et Cultures »

Nanomaterials Research Group

Site de Sévenans (UTBM) - F90010 - Belfort Cedex - France

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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.

## POST-DOC

### PROGRAMME DE RECHERCHE

<mailto:Luc.aymard@u-picardie.fr>

#### **Préparation par mécanosynthèse de nanoparticules à applications en stockage de l'énergie dans les batteries Li-ions et les piles à combustible.**

On observe ces dernières années un développement exponentiel des équipements électroniques portables, un intérêt croissant pour le véhicule électrique, ou plus récemment pour des applications embarquées ou terrestres des piles à combustibles. Les technologies Ni-MH et Li-ions couvrent actuellement le marché de l'électronique portable sans avoir convaincu dans le domaine du transport. Aussi, le récent essor du stockage de l'hydrogène dans les piles à combustibles ouvre aujourd'hui de nouvelles perspectives et suscite un réel espoir pour ces applications en présentant comme principal argument celui de respecter l'environnement

Le projet de recherche proposé consistera à mettre au point par mécanochimie et électrochimie de nouveaux matériaux nanométriques à base de magnésium et/ou de lithium pouvant être utilisés comme électrode négative pour les batteries Li-ions ou comme éponge à hydrogène dans les piles à combustibles. Du point de vue fondamental, il s'agit de mettre en lumière la chimie et les propriétés en stockage de l'énergie de ces nouvelles familles de nanomatériaux .

#### **Techniques utilisées :**

XRD, SEM, EDS, TEM, Tg, DSC, BET, IR, Electrochimie :  
cyclage galvanostatique, potentiostatique GITT.....

#### **Condition de recrutement**

Titulaire d'un Doctorat en Chimie ou Physique des Matériaux, le candidat doit avoir moins de 35 ans et ne pas avoir préparé sa thèse en France ou travailler actuellement en France.

Post-Doc rémunéré par le Ministère de la Recherche et de la Technologie,  
12 mois, 1830 € net/mois, poste à pourvoir début septembre.

Envoyer un CV détaillé comprenant un résumé de l'activité de recherche et une liste des publications et brevets.

Date limite de dépôt des candidatures : le lundi 07 avril 2003

Laboratoire d'accueil français :

Laboratoire de Réactivité et de Chimie des Solides (LRCS) UMR 6007

Université de Picardie Jules Verne (UPJV) , 33 rue Saint leu 80039 Ville : Amiens

<http://www.u-picardie.fr/labo/lrcs>

Encadrement assuré par Luc Aymard, Maître de Conférences à L'UPJV, chercheur au Laboratoire de Réactivité et de Chimie des Solides.

<mailto:Luc.aymard@u-picardie.fr>



## Post Doc Proposal

**Titre : Postdoctoral research fellowship on the elaboration of nanomaterials by supercritical fluid processing – Bordeaux (France)**

**Contact** : <mailto:cansell@icmcb.u-bordeaux.fr>

The Institut of Condensed Matter Chemistry of Bordeaux (ICMCB) proposes a post-doctoral research fellowship. ICMCB (200 people) is a French laboratory of CNRS (French National Center for Scientific Research) with research activities in Solid state chemistry, Material science and Molecular sciences.

The research project concerns the elaboration of nanomaterials by supercritical fluid processing in using a new synthesis reactor equipped with an in situ analysis system by fluorescence spectroscopy. The aim consists to synthesize ferroelectric nanomaterials with controlled size and surface properties. In particular, we plan to study the nanoparticle size evolution as a function of the synthesis process working conditions (Pressure, Temperature, residence time,...) by means of fluorescence spectroscopy. The ferroelectric properties of the obtained nanomaterials will be studied and a correlation between nanoparticle size, nanoparticle surface properties and material ferroelectric properties will be established.

The post-doctoral student will work with two well known teams of ICMCB and so, will dispose of a very important human and technical potential.

The post-doctoral student must have a good expertise in material science, more precisely, in material or nanomaterial synthesis and characterization and in material surface characterization.

The postdoctoral fellowship is supported by CNRS (2050 euros per month) for 12 or 18 months.



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**Congress and School Announcements**  
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**ENROBAGE, ENCAPSULATION, DISPERSION, DISSOLUTION**

13 et 14 mai 2003 à Nantes  
Contact : J.L. ILARI (ilari@enitiaa-nantes.fr)

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**J2IM 2003**

Vogüé, ARDECHE (France)  
19-21 mai 2003

<http://www.emse.fr/fr/actualites/j2im2003.html>

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**4th international conference for conveying and handling of particulate solids, CHoPS,**

Budapest 27-30 mai 2003  
Information et programme :  
<http://www.partconf2003.hu/>

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**Colloque "De la poudre au matériau massif",**

Ecole des Mines d'ALBI, 3-5 juin 2003  
Contact : SF2M, Les Fontenelles, 1 rue Craïova, F -92024 - Nanterre Cedex  
<mailto:sf2mcongress@wanadoo.fr>,  
<http://www.sf2m.asso.fr/>

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**NUCLEATION WORKSHOP**

17-19 juin 2003 , Ecole des Mines de Saint Etienne  
Contact Jean michel Herri (herri@emse.fr)  
Vous trouverez l'appel à contribution à l'adresse :  
<http://www.emse.fr/fr/transfert/spin/actualites/nucleation.html>  
Premier et deuxième jour consacrés à la nucléation : théorie,  
applications et étude de cas Troisième jour consacré à la nucléation des hydrates de gaz

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**The 10th Int. Symposium on Metastable, Mechanically Alloyed and Nanocrystalline Materials,**

**ISMANAM 2003,**

**will be held in Foz do Iguacu,  
Brazil, on 24-28 August 2003**

<mailto:ismanam2003@dema.ufscar.br>  
<http://www.dema.ufscar.br/ismanam2003>

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**International Conference  
NANOMATERIALS AND NANOTECHNOLOGIES (NN 2003),  
Crete, Greece; August 30 - September 6, 2003**

<http://www.ipme.ru/ipme/conf/NN2003/>

Lettre RFM N°98 - Mai 2003  
Corresp. : <mailto:Eric.Gaffet@utbm.fr>



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**INTERNATIONAL CONFERENCE**  
**"Novel Technologies in Powder Metallurgy and Ceramics"**

September 8-12, 2003

Kiev, Ukraine  
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**Fourth INTERNATIONAL CONFERENCE**  
**ON MECHANOCHEMISTRY AND MECHANICAL ALLOYING**  
**4th INCOME 2003**

Technical University of Braunschweig, Braunschweig, Germany

September 7-11, 2003

Website : <http://www.tu-bs.de/INCOME2003>  
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**9ième Congrès de la SFGP,**

Saint Nazaire, 9-10 septembre 2003

Contact :

<mailto:sfgp.carole.bezzi@gepea.univ-nantes.fr>,

<http://www.sfgp.asso/stnazaire2003>  
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**11th FORESIGHT CONFERENCE ON MOLECULAR NANOTECHNOLOGY**

October 9-12, 2003

San Francisco Airport Marriott

Burlingame, CA, USA

<http://www foresight.org/Conferences/MNT11>  
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**XV. International Symposium on Reactivity of Solids:**

Nov. 9. - 13. 2003

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

<mailto:info@ISRSKYOTO.org>  
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**XV. International Symposium on Reactivity of**  
**COLOQUE DE CRISTALLISATION INDUSTRIELLE**

12-13 Novembre 2003 à Toulouse

Contact : Béatrice Biscans ([beatrice.biscans@ensiacet.fr](mailto:beatrice.biscans@ensiacet.fr))

Inscription et soumission de votre résumé en ligne :

<http://www.ensiacet.fr/PROGEP/CRISTAL2>  
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**Journées de l'AFSIA sur les techniques de séchage innovantes**

