



**RESEAU FRANÇAIS DE
MECANOSYNTHESE**

Lettre N°94

Janvier 2003

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

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HAPPY NEW YEAR 2003 !!

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Bulletin d'adhésion 2003 / Subscription Print

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

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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 : Réseau Français de Mécanosynthèse

Lettre RFM N°94 - Janvier 2003
Corresp. : <mailto:Eric.Gaffet@utbm.fr>

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

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

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



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Congress and School Announcements
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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>

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

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

**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>

**XV. International Symposium on Reactivity of Solids:
Nov. 9. - 13. 2003**

<http://www.ISRSKYOTO.org>

All the questions are welcome to: info@ISRSKYOTO.org





PORTEFEUILLE DE TECHNOLOGIES EN LIGNE.

France Innovation Scientifique et Transfert (FIST SA) développe son service de recherche de partenaires internationale en diffusant sur son site Internet les techniques innovantes disponibles pour l'exploitation commerciale par les industriels des secteurs high-tech.

L'équipe de FIST a mis en place un service Internet qui permettra aux industriels d'accéder aux technologies récemment brevetées par le CNRS, les Institut de recherche et les Universités. Il pourra également être utilisé par les investisseurs qui recherchent des brevets à exploiter dans le développement de jeunes pousses. D'autre part tout détenteur de technologies brevetées pourra solliciter FIST et commercialiser ses innovations via le site.

Le site www.frinnov.com permettra de naviguer aisément parmi des centaines de technologies de pointe présentées dans un langage clair. Tous les domaines scientifiques seront représentés : sciences de la vie, électronique, chimie, sciences de la matière... Mises à jour régulièrement, les innovations seront accessibles par des rubriques explicites ou bien grâce à une recherche par mots clefs.

PAR LE BIAIS DE FIST, LES INDUSTRIELS POURRONT CONTACTER RAPIDEMENT LE RESPONSABLE DE LA VALORISATION COMMERCIALE DES TECHNIQUES D'INTERET, LES DETENTEURS DES TECHNOLOGIES ET LES INVENTEURS ET METTRE EN PLACE DES ACCORDS D'EXPLOITATION. CE SITE PERMETTRA A FIST DE TOUCHER UN GRAND NOMBRE D'UTILISATEURS POTENTIELS DES TECHNOLOGIES DEVELOPPEES PAR SES CLIENTS ET CREERA DE NOUVELLES OPPORTUNITES DE PARTENARIATS POUR LES LABORATOIRES DE RECHERCHE QUI SOUHAITERONT METTRE EN LIGNE LEURS NOUVELLES TECHNOLOGIES.

A PROPOS DE FIST

FIST SA, FRANCE INNOVATION SCIENTIFIQUE ET TRANSFERT, EST UNE SOCIETE DE CONSEIL AU TRANSFERT DES TECHNOLOGIES INNOVANTES, FILIALE DU CNRS ET DE L'ANVAR. L'EQUIPE DE FIST POSSEDE UNE EXPERTISE EN GESTION DE PORTEFEUILLES DE BREVETS, EN ANALYSE DU POTENTIEL DE VALORISATION DES INVENTIONS, EN NEGOCIATION DE LICENCES ET EN ACCOMPAGNEMENT DE JEUNES SOCIETES POUR TOUS LES DOMAINES DE HAUTES TECHNOLOGIES. FIST a développé un savoir-faire performant basé sur l'analyse approfondie de près de 2000 inventions et la mise en place d'un processus intégré en vue de l'exploitation industrielle des technologies innovantes. L'étendue de ses services, de l'évaluation de l'invention à la création des jeunes pousses, assure à l'inventeur un accompagnement tout au long du processus de valorisation. FIST compte parmi ses clients le CNRS, l'Institut Curie, des Universités françaises, des laboratoires de la Commission Européenne et des sociétés en création.



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

From Ch. Gras
(On the 28/10/ 2002)

POST-DOCTORAL RESEARCHERS

JOINT APPOINTMENT WITH OXFORD E-SCIENCE CENTRE
Post-doctoral Research Fellow: Grid Support (Remote Microscopy)
Grade RSII / Salary: £25,451 - £29,621 pa / Job Ref DJ02/035

The **University of Oxford** has recently won major Government funding for a core e-Science funded testbed project and seeks to appoint a Research Associate to develop Grid based software to operate a scanning electron microscope remotely in a collaboration between researchers in the department of Materials and the Oxford e-Science Centre. For further information on the project see: <http://www-em.materials.ox.ac.uk/research/remote/index.htm>

The appointee will design architecture and middleware for remote control of a scanning electron microscope, for which s/he will have or will develop expertise in GLOBUS and Web Services, and will provide Systems Administration for the IT infrastructure. The position involves interfacing with the UCSD team (<http://www.npaci.edu/Alpha/telescience.html>) and with the microscope designer JEOL.

Candidates must have a good first degree and either a higher degree or extensive experience in a scientific/engineering/computing discipline. They should also have considerable technical experience in a computing environment, knowledge of systems administration, the ability quickly to grasp and assess new software technologies, together with some knowledge of the UK e-Science initiative. Excellent communication and inter-personal skills with the ability to work with and supervise a wide variety of people are expected. Experience of using Globus and/or J2EE web services such as IBM WebSphere, and experience of real time control systems would be an advantage. Some international travel is envisaged.

The post is available initially for up to 3 years duration, although it is expected that additional funding will be sought and the prospects for extension are good. Appointment will be in the range indicated above, dependent on qualifications and experience. However, if applicants with sufficient qualifications and experience are not forthcoming, an appointment in the RSIA range (£18,265 - £27,339) may be considered with a commensurate reduction in duties and responsibilities.

Before submitting an application, candidates should obtain [<DJ02-035.pdf>](#) further particulars from the website or from the Deputy Administrator (Academic), Department of Materials, University of Oxford, Parks Road, Oxford OX1 3PH (email: posts@materials.ox.ac.uk), or telephone 01865 273750, quoting reference DJ02/035. The closing date for applications is 15 November 2002 and interviews are planned for 29 November 2002.

See also <http://www.ox.ac.uk/staff.html>

FROM marc.descamps@univ-lille1.fr
16/09/2002

Le Laboratoire de Dynamique et Structure Des Materiaux Moleculaires (LDSMM)
UMR CNRS 8024 -Université LILLE1

recherche -de toute urgence (curriculum à envoyer pour le 24 septembre)

• un post doc non français,these realisée en dehors de la france , pour 6mois, position ATER.

salaires: environ 1500 euros par mois.

thématique: mecanosynthèse de materiaux organiques et substances à interet
pharmaceutique

• un chercheur non français senior (curriculum pour le 5 Octobre) sur une position de 3 mois ou de 6 mois
en mecanosynthèse de matériaux organiques et substances pharmaceutiques

CONTACT marc.descamps@univ-lille1.fr

tel:33 (0) 3 20 43 49 79

From Ch. Gras
(On the 19/08/2002)

POST-DOCTORAL POSITION IN POWDER DIFFRACTION AT IPNS



Lettre RFM N°94 - Janvier 2003
Corresp. : <mailto:Eric.Gaffet@utbm.fr>

Argonne National Laboratory, one of the nation's premier scientific research and development organizations, located 20 miles southwest of Chicago, is now looking to fill a post-doctoral position in the Intense Pulsed Neutron Source (IPNS) Division, a facility dedicated to materials science research using neutron scattering techniques.

The successful applicant will join the General Purpose Powder Diffractometer (GPPD) instrument team at IPNS, participate in user-initiated as well as independent scientific programs, and join in commissioning the upgraded GPPD (to be completed in 2003). Ongoing scientific activities include: (1) crystal chemistry and structure-property relationships in catalytic materials; (2) crystallographic and dynamics studies of occluded zeolites - particularly directed at guest-framework interactions; and (3) residual strain/crystallographic texture measurements in alloys and composites, including strain and orientation distribution function determinations. A PhD degree awarded within the last three years in a discipline such as chemistry, materials science or physics is required. Highly desirable is a motivated scientist with a strong background in diffraction and crystallography, including familiarity with x-ray and neutron scattering, powder diffraction and structure/property relationships.

We welcome applications from candidates who can contribute to our EEO/Affirmative Action goals. Interested candidates should submit a curriculum vitae, three letters of recommendation, and a statement of research interests.

Susan M. Walker
Employment and Placement
Box No. IPNS-JWR
Argonne National Laboratory
9700 S. Cass Avenue, Argonne, IL 60439.
Fax: 630-252-9388, Email: employment@anl.gov.

Technical questions concerning this position should be addressed to J. W. Richardson (jwrichardson@anl.gov)

POSTDOCTORAL RESEARCH ASSISTANT IN MODELLING OF PHASE-CHANGE MATERIALS
Research Staff Grade RAIA / Salary: £17,626 - £26,491 / Job Ref: DJ02/033

Applications are invited for a postdoctoral position, available for up to three years, to model electron transport in phase-change materials. The project is funded by the Hewlett-Packard Laboratory (HPL) in Palo Alto as part of ongoing research into advanced data storage devices. The research will involve the extension of a highly-successful Monte Carlo model for film growth to ternary systems and the development of an in-house Tight-Binding code for electron transport to phase-change materials of interest to HPL. The research programme will be led by Professor David Pettifor FRS.

The successful applicant will be expected to interact closely with experimentalists at HPL, visiting Palo Alto at least every six months. Candidates should have a good first degree and completed a doctorate (by the time of appointment) in physics, materials or a related physical science subject, and should show evidence of the required modelling skills, together with knowledge of their fundamental concepts. Excellent verbal and written communication skills in English (the project language), and the ability to work to agreed time-scales, both independently and in a team, 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: DJ02/033. The closing date for applications is 9 August 2002 and interviews are planned for 30 August 2002. Further information on the Department may be found on the web-site: <http://www.materials.ox.ac.uk>

POSTDOCTORAL RESEARCH ASSISTANT IN MODELLING OF CARBON NANOSTRUCTURES
Research Staff Grade RAIA / Salary: £17,626 - £26,491 / Job Ref: DJ02/029

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 advance conventional computing and for the new field of quantum computing. See www.nanotech.org. Applications are invited for a postdoctoral position in the first-principles modelling of the atomic and electronic properties of endohedral fullerenes within single walled carbon nanotubes. This position is funded until 30 September 2004 and will be supervised by Professor David Pettifor FRS.

The successful applicant will be expected to interact closely with experimentalists performing HREM, STM, EELS and Raman characterization within this LINK programme. Candidates should have a good first degree and completed a doctorate (by the time of appointment) in physics, chemistry or materials, and should show evidence of the required first principles modelling skills. 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: DJ02/029. The closing date for applications is 02 August 2002 and interviews are currently planned for the 29 August 2002. Further information on the Department may be found on the web-site: <http://www.materials.ox.ac.uk>

POSTDOCTORAL RESEARCH ASSISTANT IN ATOMISTIC MODELLING



Lettre RFM N°94 - Janvier 2003
Corresp. : <mailto:Eric.Gaffet@utbm.fr>

RESEARCH STAFF RAIA GRADE / Salary £17,626 - £26,491 pa / Ref. DJ02/031

Atomistic Modelling: an immediate vacancy exists for a postdoctoral research appointment funded by the EU for the study of nanoscale amorphous layers in structural and functional ceramic materials. The project is part of an international collaboration of nine research institutions in Europe and the US, in which substantial intergroup communication and exchanges are expected, and is funded until January 2005.

This post will involve Grand Canonical Monte Carlo simulations of the equilibrium structure and composition of nanometre scale grain boundary films in silicon oxy-nitride, building on a very successful network model of non-stoichiometric glasses we have recently published in PRL. Ab initio simulations will be used to refine the structures and predict local electronic structures for comparison with experiment. This project will be led by Professors Adrian Sutton and David Pettifor.

The successful applicant will have a good first degree and have completed a doctorate (by the time of appointment) in materials, physics, or a related physical science subject. The post requires the ability to work both independently and collaboratively as part of a team. Candidates should show evidence of the required skills, and a considered interest in the particular field of research.

Before submitting an application, candidates should obtain further particulars 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: DJ02/031. The closing date for applications is 9 August 2002 and interviews are planned for 29 August 2002. Further information on the Department may be found on the web-site: <http://www.materials.ox.ac.uk>.

**MICROSTRUCTURAL CHARACTERISATION XRD & SEM SPECIALIST
Research Staff grade RAIA / Salary £17,626 - £22,522 pa / Ref: DJ02/012**

Applications are invited from experienced scientists in microstructural characterisation. The Materials Department Services to Industry Characterisation and Analysis Service provides a problem solving and advisory service based on microanalytical techniques to internal research units and external commercial customers. The post holder will provide a high quality, fast turnaround X-ray diffraction (XRD) and scanning electron microscope (SEM) advisory and analytical service. He/she will assist with maintenance and servicing of the Department's analytical facilities, advise on the use of these techniques and in the interpretation of data, and assist in the training of students and other researchers in SEM and XRD techniques. The post is available for three years in the first instance.

