



**RESEAU FRANÇAIS DE
MECANOSYNTHESE**

Lettre N°134

Mai 2006

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

Bureau du RFM :

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Bulletin d'adhésion au RFM 2006 / 2006 RFM 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

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The check has to be to the order to : Reseau Francais de Mecanosynthèse

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Le site web du RFM est :

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

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

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

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Congress and School Announcements
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6th International Conference on the Scientific and Clinical Applications of Magnetic Carriers,
May 17-20, 2006

Krems (Vienna Region), Austria

(Contact: WEBSITE:

http://www.magneticmicrosphere.com/other/MagMeet2006_First_Announcement.pdf)

European Society for Precision Engineering and Nanotechnology 6th International Conference
(euspen 2006),

May 28-June 1, 2006

Congress Casino in Baden (near Vienna), Austria

(Contact: WEBSITE: <http://baden2006.euspen.com/>)

16th European Conference of Fracture, Failure Analysis of Nano and Engineering Materials and Structures (ECF-16),

July 3-7, 2006

Alexandroupolis, Greece

(Contact: E. Gdoutos, School of Engineering, Democritus University of Thrace, GR-671 00, Xanthi, Greece, FAX: +30-25410-79652, EMAIL: egdoutos@civil.duth.gr , WEBSITE: <http://ecf16.civil.duth.gr/>)

6th IEEE Conference on Nanotechnology,

July 16-20, 2006

Westin Hotel, Cincinnati, Ohio

(Contact: WEBSITE: <http://www.ececs.uc.edu/~mcahay/Nano2006/index2006.html>)

International Conference on Nanoscience and Technology (ICN&T 2006),

July 30-August 4, 2006

Convention Center, Basel, Switzerland

(Contact: WEBSITE: <http://www.icnt2006.ch/>)

- **INCOME 2006**

<http://www.solid.nsc.ru/INCOME2006/default.htm>

Nanophotonics Sessions, Optics & Photonics, SPIE Event,

San Diego Convention Center, San Diego, California

August 13-17, 2006

<http://spie.org/conferences/calls/06/op/>

NANO2006: 8th International Conference on Nanostructured Materials ,

- National Science Seminar Complex, Indian Institute of Science, Bangalore, India

August 20-25, 2006

- (Contacts: K. Chattopadhyay & A. Chokshi, FAX: +91-80-23601991,

- EMAIL: nano2006@met.iisc.ernet.in

- , WEBSITE: <http://met.iisc.ernet.in/~nano2006>)

International Conference on Micro-Nano-technology Development for Aerospace Applications
(CANEUS 2006),

Toulouse, France

August 27-September 1, 2006

Contact: WEBSITE: <http://www.asmeconferences.org/CANEUS06/index.cfm>

13th ISMANAM2006



Warsaw
27-31 August 2006, Warsaw, Poland
Warsaw University of Technology
Faculty of Materials Science and Engineering
www.inmat.pw.edu.pl/ismanam2006
ismanam2006@inmat.pw.edu.pl

Trends in Nanotechnology (TNT2006) Conference,
Centre of Innovation in Micro and Nanotechnology, MINATEC, Grenoble, France
September 4-8, 2006
Contact: A. Correia, WEBSITE: <http://www.phantomsnet.net/TNT06/>

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Matériaux 2006
Nano Mat-Tech: Des NanoMateriaux aux NanoSystemes,
Dijon, France
November 13-17, 2006
Contact: E. Gaffet, EMAIL: Eric.Gaffet@utbm.fr , WEBSITE: <http://www.materiaux2006.net>

- -----
Materials Science and Materials Mechanics at the Nanoscale conference,
Politecnico di Bari, Bari, Italy
November 19-23, 2006
(Contacts: L. Lamberti, EMAIL: lamberti@poliba.it , F. Sciammarella, EMAIL: sciammarella@iit.edu ,
and S. Toyooka, EMAIL: toyooka@mech.saitama-u.ac.jp

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**SCIENCE ET TECHNOLOGIE DES POUDRES &
POUDRES ET MATERIAUX FRITES**
"De la poudre au produit fini à propriétés d'usage maîtrisées"
Ecole des Mines d'Albi, 23 – 25 mai 2007
<http://stp2007.enstimac.fr>
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Appel à Communications

Symposium I : Nano Mat - Tech : des NanoMatériaux aux NanoSystèmes

Matériaux 2006 (<http://www.materiaux2006.net>)

23 Sociétés Savantes françaises Matériaux participeront à l'organisation de :

Matériaux 2006 (Dijon, du 13 au 17 Novembre 2006)

sous l'égide de la *Fédération Française des Matériaux*

19 Colloques seront proposés pour 1000 participants.

Dont le **Symposium I Nano Mat - Tech : des NanoMatériaux aux NanoSystèmes**.

Comité Scientifique (coordination assurée par E. Gaffet – RFM & SF2M)

3AF (A Lasalmonie), GFC (G. Fantozzi, M. Boussuge), GFCC (S. Veessler), GFEC (D. Guerard, S. Bonnamy), GFA (J. BRENDLE, B. Made, F. Bergaya), GFP (JC Daniel, JP Pascault), MECAMAT (S. Castagnet, R. Seguela), RFM (E. Gaffet), SFC (B. Sillion, JC Bernier), SFGP (S. Begin – Colin), SF2M (JM Chaix), SFMu (J. Werckmann), SFP (Jean.Vannimenus), SFN (O. Isnard), SFV (M. Cantarel), Club Nano –Micro Technologie (M. Lahmani)

En 2001, le marché des nanotechnologies représentait 40 milliards d'Euros (*DGE / Minefi*) et devrait atteindre les 1.000 milliards de dollars par an en 2015 (*National Science Foundation*), soutenant l'activité de plus de 2 millions d'emplois à l'échelle mondiale. Les matériaux et les procédés nanostructurés représenteraient plus de 30% de ce marché, soit près de 340 milliards de dollars (*Hitachi Research Institute*). L'effort mondial dans le domaine de la recherche (financements privé et public) est estimé à 4 milliards de dollars en 2003 (*NanoBusiness Alliance*).

Dans ce contexte hautement compétitif, ce colloque aura pour objectifs de présenter aussi bien les innovations scientifiques que technologiques dans le domaine des Nanomatériaux (métalliques, inorganiques et organiques, polymères, semi-conducteurs, nano-composites). Les Nanomatériaux sont des matériaux ayant une taille nanométrique comme des nanopoudres et des nanoparticules, ainsi que des matériaux pour lesquels une des échelles caractéristiques d'organisation et/ou de morphologie est de 1 à 100 nm, c'est-à-dire les nanotubes, les revêtements / dépôts, les massifs nanostructurés / nanocomposites auto-organisés,

Les thématiques développées couvriront l'élaboration, la mise en œuvre et la caractérisation des nanomatériaux, ainsi que leur intégration dans la conception et le développement de nanosystèmes. Un accent particulier sera porté aux relations entre les caractéristiques granulométriques, morphologiques et structurales des nanomatériaux et leurs propriétés physico-chimiques. Les domaines tels que la thermodynamique hors équilibre, la cinétique, la modélisation et la simulation seront considérés. Les aspects « Nanomatériaux et Sécurité » seront également pris en compte.

Ce colloque aura une vocation internationale avec la participation de chercheurs et d'industriels spécialistes des domaines concernés. Les communications se feront en Français et en Anglais.



Programme Scientifique

Liste des conférenciers invités (au 13 Avril 2006)

« *Les nanotubes de carbone pour de nouvelles fibres hautes performances et multifonctionnelles* »

Ph. Poulin
Centre de Recherche Paul Pascal, CNRS-UPR 8641,
Avenue Schweitzer, 33600 Pessac.

« *Propriétés magnétiques locales d'architectures de clusters et de molécules auto-organisés sur des surfaces* »

JP Bucher
Institut de Physique et de Chimie des Matériaux de Strasbourg,
UMR 7504 CNRS - Université Louis Pasteur, - 23 rue du Loess, BP 43, F-67034 Strasbourg Cedex 2

« *Différents procédés d'obtention de céramiques nanostructurées* »

L. Montanaro
Politecnico di Torino – Dipartimento di Scienza dei Materiali e Ingegneria Chimica
Corso Duca degli Abruzzi, 24 – 10129 Torino Italia

« *Propriétés mécaniques des nanomatériaux* »

G. Le Caër
GMCM
UMR-CNRS 6626, Université de Rennes-I, Campus de Beaulieu, - Bât. 11A, 35042 Rennes Cedex

« *Nanostructures fonctionnelles à partir d'aggrégats préformés en phase gazeuse : synthèse et propriétés* »

A. Perez
Laboratoire de Physique de la Matière Condensée et Nanostructures -
UMR 5586 CNRS - Université Claude Bernard - Lyon I
43 Bd du 11 novembre 1918
F-69622 Villeurbanne Cedex, France

« *L'Après Gutenberg: Les Encres Electrophorétiques pour Papier Electronique* »

G. Hadziioannou
Laboratoire d'Ingénierie des Polymères pour les Hautes Technologies (LIPHT)
UMR 7165 CNRS - ECPM – University Louis Pasteur Strasbourg

Thèmes Scientifiques du Symposium

I. Elaboration (0D à 3D)

0D à 1 D – NanoPoudres, Nanocharges et NanoFils

Elaboration par voies chimiques, Elaboration par voies physiques, Elaboration par voies mécaniques (Hypercorroyage, Mécanosynthèse, Extrusion, Injection, Compactage, Films, Activation Mécanique) & Biomimétisme

2D – 3D - Revêtements et Nanocomposites Massifs

(Métalliques, Polymères, Semiconducteurs, Inorganiques et Organiques, NanoCharges)
Assemblage, Auto – Organisation, Structures hiérarchisées, Des hybrides aux nanocharges, Biomimétisme, Nouveaux procédés de Consolidation / Frittage : SPS, HyperCorroyage, Micro-onde... & Mise en forme

Systemes Nano, Méso et HyperPoreux

Nanotubes, Fullerènes, NanoCages & Aérogels

NanoMatériaux Dispersés

Colloïdes & NanoFluides

II. Propriétés et Techniques de Caractérisations

Mécaniques (nano – micro, amorphe – nano...), Electriques / Diélectriques, Magnétiques, Optiques, optoélectroniques, Chimiques (Réactivité, Catalyse, Stockage de gaz), biochimiques (bio –compatibilité)

La modification des propriétés induite par la nanostructure sera particulièrement discutée

III. Modélisation – Simulation

IV. Thermodynamique / Cinétique

V. Industrialisation / Applications - Conception et développement de nanosystèmes – Formation

Mécanique, Electro - Magnétisme, Electronique, Chimie, Nanocomposants, Nanosystèmes, Textile, Optique, Biologie, Médecine

VI. NanoMatériaux et Sécurité

Techniques de détection, toxicologie, intégration de systèmes sécurisés, aspects sociétaux et environnementaux



**SCIENCE ET TECHNOLOGIE DES POUDRES
& POUDRES ET MATERIAUX FRITTES**
"De la poudre au produit fini à propriétés d'usage maîtrisées"

Ecole des Mines d'Albi, 23 – 25 mai 2007
<http://stp2007.enstimac.fr>

*Groupe Science et Technologie des Poudres
Commission Poudres et Matériaux Frittés (SF2M-GFC-RFM)
Société Française de Métallurgie et de Matériaux
Groupe Française de Céramique
Réseau Française de Mécanosynthèse
GDR Midi Milieux Divisés
Groupes SFGP : Solides Divisés et Formulation
de la Société Française de Génie des Procédés*

La science et la technologie des poudres réunissent les chercheurs et les ingénieurs du génie des procédés et du génie des matériaux travaillant dans les domaines utilisant des poudres pour l'élaboration des produits industriels : chimie fine, pharmacie, industrie céramique, métallurgie des poudres, industrie minérale, agro-alimentaire, ...

Ces systèmes intéressent également les spécialistes de la physique car les milieux granulaires font actuellement l'objet de recherches actives.

Ce colloque, co-organisé par les deux communautés «Science et Technologie des Poudres» et «Poudres et Matériaux Frittés» qui tenaient séparément leurs colloques, a pour objectif de faire le point sur les connaissances actuelles et de favoriser les échanges entre chercheurs, équipementiers et industriels. Il concernera tous les aspects de la technologie des poudres et des milieux granulaires: génération, réactivité, formulation, traitement, manipulation et mise en forme des poudres par granulation, enrobage, encapsulation, compression et frittage.

Une partie importante du colloque sera consacrée à la caractérisation des poudres, la physique et la chimie de leur comportement ainsi qu'à l'hygiène et la sécurité depuis leur synthèse jusqu'à la fin de la vie du produit.