14 novembre 2003 à Paris

contact :e.mail : <mailto:andrieu@lagep.univ-lyon1.fr>

<http://www-lagep.univ-lyon1.fr/>  
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**MELANGE DES POUDRES, ECHANTILLONNAGE, DISTRIBUTION DE TEMPS DE SEJOUR**

11-12 Décembre 2003 à Ecole des Mines d'ALBI

Contact : Henri Berthiaux

(<mailto:berthiau@enstimac.fr>)  
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Third International Symposium on Ultrafine Grained Materials

The 2004 TMS Annual Meeting, March 14-18, 2004, Charlotte, NC, US

<http://www.cms.tms.org/>



# Third International Symposium on Ultrafine Grained Materials

## The 2004 TMS Annual Meeting, March 14-18, 2004, Charlotte, NC, US

This is the third international symposium that focuses on all aspects of science and technology of ultrafine-grained (UFG) materials produced by Severe Plastic Deformation (SPD) techniques. It provides a forum on the topics of fundamental issues in SPD processing and SPD-processed materials, processing and microstructures, microstructural evolution, mechanical and physical properties, superplasticity, computational and analytical modeling, new SPD technologies and advances, structural applications, *etc.* A panel discuss is planned at the end of the symposium. A TMS proceedings book will be available at the meeting.

**Abstracts due by July 1, 2003, to [cms.tms.org](http://cms.tms.org)** (you can also check [cms.tms.org](http://cms.tms.org) website for more information).

**Full paper due by August 15, 2003.** The paper should be in MS Word and sent to [yzhu@lanl.gov](mailto:yzhu@lanl.gov). Instructions for paper preparation will be available later.

**Awards:** Two Gold Medals for oral presentation will be awarded to students or posdtocs within 3 years of their Ph.D. One Gold Medal and five Silver Medals will be awarded to the best posters (all poster presenters are eligible). The awards will be decided by a committee and all awards are intended to be accompanied by a check that helps pay for the recipient's travel expense.

### Sponsoring Committees and Organization:

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Yuntian T. Zhu MS G755 Mater. Sci. & Tech. Division Los Alamos National lab. Los Alamos, NM 87545 USA <a href="mailto:yzhu@lanl.gov">yzhu@lanl.gov</a> v	Terence G. Langdon Depts. of Aerospace & Mech Eng. and Mater. Sci. U. of Southern California Los Angeles, CA 90089 USA <a href="mailto:langdon@usc.edu">langdon@usc.edu</a>	Ruslan Z. Valiev Inst. of Phv. of Adv. Mater. Ufa State Aviation Tech. Univ. 12 K.Marx St. Ufa 450000, Russia <a href="mailto:RZValiev@mail.rb.ru">RZValiev@mail.rb.ru</a>	S. Lee Semiatin Air Force Research Laboratory Mater. & Manufact. Directorate WPAFB, OH 45433 USA <a href="mailto:Lee.Semiatin@wpafb.af.mil">Lee.Semiatin@wpafb.af.mil</a>	Dong H. Shin Dept. Metall. & Mater. Sci. Hanyang University Ansan, Kyunggi-Do 425-791 Korea <a href="mailto:dhshin@hanyang.ac.kr">dhshin@hanyang.ac.kr</a>	Terry C. Lowe Metallicum, LLC 1207 Callejon Arias Santa Fe, NM 87501 <a href="mailto:tlowe@lanl.gov">tlowe@lanl.gov</a> v
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## Périodiques / Congrès

### [45] NANOSTRUCTURES IN THERMAL SPRAY COATINGS

Gang Ji a, Jean-Paul Morniroli, Thierry Grosdidier - Scripta Materialia, to be published

The nature of the nanograins formed by high velocity oxy-fuel thermal spraying of (FeAl) milled powder has been investigated using transmission electron microscope on cross-sectional thin foils. Equiaxed 3D nanometer crystallites are formed by recrystallization in the unmelted powder particles while 2D nanometer columnar grains are produced by rapid solidification within the fully molten splats.

### [44] IMPROVED TRANSPORT CRITICAL CURRENT AND IRREVERSIBILITY FIELDS IN MONO- AND MULTIFILAMENTARY FE/MGB2 TAPES AND WIRES USING FINE POWDERS

Flukiger R. Lezza P. Beneduce C. Musolino N. Suo HL. - Superconductor Science & Technology. 16(2):264-270, 2003  
Mono- and multifilamentary Fe/MgB<sub>2</sub> tapes and wires with high transport critical current densities have been prepared by the powder-in-tube technique using fine powders. The influence of the initial MgB<sub>2</sub> grain size on critical current density, upper critical and irreversibility fields has been studied. After reducing the MgB<sub>2</sub> grains to micrometer size by ball milling the critical current density, J(c), was enhanced, while the upper critical field,  $\mu(0)H(c_2)$ , remained unchanged. The anisotropy ratio between the upper critical fields parallel and perpendicular to the tape surface was determined to be 1.3, reflecting a deformation induced texture. A good agreement has been found between resistive and inductive J(c) values, measured at various temperatures between 4.2 and 25 K. On monofilamentary tapes, J(c) values close to 10(5) A cm(-2) were measured at 25 K/1 T, while J(c) values approximate to 10(6) A cm(-2) were extrapolated for 4.2 K/0 T. Fe/MgB<sub>2</sub> tapes exhibit high exponential n factors for the resistive transition: n values of 60 and 30 were found at 4 T and 6 T, respectively. Multifilamentary wires (with seven filaments) show slightly lower J(c) values, 1.1 x 10(5) A cm(-2) at 4.2 K/2 T. The improvement of thermal and mechanical stability of MgB<sub>2</sub>/Fe tapes and wires appears clearly as a challenge for future developments

### [43] LOW TEMPERATURE PREPARATION OF MGB2 TAPES USING MECHANICALLY ALLOYED POWDER

Hassler W. Rodig C. Fischer C. Holzapfel B. Perner O. Eckert J. Nenkov K. Fuchs G. - Superconductor Science & Technology. 16(2):281-284, 2003

Instead of commercially available MgB<sub>2</sub> powder, we have used partially reacted powder prepared by mechanical alloying. This precursor powder consists of grains with a size of only a few nanometres and contains reacted MgB<sub>2</sub>, and also the starting material Mg and B, and is, therefore, more reactive than fully reacted commercial powders. Using copper as a sheath material, tapes were prepared by the usual powder-in-tube process. After annealing at relatively low temperatures (770-870 K) in inert atmosphere, the tapes have good superconducting properties. Magnetically we have measured a critical current density of 400 kA cm(-2) at 4.2 K

### [42] MECHANICAL MILLING-INDUCED DEFORMATION TWINNING IN FCC MATERIALS WITH HIGH STACKING FAULT ENERGY

He JH. Chung KH. Liao XZ. Zhu YT. Lavernia EJ. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 34(3):707-712, 2003

### [41] EFFECTS OF LIQUID MEDIUM AND BALL-MILLING ON THE SURFACE GROUP AND AQUEOUS DISPERSIBILITY OF Si3N4 POWDER

Huang Y. Dai JQ. Xie ZP. Ma T. Yang JL. Ma JT. - Journal of the European Ceramic Society. 23(6):985-990, 2003  
Influences of liquid medium and ball-milling process on the particle surface group and aqueous dispersibility of silicon nitride powder were investigated in this paper. After ball-milling in different liquids, the particle surface group and aqueous dispersibility of silicon nitride powder have different alternations. For the powders treated with identical ball-milling process in different liquids, the subsequent calcination in static air recovers the same surface characteristics and hence the same aqueous dispersibility. Ball-milling process has significant effect on the particle surface group and its aqueous dispersibility. Under conditions of high weight ratio of milling-balls to powders and large rotary speed, the reaction of ruptured Si-O-Si surface groups with ethanol is dominant and the large amount of Si-O-C-R structures depresses its aqueous dispersibility remarkably

