



**RESEAU FRANÇAIS DE
MECANOSYNTHESE**

Lettre N°97

Avril 2003

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

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Bulletin d'adhésion 2003 / Subscription Print

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Eric GAFFET

CNRS UMR5060 « Métallurgies et Cultures »

Nanomaterials Research Group

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

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Lettre RFM N°97 - Avril 2003
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Press Release

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FRITSCH is the world-wide leading manufacturer of instruments for sample preparation and particle size analysis in the laboratory. Since 80 years now, the company is concentrating on the three product groups and identified with the brand names of "pulverisette", "analysette" and "laborette" in research and industrial laboratories. The FRITSCH name is synonymous with technical competence and economical efficiency in all matters in the field of

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**8èmes Journées du Réseau Français de Mécanosynthèse Amiens Pôle Scientifique
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PROGRAMME DE RECHERCHE

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

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

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

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

Techniques utilisées :

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

Condition de recrutement

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

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

Laboratoire d'accueil français :

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

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

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

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



Post Doc Proposal

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

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

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

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

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

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

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



Congress and School Announcements

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Vogüé, ARDECHE (France)
19-21 mai 2003

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

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XV. International Symposium on Reactivity of Solids:

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[44] MECHANICAL BEHAVIOR AT 300-4.2 K OF BULK NANOSTRUCTURED TITANIUM PROCESSED BY SEVERE PLASTIC DEFORMATION

Bengus VZ. Tabachnikova ED. Natsik VD. Miskuf J. Csach K. Stolyarov VV. Valiev RZ. - Physics of Metals & Metallography (English Translation of Fizika Metallov i Metallovedenie). 94(Suppl 1):S11-S23, 2002.

The low temperature plasticity and failure of polycrystals of coarse-grained (CG) and nanostructured (NS) titanium of commercial purity (VT1-0) were studied. The NS titanium was prepared by equal-channel angular pressing (ECAP) and subsequent thermomechanical treatment. Two structural modifications were prepared, which had 0.3- μm and 0.1 μm grains. The measurements were made at 300, 77, and 4.2 K under uniaxial compression at a strain rate of $4 \times 10^{-4} \text{ s}^{-1}$. The work-hardening stress-strain curves were obtained; the macroscopic yield stress and the plasticity reserve were measured for samples compressed along (parallel to) and across (perpendicular to) the ECAP axis. The yield stress increased by a factor of 1.5-2 on changing from CG to NS titanium and on cooling from 300 to 4.2 K. Plasticity anisotropy was also observed in NS titanium when the orientation of the compression axis was changed from parallel (parallel to) to perpendicular (perpendicular to); the yield stress increased by a factor of 1.2-1.5. These changes in the sample structure and experimental conditions systematically reduced the plasticity reserve, but the prefailure strain was still above 4%. No cold brittleness was observed in NS titanium down to liquid-helium temperature, although at 4.2 K the plastic flow became jumplike, as in CG titanium. It is found that, under low-temperature uniaxial compression, the failure of NS titanium is caused by the unstable plastic shear, leading to local adiabatic heating of the material. This is not characteristic for CG titanium. Scanning electron microscopy of the shear-failure surface morphology detected a characteristic "vein" pattern indicative of local heating to 800degreesC and higher. An analysis of the experimental results prompts the following conclusions: (i) at low temperatures, plastic deformation in NS titanium is a thermally activated process; (ii) the yield stress is influenced significantly by the microstructural internal stresses induced by thermal anisotropy and by probable microtwinning; (iii) the grain-boundary shears play an important role in deformation and failure processes

[43] DEVELOPMENT OF METHODS OF SEVERE PLASTIC DEFORMATION FOR THE PRODUCTION OF HIGH-STRENGTH ALLOYS BASED ON TITANIUM NICKELIDE WITH A SHAPE-MEMORY EFFECT

Pushin VG. Stolyarov VV. Valiev RZ. Kourouov NI. Kuranova NN. Prokofev EA. Yurchenko LI. - Physics of Metals & Metallography (English Translation of Fizika Metallov i Metallovedenie). 94(Suppl 1):S54-S68, 2002.