Des communications sont souhaitées dans tous les domaines de la technologie des poudres et notamment :

- Procédés d'élaboration des poudres
- Caractérisation des poudres
- Comportement et applications des poudres sèches, en suspension et en pâte
- Physique des milieux granulaires
- Réactivité, biodisponibilité des poudres
- Procédés de mise en forme et propriétés d'usage
- La manipulation et la mise en œuvre des poudres
- Nanopoudres et matériaux nanostructurés
- Les poudres et le développement durable
- Hygiène, sécurité et explosions de poudres
- Frittage, consolidation et maîtrise des microstructures
- Poudres et frittage non conventionnel : micro-ondes, SPS, ...etc

Comité Local d'Organisation

EMAC

Michel Baron
John Dodds
Anne-Marie Fontes
Rita Franco
Laurence Galet
Driss Oulahna
Elisabeth Rodier

Comité Scientifique

Michel Baron (EMAC)
Claude Carry (PMF)
John Dodds (EMAC)
Eric Gaffet (PMF & RFM)
Laurence Galet (EMAC)
Pierre Guigon (SFGP)
Hervé Muhr (SFGP)
Guy Nicolas (PMF)
Jean-Claude Niepce (PMF)
Driss Oulahna (EMAC)
Olivier Pouliquen (GDR Midi)
Farhang Radjai (GDR Midi)
Elisabeth Rodier (EMAC)



Nanotech Jobs And / Or Post Doc Position - Contract Proposal

Looking for Post Doc Position

Hamzaoui Rabah

Date of birth 06/01/1974

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90800 Belfort Cedex France.

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Email: rabah.hamzaoui@utbm.fr

Dear Sir or madame

I'am fulfilling a PHD degree at the University of Belfort dealing with nanostructured materials (Nanomaterials Research Group – NRG / UMR 5060 CNRS / UTBM). I have finished my PHD in December 2004. My work thesis is about: mechanical alloying and magnetic properties of Fe-Ni alloys.

I have 8 papers published, 4 participations at international conferences and 5 participations at national conferences.

I have the opportunity to work with Professor O. Elkedim and Dr. E. Gaffet on nanostructured powder during my thesis and I have also the occasion to have Dr. J. M. Grenèche and Dr. G. Le Caër as the rapporteurs (referees) of my thesis. Their great open minds on this field have proved to me that there are still several answered questions and so much e sill to be done. The post doc is a great opportunity for me to have an enlarged vision on the field of nanomaterials specially nanomagnetic materials. Therefore, my enthusiasm is great and my motivation is so high to be able to work in this domain. Thus I would like to ask you the possibility of have a grant to be able to give me the opportunity to expend my skills.

Sincerely Yours

Skills

- Mechanical alloying and Nanomaterials (magnetic and physic-chemical properties)
- Materials sciences (powders metallurgy, dielectric materials)
- Metallization techniques (evaporation, magneto sputtering)
- Characterisation techniques (RX, SEM, TEM, DSC, Mössbauer spectra, Acoustic..)



Post Doc Position Proposal

From Ch. Gras (September 2005)

Postdoctoral Researcher in Electron Microscopy of Multifunctional Ceramics

Grade RAlA / Salary: £19,460 to £29,128 / Job Ref No DJ05/018

Applications are invited for a postdoctoral researcher to join the Electron Microscopy Group of Professor David Cockayne FRS to work on high resolution electron microscopy and energy loss studies of SrTiO₃ and BaTiO₃ multifunctional ceramics, with a particular focus on grain boundary structures. This is part of an international collaborative programme ("INCEMS") funded by the EC, and involves research groups from Germany, Ireland, France, Slovenia and the UK. Your role within the project will be to use the unique aberration corrected TEM/STEM, housed in the Department, to investigate the microstructure of interfaces at the highest possible resolution, and to interact with growers and modellers within the collaboration. The characterization will be by a range of techniques including high-resolution TEM (HREM, HAADF) and EELS. The post is available from October 2005 (or as soon as possible thereafter) for up to 33 months. Applicants should have a doctorate in materials science, physics or a related discipline and have demonstrated experience in high resolution electron microscopy and in sample preparation of crystalline materials. Experience with EELS would be an advantage. Evidence of ability in the use of computer software, preferably for image simulation, is required. Proven ability to identify research objectives and meet agreed deadlines, self-motivation and flexibility are essential. Excellent written and interpersonal communication skills, the ability to work effectively as part of a team, and evidence of a good publication record are expected. Willingness to travel for short periods to work with collaborators in Europe is required.

Further particulars, including instructions on applying for this post, are available from the web-site: <http://www.materials.ox.ac.uk> or from Mrs K Fewings, Department of Materials, University of Oxford, Parks Road, Oxford OX1 3PH (email: posts@materials.ox.ac.uk), or telephone 01865 273680 (post reference DJ05/018). The closing date for applications is 30 September 2005 with interviews currently planned for the week beginning 17 October 2005.

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

Academic Staff

Professorship of Materials Modelling

Applications are invited for the above post, tenable from 1 January 2006, or such later date as may be arranged. The University seeks to appoint a person with a record of outstanding international excellence in research in materials modelling who, through leadership and the distinction of his or her contribution to the field, will ensure the pursuit of the highest standards in teaching and research in materials modelling at Oxford and its wide recognition outside. The Professorship of Materials Modelling is held in the Department of Materials. A non-stipendiary fellowship at St. Anne's College is attached to the professorship. Further particulars, including details of how to apply, are available from <http://www.admin.ox.ac.uk/fp/> or from the Registrar, University Offices, Wellington Square, Oxford OX1 2JD (Tel: 01865 270200). The closing date for applications is Monday 3



Bibliographie

Mechanosynthesis references

Références NanoMatériaux / Mécanosynthèse

Synthesis of Fe-Co based nanomagnetic materials

Li-HF; Ramanujan-RV

TRANSACTIONS-OF-THE-INDIAN-INSTITUTE-OF-METALS. DEC 2005; 58 (6) : 965-970

Processing of FeCo based nanomagnetic materials was carried out by chemical synthesis, crystallization from amorphous precursors and mechanical alloying. It was found that the products from chemical synthesis depended on the concentration of reaction solution. Uniform fiber like and spheroidal nanopowders were produced using Fe+2 rich and Co+2 rich reaction solutions respectively. The chemically synthesized FeCo based alloys was found to be amorphous. Through crystallization of melt spun amorphous precursors, high density nanocrystalline Fe_{44.5}Co_{44.5}Zr₇B₄ alloy was obtained; the nanocrystals had a compacted dendritic morphology. Spheroidal nanocrystalline structure was successfully obtained after mechanical milling of Fe_{44.5}Co_{44.5}P₇B₄ for more than 20 h at 300 rpm. The fiber like and compact dendritic morphology was not suitable for soft magnetic properties. Due to the pinning of magnetic domains, the powder nanostructured FeCo based alloys had inferior soft magnetic properties to the ribbon form alloys. The fiber like chemically synthesized and mechanically alloyed FeCo based alloys powders showed large coercivities. The magnetization of chemically synthesized FeCo alloy powders was low, which was considered to be due to solid solution contaminations, amorphous phase formation and the oxidation layer.

Strain induced cementite dissolution in pearlitic steels as a classical example of mechanical alloying

Lojkowski-W; Ivanisenko-Y; Fecht-HJ

TRANSACTIONS-OF-THE-INDIAN-INSTITUTE-OF-METALS. DEC 2005; 58 (6) : 993-1001

Severe plastic deformation (SPD) is widely used now to produce a nanocrystalline structure in metals. Being applied to alloys it allows not only to refine the microstructure, but also lead to dissolution of second phases. An example of such effect of SPD is complete dissolution of cementite in eutectoid steel. In the present paper the mechanism of the strain induced cementite dissolution was analysed in terms of a model where a plastic phase (ferrite) flows under high pressure and high external stress around hard precipitates like a viscous fluid. The friction at the precipitate/matrix interface leads to two effects. One is a high strain energy accumulated in the carbides, which may strongly contribute to their thermodynamic instability, and the second is their wear due to the flow of the ferrite. The dissolution of carbon with the ferrite phase can be considered as a driven transformation, where two driving forces for mass transport flow are competing.. One is the mechanically induced drag of carbon atoms, which depends mainly on the deformation rate. The second is diffusion of carbon induced by thermodynamic energy gradients, which are connected with high strains in the precipitates.

Characteristics of mechanically alloyed nanostructured Ni-Al catalysts in H₂O₂ decomposition

Dey-PK; Gupta-MD; Pabi-SK

TRANSACTIONS-OF-THE-INDIAN-INSTITUTE-OF-METALS. DEC 2005; 58 (6) : 1037-1044

Catalytic characteristics of mechanically alloyed nanostructured phases of Ni-Al system in H₂O₂ decomposition were investigated. The catalysts were characterized by XPS, XRD, SEM, surface area measurement, and particle size analysis. The activity, activation energy, and the deactivation characteristics of each catalyst were studied for H₂O₂ decomposition at temperatures 20 - 40 degrees C controlled to +/- 0.02 degrees C. The chemical states of Ni and Al of the nanostructured catalysts were analyzed by XPS and correlated with the catalytic performances of these materials. The alloyed-Ni (i.e. Ni bonded to Al) content on the surface of the nanostructured NiAl phase generally decreased with the increase in Ni-content from 30 to 65 at. % Ni, whereas the surfaces of the Ni(Al) solid solution of Ni₉₀Al₁₀ composition or the Al₃Ni phase were relatively depleted of alloyed-Ni content and enriched with aluminum oxide. The nanocrystalline non-equilibrium NiAl phase of Ni₃₀Al₇₀ composition showed the most pronounced catalytic activity, while the conventional microcrystalline alloy powder of the same chemical composition obtained through melting route was catalytically inactive. Deactivation of the catalysts was accompanied with the depletion of alloyed-Ni, formation of Al₂O₃ and metallic-Ni on the catalyst surface, which indicated that Ni bonded to Al were the effective specie for the catalytic activity.

On the evolution of a nanocrystalline phase from the Al-Cu-Fe quasicrystalline alloy during high energy ball milling

Yadav-TP; Mukhopadhyay-NK; Tiwari-RS; Srivastava-ON

TRANSACTIONS-OF-THE-INDIAN-INSTITUTE-OF-METALS. DEC 2005; 58 (6) : 1169-1176

Mechanical milling of an Al-Cu-Fe quasicrystalline alloy was performed in a high energy ball mill (Szegevari attritor mill) at a constant speed of 400 rpm for various milling time (from 0.5 to 40 h) under liquid hexane medium with a ball to powder ratio



of 40:1. Scanning electron microscopy (SEM), transmission electron microscopy (TEM) and x-ray diffraction (XRD) techniques were employed for characterizing the milled and unmilled samples. The evolution of nanocrystalline (NC) phase (bcc, $a = 0.29$ nm) was observed from the quasicrystalline phase after 5h of milling. This nanocrystalline phase, which was quite stable up to 40h of milling, did not transform to amorphous or any other metastable phase. The size of crystalline particles was found to vary from 60nm to minimum 10nm for different milling durations. It is interesting to note that the strain induced in the milled samples tends to increase along with milling time. The possible mechanisms for the formation of nanocrystalline phase will be put forward based on the evolution of the structural and microstructural features.

Fabrication of bulk nanocrystalline Fe-C alloys by spark plasma sintering of mechanically milled powder (vol 53, pg 863, 2005)

Zhang-HW; Gopalan-R; Mukai-T; Hono-K
SCRIPTA-MATERIALIA. MAY 2006; 54 (10) : 1827-1828

Mechanochemical synthesis and electrochemical properties of nanosized SnS as an anode material for lithium ion batteries

Li-Y; Tu-JP; Wu-HM; Yuan-YF; Shi-DQ
MATERIALS-SCIENCE-AND-ENGINEERING-B-SOLID-STATE-MATERIALS-FOR-ADVANCED-TECHNOLOGY.
MAR 15 2006; 128 (1-3) : 75-79

As an alternative of carbon anode for lithium ion batteries, SnS nanoparticles were synthesized by mechanical milling. The morphology and microstructure of SnS particles were characterized by scanning electron microscopy (SEM) and X-ray diffraction (XRD). With increasing the milling time, the amount of SnS increased due to the intensive alloying between Sn and S powders, while the particle size decreased gradually. Electrochemical properties of the synthesized materials were investigated using, Li ion model cells. The irreversible capacity loss in the first discharge-charge cycle is attributed to the formation of Li₂S originated from the reaction of SnS and Li⁺. The ratio of Li₂S and S was suggested to have immense impacts on the cycling performance. Li₂S was considered to be more appropriate for buffering the volume change than S. In the following cycles, the nanosized SnS anode material exhibited good cycle stability and delivered a discharge capacity of 400 mAh/g after 40 cycles

Fabrication of nanocrystalline Mg₃X₂ (X = Bi, Sb) with supersaturated solid solubility by mechanical alloying

Xin-HX; Qin-XY; Zhu-XG; Zhang-J; Kong-MG
MATERIALS-SCIENCE-AND-ENGINEERING-B-SOLID-STATE-MATERIALS-FOR-ADVANCED-TECHNOLOGY.
MAR 15 2006; 128 (1-3) : 192-200

Mechanical alloying plus hot-pressing was employed to prepare nanocrystalline Mg₃X₂ (X=Bi, Sb) compounds that were characterized by microstructural examinations and dc electrical resistance measurements. The results indicated that Mg₃Sb_{2+y} compounds with mean grain size similar to 30 nm and with solute Sb atoms as much as 5 at.% in the powder specimens were successfully prepared. By comparison, Mg_{3+x}Bi₂ compound with solute Mg atoms as much as 13 at.% could be fabricated with similar grain sizes. Hot-pressing at 673 K (for 20-50 min) only led to a limited grain growth (< similar to 10 nm) for both compound systems. However, experiments showed that hot-pressing could further facilitate alloying of the elements in Mg-Bi system. In contrast, elementary Sb phase formed gradually in the Mg₃Sb_{2+y} system with supersaturated solute Sb atoms during hot-pressing presumably due to its high ionicity and large number of intragranular imperfections, as revealed by large lattice strain. dc resistance measurements indicated that a semimetal-metal transition occurred in Mg_{3+x}Bi₂ system as solute Mg x reached similar to 0.72; while in Mg₃Sb_{2+y} system a transition of its conduction behavior from logarithmic law to Mott's T^{-1/4} law was observed at T < similar to 190 K as solute Sb content y > similar to 0.097, suggesting that phonon-assisted hopping Occurred in the Mg₃Sb_{2+y} at the low temperatures.

