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

Congress and School Announcements

Colloque

"De la poudre au matériau massif"

Ecole des Mines d'ALBI, 3-5 juin 2003

Contact :SF2M, Les Fontenelles, 1 rue Craïova, F -92024 - Nanterre Cedex

<mailto:sf2mcongress@wanadoo.fr>,

<http://www.sf2m.asso.fr/>

NUCLEATION WORKSHOP

17-19 juin 2003 , Ecole des Mines de Saint Etienne

Contact Jean michel Herri (herri@emse.fr)

Vous trouverez l'appel à contribution à l'adresse :

<http://www.emse.fr/fr/transfert/spin/actualites/nucleation.html>

Premier et deuxième jour consacrés à la nucléation : théorie,
applications et étude de cas Troisième jour consacré à la nucléation des hydrates de gaz

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

ISMANAM 2003,

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

<mailto:ismanam2003@dema.ufscar.br>

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

International Conference

NANOMATERIALS AND NANOTECHNOLOGIES (NN 2003),

Crete, Greece; August 30 - September 6, 2003

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

INTERNATIONAL CONFERENCE

"Novel Technologies in Powder Metallurgy and Ceramics"

September 8-12, 2003

Kiev, Ukraine

**Fourth INTERNATIONAL CONFERENCE
ON MECHANOCHEMISTRY AND MECHANICAL ALLOYING**

4th INCOME 2003

Technical University of Braunschweig, Braunschweig, Germany

September 7-11, 2003

Website : <http://www.tu-bs.de/INCOME2003>



9ième Congrès de la SFGP,
Saint Nazaire, 9-10 septembre 2003
Contact :

<mailto:sfgp.carole.bezzi@gepea.univ-nantes.fr>,
<http://www.sfgp.asso/stnazaire2003>

11th FORESIGHT CONFERENCE ON MOLECULAR NANOTECHNOLOGY

October 9-12, 2003
San Francisco Airport Marriott
Burlingame, CA, USA
<http://www.foresight.org/Conferences/MNT11>

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

Website : <http://www.ISRSKYOTO.org/>
<mailto:info@ISRSKYOTO.org>

XV. International Symposium on Reactivity of
COLOQUE DE CRISTALLISATION INDUSTRIELLE

12-13 Novembre 2003 à Toulouse
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Journées de l'AFSIA sur les techniques de séchage innovantes

14 novembre 2003 à Paris
contact :e.mail : <mailto:andrieu@lagep.univ-lyon1.fr>
<http://www-lagep.univ-lyon1.fr/>

MELANGE DES POUDRES, ECHANTILLONNAGE,
DISTRIBUTION DE TEMPS DE SEJOUR

11-12 Décembre 2003 à Ecole des Mines d'ALBI
Contact : Henri Berthiaux
(<mailto:berthiau@enstimac.fr>)

Third International Symposium on Ultrafine Grained Materials

The 2004 TMS Annual Meeting, March 14-18, 2004, Charlotte, NC, US
<http://www.cms.tms.org/>



Press release

VARIO-PLANETARY MILL "pulverisette 4"

The "pulverisette 4" vario-planetary mill is capable of emulating ball mills of conventional design, precisely simulating the types of stress entailed and thus reproducing or optimising grinding processes. Due to the high flexibility available for selecting the grinding parameters, it is possible to achieve results unattainable with any other ball mills.

This is the ideal mill for mechanical activation and alloying. The main applications are in the field of materials research and, of course, wherever a powerful, innovative planetary mill is required.

When particles ≤ 10 mm are fed in, a final fineness up to $0.1 \mu\text{m}$ can be achieved. The useful capacity is between 2×30 ml in the case of 80 ml grinding bowls and 2×225 ml when 500 ml grinding bowl are used.

Method of operation:

With standard planetary ball mills the grinding bowls are rotating and mounted eccentrically on a rotating support disc. The rotational speed of the supporting disc can be selected at will; the grinding bowl rotates at a fixed transmission ratio.

Due to the overlapping of grinding bowls and supporting disc, the material to be ground and the grinding balls execute movements and trajectories in the grinding bowl, which are defined by the transmission ratio.

With the "pulverisette 4" vario-planetary mill the rotational speeds of grinding bowls and supporting disc can be adjusted completely independently of each other. By varying the transmission ratio it is possible to control the movements and trajectories of the grinding balls at will so that the balls strike the inner wall of the bowl vertically (high impact energy), approach each other tangentially (high friction) or just roll down the inner wall of the bowl (centrifugal mills).

All intermediate levels and combinations of frictional and impact pressures can be set as required. By changing the transmission ratio it is therefore possible for the first time to carry out mechanical activation as well as mechanical alloying.

Furthermore, it is also possible for the first time to optimally adjust a planetary ball mill to the material to be ground, the size of the grinding bowls and the grinding balls.



Features of performance:

- for the first time, all grinding parameters can be selected at will for optimal preparation of sample
- Programming of the grinding parameters by PC software as desired
- RS232 interface for programming and to transfer grinding parameters to the PC (validation) as well as for controlling the “pulverisette 4”
- Real-time display of the speeds to monitor the grinding process
- Reversing option (direction of rotation reversed periodically) to improve the grinding results
- Emulation of various ball mills
- Variably adjustable pressure on sample (friction and/or impact)
- Final fineness < 1 µm
- Simultaneous grinding in up to 4 small or 2 large grinding bowls
- Quick, secure fastening of the grinding bowls
- Ease of cleaning

Please ask for our new up-dated leaflet Vario-Planetary Mill “pulverisette 4” with grinding bowls in 9 different materials, grinding bowls in 6 different sizes and grinding balls in 7 different sizes.

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9 èmes Journées du Réseau Français de Mécanosynthèse - Albi

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Périodiques / Congrès

[81] LOW-ENERGY BALL MILLING ASSOCIATED WITH A MONOMODE MICROWAVE: A NEW ROUTE TO SYNTHESIZE MANGANESE HEXATHIOHYPODIPHOSPHATE AND TO INTERCALATE IONIC SPECIES

M. Gasgnier*, H. Szwarc, A. Petit, R. Clément and E. Rivière - Powder Technology, 131 (2003) 241-249.

To synthesize manganese hexathiohypodiphosphate, stoichiometric mixtures of manganese, red phosphorus, and sulfur, together with potassium chloride, underwent low-energy ball milling under ambient conditions followed by exposure to the electromagnetic beam of a monomode microwave. X-ray diffraction patterns show that $Mn_2P_2S_6$ (structure MI, or H) has formed, as well as small amounts of intercalated KCl [these findings are supported by infrared (IR) observation]. Also some MnS have been formed. Electron diffraction patterns show the existence of structure H. They also reveal the emergence of the (up to now hypothetical) orthohexagonal structure and of some «turbostratic» layered material whose nature is not yet understood. Superconducting quantum interference device (SQUID) measurements show a ferromagnetic behavior, which can be assigned to the formation of some manganese monophosphide. Tetraethyl chloride ammonium is then easily intercalated as revealed by X-ray patterns and IR measurements. Finally, it has been shown that all three parameters (ball milling, microwave exposure, and presence of KCl as a catalyst) are necessary to form manganese hexathiohypodiphosphate. Filtrations and decantation lead to a pure hexathiohypodiphosphate compound.

[80] THE INFLUENCE OF MECHANICAL ALLOYING ON THE STRUCTURAL AND PHYSICAL PROPERTIES OF YNi4B COMPOUND

Timko-M; Kowalczyk-A; Szlaferek-A; Kovac-J; Zentko-A; Tolinski-T; Briancin-J - PHYSICA-STATUS-SOLIDI-A-APPLIED-RESEARCH. 196 (1) : 201-204.

We have investigated the influence of mechanical milling on structural changes, magnetic and superconducting properties of YNi₄B compound. This compound crystallizes in a hexagonal structure with the P6/mmm space group. Magnetic measurements have shown that originally prepared sample exhibits superconducting behaviour below 12 K and above this temperature the sample is paramagnetic. The great decrease of intensity and broadening of diffraction lines have been detected after milling as a consequence of sample amorphisation.

[79] THE ELECTRONIC AND ELECTROCHEMICAL PROPERTIES OF THE LANi5-BASED ALLOYS

Szajek-A; Jurczyk-M; Nowak-M; Makowiecka-M - PHYSICA-STATUS-SOLIDI-A-APPLIED-RESEARCH. 196 (1) : 252-255

Nanocrystalline La(Ni,M)(5)-type alloys were prepared by mechanical alloying (MA) and subsequent annealing. The alloying elements of 3d transition metals, Mn, Al and Co were substituted for Ni in LaNi₅, and the structural, electrochemical as well as electronic properties were studied. It was found that the partial substitution of Ni by Al or Mn in nanocrystalline La(Ni,M)(5) alloy leads to an increase in the discharge capacity. On the other hand, cobalt substituting nickel in LaNi_{4-x}Mn_{0.75}Al_{0.25}Cox alloy greatly improved the discharge capacity and cycle life of LaNi₅ material. The electronic structure has been studied by the tight binding version of the linear muffin-tin orbital method (TB LMTO) for La(Ni_{0.8-x}CoxAl_{0.1}Mn_{0.1})(5) systems, where x = 0, 0.1, 0.2, and 0.3.

[78] THE ELECTRONIC AND ELECTROCHEMICAL PROPERTIES OF THE TiFe1-xNiX ALLOYS

Szajek-A; Jurczyk-M; Jankowska-E - PHYSICA-STATUS-SOLIDI-A-APPLIED-RESEARCH. 196 (1) : 256-259

Mechanical alloying (MA) process was introduced to produce nanocrystalline TiFe_{1-x}Ni_x alloys (0 less than or equal to x less than or equal to 1). XRD analysis showed that, firstly, after 25 h of milling, the starting mixture of the elements had decomposed into an amorphous phase and, secondly, the annealing in high purity argon at 750 degreesC for 0.5 h led to formation of the CsCl-type (B2) structures with a crystallite sizes of about 30 nm. These materials, used as negative electrodes for Ni-MH batteries, showed an increase in discharge capacity with a maximum for x = 3/4. The band structure has been studied by the Tight Binding version of the Linear Muffin-Tin method in the Atomic Sphere Approximation (TB LMTO ASA). Increasing content of Ni atoms intensified charge transfer from Ti atoms, extended valence bands and increased the values of the densities of electronic states at the Fermi level.

[77] ELECTRONIC PROPERTIES OF NANOCRYSTALLINE AND POLYCRYSTALLINE TiFe0.25Ni0.75 ALLOYS

Smardz-K; Smardz-L; Jurczyk-M; Jankowska-E - PHYSICA-STATUS-SOLIDI-A-APPLIED-RESEARCH. 196 (1) : 263-266

Nanocrystalline and polycrystalline TiFe_{0.25}Ni_{0.75} alloys were prepared by mechanical alloying (MA) followed by annealing and arc melting method, respectively. The amorphous phase of MA samples forms directly from the starting mixture of the elements, without other phase formation. Heating the MA powders at 750 degreesC for 0.5 h resulted in the creation of CsCl-type nanocrystalline compound with mean crystallite size of about 50 nm. The substitution of Fe in TiFe by Ni leads to significant modifications of the electronic structure of the polycrystalline sample. On the other hand, the XPS valence band of the MA nanocrystalline TiFe_{0.25}Ni_{0.75} alloy is considerably broader compared to that measured for the polycrystalline sample. The strong modifications of the electronic structure in the nanocrystalline TiFe_{0.25}Ni_{0.75} alloy could significantly influence its hydrogenation properties.