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: DJ02/012. The closing date for applications is 19 July 2002 and interviews are planned for the week beginning 29 July 2002. Further information on the Department may be found on the web-site: <http://www.materials.ox.ac.uk>

**MICROSTRUCTURAL CHARACTERISATION EPMA & SEM SPECIALIST
Research Staff grade RAIA / Salary £17,626 - £26,491 pa / Ref: DJ02/013**

The post of Microstructural Characterisation EPMA & SEM Specialist in the Department of Materials is available for three years in the first instance, to start as soon as possible. Working as part of a small team, the post holder will be involved in providing a high quality, fast turnaround optical microscopy, EPMA & SEM advisory and analytical service to both internal and external customers, assisting with maintenance and servicing of the Department's analytical facilities and advising on the use of these techniques and in the interpretation of data, and participating in the training of students and other researchers in SEM, optical microscopy and EPMA techniques.

Applicants should hold a Materials Science degree and have a detailed understanding of crystallography and a proven, high-level competence in EPMA, with at least five years of sophisticated/advanced analytical expertise. They should be well-organised, have good inter-personal verbal and written communications skills, and an awareness of the financial implications of working with industry, be able to liaise with academic users and industrial customers, and be willing, on occasions, to travel within the UK.

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: DJ02/013. The closing date for applications is 19 July 2002 and interviews are planned for the week beginning 29 July 2002. Further information on the Department may be found on the web-site: <http://www.materials.ox.ac.uk>



Périodiques / Congrès

[81] CHARACTERIZATION OF MECHANICALLY ALLOYED TI-AL-SI POWDER BLENDS AND THEIR SUBSEQUENT THERMAL STABILITY

Rao KP. Zhou JB. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 338(1-2):282-298, 2002

With the aim of producing metastable precursors for tailoring TiAl based composite, five compositional powder blends of Ti-Al-Si system, i.e. Ti₆₅Al₁₇Si₁₈, Ti₆₅Al₂₁Si₁₄, Ti₇₂Al₁₄Si₁₄, Ti₆₅Al₁₄Si₂₁ and Ti₅₈Al₂₁Si₂₁, were prepared by mechanical alloying (MA). The structural and compositional evolution during MA has been investigated using X-ray diffraction method (XRD), energy-dispersive spectrometer (EDS) and transmission electron microscope (TEM). MA achieved a significant increase in solid solubility limits. Supersaturated solid solution of Ti(Al,Si) was produced with prolonged milling time which was strongly dependent on the starting compositions of blends. No fcc phase or ordered and disordered intermetallics were produced during MA process. With higher and balanced Al and Si contents, for example Ti₅₈Al₂₁Si₂₁ blend, the time required for solid solution formation was reduced drastically. Thermal stability of the mechanically alloyed powders was systematically examined by subsequent annealing process according to the results of differential thermal analyzer (DTA) curves. From XRD patterns of the annealed blends, it was observed that annealing at temperatures just below 600 degreesC leads to crystalline phases of mainly Al₃Ti and TiSi₂, while more stable Ti-5(Al,Si)₃, (Ti,Si)Al and (Ti,Si)₃Al phases were formed at higher annealing temperatures of above 750 degreesC

[80] FORMATION OF GLASSY Ti₅₀Cu₂₀Ni₂₄Si₄B₂ ALLOY BY HIGH-ENERGY BALL MILLING

Zhang LC. Xu J. - Materials Letters. 56(5):615-619, 2002

The elemental powder mixtures with a nominal composition of Ti₅₀Cu₂₀Ni₂₄Si₄B₂ can be amorphized under high-energy ball milling. A well-defined glass transition before crystallization appears in the obtained amorphous phase during heating in a differential scanning calorimeter. A wide supercooled liquid region $\Delta T(x)$ is available to be about 57 K. Crystallization of the ball-milled amorphous phase was performed through one step to form the metastable (Ni, Cu)Ti phase with cubic structure and an unknown phase, followed by subsequent grain growth. The reduced glass transition temperature of the alloy was determined to be 0.56.

[79] FABRICATION OF NANO-SIZED TiO₂ POWDER VIA AN ETHYLENE GLYCOL ENTRAPMENT ROUTE

Lee SJ. Lee CH. - Materials Letters. 56(5):705-708, 2002

Pure and nano-sized TiO₂ powder was synthesized by an ethylene glycol entrapment route. Titanium isopropoxide was dissolved in liquid-type ethylene glycol without any precipitation. At the optimum amount of the polymer, the titanium ions are dispersed in solution and a homogeneous polymeric network was formed. The stable precursor was crystallized at low temperature and the crystalline powder was planetary ball milled to nano-size.

[78] LITHIUM STORAGE PROPERTIES OF NANOCRYSTALLINE Ni₃Sn₄ ALLOYS PREPARED BY MECHANICAL ALLOYING

Lee HY. Jang SW. Lee SM. Lee SJ. Baik HK. - Journal of Power Sources. 112(1):8-12, 2002

Nanocrystalline Ni₃Sn₄ alloy powders prepared by high energy ball milling are examined as an anode for lithium-ion batteries. Ex situ X-ray diffraction (XRD) and differential capacity plots show that crystalline Ni₃Sn₄ has a low affinity for lithium. In the case of well-developed nanocrystalline Ni₃Sn₄, the lithium atoms reversibly react with tin atoms in the grain boundaries with no capacity fading for extended cycling.

[77] EFFECT OF MECHANICAL ALLOYING ON THE SOLID STATE REACTION PROCESSING OF Ni-36.5 AT.% AL ALLOY

Chung CY. Zhu M. Man CH. - Intermetallics. 10(9):865-871, 2002

The solid state reaction of the mechanical alloyed NiAl elemental powders has been studied using differential scanning calorimetry, X-ray diffraction and transmission electron microscopy. NiAl compacts containing 36.5 at.% Al are within the shape memory range and were prepared by mechanical alloying for various periods. In specimens that were mechanical alloyed for less than 2 h, the grain sizes of Ni and Al were above 200 and 100 nm respectively and a two steps solid-state reaction took place during heating. In specimens which were mechanical alloyed for longer than 2 h, the corresponding grain sizes of Ni and Al were below 200 and 100 nm and in this case, a three step solid state reaction took place during heating. Thus the initial state of the alloy system before heating plays an important role in the phase reactions.

Author e-mail Address schubert@epw.ifam.fhg.de

[76] EFFECTS OF HIGH ENERGY MILLING ON DENSIFICATION BEHAVIOUR OF MO-SI POWDER MIXTURES DURING PRESSURELESS SINTERING

Schubert T. Bohm A. Kieback B. Achtermann M. Scholl R. - Intermetallics. 10(9):873-878, 2002

The present paper reports on basic investigations of the controlled reaction sintering of MoSi₂-based materials, including particle reinforced composites. It is shown, that certain amounts of pre-formed silicide phase in the powder mixtures are useful to achieve high densities by pressureless sintering of green compacts. This partial phase formation of α -MoSi₂ can already take place during the mechanical treatment of the elemental powder mixtures by milling. In this context the role of volume changes linked with the phase formations is discussed.

[75] MGAL₂O₄ SPINEL PHASE DERIVED FROM OXIDE MIXTURE ACTIVATED BY A HIGH-ENERGY BALL MILLING PROCESS

Kong LB. Ma J. Huang H. - Materials Letters. 56(3):238-243, 2002



High-energy ball milling process has been applied to MgO and Al₂O₃ system. The reaction of MgO and Al₂O₃ is significantly enhanced by the high-energy ball milling process. MgAl₂O₄ spinel phase with an average grain size of about 100 nm is formed at 900 degreesC from the mixture of MgO and Al₂O₃ after milling for 12 h. Dense MgAl₂O₄ ceramics with 98% of the theoretical density and grain size of 2-5 µm can be obtained by sintering the milled mixture at 1550 degreesC for 2 h.

[74] REACTIVE SYNTHESIS OF TITANIUM MATRIX COMPOSITE POWDERS

Joshi PB. Marathe GR. Murti NSS. Kaushik VK. Ramakrishnan P. - Materials Letters. 56(3):322-328, 2002

Mechanical activation of materials has emerged as a promising alternative to conventional thermally activated processes. The present work deals with the attrition of titanium powders to produce Ti-TiO₂ composite powders by reactive milling (RM). The phase transformation/new phases formed during the course of milling have been characterized by sophisticated techniques such as X-ray diffraction (XRD), electron spectroscopy for chemical analysis (ESCA) and differential thermal analysis (DTA). The microhardness measurements showed progressive increase in hardness with increasing milling time.

[73] LOW TEMPERATURE FORMATION OF YTTRIUM ALUMINUM GARNET FROM OXIDES VIA A HIGH-ENERGY BALL MILLING PROCESS

Kong LB. Ma J. Huang H. - Materials Letters. 56(3):344-348, 2002

Mechanochemical process has been applied to the mixture of Y₂O₃ and Al₂O₃ in order to increase its reactivity. Yttrium aluminate garnet (YAG, Y₃Al₅O₁₂) has been synthesized at 1000 degreesC from the milled mixture, which is significantly lower than that required by the conventional solid-state reaction process and comparable with that required by most of the chemical-based synthesis routes. The reduced formation temperature of the YAG phase is believed to be beneficial from the refined Y₂O₃ and Al₂O₃ as a result of the high-energy ball milling.

[72] FORMATION OF Ba₂Ti₉O₂₀ CERAMICS FROM EDTA-GEL-DERIVED POWDERS

Wang HW. Chung MR. - Materials Chemistry & Physics. 77(3):853-859, 2003

Reactive powders have been prepared by the ethylenediaminetetraacetic acid-gel (EDTA) method. Single-phase Ba₂Ti₉O₂₀ and approximate to 98% dense ceramic are readily obtained by sintering the fine powders at 1275 degreesC for 3 h. Powder derived from EDTA-gel precursor results in Ba₂Ti₉O₂₀ and BaTi₄O₉ mixed phases when sintering was performed below 1200 degreesC, and only become single Ba₂Ti₉O₂₀ phase above 1250 degreesC. However, single-phase Ba₂Ti₉O₂₀ were obtained at 1150 degreesC if ball-milling process was applied to this powder. This is considered to be due to the deagglomeration and improved mixing effects of ball-milling. One batch, the EDTA-gel precursor with ethylene glycol, showing improved decomposition behavior, results in single Ba₂Ti₉O₂₀ phase at 1200 degreesC. It was observed that the surface areas of derived powders are in the order of precursors from EDTA-ball-milled (13M) > EDTA-ethylene glycol (EG) > EDTA. The formation of single Ba₂Ti₉O₂₀ phase is enhanced by the high surface area. The microwave properties of Ba₂Ti₉O₂₀ ceramics obtained from three EDTA-derived powders are discussed and compared

[71] MICROSTRUCTURE CHARACTERIZATION OF HIGH ENERGY BALL-MILLED NANOCRYSTALLINE V₂O₅ BY RIETVELD ANALYSIS

Dutta H. Pradhan SK. - Materials Chemistry & Physics. 77(3):868-877, 2003

Nanocrystalline vanadium pentoxide has been processed with high energy planetary ball-mill at two different ball-to-powder mass ratio (BPMR) for different lengths of time (0-35 h). Microstructure characterization of ball-milled samples is made by employing Rietveld's X-ray powder structure refinement analysis in terms of lattice imperfections. The particle sizes and lattice strain values are found to be anisotropic. The plastic deformation induced in the process of ball-milling caused a massive decrease in particle size and increase in lattice strain within 15 min of milling, and a saturation of particle size of similar to 6.5 nm is reached within 10 h of milling. The shape of milled particle is presumed to be parallelepiped and the influence of BPMR predominated in initial size reduction. The initial change in intensities of two major X-ray reflections is found to be due to insignificant change in atomic coordinates on the event of severe high energy impact. The peak broadening at initial stage of milling is due to fracture of particles, but later (after particle size saturation), dominated by increase in anisotropic lattice strains particularly along [0 0 1]

[70] STRUCTURE OF AMORPHOUS P-SE PREPARED BY MECHANICAL ALLOYING

Itoh K. Misawa M. Fukunaga T. - Journal of Non-Crystalline Solids. 312:561-565, 2002.

The short- and medium-range structures of amorphous P_{1-x}Se_x (x = 0.6, 0.8) synthesized by mechanical alloying (MA) have been investigated by time-of-flight neutron diffraction. The first diffraction peak, near Q = 12 nm⁻¹ in the structure factor, S(Q), which is strongly related to the medium range order, is smaller in height and broader in width than that for amorphous P_{1-x}Se_x (x = 0.6, 0.8) prepared by liquid quenching. The formation of the medium-range order is restrained when the amorphous P-Se is synthesized by MA

[69] MECHANICALLY DRIVEN SOLID STATE AMORPHIZATION REACTION OF BALL-MILLED Nb₅₀Zr₁₀Al₁₀Ni₁₀Cu₂₀ POWDERS AND THE EFFECT OF ANNEALING

El-Eskandarany MS. Matsushita M. Inoue A. - Journal of Non-Crystalline Solids. 312:622-627, 2002.