Preparation of porous BaTiO₃-based ceramics by high-energy ball-milling process

Kim-JG; Ha-JG; Lim-TW; Park-K
MATERIALS-LETTERS. JUN 2006; 60 (12) : 1505-1508

Porous BaTiO₃-based ceramics containing potato-starch (0-15 wt.%) were prepared by wet mixing and the effect of high-energy ball-milling time (1-20 h) on the electrical property and microstructure of the porous ceramics has been investigated. With increasing potato-starch content and ball-milling time, the porosity of the BaTiO₃-based ceramics containing potato-starch increased and decreased, respectively. The grain size decreased and increased, with increasing potato-starch content and ball-milling time, respectively. The room-temperature electrical resistivity of the porous ceramics slightly increased with increasing potato-starch content, and decreased with increasing ball-milling time. PTCR jump of the porous ceramic was slightly increased with increasing potato-starch content, while it was slightly decreased with increasing ball-milling time.

High-frequency induction heat sintering of mechanically alloyed alumina-yttria-stabilized zirconia nano-bioceramics

Kim-SW; Khalil-KAR



JOURNAL-OF-THE-AMERICAN-CERAMIC-SOCIETY. APR 2006; 89 (4) : 1280-1285

Nanostructured alumina-20 vol% 3-yttria-stabilized zirconia (3YSZ) powder composites were synthesized by the wet-milling technique. The starting materials were a mixture of alumina micropowder and 3YSZ nanopowders. The mixtures were optimized for good sintering behaviors, high hardness, and toughness. Nano-crystalline grains were obtained after milling for 24h. The nano-structured powders were then processed to full density at different temperatures by high-frequency induction heat sintering. Effects of sintering temperature on the hardness, toughness, and microstructure properties have been studied. Al₂O₃-3YSZ composites with higher hardness, toughness, and smaller grain size have successfully been developed at relatively low temperatures by this technique

Controlling intermetallic compound growth in SnAgCu/Ni-P solder joints by nanosized Cu₆Sn₅ addition

Kao-ST; Lin-YC; Duh-JG

JOURNAL-OF-ELECTRONIC-MATERIALS. MAR 2006; 35 (3) : 486-493

Nanosized Cu₆Sn₅ dispersoids were incorporated into Sn and Ag powders and milled together to form Sn-3Ag-0.5Cu composite solders by a mechanical alloying process. The aim of this study was to investigate the interfacial reaction between SnAgCu composite solder and electroless Ni-P/Cu UBM after heating for 15 min. at 240 degrees C. The growth of the IMCs formed at the composite solder/EN interface was retarded as compared to the commercial Sn3Ag0.5Cu solder joints. With the aid of the elemental distribution by x-ray color mapping in electron probe microanalysis (EPMA), it was revealed that the SnAgCu composite solder exhibited a refined structure. It is proposed that the Cu₆Sn₅ additives were pinned on the grain boundary of Sn after heat treatment, which thus retarded the movement of Cu toward the solder/EN interface to form interfacial compounds. In addition, wetting is an essential prerequisite for soldering to ensure good bonding between solder and substrate. It was demonstrated that the contact angles of composite solder paste was < 25 degrees, and good wettability was thus assured.

Influence of Ni concentration and Ni₃Sn₄ nanoparticles on morphology of Sn-Ag-Ni solders by mechanical alloying

Lee-HY; Duh-JG

JOURNAL-OF-ELECTRONIC-MATERIALS. MAR 2006; 35 (3) : 494-503

The mechanical alloying (MA) process was employed as an alternative method to produce the lead-free solder pastes of Sn-3.5Ag-xNi (x = 0.1, 0.5, 1.0, 1.5, and 2.0) in this study. When the Ni concentration was low (x = 0.1, 0.5), MA particles agglomerated to a flat ingot with particle sizes > 100 μm. For higher Ni concentration (x = 1.0, 1.5, and 2.0), MA particles turned into fragments with particle sizes < 100 μm. The particle size of the solders appeared to be dependent on the Ni concentration. To reduce the particle size of SnAgNi alloys with low Ni concentration, Ni₃Sn₄ nanoparticles were doped into Sn and Ag powders to derive a Ni₃Sn₄-doped solder. For the Ni₃Sn₄-doped solder, the particle size was smaller than that doped by the pure Ni. The distinction of milling mechanism between Ni₃Sn₄-doped solder and the pure Ni-doped solder by MA process was probed and discussed. In addition, differential scanning calorimetry (DSC) results ensured its feasibility in applying the solder material in the reflow process. Wettability tests between solders and Cu substrate also revealed that the wetting angles for Ni₃Sn₄-doped solder with low Ni concentration (0.1 and 0.5 wt.%) were smaller than those for pure Ni-doped solder. The wetting angles on both Cu substrate and electroplated Ni metallization for SnAgNi solders were also comparable with commercial Sn-3.5Ag and Sn-3.0Ag-0.5Cu solders. Favorable wettability of the as-derived solder in this study was clearly demonstrated.

Nanocrystalline Fe₈₄Nb₇B₉ alloys prepared by mechanical alloying and ultra-high-pressure consolidation

Lu-W; Yang-L; Yan-B; Huang-WH; Lu-B

JOURNAL-OF-ALLOYS-AND-COMPOUNDS. MAR 9 2006; 413 (1-2) : 85-89

Nanocrystalline soft magnetic alloys such as Nanoperm have attracted a great deal of attention over the past 10 years. In the present paper, the mechanical alloying process followed by ultra-high-pressure consolidation was used to produce nanocrystalline Nanoperm (Fe₈₄Nb₇B₉) alloy. An elemental powder mixture of Fe₈₄Nb₇B₉ (in at.%) was milled in a planetary ball mills. The structural evaluation of the powder during alloying was studied with the use of X-ray diffraction (XRD) and scanning electron microscopy (SEM). The results show that the average grain size of the milled powder was about 5.5 nm after milling for 35 h and it accorded with the relation: $D = Kt^{-0.6}$ where D is the average grain sizes, t the milling time, and K is a coefficient. Subsequently, the milled powder was consolidated under a high pressure (5.5 GPa). The XRD investigations of the consolidated samples revealed that after consolidating, the bulk samples remained nanocrystalline with grain size about 8-10nm and there is no phase change during consolidating. The relative density of the bulk sample was about 98.3%. The spontaneous magnetization M_s and coercivity H_c of the bulk sample was 155 emu/g and 360 A/m, respectively. The high coercivity is mainly due to the internal strain and anti-size defects developed during mechanical alloying and consolidation. In addition, the coercivity H_c decreased to 120A/m after annealing at 350 degrees C for 30min. On the basis of the results of the present study, the mechanical alloying technique accompanied by ultra-high-pressure consolidation can provide powerful tools for the fabrication of nanocrystalline materials.



Influence of the Co-2-W coating prepared by mechanical alloying on the electromagnetism parameter of Fe_{0.7}Ni_{0.3}

Wang-Q; Guan-JG; Liu-SQ; Wang-W; Zhang-QJ

JOURNAL-OF-ALLOYS-AND-COMPOUNDS. MAR 9 2006; 413 (1-2) : 155-158

Core-shell particles of Co-2-W coated Fe_{0.7}Ni_{0.3} were prepared by mechanical alloying and characterized by TEM, XRD and SEM. The effect of the Co-2-W coating on the electromagnetism parameter of Fe_{0.7}Ni_{0.3} has been investigated. The mechanism of the interaction between the electromagnetism parameter of the core and the shell materials has been discussed. The result shows that the permittivity of Fe_{0.7}Ni_{0.3} is greatly reduced after the formation of the Co-2-W coating, but the permeability is not influenced on the whole. The electromagnetism parameter of the core-shell particles can be adjusted by controlling the electromagnetism parameter of the shell material.

High-energy ball-milled (alpha-Fe₂O₃)(alpha-Al₂O₃) system: A study on the milling time effects

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JOURNAL-OF-ALLOYS-AND-COMPOUNDS. MAR 9 2006; 413 (1-2) : 265-272

The system (alpha-Fe₂O₃)_x(alpha-Al₂O₃)_(1-x) was subjected to high-energy ball-milling, with the milling time varying between 3 and 72 h. The precursor powders, alumina and hematite, were pre-mixed in nominal concentrations $x=0.10$, 0.25 and milled in a planetary ball-mill under specific milling Conditions. The milled samples were characterized through scanning electron microscopy, X-ray diffraction and Mossbauer spectroscopy. As a result of the above analyses, the formation of two isostructural solid solutions, alpha-(Fe_{1-x}Al_x)₂O₃ and alpha-(Fe_{1-x}Al_x)₂O₃, which evolve peculiarly with the milling time, was observed. For samples with $x = 0.10$, alpha-Fe and the FeAl₂O₄ compound were identified, in addition to the solid solutions. The presence of the spinel and metallic iron phases is attributed to the abrasion of the vial and balls. For $x = 0.25$ samples, the iron content tends, for increasing milling times, to be equally shared between both solid solutions, verifying the driving force of the process to homogenize the system under milling,

Application of mechanical milling to synthesize a novel quarterly hydride

Mulana-F; Nishimiya-N

JOURNAL-OF-ALLOYS-AND-COMPOUNDS. MAR 9 2006; 413 (1-2) : 273-280

Ball milling of lithium, aluminum, nickel and graphite or carbon black was performed under 0.5 MPa of hydrogen in order to synthesize a novel quarterly hydride and quarterly hydride-carbonaceous composite. Although the intended quarterly hydride, Li_{1.6}Al_{0.8}Ni_{0.2}H₄ was not successfully synthesized as that, some modified lithium aluminohydride was obtained which might contain a little amount of nickel and reversibly absorb hydrogen. The reversible hydrogen capacity of modified lithium aluminohydride reached 2.2 wt.% at 297 K under 3.4 MPa of hydrogen. Since the lower equilibrium pressure for the Li-Al-H₂ system is estimated as 43 MPa at 297 K, the observed hydrogen capacity should be brought about by some modification of LiAlH₄ by nickel. When commercial lithium hydride was used as a starting material instead of lithium granules, modified lithium aluminohydride was not formed. The effect of the composite formation was resistance to air and anti-sticking characteristics to balls and the wall of the vial during the ball milling. The hydrogen capacity of SCTH-8h (hydrogenated SCT (stoichiometric tertiary alloy formulation), Li_{1.6}Al_{0.8}Ni_{0.2} milled for 8 h)-30 wt.% carbon black was higher than expected as calculated from linear combination of that for SCTH and that for carbon black. The cooperative effect was not found in SCTH-graphite system.

Electrochemical properties of amorphous Mg-Fe alloys mixed with Ni prepared by ball-milling

Xiao-XZ; Wang-XH; Gao-LH; Wang-L; Chen-CP

JOURNAL-OF-ALLOYS-AND-COMPOUNDS. MAR 9 2006; 413 (1-2) : 312-318

Amorphous 2Mg-Fe+x wt.% Ni alloys were synthesized by mechanical alloying of Mg and Fe elemental powders. Two kinds of ballmilling atmospheres, namely, (1) under argon and (2) first under argon and then in tetrahydrofuran (THF), were examined in this study. The effects of the Ni addition and ball-milling time and atmosphere on the microstructure and electrochemical properties of the prepared alloys have been investigated systematically. The results show that the ball-milled (2Mg-Fe) nickel-free mixture still maintains Mg and Fe elemental phases, and its discharge capacity is only 19.2 mAh g⁻¹. The Ni powder addition is advantageous for the formation of Mg-Fe amorphous structure and for the improvement of the electrochemical properties of the alloys. For the alloys milled under argon, with the Ni content x increasing, the discharge capacity of the alloys increases first and then decreases, and reaches a maximum value of 455.3 mAh g⁻¹ as $x=100$. With the milling-time extending, the discharge capacity of the alloy increases first and then decreases too. For the alloys milled under argon and then in THF, the alloys exhibit higher degree of amorphorization, bigger specific surface area and more active surfaces, and thus have better electrochemical properties than those milled under argon. With the Ni increasing, the discharge capacity increases from 249.7 mAh g⁻¹ ($x = 50$) to 565.2 mAh g⁻¹ ($x = 150$) and then decreases to 497.9 mAh g⁻¹ ($x = 200$), and the exchange current density I_0 increases from 104.8 mA g⁻¹ ($x = 50$) to 251.1 mA g⁻¹ ($x = 200$), which is consistent with the variation of the HRD of the alloy electrodes in the investigation range.