[76] ANODE BEHAVIORS OF ALUMINUM ANTIMONY SYNTHESIZED BY MECHANICAL ALLOYING FOR LITHIUM SECONDARY BATTERY

Honda-H; Sakaguchi-H; Fukuda-Y; Esaka-T - MATERIALS-RESEARCH-BULLETIN. 2003; 38 (4) : 647-656.

AlSb was synthesized as an anode active material for lithium secondary battery using mechanical alloying (MA). Electrochemical performance was examined on the electrodes of AlSb synthesized with different MA time. The first charge (lithium-insertion) capacity of the AlSb electrodes decreased with increasing the MA time. The discharge capacity on repeating charge-discharge cycle, however, did not show the same dependence. The electrode, consisting of the 20 h MA sample exhibited the longest charge-discharge life cycle, suggesting that there is the optimum degree of internal energy derived from the strain and/or the amorphization due to mechanical alloying. These results were evaluated using ex situ X-ray diffraction and differential scanning calorimetry.

[75] DEVELOPMENT OF MAGNETIC SOFTNESS IN HIGH-ENERGY BALL MILLING ALLOYED Fe50B50



Gonzalez-JM; Alcazar-GAP; Zamora-LE; Tabares-JA; Bohorquez-A; Gancedo-JR - JOURNAL-OF-MAGNETISM-AND-MAGNETIC-MATERIALS. 2003; 261 (3) : 337-346

Results are reported about the phase distribution and magnetic properties of high-energy ball milled samples prepared from pure Fe and B powders and having nominal equiatomic composition. After milling the precursor powders for times from 40 to 270 h, the milling product consists of a majority amorphous phase and of milling-time-dependent small percentages of alpha-Fe, Fe₂B and FeB. The coercivities measured in the as-milled samples were of the order of thousands of A/m and decreased to tens of A/m after a short time; low temperature treatments decreased the coercivity. We propose that this softening process is linked to a combination of stress relaxation and of enhancement of the exchange coupling between the minority crystalline phases and amorphous matrix, this last fact leading to the elimination of hindrances to the domain wall motion. (C) 2002 Elsevier Science B.V. All rights reserved.

[74] A MOSSBAUER EFFECT AND X-RAY DIFFRACTION INVESTIGATION OF TI-SN INTERMETALLIC COMPOUNDS: II. NANOSTRUCTURED PHASES PREPARED BY BALL MILLING WITH Al₂O₃ AND TIN

O'Brien-JW; Dunlap-RA; Dahn-JR SO: JOURNAL-OF-ALLOYS-AND-COMPOUNDS. 2003; 353 (1-2) : 65-73.

A thorough X-ray diffraction and Sn-119 Mossbauer effect investigation of Ti₃Sn, Ti₂Sn, Ti₅Sn₃, Ti₆Sn₅, and Ti₂Sn₃ phases ball milled with Al₂O₃, and TiN has been made. The starting compounds were verified to have the same crystal structures as those reported in the literature. X-ray diffraction data showed a decrease in grain size of the Ti-Sn phase with increased milling time and no interactions between the Ti-Sn phase and the Al₂O₃ or TiN. The two samples with the highest Sn concentrations (Ti₂Sn₃ and Ti₅Sn₆) were observed to precipitate elemental Sn when ball milled. This Sn precipitation was observed qualitatively by X-ray diffraction patterns and quantitatively by site populations reported in the Mossbauer spectroscopy data.

[73] PHASE TRANSFORMATION STUDY OF Pb-TE POWDERS DURING MECHANICAL ALLOYING

Bouad-N; Marin-Ayral-RM; Nabias-G; Tedenac-JC - JOURNAL-OF-ALLOYS-AND-COMPOUNDS. 2003; 353 (1-2) : 184-188.

In this study PbTe thermoelectric material was prepared by mechanical alloying using a high energy planetary ball mill. The alloy formation and phase transformation during mechanical alloying was followed by X-ray diffraction (XRD) and differential scanning calorimetry (DSC), and the microscopic morphology was studied by scanning electron microscopy (SEM). Results show that mechanical alloying is associated with an exothermic reaction between lead and tellurium which promotes the rapid formation of lead telluride and leads to a reasonable milling time (3 h) to obtain PbTe stoichiometric samples.

[72] STEADY STATE PRODUCTS IN THE FE-GE SYSTEM PRODUCED BY MECHANICAL ALLOYING

Kwon-YS; Gerasimov-KB; Lomovsky-OI; Pavlov-SV - JOURNAL-OF-ALLOYS-AND-COMPOUNDS. 2003; 353 (1-2) : 194-199

Prolonged mechanical alloying of elemental blends and mechanical milling of individual intermetallics of the same chemical composition in the Fe-Ge system lead to the same steady state. The phase composition of this steady state was investigated in the entire composition range using a conventional XRD technique. A map reflecting the steady-state phase composition for the different chemical composition was constructed. Mechanical alloying and grinding form products of the following composition (in sequence of increasing Ge content): alpha (alpha(1)) bcc solid solution, alpha+beta-phase (Fe₂-xGe), beta-phase, beta+FeGe(B20), FeGe(B20), FeGe(B20)+FeGe₂, FeGe₂, FeGe₂+Ge, Ge. The incongruently melting intermetallics Fe₆Ge₅ and Fe₂-xGe, decompose on milling. Fe₆Ge₅ produces a mixture of the beta-phase and FeGe(B20) while Fe₂Ge₃ produces a mixture of the FeGe(B20) and FeGe₂ phases. These facts are in good agreement with a model that implies local melting as mechanism for new phase formation during mechanical alloying. The stability of the FeGe(B20) phase, which is also an incongruently melting compound, is explained as a result of the highest density of this phase in the Fe-Ge system.

[71] STRUCTURAL EVOLUTION AND METASTABLE PHASE DETECTION IN MgH₂-5%NBH NANOCOMPOSITE DURING IN-SITU H-DESORPTION IN A SYNCHROTRON BEAM

Yavari-AR; de-Castro-JFR; Vaughan-G; Heunen-G - JOURNAL-OF-ALLOYS-AND-COMPOUNDS. 2003; 353 (1-2) : 246-251

Hydrogen sorption in magnesium and magnesium hydrides has been shown to accelerate in sub-micron structures generated by mechanical attrition. It further accelerates when transition metal nanoparticle hydrides such as Nb are added. These hydrides are reported to trigger the desorption during heating. Metastable orthorhombic gamma-MgH₂ also forms during attrition and has been shown to desorb first during heating. In this work, using a high brilliance high energy beam for diffraction in transmission, we find that indeed the desorption sequence during heating of mechanically milled MgH₂-5%NbH nanocomposite proceeds through at least two major steps. First the desorption of the metastable orthorhombic gamma-MgH₂ into hexagonal Mg triggers the decomposition of part of the tetragonal beta-MgH₂ and the orthorhombic beta-NbH. The second stage involving the desorption of hydrogen from the remaining beta-MgH₂ appears to be linked to a NbH_x metastable phase previously reported to be NbH_{0.6}. The present results however, using more extensive diffraction data, indicate that this metastable phase is a supersaturated Nb solid solution as seen likely from the phase diagram.

[70] PROCESSING OF YTTRIUM-ALUMINUM GARNETS UNDER NON-EQUILIBRIUM CONDITIONS

Patankar-SN; Zhang-D; Adam-G; Froes-FH - JOURNAL-OF-ALLOYS-AND-COMPOUNDS. 2003; 353 (1-2) : 307-309.

Polycrystalline yttrium-aluminum garnet (YAG) was processed via mechanical alloying (MA). The powder mixture comprising equal proportions of elemental aluminum and yttria was mechanically alloyed (MA'd) for 5 h and the MA'd powder mixture was heat treated. While MA resulted in aluminum-yttrium solid solution, the reaction leading to the formation of YAG occurs during the subsequent heat treatment of the powder mixture.

[69] ELECTROCHEMICAL CHARACTERISTICS OF Mg₂Ni-TYPE ALLOYS PREPARED BY MECHANICAL ALLOYING

Yuan-HT; Li-QD; Song-HN; Wang-YJ; Liu-JW - JOURNAL-OF-ALLOYS-AND-COMPOUNDS. 2003; 353 (1-2) : 322-326.

The electrochemical characteristics of Mg₂Ni-type hydrogen storage alloys prepared by mechanical alloying (MA) using a planetary ball mill were investigated. The structures of three alloys were characterized by XRD and SEM. Results of XRD indicated that the structure of the synthesized alloys changed with milling time from polycrystalline to nanocrystalline or amorphous. In charge-discharge cycle tests, the partial substitution of Al and V for Mg increased the discharge capacity of



Mg₂Ni alloy at 25degreesC. The discharge capacity of the 80-h milled Mg_{1.5}Al_{0.3}V_{0.2}Ni at 25-mAg(-1) discharge current density reached 435 mAhg(-1). The poor cycle life of Mg-based alloys is a critical problem in spite of their high capacity. It was found that the addition of Al could lead to an improvement of the cycling stability.

[68] MAGNESIUM-BASED NANOCOMPOSITES CHEMICAL HYDRIDES

Huot-J; Liang-G; Schulz-R - JOURNAL-OF-ALLOYS-AND-COMPOUNDS. 2003; 353 (1-2) : L12-L15

The hydrolysis reaction of nanostructured MgH₂ and nanocomposites MgH₂-X (X=Ca, Li, LiAlH₄) prepared by ball-milling was studied as a function of milling time and component proportion. It was found that nanocrystallinity greatly enhances the hydrolysis kinetics. Moreover, in this new class of chemical hydrides, the reaction also proceeds to full completion, contrary to some conventional chemical hydrides where the reaction stops before total completion due to the formation of passivation layers. The effect of addition of acidic solutions was also investigated.

[67] P/M PROCESSING ROUTES FOR HIGH NITROGEN MARTENSITIC STAINLESS STEELS

Toro-A; Alonso-Falleiros-N; Rodriguez-D; Ambrozio-F; Liberati-JF; Tschiptschin-AP - TRANSACTIONS-OF-THE-INDIAN-INSTITUTE-OF-METALS. OCT 2002; 55 (5) : 481-487

High nitrogen martensitic stainless steels were obtained through four different P/M processing routes namely: (1) die-compaction + simultaneous nitriding/sintering + hot isostatic pressing (2) nitriding of the uncompressed powder + hot isostatic pressing, (3) nitriding of the uncompressed powder+hot pressing, and (4) mechanical alloying (Fe+Cr+chromium nitride)+sintering +hot isostatic pressing. Nitrogen addition and hot pressing treatments were made at temperatures between 1273 and 1473 K under N₂ pressure, while hot isostatic pressing was carried out at 1423 K under 150 MPa argon pressure. Stainless steels with relative densities of 88% to 99.5% and nitrogen contents ranging from 0.47 to 2.9 wt% N was obtained. The microstructure of heat-treated specimens with less than 0,73 wt%N was composed by martensite plus retained austenite, while increasing amounts of precipitated nitrides were observed in the alloys with higher nitrogen contents. The steels were ranked according to their corrosion and corrosion-erosion properties, which were measured through slurry wear and polarization tests. The negative effect of porosity was greater on corrosion-erosion resistance (measured in slurry wear tests) than in electrochemical corrosion resistance in acid solution (measured in polarization tests). On the other hand, increasing the nitrogen content of the specimens beyond the nitrogen solubility limit strongly reduced their localized corrosion resistance, although practically no effect was observed in corrosion-erosion tests.

[66] SYNTHESIS AND OPTICAL CHARACTERIZATION OF CDTE NANOCRYSTALS PREPARED BY BALL MILLING PROCESS

Tan-GL; Hommerich-U; Temple-D; Wu-NQ; Zheng-JG; Loutts-G - SCRIPTA-MATERIALIA. 2003; 48 (10) : 1469-1474.