### [40] CONSOLIDATION OF AMORPHOUS COPPER BASED POWDER BY EQUAL CHANNEL ANGULAR EXTRUSION

Robertson J. Im JT. Karaman I. Hartwig KT. Anderson IE. - Journal of Non-Crystalline Solids. 317(1-2):144-151, 2003  
Cu<sub>50</sub>Ti<sub>32</sub>Zr<sub>12</sub>Ni<sub>5</sub>Si<sub>1</sub> gas-atomized powder was consolidated by equal channel angular extrusion (ECAE). Powder was vacuum encapsulated in copper cans and extruded at a temperature above the glass transition temperature (T-g), but below the crystallization temperature (T-x). Five samples were subjected to one extrusion pass, each with a different temperature and extrusion rate. Microstructure, thermal stability, X-ray diffraction measurements and hardness of the ECAE consolidates were examined and compared with those of the initial powder and with a conventional extrusion (CE) consolidate. All consolidates exhibit a supercooled liquid region slightly narrower than that of the starting powder. No significant crystallization peaks are observed in XRD measurements; however, changes in peak shape and the total enthalpy of crystallization in differential scanning calorimetry measurements are attributed to nanocrystallization that is not easily detected by these methods. Greater microhardness values in ECAE consolidates in comparison with the starting powder also support the probability of nanocrystallization. The brittle behavior exhibited by all consolidates is attributed to an initial high oxygen contamination of the powder (similar to 2000 ppm) and the possibility of crystallization due to long exposure to temperatures above T-g during consolidation. Microstructural examination of the ECAE consolidates shows significant shear deformation of the particles with one ECAE pass. The results of the present study encourage further work on the fabrication of bulk metallic glass from powder by ECAE consolidation



**[39] MECHANICAL PROPERTIES OF A CRYOGENICALLY MECHANICALLY ALLOYED POLYCARBONATE-POLY(ARYL ETHER ETHER KETONE) SYSTEM**

Martin JP. Kander RG. - Journal of Applied Polymer Science. 88(5):1196-1202, 2003

The processing-property relationship of a model cryogenically mechanically alloyed polymer-polymer system [polycarbonate (PC) and poly(aryl ether ether ketone) (PEEK)] was investigated. PC and PEEK powders were cryogenically mechanically alloyed for 10 h, and the resulting two-phase powder particles were processed into testable coupons with a miniature ram-injection molder. The bulk mechanical properties of the coupons made from the mechanically alloyed powders and nonmechanically alloyed powders were investigated as a function of mechanical alloying and injection-molding parameters. The injection-molded coupons were mechanically tested in the three-point-bending mode. The results demonstrated that no measurable improvement was achieved in the energy to break, strain at failure, or failure strength in the coupons made from the mechanically alloyed materials in comparison with those of the coupons made from the nonmechanically alloyed powders.

**[38] SINTERING OF CORDIERITE BASED MATERIALS**

Camerucci MA. Urretavizcaya G. Cavalieri AL. - Ceramics International. 29(2):159-168, 2003.

The sintering behavior of cordierite based materials obtained from powders with different granulometric characteristics (single fractions and binary granulometric mixtures) has been studied. Commercially available cordierite powder was used to prepare these materials by attrition milling, uniaxial pressing and sintering at 1450 degreesC. The employed cordierite powders were classified as coarse, medium and fine single granulometric fractions. Binary mixtures of them with 30, 50 and 70 wt.% of the smaller component were prepared. Densification degree and kinetics of sintering were studied through density measurements and microstructural analysis (SEM). Phases were determined by XRD and FTIR techniques and examining the isothermal section at the Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>-MgO system at sintering temperature. The contact angle of the glassy phase present at this temperature was measured using the heating microscopy technique. The characteristics of the starting powders and the particle packing were correlated to the developed microstructures and a sintering mechanism was proposed.

**[37] LOCAL STRUCTURE OF DEUTERATED TI-ZR ALLOY**

Fukunaga T. Itoh K. Hashi K. Aoki K. - Applied Physics a (Materials Science Processing). 74(Part 2 Suppl S):S957-S959, 2002

The Ti-Zr alloy system is isomorphous over the total concentration range. A neutron zero-scattering alloy can be obtained at the composition Ti<sub>0.676</sub>Zr<sub>0.324</sub> because of negative and positive coherent neutron scattering amplitudes of Ti and Zr respectively. A (Ti<sub>0.676</sub>Zr<sub>0.324</sub>)D-0.31 amorphous alloy was synthesized by mechanical alloying (MA) under a deuterium-gas atmosphere of 0.08 MPa. In contrast, it is found that the MA of Ti and Zr powders under a deuterium-gas atmosphere of 2.0 MPa forms a nanocrystalline (Ti<sub>0.676</sub>Zr<sub>0.324</sub>)D-1.54 alloy, which is composed of TiH<sub>2</sub> and ZrH<sub>2</sub> crystalline compounds.

**[36] VIBRATIONAL AND MAGNETIC PROPERTIES OF SUPERSATURATED CU100-XFEX**

Juranyi F. Suck JB. Janssen S. - Applied Physics a (Materials Science Processing). 74(Part 2 Suppl S):S972-S974, 2002

We investigated the atomic dynamics and the magnetic properties of the Cu<sub>100-x</sub>Fe<sub>x</sub> system. Samples with nominal Fe concentrations of 5, 17.5, 30 and 45 at. % were prepared in the same manner by mechanical alloying in a planetary ball mill. All samples are nanocrystalline supersaturated solid solutions, without crystalline bcc Fe phase or amorphous matrix. They have both ferromagnetic and antiferromagnetic order at 5 K, but the antiferromagnetic state transforms to a paramagnetic one well below room temperature. The inelastic neutron scattering measurements were performed at room temperature at FOCUS (PSI, SINQ). With increasing Fe concentration we additionally observed low-energy, most likely transverse modes in the dynamic structure factor

**[35] STUDY OF THE NANOCRYSTALLINE BALL-MILLED CU-80(Fe0.3Co0.7)(20) COMPOUND**

Galdeano S. Mathon MH. Chaffron L. Andre G. Vincent E. Traverse A. de Novion CH. - Applied Physics a (Materials Science Processing). 74(Part 2 Suppl S):S1046-S1048, 2002

The influence of the ball-milling temperature and intensity has been studied on the nanostructure of the Cu-80(Fe<sub>0.3</sub>Co<sub>0.7</sub>)(20) compound. The ball milling of mixed powders of Cu-Fe and Cu-Co supersaturated solid solutions allows the formation of a very fine Fe<sub>30</sub>Co<sub>70</sub> precipitation in the Cu matrix, interesting for its magnetoresistive properties. Powder neutron diffraction and SANS, coupled to magnetic measurements and EXAFS experiments, allowed us to correlate the ball-milling conditions to the nanostructure and the magnetic properties of the ternary compound

**[34] MECHANOCHEMICAL TREATMENT OF ALPHA-Fe2O3: A NEUTRON DIFFRACTION STUDY**

Hofmann M. Campbell SJ. Kaczmarek WA. - Applied Physics a (Materials Science Processing). 74(Part 2 Suppl S):S1233-S1235,

In agreement with earlier studies, neutron diffraction measurements show that off-stoichiometric Fe<sub>3-x</sub>O<sub>4</sub> is the main product of wet-milling haematite, alpha-Fe<sub>2</sub>O<sub>3</sub>, in vacuum with defect magnetite, similar to Fe<sub>2.8</sub>O<sub>4</sub>, being obtained after milling for 72 and 144 h. Rietveld refinements of the room-temperature neutron diffraction patterns show that similar to Fe<sub>2.8</sub>O<sub>4</sub> exhibits reduced lattice parameters (similar to 0.1%) and magnetic moment values (similar to 5%) compared with stoichiometric Fe<sub>3</sub>O<sub>4</sub>. Comparison of the Fe<sub>2</sub>O<sub>3</sub> content as determined from the phase fractions of the 72-h milled sample (similar to 7%) and following heat treatment at similar to 950 K (similar to 37%) reveals that a significant fraction of alpha-Fe<sub>2</sub>O<sub>3</sub> is present in the milled product in an amorphous-like or disordered state.