Mechanical activation of barium aluminate formation from BaCO₃-Al₂O₃ mixtures



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Chen-GH; Niu-DC

JOURNAL-OF-ALLOYS-AND-COMPOUNDS. MAR 9 2006; 413 (1-2) : 319-322

The effect of high-energy ball milling, and subsequent annealing on a mixture of Al_2O_3 and BaCO_3 has been investigated. DTA results show that the initiator decomposition temperature of BaCO_3 decreases with the increase of ball milling time duration. X-ray diffraction (XRD) measurement indicates that the nanocrystalline powder mixture is produced and no BaAl_2O_4 is formed during a high-energy ball milling process after milling for 30 h. Significant crystallization of BaAl_2O_4 from the nanocrystalline powder mixture milled for 5 h, 15 h and 30 h is observed after annealing at 900 degrees C, while a trace of BaCO_3 exists at this temperature. Single phase BaAl_2O_4 can be obtained for the sample milled for 5 h at 1000 degrees C, which is at least 200 degrees C lower than that used in the traditional solid-state method, while the secondary exist for the physical mixture at 1000 degrees C. No significant grain is observed when the milled powders crystalline phases $\text{Ba}_5\text{Al}_2\text{O}_8$ and BaCO_3 were annealed at 1000 degrees C with different milling time and the average size of the grain is about 0.3-0.5 μm .

Characterisation of lignin-carbohydrate complexes (LCCs) of spruce wood (*Picea abies* L.) isolated with two methods

Lawoko-M; Henriksson-G; Gellerstedt-G

HOLZFORSCHUNG-. MAR 2006; 60 (2) : 156-161

A method for the quantitative isolation of lignin-carbohydrate complexes (LCCs) in a softwood is presented. The isolation steps involve partial enzymatic hydrolysis of cellulose, subsequent swelling in urea, and quantitative dissolution into four major fractions: (1) a galacto-glucomannan LCC containing similar to 8% of the wood lignin; (2) a glucane LCC containing similar to 4% of the wood lignin; (3) a xylan-lignin-glucomannan network LCC (xylan > glucomannan) containing similar to 40% of the wood lignin; and (4) a glucomannan-lignin-xylan network LCC (glucomannan) xylan) containing similar to 48% of the wood lignin. Endo-glucanase Novozyme 476, with only cellulase activity, and Ecopulp XM, with only xylanase and mannanase activities, were used as an enzymatic tool. From mildly ball-milled wood, all the lignin was isolated as LCCs. As a control, LCC was prepared from partially chlorite-delignified wood meal without ball milling, also in a mild procedure. The results were very similar to those obtained after ball milling. Thus, it can be safely concluded that the formation of new chemical linkages between lignin and carbohydrates during ball milling is improbable. Studies on isolated milled wood lignin (MWL) supported this conclusion and clearly showed that covalent linkages between lignin and carbohydrates are present. The study provide conclusive evidence of covalent linkages between lignin and carbohydrates in the native lignin in wood. It is concluded that carbohydrate-free lignin, i.e., lignin without covalent bonds to carbohydrates, probably cannot be present in spruce wood.

A novel and facile route of ink-jet printing to thin film SnO_2 anode for rechargeable lithium ion batteries

Zhao-YM; Zhou-Q; Liu-L; Xu-J; Yan-MM; Jiang-ZY

ELECTROCHIMICA-ACTA. MAR 5 2006; 51 (13) : 2639-2645

Thin film SnO_2 electrode has been prepared for the first time by using a novel facile and low-cost ink-jet printing technique. Wet ball-milling was employed to stabilize SnO_2 nano particles and conducting agent acetylene black (AB) using two kinds of polymeric hyperdispersants CH10B and CH12B, respectively, to prepare the stable colloid as "ink". The morphology, structure, composition and electrochemical performance of SnO_2 thin film electrodes were investigated in detail by SEM, TEM, XRD, EDX, cyclic voltammograms (CV) and galvanostatic charge-discharge measurements. SEM images show uniform distribution of as-printed SnO_2 thin film electrodes. The thickness of monolayer thin film electrode was about 770-780 nm by TEM observation. The thickness of SnO_2 thin film could be increased by repeating the printing procedure on the Cu foil substrate. The average thickness of 10-layer SnO_2 thin-film electrode after compression for electrochemical measurement was about 2.3 μm . High initial discharge capacity about 812.7 mAh/g was observed at a constant discharge current density of 33 $\mu\text{A}/\text{cm}^2$ in a potential range of 0.05-1.2 V. It is expected that ink-jet printing is a very feasible, simple, convenient and inexpensive way to prepare thin film electrode for lithium ion batteries.

Soft magnetic properties of bulk amorphous Co-based samples

Fuzer-J; Bednarcik-J; Kollar-P

ACTA-PHYSICA-SLOVACA. APR 2006; 56 (2) : 119-122

Ball milling of melt-spun ribbons and subsequent compaction of the resulting powders in the supercooled liquid region were used to prepare disc shaped bulk amorphous Co-based samples. The several bulk samples have been prepared by hot compaction with subsequent heat treatment (500 degrees C - 575 degrees C). The influence of the consolidation temperature and follow-up heat treatment on the magnetic properties of bulk samples was investigated. The final heat treatment leads to decrease of the coercivity to the value between the 7.5 to 9 A/m.

Water-solubilization of nucleotides-coated single-walled carbon nanotubes using a high-speed vibration milling technique

Ikeda-A; Hamano-T; Hayashi-K; Kikuchi-J



ORGANIC-LETTERS. MAR 16 2006; 8 (6) : 1153-1156

Pristine single-walled carbon nanotubes (SWNTs) and nucleotides mixed using a mechanochemical high-speed vibration milling technique (HSVM) are soluble in an aqueous solution, and the solubilities of SWNTs depend significantly on the number of phosphate groups and the kinds of bases employed.

Influence of Fe contamination and temperature on mechanically alloyed Co-Ni-Mo electrodes for hydrogen evolution reaction in alkaline water

Dominguez-Crespo-MA; Plata-Torres-M; Torres-Huerta-AM; Ortiz-Rodriguez-IA; Ramirez-Rodriguez-C; Arce-Estrada-EM
MATERIALS-CHARACTERIZATION. MAR 2006; 56 (2) : 138-146

Ni-Co-Mo-Fe solid solutions, such as Co₃₀M₇₀, Co₃₀Mo₇₀, Ni₃₀Mo₇₀, Co-10,Ni-20,Mo-70, Fe₁₀Co₂₅Ni₆₅, Fe-20)Co-20,Ni-60 and Fe₃₀Co,Ni-15(55) wt.% alloys, have been produced by mechanical alloying using commercial Ni, Co, Mo and Fe powders. The electrocatalytic properties of these nanocrystalline materials have been studied for hydrogen evolution reaction (HER) in 30 wt.% KOH aqueous solution at three different temperatures (308, 323 and 343 K) to determine the effect of Fe contamination. The methods employed were cyclic voltammetry, steady-state polarization (Tafel) techniques and ac impedance. For comparison, it was found that iron improved electrocatalytic activity for the hydrogen evolution reaction. Important changes in the activity were obtained when the temperature was increased. The electrocatalytic effect of Mo became important at high overvoltages and temperatures. The MA Fe₃₀Co₁₅Ni₅₅ and Co₁₀Ni₂₀Mo₇₀ alloyed powders showed the best catalytic activities for HER.

Fabrication of boron nitride dispersed nanocomposites by chemical processing and their mechanical properties

Kusunose-T

JOURNAL-OF-THE-CERAMIC-SOCIETY-OF-JAPAN. FEB 2006; 114 (1326) : 167-173

Hexagonal boron nitride (h-BN) dispersed nanocomposites were fabricated through a chemical process involving sintering of matrix ceramic powders covered partly with turbostratic BN (t-BN), which was synthesized by the reduction of a mixture of boric acid and urea precipitating on the matrix ceramic powder during drying after wet-ball milling. Silicon carbide (SiC), silicon nitride (Si₃N₄), alumina (Al₂O₃) and aluminum nitride (AlN) were employed as matrix for h-BN dispersed nanocomposites. The nano-sized h-BN particles were found to be homogeneously dispersed within the matrix grains as well as at grain boundaries, which is typical of nanocomposite structures. As a result, the present nanocomposites showed unique and excellent properties such as high strength, low Young's modulus, high thermal shock resistance, and good machinability.

Fabrication of submicron alumina ceramics by pulse electric current sintering using Mg²⁺-doped transition alumina powders

Hida-M; Yajima-Y; Yamaguchi-T; Taruta-S; Kitajima-K

JOURNAL-OF-THE-CERAMIC-SOCIETY-OF-JAPAN. FEB 2006; 114 (1326) : 184-188

Dense submicron-grained alumina ceramics were fabricated by pulse electric current sintering (PECS) using Mg²⁺-doped transition alumina powders at 1200-1350 degrees C under a uniaxial pressure of 40 or 80 MPa. The Mg²⁺-doped transition alumina powders (0-0.50 mass% MgO base) were prepared through a new sol-gel route using high-purity polyhydroxoaluminum (PHA) and MgCl₂ solutions as starting materials. The composite gels obtained were calcined at 900 degrees C and ground by planetary ball-milling. Upon heating, the composite gels transformed into a single-phase gamma-alumina or mixed phase of gamma- and chi-aluminas, depending on the MgO content. The resultant transition alumina powders were solid solutions, in which Mg²⁺ cations were substituted into the crystal lattice. The powders were re-calcined to increase the content of alpha-alumina particles, which act as seeding for low-temperature densification. Densification depended on the MgO content and loading pressure. The critical Mg²⁺-doping for suppressing grain growth was found to be 0.10 mass% MgO. Higher loading pressures led to full densification at lower temperatures, resulting in a more uniform and finer microstructure. Thus, dense alumina ceramics (relative density >= 99.6%) with a uniform microstructure composed of fine grains with an average size of 0.47 mu m could be obtained by PECS at 1250 degrees C under 80 MPa.

Fe³⁺-assisted formation of alpha-Al₂O₃, starting from sol-gel precursors

Stosser-R; Nofz-M; Feist-M; Scholz-G

JOURNAL-OF-SOLID-STATE-CHEMISTRY. MAR 2006; 179 (3) : 652-664

The role of Fe³⁺ ions in the transformations from boehmites and pseudoboehmite xerogels via transition aluminas to corundum was studied here. Especially, the active iron species responsible for the decrease of the temperature of transformation to corundum were looked for. To enable the formation of various Fe³⁺ and Fe²⁺ species, samples were subjected to thermal treatments in different atmospheres as well as mechanically activated. Thermal analysis and ESR spectroscopy served to follow the processes and to characterise the resulting products. It was found that (i) isolated Fe³⁺ ions can indicate local structural changes but have (almost) no influence on the temperature of corundum formation, (ii) the temperature of corundum formation decreases in the result of action of small alpha-Fe₂O₃ particles and (iii) during thermal treatments Fe³⁺ ions are distributed between different phases or precursors thereof. transition aluminas, corundum, Fe₂O₃,



and a Fe³⁺ pool.

Room-temperature synthesis and conductivity of the pyrochlore type Dy-2(Ti_{1-y}Zr_y)(2)O-7 (0 ≤ y ≤ 1) solid solution

Moreno-KJ; Guevara-Liceaga-MA; Fuentes-AF; Garcia-Barriocanal-J; Leon-C; Santamaria-J

JOURNAL-OF-SOLID-STATE-CHEMISTRY. MAR 2006; 179 (3) : 928-934

Different compositions in a solid solution of general formula Dy-2(Ti_{1-y}Zr_y)(2)O-7, showing high oxygen ion conductivity, have been successfully prepared at room temperature via mechanochemical synthesis. Stoichiometric mixtures of the constituent oxides were dry milled together in a planetary ball mill by using zirconia vials and balls. Chemical changes in the powder mixtures as a function of composition and milling time were followed by X-ray diffraction and revealed that, in all cases and after milling for 19 h, the powder mixtures consisted of a single phase. Electrical properties were measured on sintered pellets as a function of frequency, temperature and zirconium content, revealing an increase in conductivity of more than one order of magnitude for y ≥ 0.4, which, as observed in the similar Y-2(Ti_{1-y}Zr_y)(2)O-7, has been related with the onset of disordering of the anion sublattice. Despite increasing structural disorder with increasing Zr content, conductivity remains almost constant for y > 0.6, reaching a maximum value of similar to 5 × 10⁻³ for Dy₂Zr₂O₇ at 900 degrees C.

Magnetic coupling and spin structure in nanocrystalline iron powders

Slawska-Waniewska-A; Grafoute-M; Greneche-JM

JOURNAL-OF-PHYSICS-CONDENSED-MATTER. FEB 22 2006; 18 (7) : 2235-2248

Pure single-phase iron nanostructured particles with pseudo-cubic shape crystalline grains and linear dimensions of around 11 nm can be produced by the low energy ball milling of microcrystalline Fe under argon atmosphere. The long range ferromagnetic correlation of exchange coupled crystallites extending across grain boundaries leads to a reduction of the effective anisotropy, as expected from the generalized random anisotropy model. This ferromagnetic network of correlated grains is preserved at low temperatures. No spin-glass freezing process is detected. Slight oxidation of the particles with formation of an FeO phase is achieved with deliberately prolonged milling. This FeO phase leads to non-collinear spin structure at the interfaces that suppresses the intergrain correlations and enhances the role of long range dipolar interactions. The interface spin disorder and the complex state of the intergrain interactions are the sources of the spin-glass-like behaviour found in these Fe-FeO nanocomposites.