CdTe nanocrystals have been successfully synthesized by mechanical alloying (MA) process. XRD pattern and HRTEM images confirmed the formation of cubic structural CdTe Nan crystals (2-40 nm). The capped CdTe nanocrystals show absorption peaks locating within the visible range of 500-700 nm, when uncapped ones locating within ultraviolet range.

[65] FEAL-TIN NANOCOMPOSITE PRODUCED BY REACTIVE BALL MILLING AND HOT-PRESSING CONSOLIDATION

Krasnowski-M; Kulik-T - SCRIPTA-MATERIALIA. 2003; 48 (10) : 1489-1494.

The FeAl-TiN nanocomposite was produced by milling an elemental powder mixture of Al-Fe-Ti in nitrogen, followed by hot-pressing consolidation under 8 GPa of pressure. The average hardness of the nanocomposites consolidated at temperatures of 750 and 950 degreesC are 1424 HV0.2 and 1461 HV0.2 respectively, and the density is 97% of the theoretical value in both cases.

[64] HOT ROLLING EFFECT ON MECHANICAL PROPERTIES OF COPPER ALLOYS STRENGTHENED BY DISPERSION AND UNIAXIALLY HOT PRESSED

Camurri-C; Lopez-M; Inostroza-J; Guzman-M; Jimenez-JA - REVISTA-DE-METALURGIA. 2003; 39 (1) : 35-40.

Copper powder of 140 mum mean size was mechanical alloyed with several Compounds, 2 % B₄C, 2 % ZrC, 1 % CrB, 2 0%. Cr₃C₂, 1 and 2 vol. % ZrB₂ in a RETSCH PM high energy balls mill. The alloying process was carried out in stainless steel containers during 6 or 8 h of milling, under argon atmosphere, and the ball to powder charge ratio was 5:1. Instead of HIP processing, the alloyed powders were consolidated by uniaxial hot pressing at 650 degreesC for 2 h at a pressure of 90 MPa in argon atmosphere. Afterward the compact samples of 30 x 10 x 10 mm were hot rolled at 850 degreesC with area reduction from 10 to 40 %. If an homogeneous hot pressing process is obtained, the subsequent hot rolling can be avoided as only an annealing effect is produced, characterized by a small decrease in hardness and tensile strength with a small increase in ductility

[63] MECHANICALLY ALLOYED MG₂NI FOR METAL-HYDRIDE-AIR SECONDARY BATTERY

Mohamad-AA; Mohamed-NS; Alias-Y; Arof-AK - JOURNAL-OF-POWER-SOURCES. 2003; 115 (1) : 161-166.

Mechanically alloyed Mg₂Ni and a single air (oxygen) electrode are used as the anode and cathode, respectively, in a Mg(2)Ni₆ M KOHIO₂ rechargeable metal-hydride-air (MH-air) battery. The battery is tested for self-discharge by measuring the open-circuit voltage (OCV) and cycling characteristics. Battery degradation after charge-discharge cycling is characterized by means of X-ray diffraction (XRD) and scanning electron microscopic (SEM) analyses.

[62] LEAD-FREE SN-AG AND SN-AG-BI SOLDER POWDERS PREPARED BY MECHANICAL ALLOYING

Lai-HL; Duh-JG - JOURNAL-OF-ELECTRONIC-MATERIALS. 2003; 32 (4) : 215-220.

A mechanical alloying (MA) process was used to produce lead-free solder pastes of Sn-3.5Ag and the Sn-3.5Ag-4Bi system. Because of the high energy induced by repeated fracturing and welding, the grinding media played an important role during the MA process. A ceramic container was used to provide stronger impact force, which could induce phase transformation better than a Teflon container. In addition, it was found that 1-cm balls could fracture Bi particles and promote their dissolution into the Sn matrix. On the contrary, the milling process tended to achieve homogeneous mixing when using 3-mm balls. The MA powders, after milling with 3-mm balls, showed a small endothermic peak from the differential scanning calorimetry (DSC) profile at around 138degreesC, which was the eutectic temperature of Sn-Bi. The melting points of the MA powders in the ceramic container were measured to be 221degreesC and 203degreesC, respectively, for Sn-3.5Ag and Sn-3.5Ag-4Bi from the DSC curves. The reduced melting point ensured the complete melting during reflow with a peak temperature of 240degreesC. The formation of Ag₃Sn was also observed from the x-ray diffraction peaks, indicating successful alloying by MA. The solder pastes could, thus, be produced by adding flux into the MA powders. The wetting



property of the solder joint was also evaluated. The as-prepared solder pastes on electroless Ni-P/Cu/Si showed good metallurgical bonding with a contact angle less than 20degrees.

[61] SOLID-STATE REACTIONS IN THE FE(68)GE(32) SYSTEM UPON MECHANICAL ALLOYING

Elsukov-EP; Dorofeev-GA; Ul'-yanov-AL; Nemtsova-OM; Porsev-VE - PHYSICS-OF-METALS-AND-METALLOGRAPHY. FEB 2003; 95 (2) : 164-168.

X-ray diffraction and Mossbauer spectroscopy were used to study solid-state reactions that occur upon mechanical alloying of iron and germanium powders taken in an atomic ratio of 68 : 32. The mechanical alloying was found to proceed in two stages and occur only if alpha-Fe is in a nanostructural state.

[60] SYNTHESIS AND MECHANICAL PROPERTIES OF MECHANICALLY ALLOYED AL-CU-FE QUASICRYSTALLINE COMPOSITES

Schurack-F; Eckert-J; Schultz-L - PHILOSOPHICAL-MAGAZINE. 2003; 83 (11) : 1287-1305.

Al-Cu-Fe alloys were prepared by mechanical alloying starting from elemental powders in a high-energy planetary ball mill. Three different alloy compositions with the same c(Cu) : c(Fe) ratio of 2: 1 but different aluminium contents, that is Al55Cu30Fe15, Al63Cu25Fe12 and Al70Cu20Fe10, were investigated. A sequence of solid-state reactions resulting in quasicrystalline phase formation in Al63Cu25Fe12 proceeds during milling and during annealing of the as-milled powder. These reactions were studied by X-ray diffraction, transmission electron microscopy and differential scanning calorimetry. In order to form an aluminium-matrix composite, Al63Cu15Fe12 single-phase quasicrystalline powders were blended with different amounts of aluminium. In an intermediate milling step the powder blend was homogenized. The powders were consolidated by hot extrusion. The bulk samples revealed a homogeneous dispersion of the particles in the matrix but a rather heterogeneous size distribution. The mechanical properties at room temperature were tested by constant-rate compression tests. A rule-of-mixtures dependence of the ultimate strength and the yield strength on the volume fraction of the quasicrystalline particles was found.

[59] MECHANICALLY INDUCED SOLID-STATE DEVITRIFICATIONS OF ZR70PD20NI10 GLASSY ALLOY POWDERS

El-Eskandarany-M; Saida-J; Inoue-A - METALLURGICAL-AND-MATERIALS-TRANSACTIONS-A-PHYSICAL-METALLURGY-AND-MATERIALS-SCIENCE. 2003; 34A (4) : 893-898.

A single glassy phase of Zr70Pd20Ni10 alloy powder was synthesized by mechanical alloying the elemental powders for 48 hours, using a high-energy ball-milling technique. The obtained glassy phase transformed into a metastable big-cube phase upon increasing the ball-milling time (100 hours). After 150 hours of milling, a complete glass-metastable-phase transformation was achieved, and the end product was nanocrystalline big-cube powder, which has a lattice constant of 1.23 nm. As the ball-milling time was further increased the big-cube phase could no longer withstand the mechanical deformation that was generated by the milling media and transformed into a new metastable phase of nanocrystalline fcc Zr70Pd20Ni10. The lattice constant of this metastable phase was calculated to be 0.455 nm. The reported metastable phases here are new and have never been, so far as we know, reported for the ternary Zr

[58] CHANGE IN PRIMARY PHASE FROM ICOSAHEDRAL QUASICRYSTAL TO FCC ZR2NI BY MECHANICAL DISORDERING IN ZR-AL-NI-CU-PD GLASSY ALLOY

Saida J. El-Eskandarany MS. Inoue A. - Scripta Materialia. 48(9):1397-1401, 2003

Change in the primary crystallization from a single icosahedral quasicrystalline phase into the fcc Zr2Ni phase by mechanical disordering was investigated in a melt-spun Zr65Al7.5Ni10Cu12.5Pd5 glassy alloy. The transition of the primary phase is attributed to the mechanical strain induced in the icosahedral local structure in the glassy state.

[57] SOLID-STATE REACTIONS IN THE FE(68)GE(32) SYSTEM UPON MECHANICAL ALLOYING

Elsukov EP. Dorofeev GA. Ul'yanov AL. Nemtsova OM. Porsev VE. - PHYSICS OF METALS AND METALLOGRAPHY. 95(2):164-168, 2003

X-ray diffraction and Mossbauer spectroscopy were used to study solid-state reactions that occur upon mechanical alloying of iron and germanium powders taken in an atomic ratio of 68 : 32. The mechanical alloying was found to proceed in two stages and occur only if alpha-Fe is in a nanostructural state.

[56] MECHANICALLY INDUCED SOLID-STATE DEVITRIFICATIONS OF ZR70PD20NI10 GLASSY ALLOY POWDERS

El-Eskandarany M. Saida J. Inoue A. - METALLURGICAL AND MATERIALS TRANSACTIONS A-PHYSICAL METALLURGY AND MATERIALS SCIENCE. 34A(4):893-898, 2003

A single glassy phase of Zr70Pd20Ni10 alloy powder was synthesized by mechanical alloying the elemental powders for 48 hours, using a high-energy ball-milling technique. The obtained glassy phase transformed into a metastable big-cube phase upon increasing the ball-milling time (100 hours). After 150 hours of milling, a complete glass-metastable-phase transformation was achieved, and the end product was nanocrystalline big-cube powder, which has a lattice constant of 1.23 nm. As the ball-milling time was further increased the big-cube phase could no longer withstand the mechanical deformation that was generated by the milling media and transformed into a new metastable phase of nanocrystalline fcc Zr70Pd20Ni10. The lattice constant of this metastable phase was calculated to be 0.455 nm. The reported metastable phases here are new and have never been, so far as we know, reported for the ternary Zr-Pd-Ni system, or its binary-phase relations.

[55] OXIDATION BEHAVIOUR OF Ti3Al-TiC COMPOSITES

Li Z. Gao W. Liang J. Zhang DL. - Materials Letters. 57(13-14):1970-1976, 2003

Ti3Al-TiC composites were fabricated through mechanical milling and hot isostatic pressing (HIPing) route using Ti, Al, and TiC as starting materials. Their isothermal oxidation resistances were tested at 700 and 800 degreesC in air. The results showed that the composite samples exhibited a lower oxidation rate than the cast Ti3Al at both temperatures. It appeared that the total oxidation mass gains were affected by the ball milling time, following the order of 4 < 2 < 8 < 16 h from low to high. It was also found that all composite samples exhibited superior scale spallation resistance, except for the sample with 4 h milling at 800 & DEG;C. It is believed that the oxide scales with the properties of dense, easy to deform, strong adherence, and good interfacial connections have better protective ability.