A brief review of the investigations of the authors concerning the application of methods of severe plastic deformation (SPD) for the production of nanostructured alloys on the basis of titanium nickelide with a thermomechanical memory is presented. For the first time, high-strength nanostructured TiNi-based alloys were obtained using several different SPD techniques, including (1) multiple torsion under a high pressure, (2) multiple equal-channel angular pressing, and (3) multiple deformation by rolling and drawing with intermediate annealings. It is shown that a special place among these techniques belongs to the process of controlled nanocrystallization during annealing of alloys in the initially amorphous state that is ensured by severe cold plastic deformation. The main features of structural states of SPD-processed alloys and their thermal stability are analyzed using X-ray diffraction and transmission electron microscopy, including experiments in situ. The time temperature conditions of nanocrystallization of amorphous TiNi-based alloys and the critical temperatures and sequence of martensitic transformations were established and their dependence on the mode and amount of deformation and average grain size was determined. The main structural mechanisms of the nucleation and growth of martensitic phases (R, B19', and B19) and their morphology in nanostructured SPD-processed alloys based on TiNi were revealed. The temperature dependences of the electrical resistivity and the mechanical properties of nanostructured alloys subjected to severe plastic deformation were measured

[42] SEVERE PLASTIC DEFORMATION OF R-FE-B (R = PR OR ND) HARD MAGNETIC ALLOYS

Popov AG. Gaviko VS. Shchegoleva NN. Puzanova TZ. Ermolenko AS. Stolyarov VV. Gunderov DV. Raab GI. Valiev RZ. - Physics of Metals & Metallography (English Translation of Fizika Metallov i Metallovedenie). 94(Suppl 1):S75-S81, 2002.

The results of investigations of the effect of severe plastic deformation on the structure and magnetic hysteresis properties of R-Fe-B (R = Pr or Nd) alloys are reviewed. Apart from grain refinement, the R2Fe14B phase decomposes to form an R-rich amorphous phase and nanocrystalline (α -Fe). Annealing of heavily deformed alloys at temperatures $T_a > 500\text{degreesC}$ recovers the R2Fe14B phase in its nanocrystalline state, which significantly increases the coercive force of such alloys. Annealing at $300 < T_a < 500\text{degreesC}$ of alloys with intermediate degrees of deformation, which mainly consist of elastically stressed grains of the R2Fe14B phase, induces additional decomposition of this phase and a corresponding decrease in H-c. To fabricate large bulk magnets, equal-channel angular pressing is used.

[41] STUDY OF A NANOCRYSTALLINE FE-C ALLOY COMPACTED BY SEVERE PLASTIC DEFORMATION OF POWDER AFTER BALL MILLING

Krasil'nikov NA. Ivanisenko YV. Raab GI. Valiev RZ. - Physics of Metals & Metallography (English Translation of Fizika Metallov i Metallovedenie). 94(Suppl 1):S91-S97, 2002.

Nanostructured Fe-1 wt % C samples characterized by a nearly theoretical density and a high strength were compacted by severe plastic deformation of the powder obtained by ball milling. Study of the samples showed that their high density and strength are determined by the compacting conditions, the nanocrystalline structure of the powder, and phase transformations occurring upon severe plastic deformation



[40] STRUCTURAL FEATURES, STRENGTH, AND MECHANISMS OF DEFORMATION OF NANOCRYSTALLINE MATERIALS

Noskova NI. - Physics of Metals & Metallography (English Translation of Fizika Metallov i Metallovedenie). 94(Suppl 1):S119-S130, 2002.

Results of recent original studies of the structure and properties of nanocrystalline metals and alloys produced by severe plastic deformation and by nanocrystallization of amorphous alloys are considered. High resolution transmission electron microscopy, scanning electron microscopy, and in situ deformation in the column of an electron microscope were used to analyze the structural features and the mechanisms of plastic deformation of nanocrystalline materials.

[39] TOUGHENING OF MOSI₂ DOPED BY LA₂O₃ PARTICLES

Zhang H. Wang DZ. Chen SP. Liu XY. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 345(1-2):118-121, 2003

Molybdenum disilicide (MoSi₂) with or without La₂O₃ particles were synthesized by mechanical alloying (MA) and HP techniques. The fracture toughness at room temperature was measured by Anstis' mode and the responded microstructures were analyzed by using SEM and X-ray. The results show that the addition of La₂O₃ could obviously toughen the matrix. When the content of La₂O₃ in MoSi₂ is about 0.9%, the fracture toughness value increases by about 50% more than that of pure MoSi₂. We think, the corresponding toughening mechanisms depend on the following: fine-grain, microcracking, crack deflection, crack microbridging, crack bowing and branching.