A single amorphous phase of Nb₅₀Zr₁₀Al₁₀Ni₁₀Cu₂₀ alloy powders was synthesized by ball milling the elemental powders at room temperature. During the first stage of milling, the atoms of Zr, Al, Ni and Cu migrated and diffused into the Nb (base material) lattice to form a bee supersaturated solid solution that transformed into an amorphous phase with the same composition upon post-annealing at 725 K. As the milling time increased, the powders were subjected to continuous lattice imperfections that lead to gradual changes in the free energy, so that the solid solution phase is transformed to an amorphous



phase. At the final stage of milling (720 ks) the crystallization temperature and the enthalpy change of crystallization for the obtained amorphous alloy were 963 K and -8.44 kJ mol⁻¹, respectively

[68] PHASE TRANSFORMATION AND MAGNETIC PROPERTIES OF ND-FE-V-NB ALLOYS PREPARED BY MECHANICAL ALLOYING

Cui BZ. Sun XK. Sui YC. Geng DY. Zhang ZD. - Journal of Magnetism & Magnetic Materials. 250(1-3):212-218, 2002
The phase transformation and magnetic properties of the NdFe_{11.35-x}VxNb_{0.65} (x = 0-1.35) alloys prepared by mechanical alloying (MA) have been studied. Phases of 2:17, 1:7 and 1:12 are formed in the as-annealed alloys with x = 0, 0.3-0.7 and 1.0-1.35, respectively. With increasing V content in the NdFe_{11.35-x}VxNb_{0.65} (x = 0.3-0.7) alloys, the Curie temperatures of the matrix 1:7 phases monotonically increase. Upon nitrogeneration, with increasing V content, the intrinsic coercivity $\mu(0)H(c)$ of the nitrided alloys monotonically increases. The increase becomes sharp at x = 1.0 due to the formation of a great amount of Nd(Fe, V)(12)N- δ . The remanence J(r) and the maximum magnetic energy product (BH)(max) of the nitrided alloys monotonically decrease with increasing V content. The 1: 12 phases are formed in all the NdFe_{11-x}NbxV₁ (x = 0, 0.3, 0.65 1.0) alloys annealed at 850degreesC for 20 min. With increasing Nb content, the crystallization of the 1: 12 phase is improved and the relative volume fraction of the 1: 12 phase tends to be higher. While $\mu(0)H(c)$ of the nitrided alloys monotonically increase, and J(r) and (BH)(max) of the nitrided alloys monotonically decrease. With increasing Nb content in the nitrided alloys, the average grain size of alpha-Fe decrease while the nitrogen content increases

[67] SYNTHESIS AND CHARACTERIZATION OF NANOSIZED POWDERS OF YTTRIA-DOPED ZIRCONIA

Tadokoro SK. Muccillo ENS. - Journal of Alloys & Compounds. 344(1-2):186-189, 2002
Zirconia-3 mol% yttria nanosized powders have been prepared following a chemical route. The aim of this work is to optimize the techniques of solid solution synthesis and processing to obtain nanophase sintered ceramics without using milling or other special procedures. To ascertain this, several characterization techniques have been used. The electrical resistivity of polycrystalline specimens was compared to that of a single crystal of similar chemical composition. The main results show that a monomodal distribution of pore sizes has been obtained. Total stabilization of the tetragonal phase and nanophase sintered ceramics with relative densities of 98% were prepared by sintering at temperatures lower than 1200 degreesC. The main conclusion is that the optimization of the synthesis and processing techniques play a key role for improvement of chemical and physical properties of solid electrolytes prepared by chemical techniques

[66] SOLUBILITY STUDY OF FE_{0.95}PB_{0.05} ALLOY PREPARED BY HIGH ENERGY BALL MILLING

Nunes E. Passamani EC. Larica C. Freitas JCC. Takeuchi AY. - Journal of Alloys & Compounds. 345(1-2):116-122, 2002
In this work Pb_{0.05}Fe_{0.95} nanostructured alloy was prepared by high energy ball milling under argon atmosphere. X-ray diffraction (XRD), differential scanning calorimetry (DSC) and Mossbauer measurements were performed in the milled samples to study the alloying process and the structural variation with milling time. XRD and DSC results of Pb_{0.05}Fe_{0.95} alloy indicate the presence of free Ph metal for milling times shorter than 20 h. On the other hand, XRD and Mossbauer results show that the final Pb_{0.05}Fe_{0.95} alloy (sample milled for 310 h) is constituted by four nanocrystalline phases: b.c.c.-Fe, possibly with Ph impurities trapped in it, Fe/Pb phase and finally two Fe oxide phases (Fe_{1-x}O and Fe₃O₄). The relative areas corresponding to these phases in the Mossbauer spectrum are 58% for the b.c.c.-Fe, 12% for the b.c.c. Fe/Pb alloy, and 30% for the Fe oxide phases. The Fe/Pb phase, with approximate composition Fe_{0.71}Pb_{0.29}, is being reported for the first time and it is also shown that this phase is thermally stable up to 650 K.

[65] EFFECT OF EXCESS PBO ON MICROSTRUCTURE AND ELECTRICAL PROPERTIES OF PLZT7/60/40 CERAMICS DERIVED FROM A HIGH-ENERGY BALL MILLING PROCESS

Kong LB. Ma J. Huang H. Zhang RF. - Journal of Alloys & Compounds. 345(1-2):238-245, 2002
A high-energy ball milling process has been used to prepare lead lanthanum zirconate titanate (PLZT7/60/40) powders with different excess PbO levels. The microstructure development and the electrical properties of the PLZT ceramics derived from the synthesized powders were investigated. The present results demonstrate that an excess PbO exhibits a negative influence on the electrical properties of the PLZT ceramics, even though it promoted the densification of the PUT ceramics at low temperature. Fully dense PUT ceramics with a piezoelectric coefficient (d₃₃) of 702 pC/N were achieved by sintering the powders without excess PbO at 1150 degreesC for 1 h. It can be concluded that excess PbO is not necessary for lead-containing ceramics produced by a high-energy ball milling process.

[64] MECHANICAL ALLOYING OF A NEW PROMISING THERMOELECTRIC MATERIAL, SB₃ZN₄

Izard V. Record MC. Tedenac JC. - Journal of Alloys & Compounds. 345(1-2):257-264, 2002
We report in this paper a study of the mechanical alloying (MA) process for a high-performance p-type thermoelectric material, Sb₃Zn₄. Three samples belonging to the homogeneity range of Sb₃Zn₄ and one with a slightly higher content in zinc have been prepared by MA. From systematic analyses of the latter one, the mechanism of formation for Sb₃Zn₄ has been studied. The powders have been characterised by X-ray diffraction, scanning electron microscopy and differential scanning calorimetry. The results lead us to conclude that MA powder of Sb₃Zn₄ is produced by solid state diffusion. With the experimental conditions used in this work, single phase material can not be obtained directly from MA, its formation requires a further heating at low temperatures. Since this heating occurs in the industrial processes for solidification and net-shaping to thermoelectric devices, MA seems to be a good alternative method of preparation for Sb₃Zn₄.

[63] HYDROGEN SORPTION PROPERTIES OF THE NANOCOMPOSITES MG-MG₂Ni_{1-x}XFEX

Bobet JL. Grigorova E. Khrussanova M. Khristov M. Radev D. Peshev P. - Journal of Alloys & Compounds. 345(1-2):280-285, 2002



The hydrogen sorption properties of the composites 85 wt.% Mg-15 wt.% Mg₂Ni_{1-x}Fe_x (x=0, 0.1 and 0.3) prepared by high-energy ball milling were studied. It was shown that the presence of a Mg₂Ni_{1-x}Fe_x additive significantly improved the kinetics of hydrogen absorption and desorption of magnesium, the absorption capacity remaining sufficiently high even at temperatures between 423 and 573 K. The catalytic effect of the Mg₂Ni_{1-x}Fe_x intermetallics was explained as due to the formation of a second hydride, Mg₂NiH₄, to the appearance during the mechanical alloying of defects facilitating the formation of hydride nuclei and to the presence of Ni and Fe clusters on the surface of nanocomposite particles

[62] STRUCTURE AND MAGNETIC PROPERTIES OF MECHANICALLY ALLOYED SMZR(CO,FE) NANOPHASE HARD MAGNETS

Gallagher K. Le Gouil A. Venkatesan M. Coey JMD. - IEEE Transactions on Magnetics. 38(5 Part 1):2916-2918, 2002

In this paper, Sm_xZr_y(Co_{1-x}Fe_x)_{100(-y-z)} (nominal composition) powders, where 13 less than or equal to z less than or equal to 15, 0 less than or equal to y less than or equal to 5, and 0 less than or equal to x less than or equal to 0.5, were prepared by mechanical alloying and annealed using an optimized procedure. As-milled powders were amorphous, or showed some small alpha-Fe crystallites, depending on Fe composition. Initially, powders with y = 0 were prepared with a view to optimize the Fe content, and it was found that x = 0.1 was the best Fe content for all Sm compositions. Samples without Zr showed a mixture of Th₂Ni₁₇ and Th₂Zn₁₇ Structure types, as well as SmCo₅ and mu(0)H(c) was high as 1.6 T. Powders were then made varying x and y, and keeping z = 0.1. These samples also showed a mixture Of Sm₂CO₁₇ phases along with SmCo₅, dependent on Zr content. Room-temperature coercivities greater than 2.0 T were observed. Enhanced remanence (sigma(t)/sigma(s) greater than or equal to 0.65) Is achieved in all compositions, indicating intergrain exchange coupling among the fine grains

[61] EFFECT OF FE, CU, ZR, AND TI ON THE MAGNETIC PROPERTIES OF SMCO-1 : 7 MAGNETS

Venkatesan M. Rhen FMF. Gunning R. Coey JMD. - IEEE Transactions on Magnetics. 38(5 Part 1):2919-2921, 2002

The effect of Fe, Cu, Zr, and Ti substitution on magnetic properties of SmCo 1 : 7 magnets at temperatures up to 500 degreesC were studied. As-milled amorphous Sm(Co,Fe,Cu,Ti-Zr)(7) magnets crystallized in the TbCu₇-type structure after annealing at 750 degreesC for 2 h. A room-temperature intrinsic coercivity of around 2.5 T is obtained for compositions SmCo_{6.4} Zr_{0.2} Ti_{0.4} and SmCo_{6.4}Fe_{0.1}Zr_{0.1}Ti_{0.4}. The high coercivity of the ball-milled SmCo_{7-x}Ti_x magnets is attributed to the strong pinning of the walls of the "interaction domains" by the network of grain boundaries between the nanocrystalline grains. The virgin curve of the ball-milled samples is suggestive of strong domain wall pinning

[60] MICROSTRUCTURE EVOLUTION DURING DENSIFICATION OF THE NANO-STRUCTURED W-NI-FE COMPOSITE POWDERS

JL Fan, BY Huang, XH Qu - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 2301-2304 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

Systematic investigations were carried out on microstructure changes during densification of the nano-crystalline powder produced by mechanical alloying (MA). The results have shown, the nano-crystalline powder went through a series of phase and microstructure changes. They include phase and structure changes and slower grain growth at lower temperature, accelerated grain growth and grain coarsening with increase of temperature, rapid tungsten grain spherical microstructure transformation and double grain coarsening during liquid phase sintering, and tungsten solubility changes in the second phase. A significant grain growth occurred during consolidation process. Fully densification occurs at the price of speedily grain-coarsening, double-rearrangement of tungsten grain during liquid phase sintering.

[59] PROPERTIES OF MECHANICALLY ALLOYED CU-SN POWDERS AND THE SINTERED COMPACTS

W Okamura, M Kohzu, S Tanabe, K Higashi, K Yamadori - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 2309-2312 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

Cu-Sn alloy has been utilized for a long time. Even today, it is the most practical material after Fe-based one as the metal bonds of diamond sintered compact in the powder metallurgy materials. Mechanical alloying from elemental powder mixtures, which is one of the most widespread techniques, enables its mechanical properties extremely improved and sintering temperature decreased. In this study, Cu and Sn powders were mechanically alloyed under various conditions in order to improve the quality in metal bonds. These MA powders were analyzed with scanning electron microscope (SEM), X-ray diffraction (XRD), and particle size analyzer. The compacts of these MA powders were sintered by hot sizing. The measurements were conducted on traverse rupture strength (TRS) and hardness to these compacts. Moreover, additional experiments were performed after adding Co to the composition to enhance the hardness.

[58] THE EFFECTS OF MECHANOCHEMICAL TREATMENT ON THE PREPARATION OF CARBON NANOTUBE BY CATALYTIC CVD PROCESS

H Ryu, HK Yu, WK Choi, HS Yoon, KS Kim, B Lee, F Saito - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 2335-2337 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

Effect of mechanochemical treatment of catalysts and supports mixture on formation of carbon nanotubes has been investigated by XRD, SEM, TEM, etc. The structure of Al(OH)(3) is transformed into an amorphous state within about 120



minutes grinding by mixer mill under dry condition. With the unground and ground mixtures, carbon nanotubes have been prepared by the catalytic CVD process using C₂H₂ or CH₄ gas. As a result the shape and yield of carbon nanotubes gone through the process of mechanochemical treatment are different from those of untreated carbon nanotubes. It was verified that the effects of mechanochemical activation of mixtures showed a consequence of better characteristics of carbon nanotubes.