[54] MANUFACTURING AND CHARACTERIZATION OF NANO-SIZED PBTE POWDERS

HW Lee, DY Lee, IJ Kim, BC Woo - XXI INTERNATIONAL CONFERENCE ON THERMOELECTRICS, PROCEEDINGS ICT '02, 2002, pp 17-20 - 21ST INTERNATIONAL CONFERENCE ON THERMOELECTRICS (ICT 02); LONG BEACH, CALIFORNIA. AUGUST 25-29, 2002

Nano-sized PbTe powders were manufactured by the high-energy ball milling of a PbTe bulk, obtained by the water quenching of a melt. The wet milling using an ethanol were applied during the ball milling. The prepared powders were characterized by the X-ray powder diffraction pattern analysis to obtain crystallographic parameters such as the crystallite size and the misfit strain. A pseudo-Voigt function was used for the determination of the integral breadth and the full width at half maximum (FWHM) of peaks broadened by the effect of crystallite size and misfit strain. The Scherrer method, the Williamson-Hall plot and the single peak analysis were employed to calculate the size of crystallite and misfit strain with the consideration of instrumental broadening using standard sample. As results, we can observe the tendency that the crystallite size decreases and the misfit strain increases with increasing milling time. The crystallite size of powders was determined at the range of 15 similar to 100 nm in 50 similar to 150 hours milling time. The crystallite size and the misfit strain were different from crystallographic directions in this material, according to the Williamson-Hall plot and the $\sin^2 \psi$ peak analysis.

[53] PHASE TRANSFORMATION IN AN N-TYPE FeSi₂ PROCESSED BY MECHANICAL ALLOYING

SC Ur, IH Kim, JI Lee, KW Cho, P Nash - XXI INTERNATIONAL CONFERENCE ON THERMOELECTRICS, PROCEEDINGS ICT '02, 2002, pp 114-117- 21ST INTERNATIONAL CONFERENCE ON THERMOELECTRICS (ICT 02); LONG BEACH, CALIFORNIA. AUGUST 25-29, 2002

-type Fe_{0.98}Co_{0.02}Si₂ powders have been produced by mechanical alloying process and consolidated by vacuum hot pressing. As-milled powders were in a metastable state and fully transformed to beta-FeSi₂ phase by subsequent isothermal annealing. However, as-consolidated iron silicides consisted of untransformed mixture of alpha-Fe₂Si₅ and epsilon-FeSi phases. Isothermal annealing has been carried out to induce the transformation to a thermoelectric semiconducting beta-FeSi₂ phase. The transformation behavior of beta-FeSi₂ was investigated a modified TGA, SEM and XRD analyses. Isothermal annealing at 830 degreesC in vacuum led to the beta-FeSi₂ phase transformation, but some residual metallic alpha- and epsilon-phases were unavoidable. Microstructures of iron silicides were investigated using SEM. Microharness before and after isothermal annealing was evaluated and correlated with the phase transformation as well as thermoelectric proper-ties.

[52] PREPARATION AND CHARACTERIZATION OF IRON OXIDE-ZIRCONIA NANO POWDER FOR ITS USE AS AN ETHANOL SENSOR MATERIAL

CVG Reddy, SA Akbar, W Cao, OK Tan, W Zhu - CHEMICAL SENSORS FOR HOSTILE ENVIRONMENTS (Series: CERAMIC TRANSACTIONS), 2002, Vol 130, pp 67-78 - SYMPOSIUM ON CHEMICAL SENSORS FOR HOSTILE ENVIRONMENTS; INDIANAPOLIS, INDIANA. APRIL 22-25, 2001

Powders of composition xZrO(2)-(1-x)Fe₂O₃ were prepared by several methods such as high-energy ball milling, co-precipitation and hydrazine methods. This paper presents the effect of the preparation methods and annealing temperatures on the ethanol gas sensitivity. Noble metals such as Pt and Pd were added in order to examine the effects on ethanol gas sensitivity. Sensors based on 1 wt.% Pt + xZrO(2)- (1-x)Fe₂O₃ demonstrated an excellent sensing performance at 230 degreesC for 1000 ppm of ethanol. The gas-sensing behavior of these materials to various reducing gases like CO, CH₄ and H₂ was also studied. This sensor showed good selectivity toward ethanol and thus could effectively be used as a breath sensor.

[51] SI/TIN NANOCOMPOSITE ANODES BY HIGH-ENERGY MECHANICAL MILLING

IS Kim, PN Kumta, GE Blomgren - MATERIALS FOR ELECTROCHEMICAL ENERGY CONVERSION AND STORAGE (Series: CERAMIC TRANSACTIONS), 2002, Vol 127, pp 35-43 - 103RD ANNUAL MEETING OF THE AMERICAN-CERAMIC-SOCIETY; INDIANAPOLIS, INDIANA. APRIL 22-25, 2001

Nanocomposites containing silicon and titanium nitride were synthesized by high-energy mechanical milling (HEMM). The process results in very fine Si particles distributed homogeneously inside the TiN matrix. The Si/TiN nanocomposites synthesized using different experimental conditions were evaluated for their electrochemical properties. Results indicate that Si in the composite alloys and de-alloys with lithium during cycling while TiN remains inactive providing the desired structural stability. The composite containing 33.3 mol% Si obtained after 12 h milling exhibited a capacity of approximate to 300 mAh/g, reflecting its promising nature. Preliminary cycling data show good capacity retention indicative of good phase and micro-structural stability as verified by XRD and SEM analyses.

[50] NEW NANOSTRUCTURED SILICON AND TITANIUM NITRIDE COMPOSITE ANODES FOR LI-ION BATTERIES

IS Kim, PN Kumta, GE Blomgren - MATERIALS FOR ELECTROCHEMICAL ENERGY CONVERSION AND STORAGE (Series: CERAMIC TRANSACTIONS), 2002, Vol 127, pp 249-258- 103RD ANNUAL MEETING OF THE AMERICAN-CERAMIC-SOCIETY; INDIANAPOLIS, INDIANA. APRIL 22-25, 2001

Silicon based nanocomposites containing TiN were synthesized by high-energy mechanical milling (HEMM). Mechanical milling leads to very fine amorphous silicon particles distributed homogeneously inside the TiN matrix. The Si/TiN nanocomposites synthesized using different experimental conditions were evaluated for their electrochemical properties. Results indicate that silicon in the composite alloys and de-alloys with lithium during cycling, while TiN remains inactive providing structural stability. The composite containing 33.3 mol% silicon obtained after milling for 12 h exhibited a high capacity, approximate to 300 mAh/g with good capacity retention reflective of the good phase and micro-structural stability



as verified by XRD and SEM analyses. Conventional and high-resolution electron microscopy coupled with electron energy-loss spectroscopy conducted on the composites validated the existence of amorphous silicon in a nanocrystalline TiN matrix.

[49] NONLINEAR REFRACTION AND NONLINEAR ABSORPTION MEASUREMENTS OF CDTE NANO-SCALE MATERIALS EMBEDDED IN PMMA USING ULTRAFAST LASER QG YANG, JT Seo, SJ Creekmore, G Tan, DA Temple, SS Jung, JH Kim, M Namkung, A Mott - MULTIPHOTON ABSORPTION AND NONLINEAR TRANSMISSION PROCESSES: MATERIALS, THEORY, AND APPLICATIONS (Series: PROCEEDINGS OF THE SOCIETY OF PHOTO-OPTICAL INSTRUMENTATION ENGINEERS (SPIE)), 2003, Vol 4797, pp 125-131 - CONFERENCE ON MULTIPHOTON ABSORPTION AND NONLINEAR TRANSMISSION PROCESSES - MATERIALS, THEORY AND APPLICATIONS; SEATTLE, WASHINGTON. JULY 8-9, 2002

Experimental investigations have shown that CdTe semiconductor microcrystals possess large third-order susceptibilities and short response times at both resonant and non-resonant wavelengths (580 nm and 1064 nm). These excellent properties indicate their potential applications in nonlinear photonic devices. In this work, we measured the nonlinear refraction and nonlinear absorption coefficients of CdTe nanocrystals using Z-scan method at 800 nm. Application in optical limiting of the sample was also demonstrated. The samples used were made by ball milling process and then embedded in polymethylmethacrylate (PMMA). The two photon absorption (TPA) and nonlinear refraction were evaluated from the normalized transmittance with open aperture and with closed-aperture, respectively. Optical limiting studies were carried out as a function of input intensity at 800 nm. The input intensities were varied from 5 to 70 kW/cm². The transmitted power was collected by a photo-detector through a 2-mm diameter aperture. We found that the transmitted power decreased significantly over the input intensity range of 10-20 kW/cm².

[48] COMBUSTION SYNTHESIS OF EU²⁺-ACTIVATED BAMGAL10017 PHOSPHOR

Park S. Kang SH. - Journal of Materials Science-Materials in Electronics. 14(4):223-228, 2003

The combustion synthesis of a Eu²⁺-doped BaMgAl₁₀O₁₇ phosphor was performed using urea and carbonylhydrazide (CH) as combustion fuel. The concentration of activator ion was fixed at 10 mol %, namely Ba_{0.9}Eu_{0.1}MgAl₁₀O₁₇. Powders with an excellent degree of crystallinity were obtained within a few minutes at 500°C when urea was used as the fuel and at 400°C for the case of CH. This as-synthesized phosphor obtained from using CH exhibited a larger surface area, a rougher surface and a higher PL brightness than that using urea. Particle characteristics and luminescent properties varied with the type of fuel and the amount of added NH₄NO₃ addition. However, the combustion reaction led to severe agglomeration of the particles, causing an irregular shape and a broad distribution of particle size regardless of the fuel used. Post-treatment such as ball milling and annealing was found to be beneficial to improving the crystallinity and morphology of the synthesized powder, but at the loss of PL brightness

[47] MOSSBAUER AND X-RAY DIFFRACTION INVESTIGATION OF NANOCRYSTALLINE FE-O ALLOYS

Lileev AS. Yagodkin YD. Reissner M. Steiner W. - JOURNAL OF MAGNETISM AND MAGNETIC MATERIALS. 258(Special Issue SI):504-506, 2003

X-ray and Mossbauer investigations were carried out on powders produced by milling of Fe₂O₃+α-Fe mixtures in a high-energy ball mill and subsequent low-temperature annealing. The nanocrystalline composite alloys obtained as a result of the milling, contained FeO and α-Fe with an average crystallite size of 15-20 nm as well as an amorphous phase. Alloys subjected to subsequent annealing contained, however, only α-Fe+Fe₃O₄ with an average crystallite size of about 20 nm. Unlike the starting materials the produced powders had properties, which are characteristic for hard magnetic materials.

[46] HYSTERESIS MAGNETIC PROPERTIES OF THE FE(100-X)C(X); X=5-25 AT% NANOCOMPOSITES AS-MECHANICALLY ALLOYED AND AFTER ANNEALING

Yelsukov EP. Ul'yanov AI. Zagainov AV. Arsent'yeva NB. - JOURNAL OF MAGNETISM AND MAGNETIC MATERIALS. 258(Special Issue SI):513-515, 2003

The paper presents the results on coercive force and specific saturation magnetization depending on the phase composition and grain size of the Fe-C powders after mechanical alloying and subsequent annealing

[45] SPECIFIC COMPLEXATION OF URSODEOXYCHOLIC ACID WITH GUEST COMPOUNDS INDUCED BY CO-GRINDING. II. EFFECT OF GRINDING TEMPERATURE ON THE MECHANOCHEMICAL COMPLEXATION

Oguchi T. Kazama K. Fukami T. Yonemochi E. Yamamoto K. - BULLETIN OF THE CHEMICAL SOCIETY OF JAPAN. 76(3):515-521, 2003

Ursodeoxycholic acid (UDCA) formed an inclusion complex with phenanthrene or with anthrone when being ground at an ambient temperature, while grinding at lower temperatures did not provide the complex but rather a mixture of amorphous UDCA and the guest compound crystals. Since heat treatment of the sample ground at low temperature did not provide the complex, it was concluded that the interplay of mechanochemical and thermal factors is responsible for the complexation of UDCA with the guest compound.