**[33] FORMATION OF FINE CEMENTITE PRECIPITATES IN AN ULTRA-FINE GRAINED LOW CARBON STEEL**

Shin DH. Park KT. Kim YS. - Scripta Materialia. 48(5):469-473, 2003



Low-carbon steel containing 0.06 wt.% vanadium was deformed by a severe plastic deformation technique and annealed in a temperature range from 420 to 600 degreesC. The treatment resulted in a formation of nano-sized cementite precipitates as well as refinement of ferrite grains to a submicrometer size. This phenomenon is discussed based on dislocation-cementite interactions

**[32] EFFECT OF STRUCTURE AND PHASE COMPOSITION ON COERCIVE FORCE AND SATURATION MAGNETIZATION OF MECHANICALLY ALLOYED FE100-XCX (X=25,32) POWDERS**

Ul'yanov AI. Gorkunov ES. Zagainov AV. Elsukov EP. Dorofeev GA. Konygin GN. Arsent'eva NB. Fomin VM. - Russian Journal of Nondestructive Testing. 38(7):528-536,

Mechanical fracture of iron and graphite powders leads to formation of the amorphous Fe-C phase and subsequent precipitation of the Fe<sub>3</sub>C carbide in the Fe(75)C(25) system and Fe<sub>3</sub>C $\rightarrow$ Fe<sub>7</sub>C<sub>3</sub> carbide in the Fe(68)C(32) system, which are in strongly strained states. After annealing, carbides with undistorted structures are formed, and their content is higher. On the initial stage of this mechanical alloying, the magnetic characteristics of these structures are determined by their defectiveness, and on the subsequent stages of fracturing and annealing by the types, quantities, and structural conditions of the resulting carbide phases

**[31] RECYCLING OF SILICONE RUBBER WASTE: EFFECT OF GROUND SILICONE RUBBER VULCANIZATE POWDER ON THE PROPERTIES OF SILICONE RUBBER**

Ghosh A. Rajeev RS. Bhattacharya AK. Bhowmick AK. De SK. - Polymer Engineering & Science. 43(2):279-296, 2003

The silicone rubber vulcanizate powder (SVP) obtained from silicone rubber by mechanical grinding exists in a highly aggregated state. The particle size distribution of SVP is broad, ranging from 2  $\mu$ m to 110  $\mu$ m with an average particle size of 33  $\mu$ m. X-ray Photoelectron Spectroscopy (XPS) and Infrared (IR) Spectroscopy studies show that there is no chemical change on the rubber surface following mechanical grinding of the heat-aged (200degreesC/10 days) silicone rubber vulcanizate. Addition of SVP in silicone rubber increases the Mooney viscosity, Mooney scorch time, shear viscosity and activation energy for viscous flow. Measurement of curing characteristics reveals that incorporation of SVP into the virgin silicone rubber causes an increase in minimum torque, but marginal decrease in maximum torque and rate constant of curing. However, the activation energy of curing shows an increasing trend with increasing loading of SVP. Expectedly, incorporation of SVP does not alter the glass-rubber transition and cold crystallization temperatures of silicone rubber, as observed in the dynamic mechanical spectra. It is further observed that on incorporation of even a high loading of SVP (i.e., 60 phr), the tensile and tear strength of the silicone rubber are decreased by only about 20%, and modulus dropped by 15%, while the hardness, tension set and hysteresis loss undergo marginal changes and compression stress-relaxation is not significantly changed. Atomic Force Microscopy studies reveal that incorporation of SVP into silicone rubber does not cause significant changes in the surface morphology

**[30] MECHANOCHEMICAL TREATMENT OF RESIDUAL COMPONENTS OF THE ASTRAKHAN GAS CONDENSATE**

Strakhova NA. Rozental' DA. Kortovenko LP. - Petroleum Chemistry. 43(1):52-56, 2003

The effect of an alternating electromagnetic field in a vortex-layer mill on the structure and properties of a high-wax residuum of gas condensate from the Astrakhan field was studied. The action of the electromagnetic field on the residuum at 280-300degreesC resulted in phase redistribution in the disperse system, and the new product was characterized by an increased resin-asphaltene content. Spectroscopic studies showed that the mechanocrackinc, of the residual components of the Astrakhan condensate could occur in the vortex-layer machine

**[29] MECHANICAL ACTIVATION OF NATURAL TITANITE AND ITS INFLUENCE ON THE MINERAL DECOMPOSITION**

Kalinkin AM. Kalinkina EV. Makarov VN. - International Journal of Mineral Processing. 69(1-4):143-155, 2003

Processes induced by mechanical activation of the natural titanite CaTiSiO<sub>5</sub> using a laboratory agate mechanical mortar and a planetary mill AGO-3 have been studied. Titanite consumes substantial amounts of atmospheric carbon dioxide during prolonged dry grinding in air. Carbonisation of titanite occurs alongside with its amorphization. According to FT-IR spectroscopic data CO<sub>2</sub> is present in the ground sample in the form of distorted CO<sub>3</sub><sup>2-</sup> groups resulting in the characteristic double band in the 1500-1430 cm<sup>-1</sup> region. Previously similar processes were revealed for Ca and Mg containing silicate gangue minerals such as enstatite MgSiO<sub>3</sub>, diopside CaMgSi<sub>2</sub>O<sub>6</sub>, (a) over circle kermanite Ca<sub>2</sub>MgSi<sub>2</sub>O<sub>7</sub>, and wollastonite CaSiO<sub>3</sub>. The CO<sub>2</sub> content in the ground titanite reached 4.0 and 7.0 wt.% after grinding in the laboratory agate mechanical mortar for 36 and 108 h, respectively. The amount of carbon dioxide consumed due to grinding increases with increasing CaO content in the chemical formula of minerals and correlates with Gibbs free energies of reactions of the crystalline minerals with CO<sub>2</sub>. The following sequence concerning carbon dioxide sorption ability was revealed: enstatite<diopside<titanite<<(a)over circle>kennanite<wollastonite. Preliminary mechanical activation results in considerable rise of titanite reactivity with respect to 5-20% nitric acid. According to the results obtained the release of Ti and Ca increases with increasing extent of mechanically induced crystal structure disordering and amorphization. In contrast to other titanium containing minerals such as leucosene, ilmenite, rutile, anatase preliminary mechanical activation of titanite in planetary mill for 30 min allows to reach practically complete leaching of Ti with diluted nitric acid at room temperature.