[57] MICROSTRUCTURE EVOLUTIONS IN MO-HfC COMPOSITE MATERIALS DURING HIP

K Nakai, K Watanabe, S Kobayashi, H Kurishita, Y Ohmori - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 2567-2570 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

Processes of Mo grain growth and carbide formation during hot isostatic pressing (HIP) in Mo-HfC composite materials prepared by mechanical alloying (MA) method have been investigated by optical and transmission electron microscopies. The Mo grain growth during HIP is suppressed by the precipitation of (Mo,Hf)₂C, and by increasing MA duration. The precipitation of (Mo,Hf)₂C particles depends on the grain boundary diffusion of Hf. Alpha-Mo₂C particles are also formed within a grain homogeneously by rapid diffusion of carbon.

[56] ON THE MECHANICAL BEHAVIOUR OF FINE-GRAINED 5056 ALUMINUM ALLOY PRODUCED BY EQUAL-CHANNEL ANGULAR PRESSING

K Kitagawa, K Higashi, Y Demura, V Patlan, A Vinogradov, A Washikita - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 2595-2598 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

The mechanical properties of fine-grain Al-Mg alloy (5056) produced by severe plastic deformation through equal channel angular pressing (ECAP) are assessed in terms of Vickers microhardness, tensile and fatigue behavior. Several samples with various average grain sizes were prepared by annealing of the ECAP samples at different temperatures. Comparing to conventional commercial alloy with the same composition, the ECAP material exhibits a fairly high proof stress over 400MPa, and low-cycle fatigue strength under constant stress amplitude. However, its fatigue life under strain-controlled conditions is shorter than that of O-tempered coarse-grain specimens. Hall-Petch relation was observed between proof stress and grain size. It is found that the mechanical properties after ECAP can be improved by annealing at moderate temperature after fabrication. Such annealing is supposed to recover the grain and grain boundary region, which has been heavily distorted during processing.

[55] MECHANICAL ALLOYING AND ELECTRONIC SIMULATION OF MG-BASED HYDROGEN ABSORBING MATERIALS

ZX Guo, M Bououdina, Y Song, C Shang - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 2917-2920 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

Mg-8 at % TM systems (TM is a transition metal, TM = Ni, Cu, Al and Nb) have been synthesised by Mechanical Alloying (MA). X-ray diffraction (XRD) analysis reveals the formation of new phases: Mg₂Ni and Mg₂Cu phases in the case of Mg-Ni and Mg-Cu mixtures; an Mg₁₇Al₁₂ phase for the Mg-Al system; and a (Nb, Mg) solid solution for the Mg-Nb mixture. Rietveld analysis indicates a volume contraction of Mg after MA, in agreement with atomic size reduction of the transition metal. Scanning Electron Microscopy (SEM) carried out on milled powders clearly indicate an important particle size reduction, compared with the as-received powders. The crystal structure was discussed with the nature of the transition metal. Binding energies of the systems were evaluated by first-principles electronic simulations. Among the alloying elements studied, it is noted that the Mg-Al system is the most stable, followed by Mg-Ni and Mg-Cu. However, mixing Mg with a bcc metal, such as Nb, was found to have a relatively low stability.

[54] AMORPHIZATION OF B2 INTERMETALLIC COMPOUND BY SEVERE PLASTIC DEFORMATION

K Tsuchiya, H Nakayama, ZG Liu, M Umemoto, K Morii, T Shimizu - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 39-42 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

Changes in the fine structures was investigated through the course of partial amorphization by cold rolling in TiNi B2 intermetallic compounds. Samples of Ti-50.0 at%Ni and Ti-50.2 at%Ni alloys were cold rolled to various reductions (0 similar to 70 %). DSC measurements revealed that the peaks corresponding to martensitic transformation disappeared after cold rolling to 30 similar to 40 % reduction. This suggests that the stress field introduced by the lattice defects suppress the transformations. Optical and scanning electron microscopic observations were carried out on the rolling plane and on the longitudinal section. Amorphous bands taken from longitudinal section using TEM inclined about 35 degrees similar to 50 degrees from rolling direction. This angle is in a good agreement with that of the shear band observed by optical and scanning electron microscopy. These results suggest that the a part of shear bands is the preferential site for the crystal-to-amorphous transformation by cold rolling.

[53] PROCESSING AND MECHANICAL PROPERTIES OF AUSTENITIC STAINLESS STEELS WITH GRAIN REFINEMENT BY MECHANICAL MILLING



R Ishibashi, H Arakawa, H Doi, Y Aono - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 151-154 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

304 type austenitic stainless steels with high strength were manufactured by grain refinement. The basic process was consolidation of mechanically milled powders with nano-meter-sized grains. A major objective was suppression of grain growth during the consolidation process at elevated temperatures. Dispersion of ZrC particles suppressed grain growth and improved strength. Furthermore, use of intermediate temperature holding during hot isostatic pressing (HIP) seems to be effective for the control of microstructure. This probably results from combined effect of carbide precipitation and microstructure transition in mechanically milled powders during elevated temperature processing. Use of HIP with subsequent hot forging was able to lower the consolidation temperatures in comparison to the only-HIP case. Ultra fine grains resulted from ZrC dispersion and the mentioned processing method. The manufactured material had ultra fine grains with tensile strengths higher than 1000 MPa. It also had Charpy absorbed energy of about 150 J which is almost the same as 304 wrought steels.

[52] ALTERATIONS TO POLYURETHANE MATERIAL BY THE ADDITION OF MECHANICALLY ALLOYED RUBBER

WJD Shaw - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 159-162 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

The mechanical alloying, MA, technique was used to process rubber, NR, by itself and also in conjunction with other additives such as silicon. This processed material was added to polyurethane, PUP, resin and allowed to cure. Comparisons were conducted when regular unmodified rubber particles of two, different sizes were added to the resin as well as that of MA processed rubber. These comparisons include both mechanical properties and chemical resistance behaviour. Both fractography and Fourier transform infrared, FTIR, analysis show major alterations in the material formed. The regular rubber particles are held in the PUR by adhesion, thus interacting as a regular filler. The MA rubber particles undergo a chemical reaction as the resin cures and direct chemical bonding between the rubber and the PUR occurs. This chemical reaction combined with the fine particle size resulting from the MA processing favorably alters the material and provides a number of engineering applications for this new material.

[51] INTEGRATED MATERIAL SYSTEM FOR BULK NANOCRYSTALLINE CERAMICS

H Kimura - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 163-166 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

This article describes the integrated P/M material system that consists of the amorphous powder synthesis via reaction milling, the pore free consolidation of the nanocrystalline ceramics by intelligent sintering and the model mechanics for bulk mechanical characteristics. Emphasis here is placed on the chemistry necessary for the design and development of the bulk nanocrystalline ceramics with outstanding mechanical properties. The mechanical alloying based on the non-equilibrium reaction ball milling can produce the solid state amorphization of oxide and covalent ceramics such as PSZ-Al₂O₃ and SiC. The instrumented pulse electric discharge consolidation can be used to prepare a fully dense nanocrystalline SiC product without the additive at a considerably low temperature of 1760 degreesC. The near-net-shape forming into the disk with a boss can be achieved via high-speed superplastic forging of highly dense nanocrystalline PSZ-20mol%Al₂O₃ with cubic and tetragonal phases.

[50] CONTROL OF CRYSTALLINITY AND CRYSTAL STRUCTURE IN HYDROXYAPATITE AND RELATED CALCIUM PHOSPHATES BY MECHANICAL GRINDING AND SUBSEQUENT HEAT-TREATMENT

Y Umakoshi, T Nakano, A Tokumura - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 217-220 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

Variation in crystallinity and crystal structure of calcium deficient apatite (DAP) and dicalcium phosphate dihydrate (DCPD) was examined during mechanical grinding (MG) and subsequent annealing using X-ray diffraction and FT-IR spectrum, being compared with hydroxyapatite (HAp) and related calcium phosphates. The crystallinity due to crystallite size and crystal strain in DAP decreased during MG, while the limited peaks of (040) and (060) disappeared in DCPD. The bonds in each molecule were disintegrated, especially HPO₄²⁻ in DAP was collapsed by MG. The crystallinity in DAP powder after MG was recovered by annealing at lower temperature than that in DAP without MG. Change of crystal structure from DCPD to dicalcium phosphate anhydrous (DCPA) was also observed during MG since the fracture of DCPD powder during MG preferentially occurred at the H₂O layers on (010) plane.

[49] HYDROGEN STORAGE OF MG-CONTAINING CARBON NANOCOMPOSITES OBTAINED BY HIGH ENERGY BALL MILLING

H Imamura, S Tabata, N Shigetomi, M Matsumoto, K Masanari, Y Sakata - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 457-459 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001



We have proposed the application of Mg-containing carbon nanocomposites, synthesized by high energy ball milling of graphite (G) and Mg with organic additives (tetrahydrofuran, benzene, cyclohexane, etc.), as novel hydrogen storage materials. Hydrogen storage by mechano-synthesized nanocomposites was studied, combined with characterization by XRD, TPD, DSC, TGA, TEM and Raman spectroscopy. The use of organic additives during the milling process was an important factor in determining the hydrogen-absorbing properties of the resulting nanocomposites. Besides hydriding/dehydriding by the Mg component additional hydrogen uptake by the Mg/G nanocomposites as a result of such mechanical treatments was observed.

[48] HYDROGENATION OF QUASICRYSTALLINE POWDERS PREPARED BY MECHANICAL ALLOYING

A. Takasaki, Y Furuya, KF Kelton - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 497-500 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

Mg₄₄Al₁₅Zn₄₁ and Ti₄₅Zr₃₈Ni₁₇ icosahedral quasicrystalline, (i-phase) powders, which were prepared by mechanical alloying from the elemental powder mixture directly and subsequent heat treatment in a vacuum respectively have been hydrogenated with hydrogen gas at a temperature of 573 K and at high hydrogen pressures. The hydrogen solubility limits for the Mg₄₄Al₁₅Zn₄₁ and the Ti₄₅Zr₃₈Ni₁₇ i-phase powders were estimated to be about 16 at% and 60 at% respectively. After hydrogenation, the Mg₄₄Al₁₅Zn₄₁ i-phase powder decomposed into a cubic approximant and MgZn₂ intermetallic phases, whereas the Ti₄₅Zr₃₈Ni₁₇ i-phase powder was structurally stable without forming any hydride phases. Hydrogen desorption for the Mg₄₄Al₁₅Zn₄₁ and Ti₄₅Zr₃₈Ni₁₇ i-phase powders occurred at temperatures above about 600 K and 400 K respectively. Hydrogenation of related phases, such as approximant and amorphous phases, for these powders were also investigated.

[47] MECHANICALLY SYNTHESIS, MICROSTRUCTURE AND MECHANICAL PROPERTIES OF NiAl-BASED COMPOSITES

FB Yang, JT Guo, GS Li, JY Zhou - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 835-838 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

NiAl based composites with ceramic dispersoids have been synthesized by mechanical alloying from elemental mixed powders. The formation mechanism attributed to explosive reaction, which comprised two exothermic reaction, i.e. Ni+Al->NiAl+DeltaH; Hf+C->HfC+DeltaH (Hf+B->HfB₂+DeltaH). Hot pressing and hot isostatic pressing have been applied to consolidate nanocrystalline powders to near fully dense (>96%) compacts. SEM and TEM observations show that small ceramic particles distributed in NiAl matrix homogeneously and large ones concentrated on grain boundaries. The compressive yield stress of both composites at high temperature is about 2-3 times higher than that of cast NiAl, and exhibited over 10% compressive strain at room temperature. Furthermore, the deformation behavior of composites at high temperature has also been investigated with different strain rates.

[46] EFFECT OF BALL MILLING ON ANNEALING PROCESSES IN EXTRA-FAST SOLIDIFIED Ti-AL-MO ALLOY POWDERS

S Hata, T Yoshida, N Kuwano, K Oki - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 895-898 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

The effect of ball milling on microstructures and their evolution of Ti₅₀Al₄₅Mo₅ (at%) alloy powders produced by the plasma rotating electrode process (PREP) was investigated by means of X-ray diffraction and transmission electron microscopy. After ball milling for 345.6 ks (96 h), the Ti₅₀Al₄₅Mo₅ alloy powders are composed of nanocrystalline grains of alpha(hcp-A3) or beta(bcc-A2) phase smaller than 50 nm in diameter. In the early stages of annealing at 873-1373 K, the milled powders transform into a three-phase state of alpha(2)(D0(19))+beta(2)(B2)+gamma(L1(0)), followed by the formation of gamma phase from the alpha (or alpha(2)) phase. The annealed powders have an equiaxed grain structure and exhibit a small increase in grain size after the heat treatment. The result indicates that the coexistence of fine alpha(2), beta(2) and gamma grains with the different crystal structures and compositions retards the grain coarsening in the annealing processes.