Characterization and thermoelectric properties of p-type 25%Bi₂Te₃-75% Sb₂Te₃ prepared via mechanical alloying and plasma activated sintering

Fan-XA; Yang-JY; Chen-RG; Yun-HS; Zhu-W; Bao-SQ; Duan-XK

JOURNAL-OF-PHYSICS-D-APPLIED-PHYSICS. FEB 21 2006; 39 (4) : 740-745

In the present work, starting from elemental bismuth, antimony and tellurium powders, p-type 25%Bi₂Te₃-75%Sb₂Te₃ thermoelectric materials with high density were prepared by mechanical alloying (MA) and plasma activated sintering (PAS). The single phase 25%Bi₂Te₃-75%Sb₂Te₃ alloys were obtained after MA for 12 h. The effect of sintering temperatures on microstructure and thermoelectric properties of the as-PASed samples was researched. Highly compact samples with relative density over 99% could be obtained when sintering temperature was over 653 K. A preferentially orientated microstructure with the (1 10) plane parallel to and the basal planes (0 0 1) perpendicular to the pressing direction was formed, and the orientation factors of the (0 0 1) planes changed from 0.11 to 0.12 at different sintering temperatures. The maximum power factor and figures of merit (Z) at room temperature were 3.10 × 10⁻³ W m⁻¹ K⁻² and 2.85 × 10⁻³ K⁻¹, respectively. The Vickers microhardness reached 112.7 Hv, which was twice that of the single crystal samples prepared by zone-melting.

Intrinsic paramagnetic defects probe the superionic phase transition in mechanochemically synthesized AgI nanocrystals

Mohan-DB; Sunandana-CS

JOURNAL-OF-PHYSICAL-CHEMISTRY-B. MAR 16 2006; 110 (10) : 4569-4575

Electron paramagnetic resonance (EPR) of two intrinsic paramagnetic centers generated by soft mechanochemistry of Ag and I to yield zinc blende gamma-AgI nanoparticles (similar to 38 nm) has been used for the first time to probe the gamma-alpha. (body centered cubic) superionic phase transitions in AgI at (423 ± 1) K. These results are agreeable with the differential scanning calorimetric studies. A transmission electron microscope picture shows the average crystallite size in the range of similar to 30-40 nm. A hole-type Ag-related paramagnetic center (Ag-2+) with an average g = 2.21025 value is remarkably sensitive to the first-order phase transition exhibiting sharp drops at the phase transition temperature (T_t) and complete reversibility. The T_t is characterized by a sharp, abrupt rise in the inverse paramagnetic susceptibility 1/chi by 1 order (7.4 × 10¹¹ to 3.17 × 10¹¹ in kg m⁻³) which reflects changes in the bonding of the material. Furthermore, a sharp signal at < g > = 2.0019 (Delta H-PP = 10 G) due to an electron-excess center (Ago) as a result of Ag metal nanoclusters also formed during the mechanochemical reaction (MCR) yields an abrupt and drastic decrease in the intensity observed at T_t = 423 K. From high-temperature (323 to 433 K) I-V characteristics, the evolution of nonohmic behavior is observed on the order of 10⁻⁹-10⁻⁶ A with increasing temperature until below T_t which becomes ohmic thereafter. The reason could be the creation



of an electronic defect such as Ag-0 metal nanoclusters formed during the near-equilibrium mechanochemical reaction, with the increased excess free energy favoring the formation of gamma-AgI nanoparticles.

Synthesis of MnFe₂O₄ nanoparticles by mechanochemical reaction

Osmokrovic-P; Jovalekic-C; Manojlovic-D; Pavlovic-MB

JOURNAL-OF-OPTOELECTRONICS-AND-ADVANCED-MATERIALS. FEB 2006; 8 (1) : 312-314

The influence of long-term milling of a mixture of MnCO₃ and alpha-Fe₂O₃, and of MnO₂ and alpha-Fe₂O₃ powders in a planetary ball mill on the reaction synthesis of nanosized MnFe₂O₄ ferrites was studied. The mechano-chemical reaction leading to formation of the MnFe₂O₄ spinel phase was followed by X-ray diffraction. The spinel phase was first observed after 10 h of milling and its formation was completed after 20 h in case of the MnCO₃ - alpha-Fe₂O₃ mixture. The synthesized MnFe₂O₄ ferrite has a nanocrystalline structure with a crystallite size of about 28 nm. The thermal treatment of the as-milled powder at 400 degrees C for 4 h led to formation of the MnFe₂O₄ crystalline phase in case of the mixture of MnCO₃ and alpha-Fe₂O₃, but in MnO₂ alpha-Fe₂O₃ case thermal treatment at temperatures of 400 degrees C and higher leads to formation of (Mn,Fe)₂O₃ and separation of Mn₂O₃ and Fe₂O₃.

Matrix-analyte-interaction in MALDI-MS: Pellet and nano-electrospray preparations

Horneffer-V; Gluckmann-M; Kruger-B; Karas-M; Strupat-K; Hillenkamp-F

INTERNATIONAL-JOURNAL-OF-MASS-SPECTROMETRY. MAR 1 2006; 249 : 426-432

The incorporation of analytes into matrix crystals and even more so its mechanistic aspects as a prerequisite for a successful MALDI-MS has been discussed controversially in the literature. Solventless sample preparation techniques can shed new light on this question. In order to investigate some crucial aspects of these preparation techniques, lyophilized peptides and proteins were ground or milled with the powder of two different matrices, 2,5-DHB as incorporating matrix and 2,6-DHB for which protein incorporation was definitely excluded in a prior study, and pressed into pellets. The dependence of the quality of the UV-MALDI-spectra on the mass (up to 12,360 Da) and the milling time in a ball mill is reported. For mellitin different initial axial ion velocities were found, when desorbed from 2,5-DHB-pellets as prepared and after wetting and re-drying. Velocities of 150 and 580 m s⁻¹ for dry and wetted pellets are taken as representative for hard desorption from a surface and soft desorption of matrix-incorporated analytes, respectively. Proteins labeled with either fluorescein isothiocyanate (FITC) or Texas Red (TR) were nano-electrosprayed onto a bed of ferulic acid in a 'dry' or 'wet' mode. All 'dry' deposits exhibit strong fluorescence but do not yield MALDI-ion signals. All 'wet' deposits yield MALDI-signals of the proteins; the fluorescence of FITC is quenched in 'wet' deposits because of the low matrix pH.

Review on hydrogen absorbing materials - structure, microstructure, and thermodynamic properties

Bououdina-M; Grant-D; Walker-G

INTERNATIONAL-JOURNAL-OF-HYDROGEN-ENERGY. FEB 2006; 31 (2) : 177-182

Hydrogen is a promising renewable fuel for transportation and domestic applications. Many systems have been investigated in order to improve the maximum hydrogen storage capacity (reversible), high kinetics, moderate equilibrium pressure and/or decomposition temperature and better cyclability. In this paper, a review of studies related to stability of Zr-based Laves phase system as well as in situ neutron diffraction investigation, the kinetics of TiFe, surface treatment of LaNi₅ system, mechanically alloyed Mg-based hydrides and graphite nanofibres are reported.

Hydrogen storage in Ti-based quasicrystal powders produced by mechanical alloying

Takasaki-A; Kelton-KF

INTERNATIONAL-JOURNAL-OF-HYDROGEN-ENERGY. FEB 2006; 31 (2) : 183-190

Ti-based quasicrystals belong to the second largest group of the stable quasicrystals, showing attractive properties as hydrogen storage materials. This paper summarizes our recent research results on hydrogen absorption and desorption properties of the Ti-Zr-Ni and Ti-Hf-Ni quasicrystals and the related phases produced by a combination of mechanical alloying and subsequent annealing.

Structural, solid-gas and electrochemical characterization of Mg₂Ni-rich and Mg_xNi_{100-x} amorphous-rich nanomaterials obtained by mechanical alloying

Abdellaoui-M; Mokbli-S; Cuevas-F; Lacroche-M; Guegan-AP; Zarrouk-H

INTERNATIONAL-JOURNAL-OF-HYDROGEN-ENERGY. FEB 2006; 31 (2) : 247-250

Using a planetary ball mill and starting from a mixture of Mg and Ni with an atomic ration of 2: 1, we successfully elaborated a nanocomposite material formed by the Mg₂Ni phase in high proportion, some residual Ni and an amorphous phase. The synthesis of this composite proceeded at milling intensities 7 and 10, corresponding to 3.5 and 10W/g shock power, respectively, after 18 and 4 h. The best hydrogen absorption capacity reported, 3.75 H mol⁻¹ (3.53 wt%) is for the composite synthesized for 24 h at 3.5 W/g shock power.

Using the same planetary ball mill and starting from a mixture of Mg₂Ni and Ni with a Mg atomic content ranging from 40 to



60 at %, we elaborated amorphous phase alloys with little quantities of residual Ni. The synthesis of the amorphous phase proceeded at 6.49 W/g shock power, for milling durations ranging from 8 to 10 h for Mg₄₀Ni₆₀ and Mg₆₀Ni₄₀ samples, respectively. The best electrochemical capacity (470mAh/g) was obtained for the Mg₅₀Ni₅₀ sample obtained at milling duration 10 times shorter than that reported in the literature.

Composite structure and hydrogen storage properties in Mg-base alloys

Zhu-M; Wang-H; Ouyang-LZ; Zeng-MQ

INTERNATIONAL-JOURNAL-OF-HYDROGEN-ENERGY. FEB 2006; 31 (2) : 251-257

In order to improve the hydrogen absorption/desorption kinetic properties of Mg and Mg-Ni alloys, composite hydrogen storage alloys in the form of powder and film have been synthesized and investigated. For fabricating the composite powder, Mg or Mg-Ni powder was mechanically alloyed with MmNi(3.5)(CoAlMn)(1.5) alloy. For the preparation of the film with a composite structure, evaporation deposition and magnetron sputtering methods have been used to fabricate Mg-Ni film with multi-phase structure and Mg/Mm-Ni and Mg-Ni/Mm-Ni multi-layer film. By controlling the fabrication process, the microstructure feature, such as phase constituent, grain size, interlayer distances, interlayer boundary structure, of the composite can be modified. To reveal the influence of the composite structure on the hydrogen absorption/desorption kinetic properties of Mg and Mg-Ni alloys, hydrogen storage properties of the composite were measured with their microstructure features varied systematically. The present work shows that the hydrogen sorption properties of Mg and Mg-Ni-based alloys can be substantially improved by forming composites having proper microstructure features.

Secondary emission of nanocrystalline zinc oxide

Gorelik-VS; Mikov-SN; Sokolovskii-MI; Tsuzuki-T

INORGANIC-MATERIALS. MAR 2006; 42 (3) : 282-285

The Raman and photoluminescence (PL) spectra of nanocrystalline zinc oxide produced by mechanochemical synthesis were measured using a pulsed nitrogen laser (337.1 nm) and xenon lamp (360 nm) as excitation sources in PL measurements and a cw Nd:YAG laser in Raman measurements. PL was observed in the range 400-800 nm. The Raman spectrum of nanocrystalline (90 nm) ZnO was compared to that of coarse-grained ZnO. The Raman bands of nanocrystalline zinc oxide were found to be shifted to lower frequencies and broadened. Laser radiation was shown to cause local heating of zinc oxide up to 1000 K, resulting in photoinduced formation of zinc nanoclusters. Mixtures of zinc oxide and sodium chloride powders are heated to substantially lower temperatures. Under nitrogen laser excitation, the green PL band (535 nm), characteristic of bulk ZnO, is shifted to longer wavelengths by 85 nm. The results are interpreted in terms of light confinement in zinc oxide microclusters consisting of large number of nanocrystallites. The photoinduced processes in question may be a viable approach to producing metal-insulator structures in globular photonic crystals, opals, filled with zinc oxide.

Effects of crystallinity on dilute acid hydrolysis of cellulose by cellulose ball-milling study

Zhao-HB; Kwak-JH; Wang-Y; Franz-JA; White-JM; Holladay-JE

ENERGY-AND-FUELS. MAR-APR 2006; 20 (2) : 807-811

The dilute acid (0.05 M H₂SO₄) hydrolysis at 175 degrees C of samples comprised of varying fractions of crystalline (alpha-form) and amorphous cellulose was studied. The amorphous content, based on XRD and CP/MAS NMR, and the product (glucose) yield, based on HPLC, increased by as much as a factor of 3 upon ball milling. These results are interpreted in terms of a model involving mechanical disruption of crystallinity by breaking hydrogen bonds in alpha-cellulose, opening up the structure, and making more beta-1,4 glycosidic bonds readily accessible to the dilute acid. However, in parallel with hydrolysis to form liquid-phase products, there are reactions of amorphous cellulose that form solid degradation products.

A new rapid synthesis technique for electrochemically active materials used in energy storage applications

Needham-SA; Calka-A; Wang-GX; Mosbah-A; Liu-HK

ELECTROCHEMISTRY-COMMUNICATIONS. MAR 2006; 8 (3) : 434-438

LiFePO₄ is a promising environmentally friendly and low cost alternative cathode material for use in lithium-ion batteries. The most common materials production process used to manufacture LiFePO₄ is solid-state synthesis which entails several grinding and calcination steps, occurring over many hours. We report on the synthesis of crystalline LiFePO₄ in only 10 min via a versatile process of Electric discharge assisted mechanical milling (EDAMM). Preliminary electrochemical testing of the synthesized powder demonstrates good capacity and excellent cyclability. The EDAMM technique offers an exciting opportunity to synthesize a range of new and existing materials to be used in a variety of energy storage applications that include rechargeable lithium batteries, hydrogen fuel cells, and supercapacitors.