**[28] THE EFFECTS OF OPERATING CONDITIONS ON THE MILLING OF MICROCRYSTALLINE CELLULOSE**

Kwan CC. Ghadiri M. Papadopoulos DG. Bentham AC. - Chemical Engineering & Technology. 26(2):185-190, 2003

There is little detailed work relating the physical process that occurs during milling to the mechanical properties and mechanism of particle breakage. Very often, the selection of an appropriate mill and subsequently the determination of its



optimum operating conditions are by trial and error. This paper look into optimizing the operating conditions of a ball mill through statistical analysis and the effect of temperature on the milling behavior of a common pharmaceutical excipient, microcrystalline cellulose (MCC). In addition, the bulk milling behavior of MCC is compared to its single particle breakage behavior. In this work, milling is conducted in a Retsch single ball mill where a bed of powder is subjected to impact by a steel ball in a horizontal cylindrical container. The container is vibrated horizontally at a set frequency, causing the ball to impact on the bed of particles. It is found that the finest MCC product can be achieved by milling a 2 g batch of material using a 12 mm ball size and at a frequency of 18 Hz. Temperature is found to have insignificant effect on the extent of breakage of MCC in both bulk milling and single particle impact testing. Milling and single particle impact experiments have both shown that MCC is more susceptible to breakage with increasing strain rate. In conclusion, the single impact tests could be, used successfully for predicting the bulk milling behavior of the material, as shown in the case of MCC

**[27] PHASE TRANSFORMATION AND PERCOLATION EFFECT IN  $PbO_2-1/12Ag(2)O-xC$  SYSTEM**

Li D. Zhang ZD. Geng DY. Li WF. Song XP. Qiao GW. Wang YZ. Solid State Communications. 125(9):493-497, 2003.

The structure and electronic transport properties have been investigated for composite  $PbO_2-1/12Ag(2)O-xC$  (0 less than or equal to x less than or equal to 2) materials prepared by mechanical milling in an O-2 atmosphere. The solid-state reactions result in the occurrence of abundant phase transformations, when the graphite content increases. With the phase transformations occurring, the percolation effect is observed for the electronic transport of  $PbO_2-1/12Ag(2)O-xC$  system, while its conductivity undergoes an insulator-metal transition. The percolation threshold is determined by DC conductivity measurement and analysis of X-ray diffraction patterns

**[26] SYNTHESIS AND CHARACTERIZATION OF NANOCRYSTALLINE CU-AL COATINGS**

Lau ML. He J. Schweinfest R. Ruhle M. Levi CG. Lavernia EJ. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 347(1-2):231-242, 2003

Commercially pure Cu and Al powders were blended in a 90: 10 ratio by weight and then mechanically milled in methanol or in liquid nitrogen. The milled powders, as well as as-blended (non-milled) powder, were deposited as coatings using high velocity oxygen fuel thermal spraying. Scanning and transmission electron microscopy techniques were used to investigate the microstructure of the powders and coatings. The results showed that milling of the powders in methanol induced the conversion of most of the Al into amorphous  $Al_2O_3$ , precluding the desired mechanical alloying. This experimental observation was consistent with available thermodynamic data. In contrast, cryomilling exhibited no significant oxidation and induced mechanical alloying of the powders, albeit incomplete. The non-milled powder generated a coating with a bimodal grain structure consisting of fine Cu grains and coarse Al grains. Amorphous oxide regions and coarse Al grains were observed intermixed with the finer Cu matrix in the coatings sprayed using the powders milled in methanol. Coatings based on cryomilled powders consisted primarily of equiaxed Cu grains and twinned martensite regions, with occasional inclusion of elongated amorphous  $Al_2O_3$  regions

Author e-mail Address [avc@ukc.ac.uk](mailto:avc@ukc.ac.uk)

**[25] A COMPARISON OF THE EXTENDED X-RAY ABSORPTION FINE STRUCTURE OF NANOCRYSTALLINE  $ZrO_2$  PREPARED BY HIGH-ENERGY BALL MILLING AND OTHER METHODS**

Chadwick AV. Pooley MJ. Rammutla KE. Savin SLP. Rougier A. - Journal of Physics-Condensed Matter. 15(3):431-440, 2003

We report the results of an extended x-ray absorption fine structure (EXAFS) study of a sample of  $ZrO_2$  prepared by high-energy ball milling. X-ray diffraction showed that the sample contained nanocrystals that were predominantly monoclinic with a particle size of 15 nm. The EXAFS for the sample was strongly attenuated in comparison to that for bulk monoclinic  $ZrO_2$ . This has been interpreted as the ball-milled sample containing a large level of disorder whose possible origins are discussed. In contrast, our previous EXAFS studies of nanocrystalline oxides prepared by sol-gel methods have shown that these samples contain well-ordered crystallites with grain boundaries similar to those in bulk materials. It is concluded that ball-milled samples are very different from oxide nanocrystals produced by other techniques

**[24] EVOLUTION OF STRUCTURE AND MAGNETIC PROPERTIES WITH ANNEALING TEMPERATURE IN NANOSCALE HIGH-ENERGY-MILLED NICKEL FERRITE**

Sepelak V. Baabe D. Mienert D. Schultze D. Krumeich F. Litterst FJ. Becker KD. - Journal of Magnetism & Magnetic Materials. 257(2-3):377-386, 2003

Metastable nanocrystalline nickel ferrite ( $NiFe_2O_4$ ) with crystallite size similar to 9 nm produced by high-energy milling the bulk material has been annealed at elevated temperatures to investigate the change induced in the microstructure and the magnetic properties. High-temperature X-ray diffraction, thermogravimetric measurements, Mossbauer spectroscopy, electron microscopy, and magnetisation experiments have been used for this purpose. The results show that the range of the thermal stability of the mechanically induced defects in the structure of milled  $NiFe_2O_4$  extends up to 600K. Owing to the thermally induced variability of the cation arrangement and the spin configuration, the milled  $NiFe_2O_4$  annealed at temperatures above 600 K exhibits a wide range of magnetic behaviour. The observed magnetic softening and an increase of both the saturation magnetisation and the Neel temperature with increasing annealing temperature are attributed to the cation reequilibration process and to the disappearance of the canted spin arrangement.

**[23] X-RAY AND MOSSBAUER STUDIES OF  $SM_2FE_{17}$ -XCRX MATERIALS SYNTHESIZED BY MECHANICAL ALLOYING FOLLOWED BY AN APPROPRIATE SHORT ANNEALING**



Nehdi I. Bessais L. Djega-Mariadassou C. Abdellaoui M. Zarrouk H. - Journal of Alloys & Compounds. 351(1-2):24-30, 2003

Samples of  $\text{Sm}_2\text{Fe}_{17-x}\text{Cr}_x$  ( $x = 0.5-3$ ) ball-milled and subsequently annealed at 1425 K, were studied by X-ray diffraction (XRD) using the Rietveld method coupled to Curie temperature T-c measurements and Mossbauer spectroscopy. XRD investigation have shown that for x less than or equal to 2,  $\text{Sm}_2\text{Fe}_{17-x}\text{Cr}_x$  alloys crystallize in the  $\text{Th}_2\text{Zn}_{17}$  type rhombohedral structure and in the Nd-3(Fe, Ti)(29) type monoclinic structure for  $x > 2$ . The Mossbauer studies indicate that the Cr atoms occupy 6c sites in the rhombohedral structure in all cases. For  $x = 2$ , the six (6c) Fe atoms are substituted by Cr atoms and the Fe(6c) sites disappear. The Curie temperature increases up to 447 K for  $x = 1$  and decreases to a value of 424 K for  $x = 2$ . The increase of T-c was explained by the increase of the distance between the 'dumbbell' Fe(6c) sites from 2.377 to 2.404 Angstrom. The decrease of T-c is assigned to the magnetic dilution of the  $\text{Sm}_2\text{Fe}_{17}$  lattice by substitution of strong magnetically Fe atoms by weak magnetically Cr atoms and the smaller Cr-Fe and Cr-Cr exchange coupling

#### [22] FORMATION OF NANOCRYSTALLINE STRUCTURES IN A CO-AL SYSTEM BY MECHANICAL ALLOYING AND LEACHING

Golubkova GV. Lomovsky OI. Kwon YS. Vlasov AA. Chuvilin AL. - Journal of Alloys & Compounds. 351(1-2):101-105, 2003