[44] ATOMISTIC SIMULATION OF STRAIN-INDUCED AMORPHIZATION

Lund AC. Schuh CA. - Applied Physics Letters. 82(13):2017-2019, 2003

The process of solid-state amorphization through extensive plastic straining (i.e., mechanical alloying) is studied through molecular simulation of binary Cu-Zr alloys. Without such confounding factors as impurity pick-up, or such thermal effects as melting or diffusion, we find that amorphization can be driven solely by accumulation of crystal defects and interfacial roughening between the component phases. The tendency for glass formation is also seen to depend on composition, and the results are in line with extensive prior experimental work.

[43] CARBON NANOTUBES FORMED IN GRAPHITE AFTER MECHANICAL GRINDING AND THERMAL ANNEALING



Chen Y. Conway MJ. Fitzgerald JD. - APPLIED PHYSICS A-MATERIALS SCIENCE & PROCESSING. 76(4):633-636, 2003

Multi-walled carbon nanotubes with cylindrical and bamboo-type structures are produced in a graphite sample after mechanical milling at ambient temperature and subsequent thermal annealing up to 1400 degreesC. The ball milling produces a precursor structure and the thermal annealing activates the nanotube growth. Different nanotubular structures indicate different formation mechanisms: multi-wall cylindrical carbon nanotubes are probably formed upon micropores and the bamboo tubes are produced because of the metal catalysts. A two-dimensional growth governed by surface diffusion is believed to be one important factor for the nanotube growth. A potential industrial production method is demonstrated with advantages of large production quantity and low cost

[42] STRUCTURAL AND CALORIMETRIC EVOLUTIONS OF MECHANICALLY-INDUCED SOLID-STATE DEVITRIFICATED ZR60NI25AL15 GLASSY ALLOY POWDER

El-Eskandarany MS. Saida J. Inoue A. - Acta Materialia. 51(5):1481-1492, 2003

A single phase of glassy Zr₆₀Ni₂₅Al₁₅ alloy powder was synthesized by mechanically induced solid-state reaction (MISSR) technique. The MISSR was performed in a room-temperature high-energy ball mill, using a mechanical alloying method. Whereas the glass transition temperature of the obtained glassy alloy is 681 K, the melting and liquidus temperatures are 1179 K and 1256 K, respectively. The mechanically alloyed Zr₆₀Ni₂₅Al₁₅ glassy powders maintain its unique disordered structure through a large supercooled liquid region (99 K). It, however, transforms into a mixture of Zr₅Ni₄Al and Zr₆NiAl₂ crystalline phases at 780 K with a large enthalpy change of crystallization of -81.8 J/g. The possibility of devitrification of the synthetic glassy phase upon increasing the ball milling time was investigated. The results have shown that the glassy powder obtained after 173 ks of milling is subject to heavy lattice imperfections and tends to transform into a metastable-big cube phase after further ball milling times (216-259 ks). After 446 ks of milling, a complete glassy-metastable phase transformation is achieved and the end-product of this stage of milling is nanocrystalline big-cube powders which have a lattice constant of 1.2282 nm. The big-cube Zr(60)Ni(25)Al(15) phase transforms into the same crystalline mixture of Zr₅Ni₄Al and Zr₆NiAl₂ phases at 799 K with an enthalpy change of transformation of -73.7 J/g. As the milling time increases (720 ks), the obtained big-cube phase can no longer withstand against the shear and impact stresses that are generated by the milling media and surprisingly transformed into a new metastable phase of nanocrystalline fcc-Zr₆₀Ni₂₅Al₁₅. The lattice constant of this metastable phase was calculated and found to be 0.45449 nm. The fcc-metastable phase transforms into a mixture of Zr₅Ni₄Al and Zr₆NiAl₂ crystalline phases at rather a high temperature, as high as 901 K with a heat change of transformation of -24.4 J/g. The reported metastable phase here is new and has never been, so far as we know, reported for ternary Zr-Ni-Al system, or its binary phase relations.

[41] LOWERING THE SYNTHESIS TEMPERATURE OF HIGH-PURITY BaTiO₃ POWDERS BY MODIFICATIONS IN THE PROCESSING CONDITIONS

Brzozowski E. Castro MS. - Thermochemica Acta. 398(1-2):123-129, 2003

In this work, the influence of the BaCO₃:TiO₂ ratio and milling conditions on the solid-state barium titanate formation at high temperature has been studied. An excess of TiO₂ improved the BaTiO₃ formation when the TiO₂ reactivity was low. Besides, long milling time and an excess of fine grained titanium led to agglomerate formation. In addition, mechanochemical activation performed by rigorous milling and the employment of fine particles of anatase-rich TiO₂ powder allowed the reaction to go to completions at temperatures about 150 degreesC and below than traditionally need

[40] THERMAL BEHAVIORS OF MECHANICALLY ACTIVATED PYRITES BY THERMOGRAVIMETRY (TG)

Hu HP. Chen QY. Yin ZL. Zhang PM. - Thermochemica Acta. 398(1-2):233-240, 2003

The thermal decompositions of mechanically activated and non-activated pyrites were studied by thermogravimetry (TG) at the heating rate of 10 K min⁻¹ in argon. Results indicate that the initial temperature of thermal decomposition (T-di) in TG curves for mechanically activated pyrites decreases gradually with increasing the grinding time. The specific granulometric surface area (S-G), the structural disorder of mechanically activated pyrites were analyzed by X-ray diffraction laser particle size analyzer, and X-ray powder diffraction analysis (XRD), respectively. The results show that the S-G of mechanically activated pyrites remains almost constant after a certain grinding time, and lattice distortions (epsilon) rise but the crystallite sizes (D) decrease with increasing the grinding time. All these results imply that the decrease of T-di in TG curves of mechanically activated pyrites is mainly caused by the increase of lattice distortions e and the decrease of the crystallite sizes D of mechanically activated pyrite with increasing the grinding time. The differences in the reactivity between non-activated and mechanically activated pyrites were observed using characterization of the products obtained from 1 h treatment of non-activated and mechanically activated pyrites at 713 K under inert atmosphere and characterization of non-activated and mechanically activated pyrites exposed to ambient air for a certain period

[39] MECHANOCHEMICAL SYNTHESSES OF ANTIMONY SELENIDE, TIN SELENIDES AND TWO TIN ANTIMONY SELENIDES

Shen J. Blachnik R. - Thermochemica Acta. 399(1-2):245-246, 2003

Sb₂Se₃, SnSe, SnSe₂, SnSb₂Se₄, and Sn₂Sb₆Se₁₁ were prepared from powdered elements in a planetary ball mill. The progress of the reactions was investigated in situ by difference thermoanalysis and ex situ by X-ray analyses. SnSb₂Se₄, and Sn₂Sb₆Se₁₁ are probably both positioned in the homogeneity region of the only ternary compound of the system Sn-Sb-Se.

[38] SYNTHESIS OF FE-FeAl₂O₄-Al₂O₃ BY HIGH-ENERGY BALL MILLING OF AL-Fe₃O₄ MIXTURES

Botta PM. Mercader RC. Aglietti EF. Lopez JMP. - Scripta Materialia. 48(8):1093-1098, 2003



Physicochemical and structural changes induced by mechanical activation of Al-Fe₃O₄ mixtures are studied. After 37 min, the system undergoes a self-sustained reaction with formation of alpha-Fe, FeAl₂O₄ and alpha-Al₂O₃. The evolution of the composition follows a three-step reaction. The solid solution spinel Fe[Al_{2-x}Fe_x]O₄, (0.13 < x < 0.29), is obtained through a mechanochemical-thermal route

[37] EVOLUTION OF GRAIN BOUNDARY ASSEMBLIES IN FE-0.6%O UNDER MECHANICAL MILLING FOLLOWED BY CONSOLIDATING ROLLING

Belyakov A. Sakai Y. Hara T. Kimura Y. Tsuzaki K. - Scripta Materialia. 48(8):1111-1116, 2003

Microstructure evolution was studied in Fe-0.6%O processed by mechanical milling followed by consolidating warm bar rolling. Depending on the milling time, the fraction of high-angle grain boundaries that evolved in the final bulk material can decrease or, alternatively, increase by the consolidating working.

[36] MAGNETISM OF THE NANOSTRUCTURED SPINEL ZINC FERRITE

Ehrhardt H. Campbell SJ. Hofmann M. - Scripta Materialia. 48(8):1141-1146, 2003

Nanostructured zinc ferrite has been prepared by mechanical milling. The changes in the microstructure indicate a decrease in particle size and a simultaneous increase in the inversion parameter. Along with the structural changes, a magnetic transformation from the antiferromagnetic phase to a ferrimagnetic-like behaviour is observed by neutron diffraction.

[35] MECHANOCHEMICAL FORMATION OF NOVEL CATALYST FOR PREPARING CARBON NANOTUBES: NANOCRYSTALLINE YTTRIUM ALUMINUM IRON PEROVSKITE

Guo XM. Qi JF. Sakurai K. - Scripta Materialia. 48(8):1185-1188, 2003

The first mechanochemical synthesis of nanosized YAl_{1-x}Fe_xO₃ (X = 0 to 1) perovskite powders is reported. Continuous ball-milling of the yttria, aluminum hydroxide and iron oxide powders over 24 h led to the formation of nanocrystals with a size of nearly 20 nm. It has been found that the material exhibits a catalytic property for preparing carbon nanotubes.

[34] DEFORMATION-INDUCED NANOCRYSTALLIZATION IN AN AL-BASED AMORPHOUS ALLOY AT A SUBAMBIENT TEMPERATURE

Jiang WH. Pinkerton FE. Atzmon M. - Scripta Materialia. 48(8):1195-1200, 2003

The compressive region of amorphous Al₉₀Fe₅Gd₅, bent at -40 degreesC, was investigated by transmission electron microscopy. A high density of nanocrystals is observed within shear bands. Severe plastic deformation and precipitation of nanocrystallites are observed at the fracture,surface. It is argued that deformation-assisted atomic transport leads to nanocrystallization.

[33] HYDROGEN STORAGE PROPERTIES IN NANO-STRUCTURED MAGNESIUM- AND CARBON-RELATED MATERIALS

Fujii H. Orimo S. - Physica B: Condensed Matter. 328(1-2):77-80, 2003

We review hydrogen-storage properties of two nano-structured systems; multi-layered Pd/Mg films prepared by the RF sputtering method and mechanically milled graphite under a high hydrogen atmosphere. The former prepared under optimized RF sputtering conditions absorbs hydrogen of similar to 5 mass% at 373 K under 0.1 MPa hydrogen pressure, and desorbs all the hydrogen at 360 K in vacuum. The latter prepared by milling for 80 h absorbs hydrogen up to 7.4 mass%. Hydrogen molecules are desorbed by the two processes above at 600 and 950 K, respectively

[32] MICROSTRUCTURAL ASPECTS OF THE HCP-FCC ALLOTROPIC PHASE TRANSFORMATION INDUCED IN COBALT BY BALL MILLING

Sort J. Nogues J. Surinach S. Baro MD. - Philosophical Magazine. 83(4):439-455, 2003

A detailed correlation between microstructure evolution and allotropic phase transformations occurring in Co when subjected to ball milling has been carried out. After short-term milling, the starting mixture of hcp + fcc Co develops into an almost pure hcp phase. However, for longer milling times, plastic deformation introduces large amounts of stacking faults, especially of twin type, in the hcp structure. As a consequence, some of the hcp Co is converted back into fcc and the hcp unit cell is progressively anisotropically distorted. After long-term milling, a steady 'pseudo-equilibrium' state is observed, where all microstructural parameters, including the fcc percentage, tend to level off. However, the milling intensity can still be adjusted to increase further the stacking-fault probability and, consequently, the amount of fcc Co in the milled powders. The results imply that the stacking-fault formation, rather than the local temperature rise or crystallite size reduction associated with the milling process, is the main mechanism governing the hcp-fcc transformation

[31] RIETVELD ANALYSIS OF POLYMORPHIC TRANSFORMATIONS OF BALL MILLED ANATASE TiO₂

Bose P. Pradhan SK. Sen S. - MATERIALS CHEMISTRY AND PHYSICS. 80(1):73-81, 2003

Rietveld's whole powder profile fitting method based on crystal structure refinement is applied to extract the microstructure information of several polymorphic TiO₂ phases grown simultaneously during high energy vibratory ball milling of anatase phase. Warren-Averbach's method of X-ray line profile analysis is also made for comparison of above results. The X-ray powder patterns of four TiO₂ phases along with alpha-Al₂O₃ (contamination) are simulated simultaneously for the first time to fit the experimental X-ray powder patterns of multiphase materials. The structural details of monoclinic TiO₂ (B) phase are also reported first time in this paper. The advantages of the present method of analysis over the other methods of X-ray line profile analysis are discussed in details.