[45] ADVANCES IN CUBIC TITANIUM TRIALUMINIDE INTERMETALLICS

Authors RA Varin, T Czujko, L Zbronec - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 947-950 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

Compositional modifications such as increase in titanium concentration and boron doping have been implemented with the objective of improving mechanical properties of cubic (L1(2)) titanium trialuminides. Observations of process zones at the crack tips induced in bending were carried out by scanning electron microscopy (SEM) and atomic force microscopy (AFM). Nanocrystalline powders were fabricated by ball (mechanical) milling and consolidated into compacts and their properties were investigated. In this work, the recent advances made in our laboratory in the understanding the relationships between the microstructure and properties of L1(2)-ordered titanium trialuminides will be critically discussed.



[44] PSEUD-SUPERPLASTIC DEFORMATION OF A TiC/Ti5Si3 NANO GRAIN COMPOSITE PRODUCED BY NON-EQUILIBRIUM PM PROCESS

Authors K Tagaya, Y Suehiro, K Ameyama - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 959-962 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

A non-equilibrium PM process was applied to produce a TiC/Ti5Si3 intermetallic composite. Microstructure and high temperature deformation behavior of Ti-20 mass% SiC mechanically alloyed (MA) powder compacts were investigated. An amorphous and an α -Ti phases were observed in the MA powder processed for 720ks. These phases changed to TiC and Ti5Si3 phases. After the heat treatment at elevated temperatures. A (TiC + Ti5Si3) microduplex structure was obtained in a 753 K VHP specimen, which was compressed to 25 % at 1133 K. The compression tests at 1133 K revealed that the 753 K VHP specimen show lower flow stress at initial strain rate of $2.1 \times 10(4) \text{ s}(-1)$ rather than that of $4.2 \times 10(4) \text{ s}(-1)$. The slower strain rate test produced larger amount of harder phases such as TiC and Ti5Si3. Therefore, there exists an appropriate condition for a low temperature and high strain rate forming process in the material.

[43] EQUAL CHANNEL ANGULAR PRESSING OF MG ALLOYS; PROCESSING, MICROSTRUCTURE AND MECHANICAL PROPERTIES

SY Chang, DH Shin, H Tezuka, T Sato - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 1187-1190 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

Equal channel angular pressing of difficult-to-work Mg alloys was conducted, and the pressing temperature that is one of most important processing factors in deformation of Mg alloys was established. Pure Mg and a commercial AZ91 Mg alloy were susceptible to shear localization during equal channel angular pressing; surface cracking occurred along to the direction of shear localization at lower temperatures than 573K. Uniform flow occurred at 573 K. In addition, 4 repetitive equal channel angular pressings of Mg were successfully conducted at 573K on the same sample that was rotated 180degrees around its longitudinal axis between pressings. The grain size of pure Mg was decreased from approximately 400 μm to 80 μm after 1 pressing at 573 K, and it remained almost unchanged after 4 pressings. The grains were dynamically recovered during the repetitive pressings at 573 K ($> 1/2T_m$). A marked twinning was also observed in the equal channel angular pressed Mg. There was small increase of microhardness, proof stress and tensile strength in equal channel angular pressed Mg with no decrease in elongation.

[42] GREEN-STATE FORMING OF MAGNESIUM BASE FUNCTIONAL ALLOYS

T Aizawa - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 1215-1218 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

Solid-state processing via bulk mechanical alloying is developed to synthesize magnesium functional alloys, Different from the conventional elements, magnesium has its intrinsic properties even in formation of binary compounds. This uniqueness reflects on its solid state processing. Mg-Ni and Mg-Si systems are employed as a target to show two types of green forming processes information of Mg2Ni and Mg2Si. In the former case, full reaction from the element constituent mixture to Mg2Ni takes place during the bulk mechanical alloying. Non-equilibration with nickel enrichment in this green-state forming enables us to control the hydrogen absorbing behavior. In the latter case, little or no reactions to Mg2Si take place in the bulk mechanical alloying. In-process activation of reactivity has the precursor reacted in the solid state by post-annealing. Since its grain size is preserved as the refined silicon size before reaction, the thermoelectric properties can be improved by phonon scattering across the refined grain boundaries.

[41] GAS NITRIDING OF REFINED MAGNESIUM VIA BULK MECHANICAL ALLOYING

P Visuttipitukul, T Luangvaranunt, T Aizawa, H Kuwahara - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 1283-1286 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

Magnesium alloys have been recently rediscovered as the alloys of choice for applications in automobile and handheld electronics casing. With their many favorable intrinsic properties, magnesium alloys are increasingly replacing aluminum alloys and plastics. There still is a remains some limitation in its application to automotive parts where surface quality of magnesium alloys must be sufficiently improved. Therefore, the obvious next step of technological innovation is surface modification for tribological enhancement. In this research, a simple gas nitriding was selected to achieve in formation of nitride layer hoping to increase surface hardness. The key in achieving nitride formation is the activated state of the magnesium substrate. Bulk Mechanical Alloying was employed to prepare substrate having refined microstructure with small amount of MgO on the surface. After gas nitriding GIXD result confirms the presence Of Mg3N2. Pure magnesium billet was oxidized before nitriding to confirm that Mg3N2 Cannot be formed by conventional gas nitriding if the natural oxide layer is not eliminated. However, results from XPS cannot confirm the presence of Mg3N2 due to the lack reference data. Another intriguing aspect is the stability of the nitride layer. After leaving the sample exposed to ambient atmosphere for 5 months, some Of Mg3N2 react with moisture in air and transform to Mg(OH)(2) in form of loose gray powdery and release



ammonia gas as by product. The added titanium does not have a significant effect on the formation and stability of Mg₃N₂ to magnesium-titanium alloys. Investigation of tribological properties is underway.

[40] FABRICATION OF MICRO/NANOCOMPOSITE OF ALUMINA/ZIRCONIA SYSTEM

M Nagashima, K Maki, M Hayakawa - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 2061-2064 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

An Al₂O₃-5vol%ZrO₂ micro/nano-composite was successfully fabricated by using the HE ball mill mixing. The role of HE ball milling was to reduce the crystallite size of Al₂O₃, powder into half of the as received powder, as well as introducing some strain in the particles. Thus, the refinement of Al₂O₃ powder seem to be effective to embed ZrO₂ particles in the Al₂O₃ grains by the enhanced grain growth of the Al₂O₃, matrix. The mixed powders could be sintered to a nearly full density at 1400degreesC in the ambient atmosphere. The matrix grain size of the composite was under 0.6µm. The flexural strength of the composites was 1.3 times higher than that of monolithic Al₂O₃.

[39] THERMOELECTRIC PROPERTIES OF BI-SB-TE COMPOUNDS PREPARED BY MA-PDS PROCESS

XD Liu, H Okamura, YH Park - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 2161-2164 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

In this work, we employed a new processing technique, MA-PDS (mechanical alloying followed by pulse-discharge sintering), to fabricate the bulk (Bi₂Te₃)(₂₅)(Sb₂Te₃)(₇₅) polycrystalline materials with Ag, BN and Ag&BN dopant additions. The electrical, thermal and thermoelectric properties of the doped samples were systematically investigated as a function of the doping content and temperature. Based on these studies, we identified a great potential for further improving the thermoelectric performance of the mother (Bi₂Te₃)(₂₅)(Sb₂Te₃)(₇₅) material by doping a small amount of Ag. The doping content was optimized at 0.01-0.02wt%, which corresponds to the maximum room-temperature figure of merit of 3.4x10⁽⁻³⁾K⁽⁻¹⁾. Doping of BN or Ag&BN, however, yields lower values of the figure of merit than that of the undoped analog.

[38] EFFECT OF SHORT METAL FIBER ADDITION ON THERMOELECTRIC PROPERTIES OF MG2SI

A Matsumoto, K Kobayashi, K Ozaki, T Nishio - PRICM 4: FORTH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING, VOLS I AND II, 2001, pp 2177-2180 - 4TH PACIFIC RIM INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS AND PROCESSING (PRICM4); HONOLULU, HAWAII. DECEMBER 11-15, 2001

Thermoelectric properties, electric conductivity and Seebeck coefficient, of metal fiber dispersed Mg₂Si were investigated over the temperature range from 350 to 750K. In order to decrease electric resistivity of Mg₂Si, Cu or Ti fibers were mixed. Mg-33.3at%Si powder was synthesized by mechanical alloying and consolidated by pulsed current sintering. The power factor of 1vol% Cu fiber dispersed Mg₂Si showed the maximum value of 1.2x10⁽⁻⁴⁾ WK⁽⁻¹⁾ m⁽⁻²⁾, which was 1.5 times as high as that of Mg₂Si. On the other hand, Ti fiber addition didn't improve thermoelectric properties of Mg₂Si in this investigation.

[37] FORMATION OF INTERMETALLIC COMPOUNDS WITH THE B2-TYPE STRUCTURE UPON MECHANICAL ALLOYING OF FE50AL50-XSIX

Fadeeva VI. Sviridov IA. Baldokhin YV. - Physics of Metals & Metallography (English Translation of Fizika Metallov i Metallovedenie). 94(3):298-303, 2002

X-ray diffraction and Mossbauer spectroscopy were used to study Fe₅₀Al₅₀-Si-x(x) powders prepared by mechanical alloying. The solid-state interaction between Fe, Al, and Si was provided by milling powder mixtures in a high-energy ball mill in an argon atmosphere. The formation of a partially ordered bcc solid solution occurs immediately upon milling. This is confirmed by the appearance of superlattice reflections typical of B2 ordering in X-ray diffraction patterns and by the presence of a singlet typical of the symmetrical surroundings of Fe atoms by nonmagnetic Al and Si atoms in the Mossbauer spectrum. Long-term milling of mixtures containing more than 25 at. % Si leads to the formation of two phases, Fe₅₀Al₅₀-xSi_x (B2) and FeSi (B20). For the Fe₅₀Al₅₀-xSi_x mixtures with x < 20 at. %, the degree of long-range order increases, and the lattice parameter of the bcc solid solution decreases with increasing silicon content.

[36] A THERMIONIC TUNGSTEN CATHODE ACTIVATED WITH NANTHORIA AND PREPARED BY SWAGING METHOD

Wang FZ. Zhang H. Ding BJ. Zhu RH. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 336(1-2):59-63, 2002

Using high-energy ball milling, nanosized thoria powders were obtained. After mixing the thoria nanoparticles with tungsten powders, a nanocomposite cathode was fabricated by sintering and hot swaging. The relative density of the nanocomposite material is near 100%. The microstructure of the nanocomposite cathode is quite different from that of conventional thoriated tungsten cathodes. Most of the thoria particles in the nanocomposite are less than 100 nm in diameter, and situated on the boundaries of tungsten grains. The nanocomposite cathode shows a much lower arc initiation field than that of conventional cathode, which will improve the performance of the cathode significantly

[35] ENHANCING OXYGEN RECOVERY FROM ILMENITE BY EXTENDED MILLING



Welham NJ. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 336(1-2):143-149, 2002

The generally accepted method for recovering oxygen on an extraterrestrial body is by thermally induced reduction of indigenous minerals by hydrogen. The most amenable mineral is considered to be ilmenite, FeTiO_3 . The effect of up to 400 h ball milling on the subsequent hydrogen reduction of a terrestrial beach sand derived ilmenite is examined. XRD examination of the final powders indicates that reduction of ilmenite proceeds via elemental iron and rutile which is then further reduced to sub-oxides. The recovery of oxygen as water was found to be over two and a half times greater for a sample milled for 400 h than for an unmilled sample.

[34] REACTION SINTERING OF TITANIUM CARBIDE AND TITANIUM SILICIDE PREPARED BY HIGH-ENERGY MILLING

Orthner HR. Tomasi R. Botta WJ. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 336(1-2):202-208,

Powders of titanium, silicon and carbon black with a molar ratio of 3:1:2 were milled in a high-energy ball mill in order to produce titanium silicon carbide (Ti_3SiC_2). The milling process initiated an exothermic reaction and the starting material was completely converted into a mixture of mainly titanium carbide (TiC) and minor amounts of TiSi_2 , $\text{Ti}_5\text{Si}_3\text{C}_x$ and Ti_3SiC_2 . Using off-stoichiometric mixtures or SiC as a source of silicon did not affect the amount of TiC in the product. To obtain Ti_3SiC_2 the as-milled powders were pressed to compacts and sintered for 2 h. The thermal treatment caused the formation of large amounts of Ti_3SiC_2 . Using a slight excess of silicon in the range of 10-30%, pure samples of the ternary phase could be obtained

[33] NANOSTRUCTURED COATINGS [REVIEW]

He JH. Schoenung JM. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 336(1-2):274-319, 2002

This review is based essentially on the results in the field of synthesis and characterization of nanostructured coatings obtained by the authors themselves or their colleagues. Characteristics of feedstock powders for synthesizing nanostructured coatings such as particle size and morphology, changes in chemical composition and grain size are summarized. The evolution of microstructure caused by mechanical milling in two typical powder systems, $\text{Cr}_3\text{C}_2\text{-NiCr}$ and Inconel 625, and mechanisms governing the development of the nanostructure are discussed. Mechanical properties and microstructure of several nanostructured coatings are evaluated by using microhardness testing, scanning electron microscopy, transmission electron microscopy, and X-ray diffraction. As background information, a review of the agglomeration process for milled powders and of thermal spraying technologies to synthesize nanostructured coatings are also included. In addition, the methodologies used to characterize the performance of the milled powders and nanostructured coatings, as well as the practice techniques for sample preparation, are described in detail.