Phase composition of the materials obtained by mechanical alloying of system Co-Al (Al concentration ranges from 50 to 70 at.%) and removal of aluminum from such alloys was investigated by differential dissolution, X-ray phase analysis and TEM with a resolution of 0.4 nm. The intensive mechanical alloying provides formation of the nanocomposite material containing both amorphous phase  $\text{Co}_2\text{Al}_5$  and nanocrystalline particles of phase CoAl. Leaching of amorphous phase  $\text{Co}_2\text{Al}_5$  results in the amorphous cobalt containing admixtures of alumina and hydroxide. Nanocomposite amorphous phase  $\text{Co}_2\text{Al}_5$  and CoAl convert into nanocomposite amorphous Co and b.c.c. Co

#### [21] SUPERSATURATED SOLID SOLUTION OF NIOBIUM IN COPPER BY MECHANICAL ALLOYING

Botcharova E. Heilmaier M. Freudenberger J. Drew G. Kudashov D. Martin U. Schultz L. - Journal of Alloys & Compounds. 351(1-2):119-125, 2003

Alloys with both high strength and high conductivity have been produced by mechanical alloying. In the present study, copper was mechanically alloyed with 5, 10 and 20 at.% Nb using a planetary ball mill. The Cu-Nb phase diagram shows a negligibly low mutual solubility in the solid state, but high energy ball milling can largely extend the region of solid state solution. Previously, it was observed that niobium partly dissolves in the copper lattice during milling. The present investigation demonstrates that this limit can be extended to a strongly supersaturated Cu solid solution of up to 10 at.% Nb provided the appropriate mechanical alloying method is applied. The change in the powder microstructure was followed by scanning and transmission electron microscopy (TEM) as well as by X-ray diffraction (XRD) analysis. In the case of Cu-5%Nb and Cu-10%Nb a homogeneous single-phase microstructure was obtained after 30 It of milling. Elemental Nb could no more be detected, indicating the formation of a metastable supersaturated Cu-Nb solid solution

#### [20] APPLICATION OF ATOMIC FORCE MICROSCOPY IN MICROSTRUCTURE ANALYSIS OF MECHANICALLY ALLOYED $\text{Nd}_{12.6}\text{Fe}_{81.4}\text{B}_6/37.5$ vol.% $\alpha$ -Fe-TYPE NANOCOMPOSITES

Jakubowicz J. - Journal of Alloys & Compounds. 351(1-2):196-201, 2003

The microstructure of the two-phase nanocomposites  $\text{Nd}_{12.6}\text{Fe}_{81.4}\text{B}_6/37.5$  vol.%  $\alpha$ -Fe and  $\text{Nd}_{12.6}\text{Fe}_{80.9}\text{Zr}_{0.5}\text{B}_6/37.5$  vol.%  $\alpha$ -Fe magnets has been investigated by atomic force microscopy. The required structure was achieved by mechanical alloying of elemental powders and further heat treatment at different temperatures. The nanocomposites with addition of Zr are characterized by smaller grains and a narrower grain size distribution than without Zr. Differences in grain size after different conditions of experiments were clearly and easily identified by AFM. A  $\text{Nd}_{12.6}\text{Fe}_{80.9}\text{Zr}_{0.5}\text{B}_6/37.5$  vol.%  $\alpha$ -Fe nanocomposite with an average grain size of similar to 19 nm has been produced. The grains combine together in agglomerates with a size of similar to 250 nm. The AFM height mode image presentation is more accurate for agglomerate analysis, deflection for grain analysis

#### [19] ADDITION OF NANOSIZED $\text{Cr}_2\text{O}_3$ TO MAGNESIUM FOR IMPROVEMENT OF THE HYDROGEN SORPTION PROPERTIES

Bobet JL. Desmoulins-Krawiec S. Grigороva E. Cansell F. Chevalier B. - Journal of Alloys & Compounds. 351(1-2):217-221, 2003

The combination of (i) the catalytic effects of  $\text{Cr}_2\text{O}_3$ , (ii) reactive mechanical grinding (RMG), and (iii) nanosize particles allow a huge improvement in the sorption properties of magnesium. For short milling duration, the absorption kinetics were already as good as that reported for nanocrystalline (Mg+ $\text{Cr}_2\text{O}_3$ ) mixtures. The desorption process is also improved but not in a similar manner. It is assumed that the RMG of nanosized particles of  $\text{Cr}_2\text{O}_3$  results in the formation of some Cr atoms in the mixture which greatly influence the sorption behavior.

#### [18] NICKEL SULFIDE SYNTHESIZED BY BALL MILLING AS AN ATTRACTIVE CATHODE MATERIAL FOR RECHARGEABLE LITHIUM BATTERIES

Han SC. Kim HS. Song MS. Kim JH. Ahn HJ. Lee JY. - Journal of Alloys & Compounds. 351(1-2):273-278, 2003

Nickel sulfide (NiS) powders were prepared by ball milling and melting as cathode materials for a lithium rechargeable battery which was charged and discharged at room temperature (30 degreesC). The NiS powders prepared by melting were composed of several phases such as  $\text{Ni}_3\text{S}_2$ ,  $\text{Ni}_7\text{S}_6$ ,  $\text{Ni}_9\text{S}_6$ , and  $\text{Ni}_3\text{S}_4$ , as derived from XRD. In order to synthesize a homogeneous nickel sulfide (NiS) phase, ball milling (BM) was adopted. A homogeneous NiS phase was easily formed after ball milling up to 12 h under an Ar atmosphere. The ball milled NiS particles were relatively large compared to those of the



starting materials and they had a nanocrystalline structure. The initial discharge capacity of the NiS positive electrode prepared by ball milling is 580 mAh/g-NiS, at 1.4 V vs. Li/Li+. The NiS powders synthesized by ball milling show a better cycling property than NiS prepared by melting and also had a better rate capability. It exhibited 87% of its theoretical capacity at a current rate of 2C, comparable with that of 1/6C. This may be related with the small sized grains of NiS prepared by ball milling.

**[17] NANOASSEMBLY OF CARBON NANOTUBES THROUGH MECHANOCHEMICAL NANOROBOTIC MANIPULATIONS**

Dong LX. Arai F. Fukuda T. - Japanese Journal of Applied Physics Part 1-Regular Papers Short Notes & Review Papers. 42(1):295-298, 2003

Nanoassembly of multi-walled carbon nanotubes (MWNTs) in three-dimensional space is realized by positioning building blocks with nanometer-order accuracy, joining them together through van der Waals forces, and fixing them with chemical bonds through nanorobotic manipulations. Furthermore, MWNT intramolecular junctions are constructed

**[16] OXIDATION BEHAVIOR OF FINE-GRAINED SiC-B4C/C COMPOSITES UP TO 1400 DEGREES C**

Fan ZJ. Song YZ. Li JG. Liu L. Song JR. Chen JL. Zhai GT. Shi JL. - Carbon. 41(3):429-436, 2003.

Fine-grained B4C-SiC/C composites were fabricated using a ball-milling dispersion process. The oxidation behaviors of both fine-grained B4C-SiC/C composites and coarse-grained B4C-SiC/C composites at temperatures of up to 1400 degreesC were analyzed by the differential thermal analysis technique, and the surface morphology of the composites after isothermal oxidation at 800, 1200 and 1400 T was examined by scanning electron microscopy (SEM). The results indicated that fine-grained B4C-SiC/C composites had excellent oxidation resistance with self-healing properties at 1400 degreesC. A general model and mechanism for self-protection against oxidation of carbon materials were proposed

**[15] TENSILE AND CREEP BEHAVIOR OF CRYOMILLED INCO 625**

Rodriguez R. Hayes RW. Berbon PB. Lavernia EJ. - Acta Materialia. 51(4):911-929, 2003

The tensile and creep behavior of a cryomilled Inconel 625 alloy have been investigated. The microstructure is duplex with grain sizes ranging from 200 nm at the smallest to about 10  $\mu$ m at the largest. Normal work hardening is observed in uniaxial tension and the stress-strain behavior follows a power law. Deformation of this material is accomplished by dislocation motion as evidenced by TEM examination of deformed samples. The primary source of strengthening comes from grain size refinement. Additional factors such as solid solution strengthening contribute to a lesser extent. Analysis of the high temperature creep behavior reveals that cryomilled Inconel 625 exhibits characteristics of structure controlled (Class II) as well as mobility controlled (Class I) creep behavior. The true activation energy for creep for both structure controlled and mobility controlled creep exceeds the value for lattice self-diffusion in Ni. This is explained in terms of the simultaneous deformation of second phase particles along with the Ni matrix during high temperature creep deformation.