[30] FABRICATION AND MICROSTRUCTURE CHARACTERIZATION OF Ti₃SiC₂ SYNTHESIZED FROM Ti/Si/TiC POWDERS USING THE PULSE DISCHARGE SINTERING (PDS) TECHNIQUE

Zhang ZF. Sun ZM. Hashimoto H. Abe T. - JOURNAL OF THE AMERICAN CERAMIC SOCIETY. 86(3):431-436, 2003



Ti/Si/2TiC powders were prepared using a mixture method (M) and a mechanical alloying (MA) method to fabricate Ti₃SiC₂ at 1200degrees-1400degreesC using a pulse discharge sintering (PDS) technique. The results showed that the Ti₃SiC₂ samples with <5 wt% TiC could be rapidly synthesized from the M powders; however, the TiC content was always >18 wt% in the MA samples. Further sintering of the M powder showed that the purity of Ti₃SiC₂ could be improved to >97 wt% at 1250degrees-1300degreesC, which is similar to 200degrees-300degreesC lower than that of sintered Ti/Si/C and Ti/SiC/C powders using the hot isostatic pressing (HIPing) technique. The microstructure of Ti₃SiC₂ also could be controlled using three types of powders, i.e., fine, coarse, or duplex-grained, within the sintering temperature range. In comparison with Ti/Si/C and Ti/SiC/C mixture powders, it has been suggested that high-purity Ti₃SiC₂ could be rapidly synthesized by sintering the Ti/Si/TiC powder mixture at relatively lower temperature using the PDS technique

[29] MICROSTRUCTURE AND CHEMICAL STATES OF HYDROXYAPATITE/SILK FIBROIN NANOCOMPOSITES SYNTHESIZED VIA A WET-MECHANOCHEMICAL ROUTE

Wang L. Nemoto R. Senna M. - Journal of Nanoparticle Research. 4(6):535-540, 2002

Hydroxyapatite (HAp)/silk fibroin (SF) nanocomposites were prepared via a wet-mechanochemical route at room temperature. The results reveal that the inorganic phase in the composites is carbonate-substituted HAp containing 2.9-3.1 wt% of carbonate ions. The primary HAp crystals are rod-like in shape with a typical size of 20-30 nm in length and 8-10 nm in width, and lattice parameters $a = 9.423$ Angstrom, $c = 6.888$ Angstrom. The self-assembled HAp crystals along their c-axes aggregate into bundles, which are connected with SF fibrils. Consequently, a three-dimensional porous network is formed in the composite, which is beneficial to inducing new bone formation in practical implantation

[28] ENHANCED MAGNETISATION IN NANOCRYSTALLINE HIGH-ENERGY MILLED MGFE2O4

Sepelak V. Baabe D. Mienert D. Litterst FJ. Becker KD. - Scripta Materialia. 48(7):961-966, 2003

The changes in magnesium ferrite (MgFe₂O₄) caused by high-energy milling are investigated by means of Mossbauer spectroscopy, magnetisation measurements, and electron microscopy. The observed enhancement of the magnetisation in nanoscale milled MgFe₂O₄ is discussed with respect to the mechanically induced cation redistribution and spin canting.

[27] MIXING AT MICROMETRIC AND NANOMETRIC SCALE IN MECHANICALLY ALLOYED FE60CR40

Fnidiki A. Lemoine C. Teillet J. Malandain JJ. - Physica B: Condensed Matter. 327(2-4):140-143, 2003

Mechanical alloying (MA) of iron and chromium powder mixtures was performed from 0 to 190h. SEM, transmission electron microscopy with energy-dispersive X-ray analyses and Mossbauer spectrometry were used to study the mixing at micrometric and nanometric scale. At the stationary mixing state (approximate to 20h of milling), MA of Fe₆₀Cr₄₀ powder mixtures gives particles of several micrometers consisting of magnetic nanograin cores characteristic of crystalline Fe₆₀Cr₄₀ alloy surrounded by a disordered paramagnetic grain surface (at 300 K), which increases during milling. At 85 h, the nanograins are totally disordered. For a longer time of milling, a de-mixing occurs by partial recrystallization of this disordered structure

[26] STUDIES ON AB(5) METAL HYDRIDE ALLOYS WITH CO ADDITIVE

Hang BT. Tai LT. Que LX. Hanh MD. Thuy NP. Hien TD. Thanh LTH. - Physica B: Condensed Matter. 327(2-4):378-381, 2003

Some effects of Co additive. on the magnetic and electrochemical properties of the alloy series La_{0.8}Nd_{0.2}Ni_{4.9-x}Co_xSi_{0.1} ($x = 0.1, 0.5, 0.75, 1$ and 1.5) have been studied. The results of magnetic measurements indicate that the susceptibility (χ) and the Curie-temperature (T_c) of the samples increases with Co addition. The milling and the charge-discharge process change the magnetic properties of the as-prepared samples. Electrochemical measurements show that small additions of Co improve the performance of metal hydride alloy electrodes as charge transfer facilities.

[25] MECHANOCHEMICAL PROCESSING OF SIALON COMPOSITIONS

MacKenzie KJD. Temuujin J. Smith ME. Okada K. Kameshima Y. - JOURNAL OF THE EUROPEAN CERAMIC SOCIETY. 23(7):1069-1082,

Milling for 48 h in a sealed planetary ball mill was found to facilitate sialon formation in mixtures of aluminium and silicon oxides and nitrides on further heating in nitrogen at 1200-1600 degreesC. Al-27 and Si-29 MAS NMR indicated that milling exerts more influence on the oxide components than on the nitrides. Substitution of gamma-Al₂O₃ by Al(OH)₃ as the alumina source facilitated the mechanochemical formation of Si-O-Al bonds, as monitored by MAS NMR, but the subsequent thermal conversion of these precursors to stable aluminosilicates did not enhance sialon formation. Milled precursors of beta-sialon composition formed a mixture of beta, O and X-sialon, decomposing to polytypoid sialons at 1600 degreesC. Milled O-sialon precursors formed monophase O-sialon at 1600 degreesC, while some X-sialon compositions formed X-sialon at 1400 degreesC which decomposed to mullite and corundum at 1600 degreesC. A ground mixture of Ca alpha-sialon composition formed beta, O and X-sialons at 1600 degreesC, but no Ca sialon because of the depletion of Ca by the preferential formation of a Ca feldspar. Without milling, all these mixtures were unreactive, and generally did not form sialons on heating in nitrogen up to 1600 degreesC.

[24] EFFECT OF GRINDING ON THE LEACHING BEHAVIOUR OF PYROPHYLLITE

Temuujin J. Okada K. Jadambaa TS. MacKenzie KJD. Arnarsanaa J. - JOURNAL OF THE EUROPEAN CERAMIC SOCIETY. 23(8):1277-1282, 2003

The effect of dry grinding on the leaching behaviour of pyrophyllite has been studied by XRD, FT-IR, thermal analysis and N-2 adsorption techniques. Pyrophyllite samples were ground for 3, 6, 12 and 18 h. Grinding initially causes the surface area of the pyrophyllite to increase to similar to 73 m² g⁻¹, decreasing with longer grinding because of particle agglomeration. Leaching the ground pyrophyllite with 4 M HCl at 80 degreesC for 2 h causes depletion of Al³⁺ and increases the surface



area. The highest surface area was recorded in the leached sample ground for 3 h (156.9 m² g⁻¹) with a pore volume of 0.36 ml g). Structural breakdown by grinding is thought to be the main reason for the enhanced leaching behaviour of the pyrophyllite, but grinding for extended periods decreases the leaching rate without enhancement of the porous properties.

[23] MAGNETIC PROPERTIES OF SM-Fe(Ti)-C(N)/ALPHA-Fe ALLOYS PREPARED BY MECHANICAL ALLOYING

Geng DY. Zhang ZD. Liu W. Zhao XG. Yu MH. Ren WJ. Xiao QF. Grossinger R. Hauser R. - Journal of Physics D-Applied Physics. 36(4):375-379, 2003

Magnetic properties of Sm-Fe(Ti)-C(N)/alpha-Fe nanocomposite alloys prepared by mechanical alloying (MA) have been studied. The hysteresis loops of MA Sm₁₂Fe₇₄Ti₇C₇N_{delta}, Sm₁₀Fe₇₆Ti₇C₇N_{delta} and Sm_{19.0}Fe_{66.7}C_{14.3} (i.e. Sm₂₀Fe₇₀C₁₅) made by different processes (annealing, nitriding and re-milling) were measured at the applied magnetic fields with different dH/dt at 400, 350 and 295 K. The main phase with hard magnetic properties is of TbCu₇-type. These three alloys have different behaviours of magnetization processes when the magnetic fields with dH/dt = 31.6 and 94.7 MA ms⁻¹ are applied. The magnetic viscosity S-v of Sm₁₂Fe₇₄Ti₇C₇N_{delta}, Sm₁₀Fe₇₆Ti₇C₇N_{delta} and Sm₂₀Fe₇₀C₁₅ is determined from hysteresis loops measured for different dH/dt at various temperatures. The magnetic viscosity S-v in the Sm-Fe(Ti)-C(N)/alpha-Fe alloys is approximately proportional to the coercive field iH(c) at the measuring temperature.