Ignition of aluminum-rich Al-Ti mechanical alloys in air

Shoshin-YL; Trunov-MA; Zhu-XY; Schoenitz-M; Dreizin-EL

COMBUSTION-AND-FLAME. MAR 2006; 144 (4) : 688-697

Ignition of metastable Al-Ti mechanical alloys with titanium concentrations from 10 to 25 at% was investigated



experimentally. A thin layer of powder was coated on an electrically heated filament. The ignition instant was identified from the powder's radiation measured in real time. Simultaneously, filament temperatures were measured using a high-speed infrared pyrometer to determine the ignition temperature. The experiments were conducted at different filament heating rates in the range of $3 \times 10(3)$ - $2 \times 10(4)$ K/s to determine the ignition kinetics. The ignition temperatures and kinetics were compared to the respective characteristics of the phase changes and oxidation steps observed for the same mechanical alloys using thermal analysis. It was shown that at the heating rates exceeding 103 K/s, the exothermic formation of a metastable L1(2) phase of Al₃Ti occurring during heating of the Al-Ti mechanical alloys triggers their ignition. This conclusion was confirmed by additional ignition experiments in which annealed mechanical alloys already containing this transition Al₃Ti phase did not ignite in the same temperature range as fresh mechanical alloys. The ignition kinetics identified for Al-Ti mechanical alloys based on thermal analysis and on ignition experiments enables one to predict ignition temperatures as a function of both composition and heating rate. Specifically, extrapolation is possible to higher heating rates typical for aerosol flames.

Influence of talc grain size on formation and physico-mechanical properties of cordierite

Othman-AGM

CFI-CERAMIC-FORUM-INTERNATIONAL. FEB 2006; 83 (2) : E39-E43

Cordierite batches were prepared from clay, alumina and talc. Talc was milled in a ball mill for 20, 30 and 100 h, respectively, in order to investigate the effect of talc grain size on the formation, sintering and physico-mechanical properties of cordierite. They were fired at a temperature range of 1200...1450 degrees C with 50 K intervals for 1 h soaking time. Mullite, spinel, enstatite and alumina were detected at 1200 degrees C in addition to cordierite. Low particle size of talc enhanced the reaction between mullite and enstatite to form cordierite at lower temperature. At 1400 degrees C, cordierite was the only detected phase. At 1350 degrees C, all the batches reached their lower apparent porosity, while the batch containing the lowest particle size of talc showed higher apparent porosity and larger pores compared to the other two batches. The three studied batches revealed different thermal expansion behaviors according to the grinding time of talc. Mechanical and refractory properties were not remarkably affected by the particle size of talc.

Preparation of porous material from talc by mechanochemical treatment and subsequent leaching

Yang-HM; Du-CF; Hu-YH; Jin-SM; Yang-WG; Tang-AD; Avvakumov-EG

APPLIED-CLAY-SCIENCE. MAR 2006; 31 (3-4) : 290-297

Synthesis of porous silica via mechanochemical treatment of talc and subsequent acid leaching was investigated by X-ray diffraction (XRD), Fourier transformation infrared spectroscopy (FTIR), scanning electron microscopy (SEM) and N₂ adsorption techniques. Raw talc was ground for different times and then leached with 4 M hydrochloric acid (HCl) at 80 degrees C. Grinding for 6 h 2 and subsequent leaching for 2 h of raw talc produced the porous silica with a specific surface area of 133 m²/g and total pore volume of 0.22 ml/g. The increase in specific surface area (S-BET) of the porous silica reflected the formation of micropores of 1.2 similar to 1.8 nm and mesopores of 4.0 similar to 5.5 nm in diameter inside the porous structure. The number of micropores decreased with prolonged leaching time, which can be attributed to a condensation reaction. The characteristic of hysteresis loop indicated mainly slit-shaped pores. The apparent activation energy for the leaching process was calculated to be about 21.6 kJ/mol, indicating that the kinetic process of talc leaching was the diffusion-controlled reaction. Mechanochemical treatment may promote the amorphization of talc, being greatly favorable to the subsequent leaching.

H₂S-sensing properties of SnO₂ produced by ball milling and different chemical reactions

Kersen-U; Holappa-L

ANALYTICA-CHIMICA-ACTA. MAR 9 2006; 562 (1) : 110-114

In this work, the mechanochemical synthesis of a moderately agglomerated tin oxide (SnO₂) powders and the subsequent preparation of semiconductor gas sensors as prototypes, were studied. Tin (II) chloride (SnCl₂) powder was milled with calcium hydroxide (Ca(OH)₂) and potassium carbonate, (K₂CO₃) powder, respectively, in a ball mill at room temperature and in an air atmosphere. Heat treatment of milled mixtures at 400 degrees C resulted in the formation of a tetragonal phase, confirmed by X-ray diffraction (XRD). During milling in the presence of water, a high number of hydroxide (OH) groups are formed at the surface. When SnCl₂ was milled with K₂CO₃, no water was produced and the Fourier-transform infrared spectrum (FT-IR) of the powder has no surface hydroxyl deformations. On exposure to hydrogen sulfide (H₂S) gas, the particles, prepared from anhydrous powder, have higher sensitivity than these, prepared from hydrated powder. The SnO₂ thick film, prepared from anhydrous powder may be successfully applied to a H₂S gas sensor.

Mechanochemical decompositions of halogen-containing compounds by grinding with oxides

Zhang-QW; Saito-F

AGRICULTURE-AND-SOIL-POLLUTION-NEW-RESEARCH. 2005 : 231-256



Preparation and electrochemical characteristics of amorphous Mg_{0.9}Ti_{0.1}Ni_{1-x}Co_x (x=0.05, 0.1, 0.15, 0.2) alloys

Feng-Y; Jiao-LF; Yuan-HT; Wang-YJ; Liu-Q; Liu-Y

ACTA-CHIMICA-SINICA. MAR 14 2006; 64 (5) : 423-427

Mg-based hydrogen storage alloys Mg_{0.9}Ti_{0.1}Ni_{1-x}Co_x (x=0.05, 0.1, 0.15, 0.2) were prepared by means of mechanical alloying, and the structure and the electrochemical characteristics of these Mg-based electrodes were also studied. The result of X-ray diffraction and transmission electron microscope showed that the main phase of the alloys had amorphous structures, and some trace of weak Ni peaks might co-exist. The charge-discharge cycle tests indicate that these alloys have good electrochemistry activation characteristics, and the cycle performance of these alloys were better than those of MgNi alloys. Among such alloys, the discharge capacity of Mg_{0.9}Ti_{0.1}Ni_{0.8}Co_{0.2} was the highest, up to 427.5 mAh center dot g(-1). In the process of charge-discharge cycle test, the main reason of the electrode capacity decay is that Mg may become Mg(OH)₂ on the surface of alloys. The corrosion curve test indicates that the Co addition can improve the anticorrosion performance of these alloys in the alkali solution and the cycle stability of these alloy electrodes.

Formation of detonation coatings based on titanium aluminide alloys and aluminium titanate ceramic sprayed from mechanically alloyed powders Ti-Al

Oliker-VE; Sirovatka-V; Timofeeva II; Gridasova-TY; Hrechyshkin-YF

SURFACE-AND-COATINGS-TECHNOLOGY. MAR 15 2006; 200 (11) : 3573-3581

The phase formation of the detonation coatings from the different Ti-50Al feed stock powders were investigated. The powders were prepared by milling of TiAl (gamma) ingot and mechanical alloying of the Ti and Al elementary powders. The application of the nanocomposite powder materials which were activated by the mechanical alloying made the phase formation process in the coatings more universal and adjustable due to more active and sensitive response of powder material to a gas environment. It is shown that from the mechanically alloyed powder Ti-50Al, it is possible to consolidate by the detonation spraying method the ceramic coating based on the compound Al₂TiO₅ at oxidizing influence of the working gas environment on the powder. Coating based on the titanium aluminides with inclusions TiN can be formed at nitriding influence of the working gas environment on the powder. At the use of the cast microsize powder TiAl (gamma), its phase composition is inherited by the coating.

Mechanical activation of olivine

Kleiv-RA; Thornhill-M

MINERALS-ENGINEERING. APR 2006; 19 (4) : 340-347

This paper investigates how mechanical activation of olivine can increase the mineral's surface reactivity, and illustrates how such technology can give rise to new or improved olivine products. The olivine material used in this study consisted of pure olivine crystals (Mg_{1.860}Fe_{0.134}Ni_{0.006}SiO₄) originating from North Cape Minerals dunite deposit at angstrom heim in Western Norway. Following activation in a planetary mono mill, the activated olivine products were visually inspected using scanning electron microscopy (SEM) and characterised with respect to particle size, specific surface area (BET) and X-ray diffraction (XRD) signature. The surface reactivity of the activated olivine products was determined through simple acid leaching experiments in which the initial acid consumption rates were determined. The initial phase of olivine dissolution could be modelled using first order kinetics. Prolonged dry milling of pure olivine crystals resulted in highly aggregated products that were more reactive with respect to dissolution in acid than their respective BET surface areas would suggest. Relative to olivine that had been milled for 1 min, 60 min of milling increased the initial reaction rate by a factor of 9.0, whereas the corresponding increase in specific surface area was 1.8. The results from both the leaching experiments and the XRD analysis suggest that the observed over-proportional increase in reactivity with respect to surface area is largely due to structural disordering (i.e. mechanical activation) of the olivine surfaces.

Application of electric spark generated high power ultrasound to recover ferrous and non-ferrous metals from slag waste

Wilson-MP; Balmer-L; Given-MJ; MacGregor-SJ; Mackersie-JW; Timoshkin IV

MINERALS-ENGINEERING. APR 2006; 19 (5) : 491-499

On a worldwide basis there exist large stocks of by-products from the production of ferrous and non-ferrous metals. For example, in Scandinavia there is a site which currently has 60000 tons of stainless steel trapped in slag waste. Even at current market prices this is a valuable resource. However, current technological approaches, such as ball milling, are uneconomic. High power ultrasound (HPU) is a novel approach to this problem to allow recovery of the stainless steel and a recycling path for the silicate slag as a building material. At the University of Strathclyde, pulsed power (the compression of electrical energy with time) has been used to generate HPU shock waves from spark discharges in water. Trials using a prototype HPU system have demonstrated that stainless steel metal can be separated from the slag waste by-product rapidly and with low power consumption. Glass may also be comminuted for recycling using the HPU system. The results of the trials are presented and proposed methods for industrial scale-up are discussed



Mechanical properties of ultrafine grained 5052 Al alloy produced by accumulative roll-bonding and cryogenic rolling

Song-HR; Kim-YS; Nam-WJ

METALS-AND-MATERIALS-INTERNATIONAL. FEB 2006; 12 (1) : 7-12

Mechanical properties in conjunction with microstructural evolution during annealing of 5052 Al alloy deformed at cryogenic temperature were investigated and compared with those yielded by the ARB process. ARB was conducted up to 7 cycles under conditions where the reduction in thickness per cycle was 50 % and the rolling temperature was 300 degrees C. To investigate the effect of annealing temperature, cryo-rolled sheets with 85 % reduction were annealed in a temperature range of 150 similar to 300 degrees C for one hour. Strengths of 5052 Al alloy ARB processed at 300 degrees C increased with increasing number of cycles and decreased rapidly after 6 or cycles. This indicated that, during the ARB process, work hardening proceeded at low strains and subdivision of grains and dynamic recovery occurred at high strains. Tensile strength and yield strength of cryo-rolled 5052 Al alloy decreased as the annealing temperature increased. The volume fraction of recrystallized and coarsened grains appeared to have the most significant influence on strength and ductility of sheets annealed at 250 degrees C.

Metal injection molding of W-Cu powders prepared by low energy ball milling

Kim-SW; Suk-MJ; Kim-YD

METALS-AND-MATERIALS-INTERNATIONAL. FEB 2006; 12 (1) : 39-44

Low energy ball milled W/Cu powders were used for metal injection molding (MIM) in order to overcome the low powder volume fraction of MIM parts after debinding as well as the inherently poor sinterability of the W-Cu powder compacts. Ball milling was carried out using commercial fine W and Cu powders to form a powder mixture suitable for injection molding. W powders showed no change in either size or shape during the milling process, but the ductile Cu powders were easily deformed to a three-dimensional equiaxed shape, having a particle size comparable to that of W powders. This modification of powder characteristics by ball milling resulted in an improvement of the solid loading of roughly 58 %, maintaining a high and uniform powder packing density in the feedstock. The densification behavior of W-Cu MIM parts is also discussed on the basis of the relationship between Cu composition and W particle size.

Effect of high energy ball milling on displacement reaction and sintering of Al-Mg/SiO₂ composite powders

Woo-KD; Huo-HW

METALS-AND-MATERIALS-INTERNATIONAL. FEB 2006; 12 (1) : 45-50

High-energy ball milling and low temperature sintering were successfully employed to fabricate a metal matrix composite of Al reinforced with Al₂O₃ particulate. Nano- and/or submicro-sized SiO₂ particles embedded in an Al-Mg matrix particle can be obtained by high-energy ball milling. No new phases were found in the high-energy ball milled Al-0.4 wt.%Mg-14 wt.%SiO₂ powder. Milling of the Al-Mg-SiO₂ powder increased the sintering rate and decreased the sintering temperature. The hardness of the sintered Al-Mg-SiO₂ composite using the ball-milled powder was about twice that of a sintered composite using a mixed powder due to the fine and homogeneous distribution of Al₂O₃ particles formed by the displacement reaction between Al and SiO₂ during sintering.