**[14] EFFECT OF MICROSTRUCTURE ON THE OXIDATION BEHAVIOR OF CU-40NI-20CR ALLOY [CHINESE]**

Cao ZQ. Niu Y. Wu WT. - Acta Chimica Sinica. 61(2):166-170, 2003

The thermogravimetric analysis of ternary Cu-40Ni-20Cr alloys with different microstructures was performed in order to study the effect of microstructure change on oxidation behavior of ternary multi-phase alloys. The results show that the casting Cu-40Ni-20Cr alloy is unable to form protective external scale of chromia, but a very irregular and thin continuous layer of chromia formed at the base of the mixed internal region, while mechanical alloying Cu40Ni-20Cr alloy is able to form an external Cr2O3 layer associated with a wide light zone depleted of chromium. The reduction in the alloy grain size obviously favors the formation of Cr2O3 layer on the ternary multi-phase alloy surface

**[13] SYNTHESIS OF BETA CARBON NITRIDE NANOSIZED CRYSTAL THROUGH MECHANOCHEMICAL REACTION**

Yin LW. Li MS. Liu YX. Sui JL. Wang JM. - Journal of Physics-Condensed Matter. 15(2):309-314, 2003

Nanosized beta carbon nitride (beta-C3N4), of grain size several tens of nanometres, has been synthesized by mechanochemical reaction processing. The low-cost synthetic method developed facilitates the novel and effective synthesis of nanosized crystalline beta-C3N4 (a = 6.36 Angstrom, c = 4.648 Angstrom) powders. The graphite powders were first milled to a nanoscale state, then the nanosized graphite powders were milled in an atmosphere of NH3 gas. It was found that nanosized beta-C3N4 was formed after high-energy ball milling under an NH3 atmosphere. After thermal annealing, the shape of the P-C3N4 changes from flake-like to sphere-like. The nanosized beta-C3N4 formed was characterized by x-ray diffraction, Fourier transformation infrared spectroscopy, and transmission electron microscopy. A solid-gas reaction mechanism was proposed for the formation of nanosized beta-C3N4 at room temperature induced by mechanochemical activation.

**[12] CHARACTERIZATION AND ELECTROCHEMICAL CELL CHARACTERISTICS OF MECHANOCHEMICALLY SYNTHESIZED AgI-AG2O-MOO3 AMORPHOUS SUPERIONIC SYSTEM**

Dalvi A. Shahi K. - Journal of Physics & Chemistry of Solids. 64(5):813-819, 2003

Mechanochemically synthesized amorphous and thermally stable xAgI(100 - x)[0.5Ag(2)O + 0.5MoO(3)] system for x = 40, 50, 60 and 70 shows high ionic conductivity of similar to 10(-2) - 10(-3) Ohm(-1) cm(-1) at room temperature. The highest ionic conductivity is achieved for 36 It milled sample containing 50 m/o AgI, which is more than three orders of magnitude higher than that of crystalline AgI at room temperature and comparable with the glassy fast ionic conductor of the same composition prepared by conventional quenching. The samples are thermally stable at least up to similar to 70degreesC with ionic transport number near unity. These amorphous samples are further characterized by distinct glass transition as well as amorphous double right arrow crystalline transition temperatures. The FT-IR spectra confirm the presence of MoO42- and Mo2O72- groups in the ball-milled systems. Investigations on galvanic cells of the type Ag|a-SiC|I-2 + C reveal that these



mechanochemically synthesized amorphous samples are stable under battery conditions and the cell performance parameters are comparable with those of earlier investigated Ag/I-2 cells prepared using glassy SICs.

**[11] INFERENCE MEASUREMENT OF SAG MILL PARAMETERS III: INFERENCE MODELS**

Apelt TA. Asprey SP. Thornhill NF. - Minerals Engineering. 15(12):1055-1071, 2002

This paper discusses inferential measurement models for semiautogenous grinding (SAG) mills. Inferential measurements of SAG mill discharge and feed streams and mill rock and ball charge levels are obtained utilising process measurements and recognised process simulation models. Inferential models of recirculating load and cyclone underflow split are also presented. Results for the mill inventories and process streams are validated against reference simulation model data. Uncertainty analyses are conducted to assess the influence of the various model parameters. Mill weight based estimates for mill inventory are shown to be the least uncertain. The results suggest that regular calibration of oversize crusher and primary cyclone feed instrumentation, regular measurement of the SAG mill discharge screen aperture, oversize crusher gap setting and process water specific gravity and careful fitting of the SAG mill discharge grate model parameters will minimise uncertainty in the inferential models.

**[10] CHARACTERIZATION AND PROPERTIES OF NANOSTRUCTURED SURFACE LAYER IN A LOW CARBON STEEL SUBJECTED TO SURFACE MECHANICAL ATTRITION**

Yong XP. Liu G. Lu K. Lu J. - Journal of Materials Science & Technology. 19(1):1-4, 2003

A nanostructured surface layer was synthesized on a low carbon steel by using surface mechanical attrition (SMA) technique. The refined microstructure of the surface layer was characterized by means of different techniques, and the hardness variation along the depth was examined. Experimental results show that the microstructure is inhomogeneous along the depth. In the region from top surface to about 40 µm deep, the grain size increases from about 10 nm to 100 nm. In the adjacent region of about 4080 µm depth, the grain size increases from about 100 nm to 1000 nm. The grain refinement can be associated with the activity of dislocations. After the SMA treatment, the hardness of the surface layer is enhanced significantly compared with that of the original sample, which can primarily be attributed to the grain refinement.

**[9] MECHANO-SYNTHESIS OF P/M NANOCOMPOSITE HARDMETALS**

Arcuri F. Matteazzi P. - International Journal of Powder Metallurgy. 39(1):47-52, 2003

Nanophase WC/Co based hardmetals have been prepared from powders obtained by the high energy milling of used inserts, using two alternative cycles: cold isostatic pressing/sintering and cold isostatic pressing/hot isostatic pressing. Their properties have been compared to those of the original inserts. This investigation shows that the procedure leads to high densities in the hardmetals with mechanical properties (hardness, fracture toughness, transverse rupture strength and abrasion resistance) comparable to those of new carbide inserts

**[8] EFFECT OF MECHANO-CHEMICAL ACTIVATION ON REACTIVITY OF CEMENT KILN DUST-FLY ASH SYSTEMS**

Babaian PM. Wang K. Mishulovich A. Bhattacharja S. Shah SR. - ACI Materials Journal. 100(1):55-62, 2003

Combinations of cement kiln dust and fly ash were used to develop cementitious material through mechanochemical activation. Mixture combinations made with two different proportions were subjected to various grinding regimes to activate the material. Properties, including particle size distribution, initial time of set, heat of hydration, and compressive strength of the new material were determined. Mechanical grinding resulted in mechanochemical activation of the material, with vibratory grinding more effective than ball mill grinding. Activation was confirmed through X-ray diffraction analysis and no correlation was found between activation and the mean particle size of the material. Although not all the properties of the material tested were comparable to those of portland cement, the results indicate the potential for significant improvement