[32] MONTMORILLONITE TREATED WITH RHODAMINE-6G MECHANOCHEMICALLY AND IN AQUEOUS SUSPENSIONS - SIMULTANEOUS DTA-TG STUDY

Landau A. Zaban A. Lapidis I. Yariv S. - Journal of Thermal Analysis & Calorimetry. 70(1):103-113, 2002.

The mechanochemical solid-state adsorption of the cationic dye rhodamine-6G (R6G) by montmorillonite was investigated by XRD and simultaneous DTA-TG. Five different mixtures of R6G and montmorillonite were investigated. They contained 10, 20, 35, 50 and 100 mmol R6G per 100 g clay. The solid R6G was ground with the clay for five minutes. Mixtures were ground both in the absence of water (dry grinding) and with the adding of drops of water periodically, (wet grinding). There were no differences between samples obtained by wet or dry grinding. X-ray and DTA data were compared with those of R6G-montmorillonite obtained from aqueous suspensions. The mechanochemical products were different from those obtained from aqueous suspensions. The X-ray and DTA studies suggest that the mechanochemical adsorption of organic cations takes place on the external surfaces of the clay whereas in suspensions the adsorption takes place into the interlayer space. In the latter case the final stages of oxidation occur in temperatures higher than those of the neat dye whereas in the former they occur at lower temperatures

[31] ALLOY FORMATION IN MECHANICALLY ACTIVATED MIXTURES - HYDROXOCARBONATES WITH AL-0

Wieczorek-Ciurowa K. Gamrat K. Parylo M. Shirokov JG. - Journal of Thermal Analysis & Calorimetry. 70(1):165-172, 2002.

The physical mixtures of hydroxocarbonates of Cu and Ni with aluminium were activated using a laboratory planetary mill. The chemical reactions and alloy formations as the effects of grinding were followed by the phase analysis of solid products based on the thermogravimetry and X-ray diffractometry. Experimental evidence indicates that the nature of reactions and products of mechanical activation was dependent on the amount of aluminium and time of grinding

[30] THE INFLUENCE OF BALL MILLING AND SUBSEQUENT CALCINATION ON THE FORMATION OF LiFeO_2

Widatallah HM. Johnson C. Berry FJ. - Journal of Materials Science. 37(21):4621-4625, 2002

The influence of ball milling and subsequent calcination of a 1: 1 molar mixture of $\alpha\text{-Fe}_2\text{O}_3$ and Li_2CO_3 on the formation of LiFeO_2 has been investigated. Pre-milling was found to lower the temperature of ferrite formation by ca. 200degreesC and a thermally stable $\gamma\text{-LiFeO}_2$ phase was found to form in the temperature range 500-600degreesC. Slow cooling of the pre-milled mixture calcined at higher temperatures resulted in the formation of some LiFe_5O_8 .

[29] ELECTRON MICROSCOPY NANOSCALE CHARACTERIZATION OF BALLMILLED CU-AG POWDERS. PART I: SOLID SOLUTION SYNTHESIZED BY CRYO-MILLING



S. Zghal, M.J. Hytch, J.-P. Chevalier, R. Twesten, F.Wu, P. Bellon - Acta Materialia 50 (2002) 4695-4709

We present a study of nanostructured Cu-Ag material obtained by low temperature ball milling. The microstructural characterization is carried out using a wide variety of transmission electron microscopy (TEM) techniques from conventional dark-field imaging, selected area diffraction and energy dispersive X-ray spectrometry (EDS) to more modern techniques of scanning TEM high-angle dark-field imaging (HAADF), nanodiffraction and high resolution imaging (HREM). A novel method of HREM image analysis is also presented which consists of calculating geometric phase images by Fourier filtering. Real-space maps of lattice spacings and lattice rotations are thus obtained. The analysis addresses the following essential points of nanostructural characterization: dislocation densities, grain sizes and morphologies, grain boundaries and local lattice rotations, local textures, local variations in lattice parameters. A new description of the microstructure emerges from the observations, quite different to that expected. Analytical modeling suggests that large lattice rotations can be expected in nanomaterials produced by intense deformation.

[28] ELECTRON MICROSCOPY NANOSCALE CHARACTERIZATION OF BALL MILLED CU-AG POWDERS. PART II: NANOCOMPOSITES SYNTHESIZED BY ELEVATED TEMPERATURE MILLING OR ANNEALING

S. Zghal, R. Twesten, Fang Wu, P. Bellon - Acta Materialia 50 (2002) 4711-4726

Microstructures and phases stabilized at steady state by variable temperature ball milling of Cu50Ag50 powders are characterized using transmission and scanning transmission electron microscopy. Starting from chemically mixed and cold-worked powders obtained by room temperature milling, it is shown that, upon increasing the milling temperature, the material first decomposes into Cu-rich and Ag-rich solid solutions, and then recrystallizes. A similar sequence is observed during the static annealing of the solid solution precursor. In both cases, Cu-Ag nanocomposites are synthesized, at a scale of a few nanometers in the unrecrystallized state, and at a scale ranging from 30 nm after dynamic recrystallization to 75 nm after static recrystallization. These nanocomposites exhibit high hardness values, approaching 6 GPa. Interestingly enough, recrystallization leads to an increase in the hardness of these materials.

[27] AN X-RAY ABSORPTION SPECTROSCOPY EXAMINATION OF STRUCTURAL CHANGES TO ZINC DI-ALKYL-DI-THIOPHOSPHATE (ZDDP) FOLLOWING MILLING IN THE PRESENCE OF IRON OXIDES AND SUBSEQUENT THERMAL PROCESSING

Ferrari ES. Roberts KJ. Adams D. - Wear. 253(7-8):759-767, 2002

Solid-state mixtures of the lubricating oil additive zinc di-alkyl-di-thiophosphate (ZDDP) with two different iron oxide phases (FeO and Fe₂O₃) prepared by milling are examined using X-ray absorption spectroscopy (EXAFS). The changes of the local atomic environment around the zinc atomic site within ZDDP, both in the presence of iron and following subsequent temperature profiling, are discussed. The thermal decomposition behaviour of the milled powder is in general consistent with a two-stage reaction process: an initial loss of the alkyl chains of the di-thiophosphate groups, followed by the breakdown of the ZDDP molecule. Whilst the specific iron oxide phase used does not appear to significantly influence these decomposition reactions, changes are also seen in the local atomic structure of both the ZDDP and the iron oxides following milling and after subsequent thermal reaction. Drawing down on previously published work, the results of this work are represented via a tentative molecular-scale model based upon the known structural chemistry of ZDDP and rationalised with differential scanning calorimetry (DSC) analysis.

[26] MICROSTRUCTURE AND WEAR PROPERTIES OF AL-PB-CU ALLOYS PREPARED BY MECHANICAL ALLOYING

Zhu M. Zeng MQ. Gao Y. Ouyang LZ. Li BL. - Wear. 253(7-8):832-838, 2002

In the present work, Al-10 wt.% Pb-x wt.% Cu (x = 0-5.5) powder mixtures were mechanically alloyed. Then the mechanically alloyed powders were sintered to fabricate bulk alloys. Scanning electron microscope, X-ray diffractometer and Vickers hardness indentation were used to characterize the microstructure and mechanical properties of the Al-10 wt.% Pb-x wt.% Cu alloys. Wear properties of the alloys were characterized by measuring the bulk wear under dry lubrication condition. By comparing the microstructure and wear properties of mechanically alloyed Al-10 wt.% Pb-Y wt.% Cu alloys with that of mechanically alloyed Al-Pb alloy, the effect of Cu addition on microstructure and wear properties has been discussed. The present work shows that the wear properties of Al-10 wt.% Pb-x wt.% Cu alloys are the best at the composition of Al-10% Pb-4.5% Cu under the present experiment condition

[25] STRUCTURE OF A FE-CR-MN-MO-N ALLOY PROCESSED BY MECHANICAL ALLOYING

Mendez M. Mancha H. Cisneros MM. Mendoza G. Escalante JI. Lopez HF. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 33(10):3273-3278, 2002

Fe, Cr, Mn. and Mo powders were processed by mechanical alloying to develop a nanostructured Fe-18Cr-11Mn-5Mo alloy under a N₂ atmosphere. It was found that the nitrogen contents in the as-milled powder mixture increased up to 1.6 wt pct after 190 hours processing time. The as-milled powders were then annealed under vacuum at either 1173 or 1473 K to promote the formation of the resultant equilibrium phases. In the annealed powder mixtures, depending on the temperature and nitrogen content, the phases identified by X-ray diffraction were either austenite, ferrite, or chromium nitrides. Annealing at 1173 K promoted the development of gamma-Fe, alpha-Fe. and Cr₂N for all the nitrogen contents considered (0.5 to 1.6 wt pct). The volume fractions of the various phases formed were found to be strongly influenced by the nitrogen content and annealing temperature. In addition, the levels of nitrogen absorbed during processing were retained after annealing. Finally, the outcome indicates that a fully austenitic structure can be obtained by annealing powder mixtures at 1473 K with maximum nitrogen contents of up to 1 wt pct

[24] MECHANICAL ACTIVATION OF THE DECOMPOSITION AND SINTERING OF KYANITE



Aguilar-Santillan J. Cuenca-Alvarez R. Balmori-Ramirez H. Bradt RC. - Journal of the American Ceramic Society. 85(10):2425-2431, 2002

The influence of attrition milling on the thermal decomposition of kyanite ((Al₂O₃SiO₂)-Si-) to mullite (3Al₂O₃(.)2SiO₂) and SiO₂, and its subsequent sintering, was studied. A commercial kyanite was attrition-milled for times up to 12 h. Dilatometry confirmed that as-received unmilled kyanite decomposes between 1300degrees and 1435degreesC. The decomposition reaction is slow initially and accelerates during the later stages until about one-half of the decomposition occurs in the last 35degreesC. For the attrition-milled kyanite, the onset decomposition temperature decreases, the transformation temperature interval is reduced, and both the decomposition reaction and subsequent sintering are accelerated. A dense microstructure of fine equiaxed mullite grains in the 1 µm size range, evenly dispersed in a glassy matrix, is obtained by sintering the attrition-milled kyanites. These results are explained in terms of the energy accumulated during attrition milling, a reduction of the milled kyanite particle size, and the presence of a liquid phase during sintering

[23] V2O5-SMVO4 MECHANICAL MIXTURE: OXIDATIVE DEHYDROGENATION OF PROPANE

Barbero BP. Cadus LE. - Applied Catalysis A-General. 237(1-2):263-273, 2002

V₂O₅, SmVO₄ and the mechanical mixtures of them in two different mass ratios were studied in the reaction of oxidative dehydrogenation of propane to propene. The results of the catalytic activity indicate the presence of a synergy effect in the yield to propene at reaction temperatures lower than 450degreesC. This synergy effect disappears when the reaction temperature increases. When the catalysts tested at 500degreesC were re-evaluated at low temperatures (350-400 degreesC), a significant decrease in yield to propene was observed. By the results of physicochemical characterization (BET, X-ray diffraction (XRD), temperature programmed reduction (TPR), X-ray photoelectron spectroscopy (XPS)), it can be inferred that the synergy would be due to a transient state of the catalytic system. In it, surface vanadium species adsorbed on the SmVO₄ and originated by V₂O₅ migration would constitute active and selective centers for the oxidative dehydrogenation of propane. The reason for the loss of synergy when the reaction temperature increases would be that the transformation of the catalytic system continues towards the coverage Of SmVO₄ phase by V₂O₅. Thus, the surface Of SmVO₄ is covered by conglomerates of surface VO_x, species and even recrystallized V₂O₅. This surface transformation modifies the catalytic performance of the system.