[38] MECHANOCHEMICAL SYNTHESIS OF PZT POWDERS

Brankovic Z. Brankovic G. Jovalekic C. Maniette Y. Cilense M. Varela JA. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 345(1-2):243-248, 2003

PZT ceramic powders were successfully prepared from the mixture of PbO, ZrO₂ and TiO₂ by mechanochemical synthesis in a planetary ball mill, under different milling conditions. Phase evolution during synthesis was monitored by X-ray diffraction analysis. Intensive milling resulted in formation of the nanocrystalline, perovskite PZT powders after 1 h of milling. This is a significant improvement in comparison to milling conditions reported by other authors. Depending on milling parameters the presence of some other phases, such as unreacted ZrO₂, was also detected in some samples. The changes in powder size and morphology due to intensive milling, were determined by SEM and TEM, while BET analysis was used to determine specific surface area of the powders. Conclusions about processes taking place during mechanochemical synthesis of PZT powders were made based on the results of characterization.

[37] EFFECT OF PHASE TRANSFORMATION DURING HIGH ENERGY MILLING ON FIELD ACTIVATED SYNTHESIS OF DENSE MOSI₂

Sannia M. Orru R. Garay JE. Cao G. Munir ZA. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 345(1-2):270-277, 2003

The effect of mechanical activation through high-energy ball milling of reactant powders on the subsequent synthesis of MoSi₂ by field activation was investigated. Field activated synthesis of this silicide was made through the use of the spark plasma sintering (SPS) method using a pulsed DC current. Milling (0-6 h) produced significant changes in the dispersion and crystallite size of the reactants initially, and resulted in the partial formation of the product in both the low-temperature (alpha) and high-temperature (beta) modifications when longer milling times were employed. The sequence of phase evolution during milling was determined from XRD, EDX and SEM analyses. Subsequent field-activated synthesis resulted in the formation of alpha-MoSi₂ only. The initiation of the synthesis reaction required a threshold power level (equivalent to the level of the current), with the threshold decreasing with increased milling time. However, the initiation time increased when milling resulted in the formation of a significant amount the product phase, with the increase being markedly significant at low power levels.

[36] PARTICIPATION OF DIFFUSION IN THE PROCESSES OF MECHANICAL ALLOYING

Shtremel' MA. - Metal Science & Heat Treatment. 44(7-8):324-327, 2002

[35] OXIDATION BEHAVIOR OF MECHANICALLY ACTIVATED GALENA IN THERMOGRAVIMETRY (TG)

Hu HP. Chen QY. Yin ZL. Zhang PM. Tang AD. - Thermochemica Acta. 395(1-2):139-144, 2003

The oxidation behaviors of non-activated and mechanically activated galenas were investigated by thermogravimetry method (TG) in flowing highly pure oxygen atmosphere at the heating rate of 10 K min⁻¹. It is found that the mass increase between 400 and 850 K in the TG curves rises with the increase of grinding time of galena. The difference in oxidation reactivity of non-activated and mechanically activated galenas was also discussed. The specific granulometric surface area (S-G) and the structural disorder of mechanically activated galenas were analyzed by X-ray diffraction laser particle size analyzer and X-ray powder diffraction analysis (XRD), respectively. The results show that the specific granulometric surface area (S-G) of mechanically activated galenas almost remains constant after grinding for a certain period, and lattice distortions (δ) increase, but the crystallite sizes (D) decrease with the increase of the grinding time. All the results imply that the mass increase between 400 and 850 K in the TG curves for mechanically activated galenas is mainly caused by the increase of lattice distortions (δ) and the decrease of the crystallite sizes (D) with increasing the grinding time

[34] EFFECTS OF MECHANOCHEMICAL ACTIVATION AND ALPHA-FE₂O₃ ADDITION ON THE FORMATION OF CORUNDUM IN THERMAL TRANSFORMATIONS OF GAMMA-AL(OH)₃

Tolchev AV. Kleshchev DG. Lopushan VI. - Russian Journal of Applied Chemistry. 75(9):1384-1388, 2002



Transformations of hydrargillite $\gamma\text{-Al(OH)}_3$ during mechanochemical activation in various grinding mills and further calcination of activated samples at 200-1300°C were studied. The effects of mechanochemical activation and additions of iron(III) oxide compounds on the temperature of corundum formation and on its crystal size distribution were studied