**[7] PHASE TRANSFORMATION IN Ti-CR ALLOYS BY MECHANICAL GRINDING**

Takeichi N. Takeshita HT. Tanaka H. Kiyobayashi T. Kuriyama N. - Materials Letters. 57(8):1395-1399, 2003

The effect of mechanical grinding on phase transformation in Ti - Cr alloys with nominal compositions of TiCr<sub>2-x</sub> (x = 0, 0.2 and 0.5) was studied on phase transformation by mechanical grinding (MG). An X-ray diffraction (XRD) technique was applied for the identification of the constituent phases in the samples ground for various periods ranging from 0 to 12 h. The results indicated that the constituent phases changed from the mixture of a C15 and C14 Laves phases stable at ambient temperatures to a bcc phase stable at high temperatures such as 1643 K with an increase in grinding time. It was also found that, after 12 h of grinding, we could obtain samples composed of only a bcc phase within the detection ability of the X-ray diffraction apparatus

**[6] PERSPECTIVE OF ODS ALLOYS APPLICATION IN NUCLEAR ENVIRONMENTS**

Ukai S. Fujiwara M. - Journal of Nuclear Materials. 307(Part A):749-757, 2002

Oxide dispersion strengthened (ODS) steels are the most promising class of materials with a potential to be used at elevated temperature under severe neutron exposure environment. Leading technology development of ODS steels has been conducted at the Japan Nuclear Cycle Development Institute (JNC) particularly emphasizing fuel cladding application for fast reactors. This paper reviews the JNC's activities on ODS steel development as 'nano-composite materials'. Martensitic 9Cr-ODS and ferritic 12Cr-ODS steels have been successfully developed; Y<sub>2</sub>O<sub>3</sub> oxide particles can be controlled on a nano-scale and high-temperature properties were noticeably improved through controlling the grain boundary structure on an atomic scale. The ODS-technology development achieved in the field of fast reactors should be effectively spun off to the fusion reactor first wall and blanket structural materials to allow for safe and economical reactor design.

**[5] Ni/Fe<sub>2</sub>O<sub>3</sub> MAGNETIC COMPOSITE SYNTHESIZED BY MECHANICAL ALLOYING**

Shi Y. Ding J. Tan SLH. Hu Z. - Journal of Magnetism & Magnetic Materials. 256(1-3):13-19, 2003



Mechanical alloying of Ni and Fe<sub>2</sub>O<sub>3</sub> resulted in the formation of a disordered structure, when only broadened X-ray diffraction peaks corresponding to wustite appeared. After annealing at 500-900°C, a nanocomposite mixture of ferrite and (Ni, Fe) was formed. The investigation showed that Fe atoms concentrated in the ferrite phase, while the intermetallic structure was Ni-rich.

#### [4] MECHANOCHEMICAL NITRIDATION BY BALL MILLING IRON WITH M-PHENYLENE DIAMINE

Zhuge LJ. Yao WG. Wu XM. - Journal of Magnetism & Magnetic Materials. 257(1):95-99, 2003

The solid state synthesis reactions between elemental  $\alpha$ -Fe powder and m-phenylene diamine (C<sub>6</sub>H<sub>4</sub>(NH<sub>2</sub>)<sub>2</sub>) have been studied. The different phase transformations are found in the course of milling and during subsequent thermal annealing. The single  $\epsilon$ -Fe<sub>2</sub>-3N phase with nitrogen concentration up to 11.53 wt% was obtained after 250h ball milling. Owing to the local inhomogeneity of N content, the  $\epsilon$ -Fe<sub>2</sub>-3N phase can be formed by ball milling at room temperature, even though the sample has a low average N content. The saturation magnetization M<sub>S</sub> and coercivity H<sub>C</sub> of  $\epsilon$ -Fe<sub>2</sub>-3N are 81.4 A m<sup>2</sup>/kg and 16.28 kA/m. Thermal analysis experiments show structural stability of the  $\epsilon$ -Fe<sub>2</sub>-3N phase up to 512.0°C. On comparison with  $\alpha$ -Fe ball milled in NH<sub>3</sub> atmosphere, it is evident that the nitridation process in our experiment occurs more rapidly because more active nitrogen-containing radicals can be maintained during the milling process. It illustrates that the solid-solid reaction is more efficient than the solid-gas reaction.

#### [3] FORMATION OF FACE-CENTERED-CUBIC TITANIUM BY MECHANICAL ATTRITION

Manna I. Chattopadhyay PP. Nandi P. Banhart F. Fecht HJ. - Journal of Applied Physics. 93(3):1520-1524, 2003

This study reports a metastable hcp $\rightarrow$ fcc polymorphic transformation in elemental titanium induced by high-energy mechanical attrition in a planetary ball mill. The transformation is monitored and verified by x-ray and electron diffraction and high-resolution transmission electron microscopy. The grain size decreases and lattice parameter increases with continued milling. The phase change is gradual and accompanied by about 16% increase in volume per atom. The milling intensity and deformation mode seem crucial for the completion of the change in crystal structure. The extent and influence of both substitutional and interstitial impurities in this transformation have been assessed. It is suggested that structural instability due to negative (from core to boundary) hydrostatic pressure arising out of nanocrystallization or grain refinement, increasing lattice expansion, and plastic strain/strain rate is responsible for this hcp $\rightarrow$ fcc polymorphic transformation in titanium. Thus, the present transformation is similar in nature and genesis to those in elemental niobium and zirconium earlier reported by us

#### [2] MECHANICAL ACTIVATION OF PRECURSORS FOR NANOCRYSTALLINE MATERIALS

Heegn H. Birkeneder F. Kamptner A. - Crystal Research & Technology. 38(1):7-20, 2003.

Nanostructured materials win big scientific interest and increasingly economic meaning through their specific exceptional properties. Precursors that were compacted by pressing and sintering are normally used preparation of materials. In present work, the influence of mechanical activation by grinding on the structure as well as on compacting and sintering behavior of oxides from magnesium, aluminium and silicon has been investigated. Starting materials for each metal oxide differ in microstructure, dispersity, and porosity. The influence of mechanical activation on the destruction of crystalline structure to nanocrystalline, as well as to the amorphous stage and the compaction of powders with nano-particles, as well as structures with nanoscale pores have been compared. The possibilities of the consolidation of nanostructured materials were investigated. The mechanical activation took place in a disc vibration mill. The mechanical activated materials as well as their pressing and their sintering products were characterized by density, particle-size-distribution, specific surface, pore-structure, microstructure, and crystallite size by X-ray powder diffraction (XRD). The mechanical activation of the model-substances led, in most cases, to an improvement of the compaction properties; thus, this improvement can be achieved with subsequent sintering densities up to 98% of the theoretical density. From these experiments, generalizations transferable to other materials can be made

#### [1] NANOTUBE GROWTH DURING ANNEALING OF MECHANICALLY MILLED BORON

Fitzgearld JD. Chen Y. Conway MJ. - Applied Physics a (Materials Science Processing). 76(1):107-110, 2003

Boron powder, finely ground in a tungsten carbide ball mill in an ammonia atmosphere, has been annealed at 1200 °C in flowing nitrogen to produce small quantities of cylindrical BN nanotubes, both as isolated individuals and grouped into ropes. Thick-walled conical BN tubes are abundant in specimens annealed for longer times, and their growth was catalysed once WC debris was converted into W metal particles. Some catalytic effect of small W nanoparticles could be necessary for nanotube formation, though no tip particles have been imaged here. Given the low temperature of mechanical milling and annealing, BN growth must involve surface diffusion and solid-state reconfiguration. It could be possible to engineer desirable physical and chemical properties by exploiting the variation in cylindrical versus conical BN structures as a function of annealing time