[22] OBSERVATION OF OXIDATION AND MECHANICAL STRAIN IN CR NANOPARTICLES PRODUCED BY BALL-MILLING

Abdul-Razzaq W. Seehra MS. - Physica Status Solidi A-Applied Research. 193(1):94-102, 2002

Nanoparticles of Cr, produced by ball-milling a commercial chromium powder for up to 70 h in Ar-filled glove box, are characterized by room temperature X-ray diffraction (XRD) and magnetization (M) studies as a function of temperature from 5 to 300 K. Analysis of the XRD linewidths in terms of particle size (L) and mechanical strain (eta) shows that with increase in milling-time, L decreases and eta increases. However, for milling times greater than or equal to 40 h, saturation in L (at 20 nm) and eta is reached and presence of Cr₂O₃ becomes evident in the XRD patterns. In the magnetic studies, M increases as L decreases, also reaching saturation values for milling time greater than or equal to 40 h. The variations of M versus T are indicative of nanoparticles effects such as blocking. However, it is evident that the effect of Cr₂O₃ is present even for samples for which XRD patterns do not show the presence of Cr₂O₃ (samples milled for less than 40 h). The question of the observed large magnitudes of M for Cr nanoparticles vis-a-vis bulk Cr in terms of undetected surface oxides and uncompensated surface moments is discussed

[21] EVOLUTION OF SUBMICROCRYSTALLINE IRON CONTAINING DISPERSED OXIDES UNDER MECHANICAL MILLING FOLLOWED BY CONSOLIDATION

Belyakov A. Sakai Y. Hara T. Kimura Y. Tsuzaki K. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 33(10):3241-3248, 2002

The structural changes in an Fe-0.6 pct O alloy during mechanical milling followed by consolidation through rolling were studied. The iron-iron oxide powders were mechanically milled in an argon atmosphere for various times from 20 to 300 hours. The powders were then canned into a steel pipe and multiple rolled at 700 degreesC for consolidation. The microstructure of the final product depended significantly on the milling time. The volume fraction of the dispersed oxides (10 nm in diameter) increased from about 0.3 to 2.5 pct when the milling time was increased from 20 to 300 hours. The relatively short milling time of 20 hours resulted in the evolution of elongated grains (an average Size of about 1.2 µm) with a large fraction of low-angle grain boundaries after consolidation. In contrast, much finer grains (about 0.2 µm in size) with a near random grain-boundary misorientation distribution evolved in the samples milled for 300 hours.

[20] STRUCTURE OF A FE-CR-MN-MO-N ALLOY PROCESSED BY MECHANICAL ALLOYING

Mendez M. Mancha H. Cisneros MM. Mendoza G. Escalante JI. Lopez HF. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 33(10):3273-3278, 2002

Elemental Fe, Cr, Mn, and Mo powders were processed by mechanical alloying to develop a nanostructured Fe-18Cr-11Mn-5Mo alloy under a N-2 atmosphere. It was found that the nitrogen contents in the as-milled powder mixture increased up to 1.6 wt pct after 190 hours processing time. The as-milled powders were then annealed under vacuum at either 1173 or 1473 K to promote the formation of the resultant equilibrium phases. In the annealed powder mixtures, depending on the temperature and nitrogen content, the phases identified by X-ray diffraction were either austenite, ferrite, or chromium nitrides. Annealing at 1173 K promoted the development of gamma-Fe, alpha-Fe, and Cr₂N for all the nitrogen contents considered (0.5 to 1.6 wt pct). The volume fractions of the various phases formed were found to be strongly influenced by the nitrogen content and annealing temperature. In addition, the levels of nitrogen absorbed during processing were retained



after annealing. Finally, the outcome indicates that a fully austenitic structure can be obtained by annealing powder mixtures at 1473 K with maximum nitrogen contents of up to 1 wt pct

[19] PREPARATION AND CHARACTERIZATION OF FEXOY-TIO2 VIA SONOCHEMICAL SYNTHESIS

Huang WP. Tang XH. Felner I. Koltypin Y. Gedanken A. - *Materials Research Bulletin*. 37(10):1721-1735, 2002
Sonication is used to prepare binary oxide FexOy-TiO_2 . Under sonication, the reaction of FeCl_3 with $(i\text{-C}_3\text{H}_7\text{O})_4\text{Ti}$ takes place successfully. The products are characterized by XRD, Mossbauer, Magnetic, DSC, TEM, SEM, and BET measurements. The results of characterization show that iron oxides in as-prepared samples are all amorphous and that the particles are nanometric. The product of the nonhydrolytic reaction remains amorphous at 773 K, and at higher temperatures the iron ions exist in the form of Fe_2TiO_5 . The mesopores can withstand calcination of 773 K

[18] LOW TEMPERATURE HEAT CAPACITIES OF MECHANICALLY ALLOYED LA-DOPED PBWO4 SYSTEM

Takai S. Nakanishi T. Tojo T. Kawaji H. Atake T. Esaka T. - *Journal of Thermal Analysis & Calorimetry*. 69(3):805-811, 2002.

Heat capacity measurements were carried out on $\text{Pb}_{1-x}\text{La}_x\text{WO}_{4+x/2}$ ($x=0.2$) and $\text{Pb}_{1-x}\text{La}_{2x/3}\text{WO}_4$ ($x=0.2, 0.5$) solid solutions prepared by sintering and mechanical alloying (MA) methods. For all the solid solutions, sintered samples showed slightly larger heat capacity around 100 K in comparison with MA samples, which was presumably caused by the excitation of mobile oxide ion motion. For sintered scheelite-type structured PbWO_4 s, high-temperature synthesis introduced oxide ion interstitials even for the $\text{Pb}_{1-x}\text{La}_{2x/3}\text{WO}_4$ system, which resulted in the excess heat capacity at low temperature for excitation. On the other hand, for the samples prepared by room-temperature MA technique, oxide ion seemed to occupy the regular sites rather than interstitial ones and excess heat capacities were not observed.

[17] MECHANICAL ACTIVATION OF THE DECOMPOSITION AND SINTERING OF KYANITE

Authors Aguilar-Santillan J. Cuenca-Alvarez R. Balmori-Ramirez H. Bradt RC. - *Journal of the American Ceramic Society*. 85(10):2425-2431,

The influence of attrition milling on the thermal decomposition of kyanite ($(\text{Al}_2\text{O}_3\text{SiO}_2)\text{-Si}$) to mullite ($3\text{Al}_2\text{O}_3(\cdot)_2\text{SiO}_2$) and SiO_2 , and its subsequent sintering, was studied. A commercial kyanite was attrition-milled for times up to 12 h. Dilatometry confirmed that as-received unmilled kyanite decomposes between 1300degrees and 1435degreesC. The decomposition reaction is slow initially and accelerates during the later stages until about one-half of the decomposition occurs in the last 35degreesC. For the attrition-milled kyanite, the onset decomposition temperature decreases, the transformation temperature interval is reduced, and both the decomposition reaction and subsequent sintering are accelerated. A dense microstructure of fine equiaxed mullite grains in the 1 μm size range, evenly dispersed in a glassy matrix, is obtained by sintering the attrition-milled kyanites. These results are explained in terms of the energy accumulated during attrition milling, a reduction of the milled kyanite particle size, and the presence of a liquid phase during sintering

[16] A MONTE CARLO SIMULATION OF B-SITE ORDER-DISORDER TRANSFORMATION IN $\text{PB}(\text{SC}_{1/2}\text{TA}_{1/2})\text{O}_3$ TRIGGERED BY MECHANICAL ACTIVATION

Gao XS. Lim J. Xue JM. Wang JS. Liu JM. Wang J. - *Journal of Physics-Condensed Matter*. 14(37):8639-8653, 2002
Order-disorder transformation triggered by mechanical activation in a perovskite structure was observed in $\text{Pb}(\text{Sc}_{1/2}\text{Ta}_{1/2})\text{O}_3$; it is simulated using a Monte Carlo algorithm, based on the competition between mechanical activation leading to disordering and the thermal diffusion recovering the ordering. The time evolution of the long-range order (LRO) from an initial ordered state shows a steady decrease at the initial stage and then becomes more or less stabilized over a prolonged period; while from the disordered initial state, LRO increases first and then stabilizes at a similar end value. Thermal diffusion is the dominant process at relatively high, temperatures, leading to the disorder-to-order transformation. The effect of mechanical activation becomes significant and results in order-to-disorder transformation at relatively low temperatures. Both the mechanical activation intensity and the vacancy migration energy exert an impact on the degree of ordering and the order-disorder transformation temperature at low temperatures. Snapshot images of the simulation demonstrate the competition between thermal diffusion and mechanical activation, which refines the domain size.

[15] METAL OXIDE POWDER SYNTHESIZED WITH AMORPHOUS METAL CHELATES

Saitoh H. Satoh R. Nakamura A. Nambu N. Ohshio S. - *Journal of Materials Science*. 37(20):4315-4319, 2002
Chelate powder consisting of amorphous particles was synthesized through the process in which the droplet of the chelate solution is dried in the gas phase and solidified in a moment using a splay-dry technique. To investigate the advantage of the use of amorphous chelate powder in the processing of metal oxide powder, this study provides following two routes: a conventional route of mechanically mixing of crystalline metal-ethylenediaminetetraacetic acid (EDTA) powders and spray-dry mixing of metal-EDTA. These routes were followed by calcinations of metal-EDTA powder to form metal oxide powder. In this study, morphology, crystallization and metal composition of resulting $(\text{Ba}, \text{Sr})\text{TiO}_3$ and $\text{YBa}_2\text{Cu}_3\text{O}_7$ powders were investigated. The amorphous metal-EDTA powder involving several metal elements is appropriate for successful calcination at lower temperatures rather than the mixture of crystalline powders prepared by mechanically mixing.

[14] STRUCTURAL AND MAGNETIC PROPERTIES OF ROOM TEMPERATURE MILLED $\text{CO}_{0.2}\text{Zn}_{0.8}\text{Fe}_2\text{O}_4$ SPINEL OXIDE

Bhowmik RN. Ranganathan R. - *Journal of Materials Science*. 37(20):4391-4398, 2002
We report the nanoparticle $\text{Co}_{0.2}\text{Zn}_{0.8}\text{Fe}_2\text{O}_4$ spinel oxide, synthesized by room temperature mechanical milling. The system is stabilized mainly in spinel oxide phase after 78 hrs milling time and no other phases have been observed from the XRD spectra of the 100 hrs milled sample. We have studied the particle size effects on structural as well as on magnetic properties by annealing the 100 hrs as milled sample at different temperatures for 12 hours. The XRD and TEM data show



that the particle size decreases with increasing milling time upto 60 hrs and then show an increasing trend with milling time. The particle size also increases with annealing the 100 hrs as milled sample with better crystalline structure. It is observed that the magnetic properties of the annealed samples can be correlated with the structural change without breaking the crystal symmetry of the cubic spinel phase of $\text{Co}_{0.2}\text{Zn}_{0.8}\text{Fe}_2\text{O}_4$ spinel oxide. The change in the crystal structural as revealed by the XRD spectra can be associated with the non-equilibrium to equilibrium cationic distribution between tetrahedral (A) and octahedral (B) sites of the spinel structure.

[13] HYPERFINE STUDY ON MECHANICALLY ALLOYED FE-MN SYSTEMS

Uhrmacher M. Kulinska A. Baldokhin YV. Tcherdyntsev VV. Kaloshkin SD. Maddalena A. Principi G. - Hyperfine Interactions. 136(3):327-332, 2001.

High-energy ball milling of Fe-Mn elemental powder mixtures has been carried out for different Mn concentrations, ranging from 0 to 90%. Mossbauer spectroscopy of Fe-57 and perturbed angular correlations with In-111-implanted at 400 keV into pills made from the milled powders-have been used to investigate the structure of the milled samples. Clear changes of the hyperfine spectra occur at Mn concentrations of 15-20% and above 70%, indicating extended concentration ranges of alpha-Fe and gamma-Fe(Mn), compared to the equilibrium phase diagram. Detailed information on additional lower hyperfine fields due to nearest neighbor Mn atoms has been obtained in the alpha-Fe phase containing up to 20% Mn

[12] INCREASED CHEMISORPTION ONTO ACTIVATED CARBON AFTER BALL-MILLING

Welham NJ. Berbenni V. Chapman PG. - Carbon. 40(13):2307-2315, 2002.

Activated carbon has been milled for up to 1000 h in a laboratory-scale tumbling ball mill under a vacuum. Thermogravimetric (TG) analysis of the powder in argon showed an increasing mass loss with milling time indicating the presence of chemisorbed gas. TG Fourier transform infrared spectrometry showed the as was a mixture of water, CO, and an unidentified as (probably oxygen). BET surface area measurements showed a decreasing surface area with milling time, however, this was shown to be massively, in error for the longer milling times due to the presence of the chemisorbed gas. The area occupied by the chemisorbed gas increased from 40 to 80% of the true Surface area which was almost constant at $1258 \pm 27 \text{ m}^2 \text{ g}^{-1}$ for all three powders. These results show that extremely large errors may be made when using BET analysis to determine the surface area of powders, especially those where the surface activity is Substantially increased during processing

[11] COMPARISON OF THE CHARACTERISTICS OF NANOCRYSTALLINE FERRITE IN FE-0.89C STEELS WITH PEARLITE AND SPHEROIDITE STRUCTURE PRODUCED BY BALL MILLING

Xu Y. Umemoto M. Tsuchiya K. - Materials Transactions. 43(9):2205-2212, 2002

Nanocrystalline ferrite formation by ball milling in Fe-0.89C steels with initial pearlite and spheroidite microstructures and their annealing behaviors have been studied through microstructural observation and microhardness measurement. It was found that nanocrystalline ferrite first forms near the surface of powders due to localized severe deformation. The microhardness of nanocrystalline ferrite regions is much higher than that of work-hardening regions. The dissolution of cementite was observed together with the nanocrystallization of ferrite. The nanocrystallization rate of pearlite powder is faster than that of spheroidite powder due to higher work-hardening rate and smaller cementite size. After long time ball milling, the equiaxed nanocrystalline ferrite with less than 10 nm grain size forms in the whole powders of both pearlite and spheroidite structures, and the cementite dissolves completely. By annealing the milled pearlite and spheroidite powders, recrystallization was observed in the work-hardening regions, while continuous grain growth was observed in the nanocrystalline ferrite region. After annealing, microhardness of the former nanocrystalline ferrite region is always higher than that of the former work-hardening region when compared at the same annealing condition. The grain growth rate of nanocrystalline ferrite produced from pearlite structure is lower than that of spheroidite structure due to the finer grains.