[22] SYNTHESIS OF MG-TI ALLOY BY MECHANICAL ALLOYING

Liang G. Schulz R. - Journal of Materials Science. 38(6):1179-1184, 2003

Mg-based Mg-Ti binary alloys have been synthesized by mechanical alloying of Mg and Ti powder blends. It was found that mechanical alloying of Mg and Ti results in a nanocrystalline Mg-Ti alloy and an extended solubility of Ti in Mg, due to the favorable size factor and the isomorphous structure of Mg and Ti. In the case of Mg-20at.% Ti, about 12.5% Ti is dissolved in the Mg lattice when the mechanical alloying process reaches a stable state. The rest (about 7.5 at.%) remains as fine particles in the size of 50 - 150 nm in diameter. Dissolution of 12.5 at.% Ti in the Mg lattice causes a contraction of the unit cell volume from 0.0464 to 0.0442 nm³ and a decrease of the c/a ratio from 1.624 to 1.612 of the hexagonal structure. The supersaturated solid solution Mg-Ti alloy decomposes upon thermal annealing at temperatures above 200 degreesC. Hydrogenation enhances the decomposition process at lower temperatures

[21] PHASE TRANSFORMATION KINETIC STUDY AND MICROSTRUCTURE CHARACTERIZATION OF BALL-MILLED M-ZrO₂-10 MOL% A-TiO₂ BY RIETVELD METHOD

Dutta H. Manik SK. Pradhan SK. - Journal of Applied Crystallography. 36(Part 2):260-268, 2003

High-energy ball milling of a monoclinic ZrO₂-10 mol% anatase TiO₂ mixture results in the formation of a nanocrystalline cubic ZrO₂ polymorphic phase with equimolar fraction of the starting materials. The cubic phase is presumed to have formed from the m-ZrO₂ solid solution based on the (001) plane of the m-ZrO₂ phase. In the course of milling, the most dense (111) plane of the cubic lattice became parallel to the most dense ((1 over bar 11) plane of the monoclinic lattice due to an orientation effect. Annealing of a 12 h milled sample at 773, 873 and 973 K for 1 h results in almost complete transformation of the m-ZrO₂ to the c-ZrO₂ phase. At 1273 K annealing temperature (1 h), the nanocrystalline sample decomposed into individual starting phases. This suggests that the cubic phase is a metastable one and its stability depends on particle size as well as the working temperature. Formation of the cubic phase at such a low temperature using anatase TiO₂ as a phase stabilizer has not been reported previously. The microstructures of the unmilled, all the ball-milled and the annealed samples have been characterized by employing Rietveld's X-ray powder structure refinement methodology. The particle size, root mean square (r.m.s.) lattice strain, lattice parameters, molar fraction, etc., of individual phases have been estimated from Rietveld analysis and are utilized to interpret the results

[20] REPEATABLE HYDROGEN ADSORPTION USING NANOSTRUCTURED GRAPHITE AT ROOM TEMPERATURE

Kajiura H. Kadono K. Tsutsui S. Murakami Y. - Applied Physics Letters. 82(12):1929-1931, 2003

Repeatable hydrogen adsorption and desorption by nanostructured graphite was confirmed using a high-accuracy volumetric measuring apparatus at room temperature. The nanostructured graphite was prepared from graphite powder using a mechanical milling process at a pressure of 2.0x10⁻⁴ Pa. The untreated graphite adsorbed 0.02 wt % of hydrogen, while 0.20-0.25 wt % of hydrogen can be repeatedly adsorbed by the nanostructured graphite. Measurements of the hydrogen adsorption rate at constant pressure and pore-size distribution suggest that the hydrogen molecules are adsorbed through a diffusion process into pores with a diameter less than 1 nm.

[19] MICROSTRUCTURE, HARDNESS AND INDENTATION TOUGHNESS OF C40MO(SI,AL)(2)/ZRO2 COMPOSITES PREPARED BY SPS OF MA POWDERS

Krakhmalev PV. Strom E. Sundberg M. Li C. - Scripta Materialia. 48(6):725-729, 2003

The nano- and micron-scale composite structures, Mo/Mo₅Si₃, Al₂O₃ and Mo_{0.34}Zr_{0.20}Si_{0.46} phases have been observed in spark plasma sintered (SPS) C40 Mo(Si,Al)(2)/ZrO₂ materials. The hardness of C40 Mo(Si,Al)(2)/ZrO₂ composites is around 14 GPa. The indentation toughness lies in 2.69-2.94 MPa m^{1/2} range that is approximately 50% higher than toughness of C40 Mo(Si,Al)(2) phase

[18] INCORPORATING MG INTO THE SI SUB-LATTICE OF MOLYBDENUM DISILICIDE

Woolman JN. Petrovic JJ. Munir ZA. - Scripta Materialia. 48(6):819-824, 2003

Theoretical analysis has identified Mg as the most promising element for this purpose, but its incorporation into MoSi₂ has not been achieved. Through a combination of mechanical milling and field-activated synthesis, magnesium was successfully incorporated into the silicon sub-lattice of this silicide.

[17] HOMOGENEOUS DISPERSION OF GRAPHITE IN A 6061 ALUMINUM ALLOY BY BALL MILLING



Son HT. Kim TS. Suryanarayana C. Chun BS. - MATERIALS SCIENCE AND ENGINEERING A-STRUCTURAL MATERIALS PROPERTIES MICROSTRUCTURE AND PROCESSING. 348(1-2):163-169, 2003

A composite of rapidly solidified Al-6061 alloy powder with graphite particle reinforcements was prepared by ball milling and subsequent hot extrusion. Proper choice of the processing parameters ensured a homogeneous distribution of the graphite particles in the aluminum alloy matrix. The microstructure and mechanical properties of these composites were investigated as a function of milling time. With increasing milling time, the spherical powder became elongated and acicular, which subsequently became spherical with a lamellar structure inside it. The shape change and intermixing process of the two constituent powders, and consequently the microstructure of the milled powder and extruded bars, were dependent on the milling time. The best compression and wear properties were obtained in the powder milled for 70 h, associated with the increased fine and homogeneous distribution of graphite particles in the aluminum alloy matrix.

[16] MECHANO-CHEMICAL SYNTHESIS OF AMORPHOUS SOLIDS IN THE SYSTEM AgI-AG₂PO_{3.5} AND THEIR SILVER ION-CONDUCTING PROPERTIES

Peng HF. Machida N. Nishida S. Shigematsu T. - Journal of Non-Crystalline Solids. 318(1-2):112-120, 2003

Amorphous fine powders in the system AgI-Ag₂PO_{3.5} were obtained over a wide composition range of 20-70 mol% AgI by a high-energy ball-milling process. While an amorphous sample was not obtained of the composition 80AgI . 20Ag(2)PO(3.5) (mol%) by the ball-milling process, the amorphous sample-forming region by the high-energy ball-milling process was very similar to the glass-forming region by a conventional melt-quenching method. The 60AgI . 40Ag(2)PO(3),5 (mol%) composition, at which an amorphous sample was obtained by ball milling to 48 h, was the most easily amorphous-forming composition in the system AgI-Ag₂PO_{3.5}. Ball-milled samples showed high ion conductivities at room temperature. The σ_{298} of the 60AgI . 40Ag(2)PO(3.5) sample changed with the ball-milling time. The σ_{298} steeply increased with an increase in the milling time and showed a maximum of $7.3 \times 10^{-1} \text{ Sm}^{-1}$ at around 20 h. It was noted that the maximum conductivity was higher than the conductivity of the melt-quenched glass with the same chemical composition. The σ_{298} of the samples ball-milled for longer than 30 h decreased with an increase in the milling time, and had a similar value as that of a melt-quenched glass.

[15] EXAFS, X-RAY DIFFRACTION AND MOSSBAUER STUDIES OF AN AMORPHOUS Fe₆₀Ti₄₀ ALLOY PRODUCED BY MECHANICAL ALLOYING

de Lima JC. Machado KD. Drago V. Grandi TA. de Campos CEM. Triches DM. - Journal of Non-Crystalline Solids. 318(1-2):121-130, 2003

The local atomic structure of an amorphous Fe₆₀Ti₄₀ alloy produced by mechanical alloying was investigated using the extended X-ray absorption fine structure (EXAFS), X-ray diffraction (XRD) and Mossbauer spectroscopy techniques. From the EXAFS spectra at the Fe and Ti K-edges, the radial structure functions were obtained and the EXAFS $\chi(k)$ near neighbor oscillations were isolated. By fitting these functions with a least-squares method the coordination numbers and interatomic distances were determined. Independently, the total structure factor $S(K)$ for this alloy was deduced from the XRD measurements, and, by Fourier transforming, the total radial distribution function RDF(r) was obtained. The theoretical total structure factor $S^*(K)$ predicted by the additive hard-sphere model for a mixture of Fe and Ti atoms with the same composition was calculated. There was a strong resemblance between the amorphous structure investigated and that predicted by this model, specially for the Fe-Fe and Fe-Ti pairs. A comparison between the local atomic structure found in the amorphous Fe₆₀Ti₄₀ alloy and in the FeTi compound was made

[14] EFFECTS OF MECHANICAL ACTIVATION ON THE FORMATION OF PbTiO₃ FROM AMORPHOUS Pb-Ti-O PRECURSOR

Yu T. Shen ZX. Xue JM. Wang J. - Journal of Applied Physics. 93(6):3470-3474, 2003

We investigate the effects of mechanical activation in triggering PbTiO₃ (PT) formation from an amorphous Pb-Ti-O precursor synthesized by coprecipitation. In this work, the amorphous precursor and the samples derived from it by mechanical activation were investigated using Raman spectroscopy, x-ray diffraction, and high-resolution transmission electron microscopy. Our results show that the crystallization of tetragonal PT phase can be considered as a nucleation and growth process described by the Avrami model. In the initial stage of mechanical activation, the main effect is size reduction of the constituent starting materials. For a longer period of milling, perovskite PT crystallites are formed by mechanical activation alone. These crystallites act as seeds, reducing the activation energy from 249 \pm 6 kJ/mol for the precursor to 97 \pm 7 kJ/mol for the 30-h-milled sample and enhances the crystallization kinetics, during postcalcination. Consequently, the PT phase formation temperature is dramatically lowered. In addition, our results demonstrate that the particle size affects the structure of the PT phase, where the PT shows the pseudocubic to tetragonal transition with increasing particle size

[13] PREPARATION AND CHARACTERIZATION OF TiO₂ AND V₂O₅ NANOPARTICLES PRODUCED BY BALL-MILLING

Guimaraes JL. Abbate M. Betim SB. Alves MCM. - JOURNAL OF ALLOYS AND COMPOUNDS. 352(1-2):16-20, 2003

We studied the crystalline and electronic structure of TiO₂ and V₂O₅ nanoparticles produced by ball-milling. The experimental techniques used here were X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS). The XRD diagrams show that ball-milling preserves the crystalline structure of the bulk TiO₂ and V₂O₅ materials. The particle size estimated with the Scherrer formula after 24 h of ball-milling is 6 nm for TiO₂ and 13 nm for V₂O₅. The XPS spectra exclude the possibility of impurity contamination and confirm the composition of the nanoparticles. A chemical shift in the Ti 2p XPS spectrum indicates a slight reduction of the Ti ions in the TiO, nanoparticles. The analysis of the Ti 2p satellites shows that the O 2p-Ti 3d covalence decreases in the TiO₂ nanoparticles. These effects are attributed to a reduced O



coordination of the Ti ions at the surface of the TiO₂ nanoparticles. The effects are less evident in the case of the larger V₂O₅ nanoparticles because the surface/volume ratio is smaller

[12] SYNTHESIS AND MAGNETIC PROPERTIES OF Ni₃Fe INTERMETALLIC COMPOUND OBTAINED BY MECHANICAL ALLOYING

Chicinas I. Pop V. Isnard O. Le Breton JM. Juraszek J. - JOURNAL OF ALLOYS AND COMPOUNDS. 352(1-2):34-40, 2003

Nanocrystalline Ni₃Fe intermetallic compound has been obtained by milling of iron and nickel powders under argon atmosphere. Several milling times have been used ranging from 1 h up to 52 h. The formation of the Ni₃Fe intermetallic compound by mechanical alloying was evidenced by X-ray diffraction and Fe-57 Mossbauer spectroscopy. A heat treatment of 1 h at 330°C relaxes the first and second order internal stresses and favours the solid-state reaction. The reaction becomes significant after 4 h of milling and subsequent heat treatment. A typical particle size of 12.5 ± 2.0 nm has been obtained after 52 h. Scanning electron microscopy and X-ray microanalysis studies show the evolution of the chemical homogeneity of the particles and reveal the morphology upon Ni₃Fe phase formation. Magnetic measurements have been performed in a magnetic field up to 8 Tesla between 4 and 300 K. The influence of the synthesis conditions on the magnetic properties of Ni₃Fe is discussed.