[33] APPLICATION OF EXELFS SPECTROSCOPY TO THE STUDY OF THE LOCAL ATOMIC STRUCTURE OF CARBON-CONTAINING MATERIALS

Maratkanova AN. Huang JY. Rats YV. Surnin DV. - Physics of Metals & Metallography (English Translation of Fizika Metallov i Metallovedenie). 94(6):598-605, 2002

The main aim of this work was to further develop the method of extended energy-loss fine structure (EXELFS) for the investigation of the structure of binary compounds containing light elements (C, O, S, P, etc.). The local atomic structure in different carbon modifications such as highly oriented pyrolytic graphite and amorphous graphite prepared by milling in a planetary ball mill was studied in an HF-3000 FEG TEM transmission electron microscope equipped with an electron spectrometer for recording electron-energy-loss spectra. The advantage of the EXELFS method for the study of the local atomic structure of binary compounds containing light elements was demonstrated by the example of cementite Fe_3C in a carbon steel U15.

[32] EVOLUTION OF MICROSTRUCTURE AND MECHANICAL PROPERTIES OF IN SITU CONSOLIDATED BULK ULTRA-FINE-GRAINED AND NANOCRYSTALLINE ZN PREPARED BY BALL MILLING

Zhang X. Wang H. Scattergood RO. Narayan J. Koch CC. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 344(1-2):175-181, 2003

The evolution of the microstructure and mechanical properties of ultra-fine-grained and nanocrystalline Zn induced by ball milling at room temperature are studied systematically. The yield stresses measured from miniaturized disk bend tests and tensile tests are consistent with the microhardness results and generally increase with the decrease of average grain size. A dramatic decrease of hardness during milling from 1 to 3 h is a reflection of the increase of average grain size from 80 to 240 nm due to the initial unstable grain size and therefore, grain growth in this period. Young's modulus remains almost the same for samples milled for different times and is that for conventional grain size Zn. A transition from bending to membrane stretching is observed in the force-displacement curves for Zn ball milled for less than or equal to 18 h. The variation of transition strain with milling time could be related to the evolution of grain size distribution and therefore hardness during milling

[31] COMBINATION OF MECHANICAL ALLOYING AND TWO-STAGE SINTERING OF A 93W-5.6NI-1.4FE TUNGSTEN HEAVY ALLOY

Soon HH. Ryu HJ. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 344(1-2):253-260, 2003

The microstructural evolution and mechanical properties of a mechanically alloyed and two-stage sintered tungsten heavy alloy were investigated. Elemental powders of tungsten, nickel and iron of a composition corresponding to 93W-5.6Ni-1.4Fe were mechanically alloyed in a tumbler ball mill for 72 h. Mechanically alloyed powders were solid-state sintered at 1300 °C for 1 hr in a hydrogen atmosphere followed by secondary sintering at 1445-1485 °C for a sintering time ranging from 4 to 90 min. Solid-state sintered tungsten heavy alloys exhibited full densification (above 99% in relative density) due to the enhanced sintering resulting from mechanical alloying. Secondary sintering with a rapid heating rate changed the microstructures of the solid-state sintered alloy with contiguous tungsten phases into a dispersion alloy with spherical tungsten particles embedded in the W-Ni-Fe matrix, maintaining fine tungsten particle due to the combination of a mechanical alloying and a short sintering time. The two-stage sintered tungsten heavy alloy from mechanically alloyed powders showed finer tungsten particle (about 6 μm in diameter) than in conventional liquid-phase sintered tungsten heavy alloys. The mechanical properties of a tungsten heavy alloy were found to be dependent on the microstructural parameters such as tungsten particle size, matrix volume fraction and tungsten/tungsten contiguity which are controllable through the two-stage sintering process.