[10] X-RAY SPECTROSCOPIC ANALYSIS OF SOLID STATE REACTION DURING MECHANICAL ALLOYING OF MOLYBDENUM AND GRAPHITE POWDER MIXTURE

Yamada K. Takahashi T. Motoyama M. Nagai H. - Materials Transactions. 43(9):2292-2296, 2002

Molybdenum and graphite powders were mechanically alloyed. Carbon K X-ray emission spectra of the mechanically alloyed powders were measured using electron probe microanalyzer (EPMA) in order to investigate the solid state reaction process. In the early stage of the mechanical alloying (similar to 36 ks), graphite did not react with molybdenum, but particle size of graphite became smaller. In the next stage of mechanical alloying (36 similar to 144 ks), graphite react with molybdenum gradually as the time increases. Molybdenum carbides were formed on mechanical alloying for 288 ks. The mechanically alloyed powders for 288 ks were heat-treated in a vacuum. Mo-33 at%C system heat-treated at 1273 K was a mixture Of Mo₂C and molybdenum, while Mo-50 at%C system heat-treated was a mixture Of Mo₂C, mechanically ground graphite and graphite.

[9] INFLUENCE OF P ON MICROSTRUCTURE AND THERMOELECTRIC PROPERTY OF SINTERED (BI_{0.2}Sb_{0.8})₂TE-3 ALLOY

Xue-Dong L. Nozue A. Park YH. - Materials Transactions. 43(9):2297-2302, 2002

A combination of mechanical alloying and pulse discharge sintering (MA-PDS) was employed to synthesize the bulk (B_{0.2}Sb_{0.8})₂Te-3 alloys with the P addition up to 2.0 mass%. The effect of P on the microstructure and thermoelectric performance of the compacts were investigated. The added P exists in the alloyed powders and compacts in the form of second phase particles. The dispersed P particles were found to decrease the size of (Bi, Sb)₂Te-3 crystallites and inhibit the densification of the alloyed powders. When the P content greater than or equal to 1.0 mass%, (Bi_{0.2}Sb_{0.8})₂Te-3/P nanocomposites were developed by the MA-PDS process. Inclusion of a small fraction of P (less than or equal to 0.1 mass%)



is harmless to the thermoelectric performance of the p-type (Bi_{0.2}Sb_{0.8})₂Te-3 alloy. In the samples with high P contents, in particular, greater than or equal to 1.0 mass%, however, the increase in the electrical resistivity is much greater than the decrease in the thermal conductivity, finally giving rise to degradation of the room-temperature figure of merit

[8] MICROSTRUCTURE CHARACTERIZATION OF POLYMORPHIC TRANSFORMED BALL-MILLED ANATASE TiO₂ BY RIETVELD METHOD

Dutta H. Sahu P. Pradhan SK. De M. - *Materials Chemistry & Physics*. 77(1):153-164, 2003

Rietveld's whole powder profile fitting method based on crystal structure refinement is applied to extract the microstructure information of several polymorphic TiO₂ phases grown simultaneously during high-energy planetary ball milling of anatase phase. The advantages of the present method of analysis over the other methods of X-ray line profile analysis are discussed in detail. Particle size and lattice strain values of all the phases are found to be anisotropic for the first time and the polymorphic phase transformations have been analyzed in this light.

[7] ELUCIDATION OF MECHANOCHEMICAL DECOMPOSITION PATHWAY OF DIOXINS USING 4-CHLOROBIPHENYL (4CB), A MODEL COMPOUND [JAPANESE]

Nomura Y. Nakai S. Lee BD. Hosomi M. - *Kagaku Kogaku Ronbunshu*. 28(5):565-568, 2002

Mechanochemical (MC) treatment using calcium oxide (CaO) is one of the new technologies applicable for decomposition of dioxins contaminating fly ashes. This study was aimed to reveal their decomposition pathways in the MC treatment using 4-chlorobiphenyl (4 CB) as a model compound of co-PCBs, one of the dioxin families. Ion chromatography showed that the MC treatment caused dechlorination of 4 CB in a final ratio of 100%. The finding that the chlorine balance during the MC treatment was accounted for chlorine ion and chlorine contained in the remaining 4 CB indicated that the first step of 4 CB decomposition in the MC treatment was dechlorination. Biphenyl, cyclohexyl benzene, and ter/quarterphenyls were identified as the main products of 4 CB decomposition by a GC-MS analysis, while MC treatment of biphenyl and cyclohexyl benzene yields ter/quarterphenyls and bicyclohexyl, respectively. In addition, MC treatment of p-terphenyl confirmed benzene ring cleavage, i.e., 4-pentyl biphenyl and isopropyl biphenyl were detected as p-terphenyl decomposition products. These results suggested that 4 CB decomposition progresses via dechlorination/hydrogenate-reduction or dechlorination/polymerization, and benzene ring cleavage

[8] MECHANOCHEMICAL SYNTHESIS OF NANOCRYSTALLINE TIN/TIB₂ COMPOSITE POWDER

Shim JH. Byun JS. Cho YW. - *Scripta Materialia*. 47(7):493-497, 2002

TiN/TiB₂ composite powder has been synthesized from a mixture of Ti and BN powders by high-energy ball milling. An abrupt displacement reaction between Ti and BN occurs in about 2 h. After the reaction, the crystallite sizes of TiN and TiB₂ decrease continuously and become about 10 nm after 16 h of milling.

[7] EXAFS, X-RAY DIFFRACTION, AND REVERSE MONTE CARLO SIMULATIONS OF AN AMORPHOUS Ni₆₀Ti₄₀ ALLOY PRODUCED BY MECHANICAL ALLOYING

Machado KD. de Lima JC. de Campos CEM. Grandi TA. Triches DM. - *Physical Review B*. 66(9):4205, 2002

The local atomic order of an amorphous Ni₆₀Ti₄₀ alloy produced by mechanical alloying was studied by extended x-ray absorption fine structure (EXAFS) and x-ray diffraction (XRD) techniques. The experimental total structure factor derived from the XRD measurements was simulated by using the reverse Monte Carlo (RMC) method. This simulation was used to compute the S-Ni-Ni(c)(K), S-Ni-Ti(c)(K), and S-Ti-Ti(c)(K) partial structure factors and to infer a three-dimensional structure for this alloy. The coordination numbers and interatomic distances for the first neighbors obtained from EXAFS and RMC showed a good agreement. The bond-angle distributions derived from the structure obtained by RMC simulations give evidence that the structure of amorphous Ni₆₀Ti₄₀ alloy contains distorted trigonal-prismatic units. The calculated Warren chemical short-range order parameter showed a local chemical order similar to that one found in the solid solutions and in the Ni₃Ti compound and different of that found in the NiTi compound.

[6] MICROSTRUCTURE CHARACTERIZATION OF POLYMORPHIC TRANSFORMED BALL-MILLED ANATASE TiO₂ BY RIETVELD METHOD

Dutta H. Sahu P. Pradhan SK. De M. - *Materials Chemistry & Physics*. 77(1):153-164, 2003

Rietveld's whole powder profile fitting method based on crystal structure refinement is applied to extract the microstructure information of several polymorphic TiO₂ phases grown simultaneously during high-energy planetary ball milling of anatase phase. The advantages of the present method of analysis over the other methods of X-ray line profile analysis are discussed in detail. Particle size and lattice strain values of all the phases are found to be anisotropic for the first time and the polymorphic phase transformations have been analyzed in this light.

[5] MG-NI ALLOYS FOR HYDROGEN STORAGE OBTAINED BY BALL MILLING

Urretavizcaya G. Garcia G. Serafini D. Meyer G. - *Latin American Applied Research*. 32(4):289-294, 2002

The effect of the atmosphere and milling time on the structural, morphological and hydrogen reactivity properties of the mechanically alloyed Mg-Ni system is studied. Nanocrystalline Mg₂Ni formed by ball milling in argon exhibits a higher thermal stability than the metals amorphized by milling during shorter times. It is shown that the initially fast hydrogen absorption kinetics of Mg₂Ni at 250°C strongly depends on the crystallinity degree. When the Mg-Ni mixture is mechanically alloyed under hydrogen atmosphere, MgH₂ is formed at first and Mg₂NiH₄ is obtained later. Samples prepared in argon show a higher hydrogen absorption capacity than those obtained in hydrogen.

[4] FORMATION OF MG-AL-TI/MGO COMPOSITE VIA REDUCTION OF TiO₂

Lai MO. Lu L. Chung BY. - *Composite Structures*. 57(1-4):183-187, 2002



This paper investigates the possibility of reduction of TiO₂ through the mechanical alloying of Mg and TiO₂ powders, Elemental Mg and Al powders were first mixed with TiO₂ according to the nominal composition of Mg5wt.-%-Al17.15wt.-%TiO₂. The powder mixture was mechanically milled in a planetary ball mill for different durations. Two types of DSC curves have been observed. The unmilled powders showed no reaction when heated to about 500 degreesC, while an exothermic reaction in the mechanically milled powders was observed at about 390 degreesC. Further investigation of the heat treatment of the mechanically milled powders showed the complete disappearance of TiO₂ and the formation of MgAl₂O₄ after annealing at 400 degreesC, implying that reduction of TiO₂ has occurred via the mechanical activation and annealing processes.

[3] FABRICATION OF NANOCRYSTALLINE AND AMORPHOUS CHEVREL PHASE PbMo6S8 POWDER BY BALL MILLING

Niu HJ. Hampshire DP. - *Physica C*. 372(Part 2):1145-1147, 2002

Chevrel phase PbMo₆S₈ (PMS) superconducting powder ball-milled from 5 to 200 It has been investigated using Xray diffraction (XRD) and scanning and transmission electron microscopy. Ball milling produced particles with crystallites of PMS in the range 10-100 nm and amorphous phase. From XRD data, it was found that the crystallite size decreased and lattice strain increased with increasing milling time until by 200 h the material was predominantly amorphous. The powder ball-milled for 100 h was subsequently annealed at temperatures from 400 to 1000 degreesC for 8 h, Annealing at 600 degreesC and above released the lattice strain, At 800 degreesC and above the amorphous phase crystallised and grains coarsened with sizes in the range 50-100 nm

[2] LOW-TEMPERATURE SUPERPLASTIC BEHAVIOR OF A SUBMICROMETER-GRAINED 5083 AL ALLOY FABRICATED BY SEVERE PLASTIC DEFORMATION

Park KT. Hwang DY. Chang SY. Shin DH. - *Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science*. 33(9):2859-2867, 2002

A submicrometer-grained structure was introduced in a commercial 5083 Al alloy by imposing an effective strain of similar to 8 through equal channel angular pressing. In order to examine the low-temperature superplastic behavior, the as-equal channel angular pressed (as-ECAP) samples were tensile tested in the strain rate range of 10⁻⁵ to 10⁻¹ s⁻¹ at temperatures of 498 to 548 K corresponding to 0.58 to 0.65 T-m where T-m is the incipient melting point. The mechanical data of the alloy at 498 and 548 K exhibited a sigmoidal behavior in a double logarithmic plot of the maximum true stress vs true strain rate. The strain rate sensitivity was 0.1 to 0.2 in the low- and high-strain rate regions and 0.4 in the intermediate-strain rate region, indicating the potential for superplasticity. At 523 K, instead of the sigmoidal behavior, a strain rate sensitivity of 0.4 was maintained to low strain rates. A maximum elongation of 315 pct was obtained at 548 K and 5 x 10⁻⁴ s⁻¹. The activation energy for deformation in the intermediate-strain rate region was estimated as 63 kJ/mol. Low-temperature superplasticity of the ultrafine grained 5083 Al alloy was attributed to grain boundary sliding that is rate-controlled by grain boundary diffusion, with a low activation energy associated with nonequilibrium grain boundaries. Cavity stringers parallel to the tensile axis were developed during deformation, and the failure occurred in a quasi-brittle manner with moderately diffusive necking.

[1] AMORPHOUS SILICON FORMATION BY SHEARING AT ULTRAHIGH PRESSURE

Furuichi H. Fujii N. Kanno Y. Ito E. Tanaka H. - *Journal of Non-Crystalline Solids*. 311(1):104-106, 2002

Transmission X-ray diffraction pattern taken from silicon sheared at 10 GPa revealed 8 halo rings. Their densest radii correspond to spacings of 308.4, 264.7, 187.2, 160.5, 152.8, 124.8, 104.9, and 91.5 pm, respectively. The ratios of these values correspond to those of interplanar spacings (1 11), (2 0 0), (2 2 0), (3 11), (2 2 2), (4 0 0), (3 3 1) and (4 2 0) of face-centered cubic (fcc) lattice. These facts suggest the formation of amorphous state based on fcc structure. This state remains at atmospheric pressure.



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