Mechanochemical synthesis of LaMnO₃+ δ fine powder assisted with water vapor

Sato-K; Chalchanawong-J; Abe-H; Naito-M

MATERIALS-LETTERS. MAY 2006; 60 (11) : 1399-1402

An alternative mechanical synthesis route for LaMnO₃+ δ fine powder is presented. Mechanical forces such as compression and shear stress were repeatedly applied to a mixture of La₂O₃ and Mn₃O₄, using an attrition type milling apparatus. No media balls were employed in this milling. A proper quantity of water vapor in the milling chamber induced an efficient grinding of the mixture at early stage of the milling. The formation of LaMnO₃+ δ was observed in the subsequent milling, and its synthesis was completed after only 30 min. The average diameter of the resultant LaMnO₃+ δ was 110 nm, and the amount of contamination was less than 20 ppm in mass. Influences of the water vapor on phase evolution and changes in particle size are shown, and a possible mechanism of the present mechanochemical reaction is discussed.

Structure, phase transformation, and magnetic properties of SmCo_{7-x}Cr_x magnets - art. no. 053905

Yao-Q; Liu-W; Zhao-XG; Li-D; Zhang-ZD

JOURNAL-OF-APPLIED-PHYSICS. MAR 1 2006; 99 (5) : NIL_277-NIL_281

SmCo_{7-x}Cr_x (x=0-0.6) magnets have been synthesized by mechanical alloying and subsequent annealing. A small amount of Cr favors the formation of Th₂Ni₁₇-type 2:17 phase and increasing the Cr content can extend the temperature range in which the hexagonal 2:17 phase remains stable. The coercivities of the SmCo_{7-x}Cr_x magnets are significantly enhanced by increasing the Cr content. The highest coercivity of 15.8 kOe is obtained for the SmCo_{6.4}Cr_{0.6} alloy annealed at 700 degrees C for 30 min. The transformation of Th₂Ni₁₇-type structure to Th₂Zn₁₇-type structure is observed when increasing the annealing temperature. The magnetization reversal process of the alloys has been discussed, according to the measurement of initial magnetization curves and minor hysteresis loops.



Co-pyrolysis of wood biomass and synthetic polymers mixtures - Part IV: Catalytic pyrolysis of pine wood and polyolefinic polymers mixtures in hydrogen atmosphere

Sharypov-VI; Beregovtsova-NG; Kuznetsov-BN; Baryshnikov-SV; Cebolla-VL; Weber-JV; Collura-S; Finqueneisel-G; Zimny-T

JOURNAL-OF-ANALYTICAL-AND-APPLIED-PYROLYSIS. JUN 2006; 76 (1-2) : 265-270

The pyrolysis in a hydrogen atmosphere of pine wood and synthetic polymers (polyethylene and polypropylene) mixtures was studied in a rotating autoclave. The effects of reaction temperature, wood/polymers mixture composition and catalysts, on the mixtures conversion into liquids and gases were established and discussed. The used catalysts were pyrrhotite and haematite materials activated by mechanochemical treatment.

In the co-liquefaction processes the interaction between fragments of wood and polymers thermal decomposition took place. This results in non-additive increase of the wood/polymers conversion degree by 10-15 wt.% and of the yield of distillate fractions by 14-19 wt.%. Iron ore materials were found catalytically active in the process of hydrolysis of wood/polymers mixtures. By using these catalysts a significant increase of the distillable liquids amounts (by 14-21 wt.%) and a sharp decrease of olefins and cycloparaffins content (by approximately two to three times) were observed.

Preparation of Ti+Ti(6)Si(2)B powders by high-energy ball milling and subsequent heat treatment

Silva-AN; Silva-G; Ramos-AS; Paschoal-AL; Ramos-ECT; Filgueira-M

INTERMETALLICS-. JUN 2006; 14 (6) : 585-591

The present work reports on the preparation of two-phase Ti-SS+Ti₆Si₂B alloys by high-energy milling and Subsequent heat treatment. The milled and heat-treated products were characterized by means of X-ray diffraction (XRD), scanning electron microscopy (SEM), and microanalysis via WDS. Results indicated the dissolution of silicon and boron atoms into the Ti lattice to form supersaturated solid solutions during the ball milling of Ti-10Si-5B and Ti-20Si-10B powders. TiB₂ precipitates were formed during ball milling, and the metastable structures were decomposed due to the released heat from its exothermic formation. After heat treatment at 1100 degrees C for 4 h, the equilibrium microstructures of the Ti-10Si-5B and Ti-20Si-10B alloys indicated the majority presence of the Ti and Ti₆Si₂B phases. TiB precipitates were found in Ti-10Si-5B and Ti-20Si-10B powders after heat treatment at 1200 degrees C for 16 h, indicating that the composition was moved from two-phase Ti+Ti₆Si₂B region to the three-phase Ti+Ti₆Si₂B+TiB field.

Mechanical properties of in situ Fe₃Al matrix composites fabricated by MA-PDS process

Park-BG; Ko-SH; Park-YH; Lee-JH

INTERMETALLICS-. JUN 2006; 14 (6) : 660-665

In situ intermetallic matrix composites were fabricated by an MA-PDS process with a matrix of Fe-28 at% Al and volume fractions of particle TiB₂ formulated at 0, 5, 15, and 25%. Mechanical alloying was carried out using a vibratory mill. During the mechanical alloying, Al, Ti, and B became fully embedded to the Fe lattice. Mechanically alloyed powders were sintered in a plasma discharge sintering system. As a result, Fe₃Al matrix and TiB₂ particles were successfully synthesized by this in situ process. The tensile properties of the binary alloy were significantly improved without any loss in tensile elongation. The composite also had improved tensile strength. However, composites that contained a high volume fraction showed poor tensile properties due to the presence of micro pores existed within the microstructure.

Mechanochemical preparation of molecular and supramolecular organometallic materials and coordination networks

Braga-D; Giaffreda-SL; Grepioni-F; Pettersen-A; Maini-L; Curzi-M; Polito-M

DALTON-TRANSACTIONS. 2006; (10) : 1249-1263

This Dalton Perspective deals with solvent-free reactions taking place within solids or between solids or involving a solid and a vapour. The focus is on reactions involving organometallic and coordination compounds and occurring via reassembling of non-covalent bonding, e. g. hydrogen bonds, and/or formation of ligand-metal coordination bonds. It is argued that reactions activated by mechanical mixing of solid reactants as well as those obtained by exposing a crystalline solid to a vapour can be exploited to "make crystals", which is the quintessence of crystal engineering. It is demonstrated through a number of examples that solvent-free methods, such as co-grinding, kneading, milling of molecular solids, or reactions of solid with vapours represent viable alternative, when not unique, routes for the preparation of novel molecular and supramolecular solids as well as for the preparation of polymorphic or solvate modifications of a same species. The structural characterization of the products requires the preparation of single crystals suitable for X-ray diffraction, a goal often achieved by seeding.

Structural modifications of smectites mechanically deformed under controlled conditions

Christidis-GE; Dellisanti-F; Valdre-G; Makri-P

CLAY-MINERALS. DEC 2005; 40 (4) : 511-522

SWy-1 and SAz-1 smectites and an Italian bentonite from Sardinia were mechanically deformed via high-energy ball milling for 20 h, in a controlled thermodynamic environment at constant temperature (25 degrees C) under vacuum. The deformed



smectites have a lower cation exchange capacity (CEC) and form thicker particles than the original ones, due to agglomeration of smectite crystallites. The 001 diffraction maximum shifted to lower d spacings, the intensity of the 060 reflection decreased and the background at 20-30 degrees 2 theta increased, suggesting partial amorphization of the smectite. Moreover, the layer charge of the smectites decreased. The intensity of the complex stretching band at 3625 cm(-1), and the AlAlOH, and AlFe3+OH bending bands at 916 cm(-1) and 886 cm(-1) respectively, decreased, while the band at AlMgOH bending at 849 cm(-1), disappeared. Deformation mainly disrupted the octahedral sheet and preferentially destroyed those sites occupied by Mg cations, thus explaining the observed decrease in layer charge. Octahedral sites occupied by Fe were least affected. The disruption of the octahedral sheet is substantiated further by the almost total disappearance of the dehydroxylation peak, which is more pronounced in the Mg-rich SAz-1 smectite.

Influence of mixing/milling on sintering and technological properties of anorthite based porcelainised stoneware

Taskiran-MU; Demirkol-N; Capoglu-A

CERAMICS-INTERNATIONAL. 2006; 32 (3) : 325-330

The effect of mixing/milling time on sintering and technological properties of a now porcelainised stoneware body was investigated. This stoneware body is basically based on anorthite (CaO center dot Al2O3 center dot 2SiO(2)) crystals development in the microstructure and was fabricated by using wollastonite, calcined alumina, quartz, Ukrainian ball clay and some magnesia its raw materials. Laboratory scale tests were carried out starting from mixing/milling of a reference body composition for different durations (3, 12, 24, 48 and 96 h) in a ball mill. Sintering and technological properties such its density, water absorption, firing shrinkage, flexural strength of each body were measured. X-ray diffraction (XRD) and scanning electron microscopy (SEM) studies were carried out to analyse the microstructure.

It was found that mixing/milling has a major effect on the particle size distribution, which in turn controls particles packing. High degree of close particle packing increases the densification by reducing the number and size of closed porosity in fired microstructure, which in turn contributes to the improvement of flexural strength.

Influence of powder pre-treatments and milling on dispersion ability of aqueous hydroxyapatite-based suspensions

Sadeghian-Z; Heinrich-JG; Moztarzadeh-F

CERAMICS-INTERNATIONAL. 2006; 32 (3) : 331-337

The rheological properties and the dispersion stability of highly concentrated aqueous hydroxyapatite (HAP) powder suspensions in the presence of all anionic polyelectrolyte dispersant have been investigated. Dispersed suspensions with high content of HAP powder (up to 75 wt.%), and low viscosity were obtained. HAP suspensions with solids loading as high as 75 wt.% could be prepared following a slow process of increasing the solids weight fraction in steps of 3 wt.%, starting from 66 wt.%. After each increment of solids, ball milling was conducted for periods of 6 h, which then increased LIP to 24 h with solids loading increasing. Zeta potential measurements were conducted Oil HAP powders evaluate the influence of calcination on surface-charge properties of the particles.

It was concluded that both aqueous ball milling and calcining enhance the repulsive interaction forces between particles, like the presence of the dispersant.

Statistical analysis of impact-fracture characteristics and microstructure of industrial Portland cement clinkers

Tavares-LM; Cerqueira-MC

CEMENT-AND-CONCRETE-RESEARCH. MAR 2006; 36 (3) : 409-415

Impact is the dominant breakage mode in most industrial grinding mills used in cement manufacture. The physical amenability of two industrial Portland cement clinkers to size reduction was determined through measurement of their fracture strengths under impact loading. It was found that the fracture strength of clinker is strongly dependent oil size, which is consistent with the increasing expenditure of energy in fine grinding. Also, it was observed that the measured fracture strengths could be well described by either single or Multiple Weibull distributions. The appearance of these distributions was consistent with the variability in the composition and microstructure of the clinker nodules, observed in a detailed examination under the microscope. Possible reasons for the appearance of these populations are given. It is concluded that the fracture strength of clinker is generally determined by porosity at coarser nodule sizes and by mineralogy and texture at finer sizes.

Energy efficiency of cement finish grinding in a dry batch ball mill

Toull-D; Belaadi-S; Frances-C

CEMENT-AND-CONCRETE-RESEARCH. MAR 2006; 36 (3) : 416-421

Dry grinding experiments on cement clinker were carried out using a laboratory batch ball mill equipped with torque measurement. The specific energy was found to be dependent on operating parameters and clinker environment. Additional compounds such as gypsum and pozzolanic tuff improve energy efficiency. The optimal parameters allowing maximising the energy efficiency factor were determined. Energy efficiency factors were obtained both on the crude material (size minus 2.8 mm) and on a sieved fraction (1-0.71 mm). They demonstrate that a low initial rate of breakage implies higher energy efficiency. Oil the contrary, conditions ensuring an initial maximal rate of breakage lead to all increase of the energy



consumption.

The influence of grinding mechanism on the liberation characteristics of clinker minerals

Celik-IB; Oner-M

CEMENT-AND-CONCRETE-RESEARCH. MAR 2006; 36 (3) : 422-427

This study deals with the characterization of narrowly sized fractions of clinker ground by ball mill and high-pressure grinding rolls. Chemical, physical and mineralogical characterizations were made by using XRF, laser sizing, Blaine, BET, SEM, and image analysis techniques. The emphasis was given to the preferential liberation of the constituent clinker phases. High-pressure grinding rolls gave higher degrees of liberation of mineral phases arising from the intergranular breakage along the grain boundaries compared to ball mill grinding. This is expected to influence the downstream service properties of cement.