[11] COMPOSITIONAL EFFECTS ON THE MECHANOCHEMICAL SYNTHESIS OF Fe-Ti AND Cu-Ti AMORPHOUS ALLOYS BY MECHANICAL ALLOYING

Delogu F. Cocco G. - JOURNAL OF ALLOYS AND COMPOUNDS. 352(1-2):92-98, 2003

It is well-known that the mechanochemical processing of binary mixtures of transition metals can induce the formation of nanostructured and amorphous phases. Previous kinetic studies have shown that the phase transformation in binary mixtures follows sigmoidal conversion curves irrespective of the intensity of mechanical processing. The rate of such transformations is instead strongly influenced by both the intensity of the mechanical treatment and the physical properties of the reactant powders. In the present work, we give additional information on the amorphisation kinetics of binary mixtures by exploring the effect of composition on the transformation kinetics. Cu-Ti and Fe-Ti mixtures with composition within the amorphisation ranges have been subjected to mechanical processing at fixed intensity. The kinetics of phase transformation has been followed by quantitative X-ray diffraction. The experimental data obtained demonstrate that the amorphisation rate has a strong dependence on the product of the mass fractions of the single reactants

[10] THE CRYSTALLIZATION OF AMORPHOUS Fe₂MnGe POWDER PREPARED BY BALL MILLING

Zhang L. Bruck E. Tegus O. Buschow KHJ. de Boer FR. - JOURNAL OF ALLOYS AND COMPOUNDS. 352(1-2):99-102, 2003

We synthesized for the first time the intermetallic compound Fe₂MnGe. To avoid preferential evaporation of volatile components we exploited mechanical alloying. Amorphous Fe₂MnGe alloy powder was prepared by planetary ball milling elemental starting materials. The amorphous-to-crystalline transition was studied by means of differential scanning calorimetry (DSC) and X-ray diffraction (XRD). A cubic D0(3) phase is formed at low temperature and transforms to a high-temperature hexagonal D0(19) phase. The apparent activation energy was determined by means of the Kissinger method.

[9] PROCESSING AND FRACTURE TOUGHNESS OF NANO-SIZED Cu-DISPERSED Al₂O₃ COMPOSITES

Min KH. Oh ST. Do Kim Y. Moon IH. - JOURNAL OF ALLOYS AND COMPOUNDS. 352(1-2):163-167, 2003

An optimum route for the fabrication of Al₂O₃/Cu nanocomposites with sound microstructure and enhanced fracture toughness was investigated. Cu-dispersed Al₂O₃ nanocomposites were obtained via hydrogen reduction and the pulse electric current sintering (PECS) of ball-milled Al₂O₃ and CuO powder mixtures. Microstructural observation of the reduced powder revealed that Cu particles of about 30 nm size homogeneously surrounded the Al₂O₃ powder. The nanocomposites, consolidated by PECS at 1250 °C for 5 min, exhibited full density and enhanced fracture toughness compared with monolithic Al₂O₃.

[8] TEXTURE ANALYSIS OF OXIDE DISPERSION STRENGTHENED (ODS) Fe ALLOYS BY X-RAY AND NEUTRON DIFFRACTION

Bechade JL. Mathon MH. Branger V. Regle H. Alamo A. - Journal de Physique IV. 12(PR6):155-163, 2002

The ferritic ODS alloys studied were obtained by mechanical alloying. This strengthening method is very attractive, in particular for nuclear applications. In order to ensure the alloy a good compromise between mechanical resistance and ductility at high temperatures, it is necessary to control the microstructure and in particular the evolution during the recrystallization. First, a preliminary study, performed by X ray diffraction and optical microscopy, shows several grain growth mechanisms; in particular, the "abnormal" grain growth mechanism which conducts to a large grain size [1], [2]. After annealing (3600s at 1470°C), the 30% cold-worked (swaging) alloys exhibit an heterogeneous microstructure with a large grains size (similar to 200 to 500 μm) in the heart and near the surface of the material when the intermediate zone is inhabited by small gains (similar to 1 μm). For a higher cold-work level (60%), large size gains are only present in the periphery of the material. On account of the large grain size and strong heterogeneity of the microstructure, texture analysis using laboratory x-ray beam is not well adapted and so we have decided to use neutron beam. The neutron diffraction texture analysis has been performed at the Laboratoire Leon Brillouin on the 6T1 diffractometer on 2 different rods of the alloy (corresponding to the reduction ratios of 30% and 60%). Specific samples have been machined to characterise separately the zones with a different microstructure. After deformation, the alloys exhibit a typical alpha-fibre texture (hkl) <110> whatever the area of the sample and the reduction ratio. After recrystallization, a very inhomogeneous texture is evidenced through the thickness of the sample, in particular for the rod deformed with a reduction ratio of 30% : in



the heart and in the periphery of the rod, a "single-crystal" type texture is observed; the alpha fibre remains for the intermediate diameter of the rod. For the rod cold rolled with a reduction ratio of 60%, the alpha-fibre keeps on the heart of the material and as in the precedent case, a "single-crystal" type texture is observed near the surface of the sample. EBSD measurements have been performed to explicit at a local scale this behaviour and in order to furnish some input data for the simulation of static recrystallization

[7] NANOMATERIALS: A DIFFICULT ANALYSIS [FRENCH]

Begin-Colin S. Le Caer G. Girot T. - Journal de Physique IV. 12(PR6):441-454, 2002

The field of nanomaterials is of widespread interest since the beginning of the nineties. We describe several methods of synthesis of nanophased materials and of nanostructured materials. The problems related to the characterization of nanosized particles and to the influence of the synthesis method on their properties are illustrated first from a comparison of nanometric titanoferrites synthesized by two different methods, then by a phase transformation from face-centered cubic to hexagonal which takes place in Ni at a nanometer crystallite size and finally by the Rietveld analysis of phases found in TiO₂ powders ground for short times.

[6] CORRELATION BETWEEN BALL MILLING CONDITIONS AND PLANAR EFFECTS ON CU-NANOSTRUCTURED POWDERS [FRENCH]

Bernard F. Gaffet E. Champion Y. Budarina N. Ustinov A. - Journal de Physique IV. 12(PR6):455-460, 2002

It is most often proposed that the process of ball milling introduces a variety of defects (vacancies, dislocations, grain boundaries, stacking faults,...) which raise the free energy of the system making it possible to produce metastable phases. But there are very few investigations that deal with the characterization and quantification of the defects produced in milled powders. XRD is really a valuable technique for a characterization in terms of size and morphology of crystallites and imperfections. In this paper, a new line profile analysis method is proposed in order to take into account the dependence of the crystallite size, of the residual strains as well as of the planar defects, on the line profile broadening that may be observed on ball-milled materials. Such a method will allow to understand the influence of ball-milling parameters and for controlling the synthesis of nanostructured materials. The results concern the XRD pattern simulation, in a kinematic approach, of FCC. nanocopper produced by ball milling and containing a high concentration of planar defects. Simulations are compared to the experimental data.

[5] ANALYSIS AND INTERPRETATIONS OF THE RESULTS COULD RELY ON LOCAL OBSERVATIONS OF DEFECTS USING HIGH-RESOLUTION ELECTRON MICROSCOPY

Avettand-Fenoel MN. Taillard R. Dhers J. Parmentier P. Foct J. - Journal de Physique IV. 12(PR6):461-472, 2002

High energy ball milling carried out in a planetary ball mill is generally used to alloy elementary powders and to obtain nanostructures. The latter advantages urge us to elaborate pure tungsten or blends of tungsten and yttria powders by means of this process. The obtained powders are then sintered in order to achieve creepproof materials at high temperature. The ball-to-powder ratio is constant during the experiments. The effects of different milling parameters (number of balls: 2, 16, 92; milling time: 5mn, 20mn, 40mn, 80mn, rotational speed: 200 or 300 or 400 rpm, nature of the milling system: stainless steel or tungsten carbide system) and of the composition of blends (volumetric content of second phase) are examined. The results concern nanocrystals in the powder particles, the strain level of powder particles, the formation of alloys and the oxide particles dispersion. The difficulties and advantages of the characterization techniques are developed. The consequences of structural changes are outlined and the mechanisms of the alloy evolution discussed.

[4] FEAL MATERIALS FROM INTERMETALLIC POWDERS

Godlewska E. Szupepanik S. Mania R. Krawlarz J. Kozinski S. - Intermetallics. 11(4):307-312, 2003

FeAl materials with the aluminium concentration of 40 at.% were prepared from intermetallic powders, obtained by milling and screening of the products of self-propagating high-temperature synthesis (SHS), carried out in a loosely packed mixture of elemental powders. Pressureless sintering of the compacted intermetallic powders yielded porous materials that were densified by hot forming. The properties of dense bodies were strongly related to the grain size of the starting intermetallic powders. Two fractions with grain sizes of less than 25 pm and in the range from 25 to 40 pm were investigated. The amount of an oxide phase distributed along the grain boundaries of the intermetallic matrix was remarkably higher in the former case. The oxide dispersions adversely affected the room temperature strength and ductility of the material

[3] MICROSTRUCTURE AND HIGH TEMPERATURE MECHANICAL PROPERTIES OF MECHANICALLY ALLOYED Nb₃Al-BASED MATERIALS

Dollar A. Dymek S. - Intermetallics. 11(4):341-349, 2003

The microstructure and high temperature mechanical properties of mechanically alloyed Nb₃Al-based materials have been investigated. The results for four binary Nb-Al and three ternary Nb-Al-V materials are presented. The microstructure of the present alloys consists of four phases: a disordered Nb solid solution, the Nb₃Al and Nb₂Al ordered phases and aluminum oxide dispersoids. All alloys exhibit high strength at 800 degreesC, but only ternary alloys retain the strength up to at least 1100 degreesC. The presence of vanadium, with its atomic diameter significantly smaller than those of Nb and Al, is postulated to lead to the solid solution strengthening. In the ternary alloy Nb-15 at. % Al-20 at.% V consolidated at 1500 degreesC the creep rates are significantly lower (one to two orders of magnitude) than in all other alloys investigated in the present study because of the absence of the Nb₂Al phase in its microstructure. Creep rate dependence on temperature in the present alloys is controlled by the diffusivity of Nb and Al atoms (and V atoms in the ternary alloys) in the ordered Nb₃Al phase and the creep mechanism is diffusion-assisted dislocation creep.



[2] EXTENDED HOMOGENEITY RANGE OF INTERMETALLIC PHASES IN MECHANICALLY ALLOYED MG-AL ALLOYS

Singh D. Suryanarayana C. Mertus L. Chen RH. - Intermetallics. 11(4):373-376, 2003

The structural evolution during mechanical alloying of blended elemental powder mixtures of Mg and Al has been investigated using X-ray diffraction methods. The results indicate that, in addition to achieving extended solid solubility limits, the intermetallic phases could be directly synthesized by mechanical alloying. The homogeneity range of the beta (Mg_2Al_3) and gamma ($Mg_{17}Al_{12}$) phases could be extended to lower aluminum contents.

[1] FORMATION OF SUPERIONIC CRYSTALS FROM MECHANICALLY MILLED $Li_2S-P_2S_5$ GLASSES

Hayashi A. Hama S. Minami T. Tatsumisago M. - Electrochemistry Communications. 5(2):111-114, 2003

A superionic crystal analogous to highly conductive thio-LISICON, which is a series of sulfide crystalline solid electrolytes such as $Li_4GeS_4-Li_3PS_4$, was successfully formed by the crystallization of mechanically milled $Li_2S-P_2S_5$ glasses. The thio-LISICON phases have not been obtained by solid-phase reaction in the $Li_2S-P_2S_5$ binary system so far, and this report is the first case of obtaining the thio-LISICON analogue in the binary system. The formation of this superionic crystal enhanced the conductivities of the glass, and the high ambient temperature conductivity of $7.2 \times 10^{-4} \text{ S cm}^{-1}$ was achieved in the glass-ceramics derived from the Li-rich $80Li(2)S \cdot 20P(2)S(5)$ (mol%) glass