Tribological behavior and wear mechanisms Of MOSi₂-base composites sliding against AA6063 alloy at elevated temperature

Krakhmalev-PV; Bergstrom-J

WEAR-. FEB 24 2006; 260 (4-5) : 450-457

Intermetallic Mo(Si,Al)₂, Mo(Si,Al)₂/Al₂O₃, Mo(Si,Al)₂/SiC and Mo(Si,Al)₂/ZrO₂ composites produced by spark plasma sintering of mechanically alloyed powders were tested on a block-on-cylinder apparatus, sliding against an AA6063 alloy cylinder at elevated temperature. Abrasion, micro-fracture and surface tribochemical reactions were found to be the operative wear mechanisms, producing severe wear in the investigated alloys. Abrasive wear by pull-out of Al₂O₃ and micro-fracture of Mo(Si,Al)₂ particles promotes severe wear in the Mo(Si,Al)₂/Al₂O₃ composite. In the Mo(Si,Al)₂/SiC composites, hard SiC inclusions suppressed the abrasive wear, but a tribochemical reaction was found to be the dominant wear mechanism. A combination of abrasion by pull-out of Al₂O₃ particles and a tribochemical reaction was revealed to be the main wear mechanism in the Mo(Si,Al)₂/ZrO₂ materials. The brittleness index $B = H/K-1C$ was applicable for prediction of the relative wear resistance. In agreement with the suggested model, the lowest wear rate, corresponding to $B = 5.5-6.5 \mu m(-1/2)$, was found in the Mo(Si,Al)₂/30 vol.% SiC and Mo(Si,Al)₂/30 vol.% ZrO₂ composites.

Effect of properties of titanium aluminide powders and detonation spraying conditions on phase and structure formation in coatings

Oliker-VE; Sirovatka-VL; Timofeeva II; Grechishkin-EF; Gridasova-TY

POWDER-METALLURGY-AND-METAL-CERAMICS. SEP-OCT 2005; 44 (9-10) : 472-480

We have studied phase formation in detonation coatings sprayed from Ti - 50 at.% Al powders. The powders of the alloy were obtained by various methods: crushing an ingot and mechanical alloying of Ti and Al. Using polyphase nanostructural materials activated by mechanical alloying makes the process of phase formation in the gas-thermal sprayed coatings based on them more general-purpose and controlled due to the more active and more subtle reaction of the material with the gaseous atmosphere. We have shown that from mechanically alloyed Ti - 50 at.% Al powder, using the detonation-gas spraying method we can consolidate a coating based on Al₂TiO₅ by oxidizing action of the working gas on the powder and also a coating based on titanium aluminides with TiN inclusions by nitriding action. The phase composition of the cast microstructural gamma-TiAl powder is inherited by the coating.

Coexistence of ferrimagnetic and antiferromagnetic ordering in Fe-inverted zinc ferrite investigated by NMR –

Shim-JH; Lee-S; Park-JH; Han-SJ; Jeong-YH; Cho-YW

PHYSICAL-REVIEW-B. FEB 2006; 7306 (6) : NIL_328-NIL_331

The spin structure of nanocrystalline zinc ferrite synthesized via a high-energy ball milling process was investigated by nuclear magnetic resonance obtained at various external magnetic fields and temperatures. Iron inversion induced in nanosized zinc ferrite drastically changes its magnetic property. Nanocrystalline zinc ferrite shows ferrimagnetism below 460 K, while bulk zinc ferrite shows antiferromagnetism below 10 K. We found that this antiferromagnetic order coexists with the ferrimagnetic order below 10 K in nanocrystalline samples as well. A spin of Fe ion at the tetrahedral site is ferrimagnetically coupled with that at the octahedral site and the antiferromagnetism is found among the spins at the octahedral sites. The spins that participate in both of the ferrimagnetic and antiferromagnetic orderings at the octahedral site are canted.

Thermal stability in bulk cryomilled ultrafine-grained 5083 Al alloy

Roy-I; Chauhan-M; Lavermia-EJ; Mohamed-FA

METALLURGICAL-AND-MATERIALS-TRANSACTIONS-A-PHYSICAL-METALLURGY-AND-MATERIALS-SCIENCE. MAR 2006; 37A (3) : 721-730

Thermal stability in bulk ultrafine-grained (UFG) 5083 Al that was processed by gas atomization followed by cryomilling, consolidation, and extrusion, and that exhibited an average grain size of 305 nm, was investigated in the temperature range of 473 to 673 K (0.55 to 0.79 T-m, where T-m is the melting temperature of the material) for different annealing times. Appreciable grain growth was observed at temperatures > 573 K, whereas there was limited grain growth at temperatures <



573 K even after long annealing times. The values of the grain growth exponent, n , deduced from the grain growth data were higher than the value of 2 predicted from elementary grain growth theories. The discrepancy was attributed to the operation of strong pinning forces on boundaries during the annealing treatment. An examination of the microstructure of the alloy suggests that the origin of the pinning forces is most likely related to the presence of dispersion particles, which are mostly introduced during cryomilling. Two-grain growth regimes were identified: the low-temperature region (< 573 K) and the high-temperature region (> 573 K). For temperatures lower than 573 K, the activation energy of 25 ± 5 kJ/mol was determined. It is suggested that this low activation energy represents the energy for the reordering of grain boundaries in the UFG material. For temperatures higher than 573 K, an activation energy of 124 ± 5 kJ/mol was measured. This value of activation energy, 124 ± 5 kJ/mol, lies between that for grain boundary diffusion and lattice diffusion in analogous aluminum polycrystalline systems. The results show that the strength and ductility of bulk UFG 5083 Al, as obtained from tensile tests, correlate well with substructural changes introduced in the alloy by the annealing treatment.

Research on the structures and discharge capacities of Mg-Ni alloy with different Ni contents synthesized by high-energy ball milling

Xi-SQ; Li-PL; Zhou-JG; Zhu-RH; Wang-N

MATERIALS-SCIENCE-AND-ENGINEERING-A-STRUCTURAL-MATERIALS-PROPERTIES-MICROSTRUCTURE-AND-PROCESSING. FEB 25 2006; 418 (1-2) : 81-85

The effects of different Ni contents and milling durations on the discharge capacities of Mg-Ni alloys were investigated. The samples were prepared by high-energy ball milling. Their structures were determined with X-ray diffraction and their discharge capacities were tested. The results showed that the nanocrystalline Mg₂Ni, amorphous MgNi and nanocrystalline Mg-Ni alloy could be obtained respectively when the Mg₂Ni, Ni(15)Ni and MgNi powders were milled under different conditions. The discharge capacities of milled Mg-Ni alloy powder depend on their structures. The discharge capacity of the mixed structure of nanocrystalline Mg₂Ni and Mg is higher than that of nanocrystalline Mg₂Ni phase, but the capacity of amorphous MgNi phase is not as good as that of nanocrystalline Mg-Ni alloy. The milled MgNi powder showed the best discharge capacity, 500 mAh/g, when its structure is comprised of nanocrystalline Mg-Ni alloy and nanocrystalline Ni.

High-purity amorphous Zr_{52.5}Cu_{17.9}Ni_{14.6}Al₁₀Ti₅ powders via mechanical amorphization of crystalline pre-alloys

Siegrist-ME; Siegfried-M; Löffler-JF

MATERIALS-SCIENCE-AND-ENGINEERING-A-STRUCTURAL-MATERIALS-PROPERTIES-MICROSTRUCTURE-AND-PROCESSING. FEB 25 2006; 418 (1-2) : 236-240

Fully amorphous Zr_{52.5}Cu_{17.9}Ni_{14.6}Al₁₀Ti₅ (Vit 105) powder with very low oxygen contamination was successfully produced from pre-alloyed crystalline material. A yield of over 80% was achieved without the use of a milling agent. The amorphization process was observed by differential scanning calorimetry (DSC) and X-ray diffraction (XRD). The extremely fast amorphization rate was fitted by an exponential relaxation function. A partial time-temperature-transformation (TTT) diagram was obtained from isothermal annealing experiments based on the Johnson-Mehl-Avrami (JMA) model. In addition, the microstructure of the powder was observed by scanning electron microscopy (SEM), and gas analysis was conducted at various stages of the ball-milling process.

Tribological wear behavior of diamond reinforced composite coating

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MATERIALS-SCIENCE-AND-ENGINEERING-A-STRUCTURAL-MATERIALS-PROPERTIES-MICROSTRUCTURE-AND-PROCESSING. FEB 25 2006; 418 (1-2) : 357-363

In the present study, diamond reinforced composite (DRC) coating has been applied on mild steel substrate using thermal spray coating technique. The composite powder consists of diamond, tungsten carbide, and bronze, which was mixed in a ball mill prior deposition by thermal spray. The microstructure and the distribution of diamond and tungsten carbide particle in the bronze matrix were studied. The DRC-coated mild steel substrates were assessed in terms of their high stress abrasive wear and compared with that of uncoated mild steel substrates. It was observed that when sliding against steel, the DRC-coated sample initially gains weight, but then loses the transferred counter surface material. In case of abrasive wear, the wear rate was greatly reduced due to the coating; wherein the wear rate decreased with increase in diamond content.

Structural changes in poly(ethylene terephthalate) induced by cryomilling and ambimilling

Zhu-YG; Li-ZQ; Zhang-D; Tanimoto-T

JOURNAL-OF-POLYMER-SCIENCE-PART-B-POLYMER-PHYSICS. MAR 15 2006; 44 (6) : 986-993

Poly(ethylene terephthalate) (PET) has been subjected to high energy ball milling at two different temperatures (cryogenic temperature and ambient temperature). The morphological and crystal structural evolutions of milled powders are characterized by means of scanning electron microscopy (SEM), transmission electron microscopy (TEM), and X-ray diffraction measurement. The particle size and distribution of milled powders are measured by laser diffraction particle size analyzer (LDPSA). The results indicate that the mechanisms of refining and amorphization are remarkably different between



cryomilling (mechanical milling under cryogenic temperature) and ambimilling (mechanical milling under ambient temperature). The cryomilled particles are agglomerated morphology, while the ambimilled particles are cold-welded morphology. Cryomilling induced crystalline PET translates to general amorphous, however, ambimilling induced crystalline PET transforms to oriented amorphous.

Improvement of compatibility of advanced ferritic steels with super critical pressurized water toward a higher thermally efficient water-cooled blanket system

Cho-HS; Ohkubo-H; Iwata-N; Kimura-A; Ukai-S; Fujiwara-M
FUSION-ENGINEERING-AND-DESIGN. FEB 2006; 81 (8-14) : 1071-1076

Various oxide dispersion strengthened (ODS) steels which have high Cr concentrations with and without Al addition were made by mechanical alloying method. Corrosion measurement was done in the closed system of super critical pressurized water (SCPW) at 783 K with the pressure of 25 MPa. In order to evaluate the mechanical properties and the effect of thermal aging, a miniaturized Charpy V notch (MCVN) test and tensile test were performed before and after aging at 773 K up to 1000 h. The susceptibility to stress corrosion cracking (SCC) of high Cr-ODS steels was evaluated by slow strain rate test (SSRT). The SSRT was performed in a simulated boiling water reactor (BWR) condition with high temperature oxygenated (8-10 ppm) water at 561 K and at a pressure of 7.8 MPa. Strain rates were ranging from 1×10^{-4} to 3×10^{-7} s⁻¹. The high Cr-ODS steels showed a better resistance to corrosion than SUS316L stainless steel in SCPW. The 14Cr and 16Cr-ODS steel did not suffer from aging embrittlement at 773 K for 1000 h. The ODS steels showed no susceptibility to SCC at strain rates from 1×10^{-4} to 3×10^{-7} s⁻¹.

Magnetic and electromagnetic wave absorption properties of alpha-Fe/Z-type Ba-ferrite nanocomposites

Liu-JR; Itoh-M; Machida-K
APPLIED-PHYSICS-LETTERS. FEB 6 2006; 88 (6) : NIL_194-NIL_196

The saturation magnetization values (M_s) of alpha-Fe/Ba₃Co_{1.8}Fe_{23.6}Cr_{0.6}O₄₁ nanocomposites prepared by mechanically alloying alpha-Fe with Ba₃Co_{1.8}Fe_{23.6}Cr_{0.6}O₄₁ powders increased with increasing the concentration of alpha-Fe. alpha-Fe/Ba₃Co_{1.8}Fe_{23.6}Cr_{0.6}O₄₁ nanocomposites showed higher coercivity values than alpha-Fe and Ba₃Co_{1.8}Fe_{23.6}Cr_{0.6}O₄₁ because of the effects of shape anisotropy and exchange bias. The resin compacts with 33.5 vol % alpha-Fe/Ba₃Co_{1.8}Fe_{23.6}Cr_{0.6}O₄₁ (38, 70, 85 vol % alpha-Fe) powders provided good electromagnetic wave absorption performances in ranges of 7.5-16.0, 5.4-10.5, and 4.3-8.3 GHz over the absorber thicknesses of 1.3-2.5, 1.6-3.0, and 1.7-3.2 mm, respectively.

High-performance Ag_{0.8}Pb_{18+x}SbTe₂₀ thermoelectric bulk materials fabricated by mechanical alloying and spark plasma sintering

Wang-H; Li-JF; Nan-CW; Zhou-M; Liu-WS; Zhang-BP; Kita-T
APPLIED-PHYSICS-LETTERS. FEB 27 2006; 88 (9) : NIL_142-NIL_144

Polycrystalline Ag_nPb_mSb_{Tem+2n} thermoelectric materials, whose compositions can be described as Ag_{0.8}Pb_{18+x}SbTe₂₀ were prepared using a combined process of mechanical alloying and spark plasma sintering. Electric properties of the sintered samples with different Pb contents were measured from room temperature to 700 K. The maximum power factor of 1.766 mW/mK² was obtained at 673 K for the Ag_{0.8}Pb₂₂SbTe₂₀ sample, which corresponds to a high dimensionless figure of merit, ZT=1.37. This best composition is different from that reported before.