[30] A NOVEL PROCESSING ROUTE TO DEVELOP A DENSE NANOCRYSTALLINE ALUMINA MATRIX (< 100 NM) NANOCOMPOSITE MATERIAL

Zhan GD. Kuntz J. Wan J. Garay J. Mukherjee AK. - Journal of the American Ceramic Society. 86(1):200-202, 2003 Jan. Abbreviated Source J. Am. Ceram. Soc. 86(1):200-202, 2003

A dense 3-mol%-yttria-stabilized tetragonal zirconia polycrystalline (3Y-TZP) toughening alumina matrix nanocomposite with a nanocrystalline (<100 nm) matrix grain size has been successfully developed by a novel processing method. A combination of very rapid sintering at a heating rate of 500°C/min and at a sintering temperature as low as 1100°C for 3 min by the spark-plasma-sintering technique and mechanical milling of the starting $\gamma\text{-Al}_2\text{O}_3$ nanopowder via a high-energy ball-milling process can result in a fully dense nanocrystalline alumina matrix ceramic nanocomposite. The grain sizes for the matrix and the toughening phase were 96 and 265 nm, respectively. A great increase in toughness almost 3 times that for pure nanocrystalline alumina has been achieved in the dense nanocomposite. Ferroelastic domain switching without undergoing phase transformation in nanocrystalline t-ZrO₂ is likely as a mechanism for enhanced toughness.

[29] STRUCTURAL STABILITY OF MECHANICALLY ALLOYED (MG+10NB) AND (MGH₂+L0NB) POWDER MIXTURES

Shang CX. Bououdina M. Guo ZX. - Journal of Alloys & Compounds. 349(1-2):217-223, 2003

In order to improve the hydrogen storage characteristics of magnesium, both chemical alloying by Nb and mechanical alloying (MA) of (Mg +10 wt. %Nb) and (MgH₂+10 wt.%Nb) powder mixtures were investigated, with particular attention



paid to their structural stability. Extensive powder refinement was noted for both compositions within 20 h of milling at 250 rpm. Even nano-sized particles were generated in the hydride mixture. XRD and Rietveld analyses show the formation of a bcc phase in each case. The amount of the bcc phase increases with increasing milling time to the detriment of Nb. For the (Mg +10 wt. %Nb) mixture, it is confirmed that the newly formed phase is a bcc-(Nb,Mg) solid solution, with an extended solubility of Nb in Mg. However, for the (MgH₂ + 10 wt.%Nb) powder mixture, the new bcc phase can be a Nb hydride (NbH_x, x < 1.0), or a bcc-(Nb,Mg) solid solution, or a (Nb,Mg)H-x solid solution, or even a mixture of the three.

[28] PREPARATION OF SODIUM BOROHYDRIDE BY REACTION OF MGH₂ WITH DEHYDRATED BORAX THROUGH BALL MILLING REACTION OF MGH₂ WITH AT ROOM TEMPERATURE

Li ZP. Morigazaki N. Liu BH. Suda S. - Journal of Alloys & Compounds. 349(1-2):232-236, 2003

A convenient method was developed to synthesize NaBH₂ by the reaction of MgH₂ with Na₂B₄O₇ through ball milling at room temperature. In order to improve the sodium borohydride yield, Na compounds were added to compensate the Na insufficiency in reactants when MgH₂ instead of NaH was used as the reducing agent. It was found that Na₂CO₃ addition was better than NaOH or Na₂O₂ addition in increasing the borohydride yield

[27] OPTIMISATION OF THE BALL-MILLING AND HEAT TREATMENT PARAMETERS FOR SYNTHESIS OF AMORPHOUS AND NANOCRYSTALLINE MG₂NI-BASED ALLOYS

Spasov T. Solsona P. Surinach S. Baro MD. - Journal of Alloys & Compounds. 349(1-2):242-254, 2003

Amorphous and nanocrystalline Mg_{1.9}M_{0.1}Ni (M=Ti,ZrV) alloys were synthesized by mechanical alloying (MA) and by MA followed by annealing. The phase composition, microstructure and morphology of the as-milled powder, and the milled and heat treated powders were determined by XRD and SEM/EDX. Thermal stability, crystallization and grain growth processes in the nano-/amorphous alloys were investigated too. The milling and heat treatment conditions for obtaining amorphous or nanocrystalline alloys were optimised for different alloy compositions. After milling or milling with annealing the ternary and quaternary alloys have the hexagonal Mg₂Ni crystal structure with crystallite sizes in the range of 5-15 nm, depending on the conditions of milling and annealing. The microstructure of the alloys obtained by long time milling is much finer compared to that of the shorter time milled samples, both subjected to annealing after milling. The grain size of the final nanostructures increases slightly during heating to above 400 degreesC. The nanocrystalline Mg₂Ni-based alloys prepared by extended milling show higher thermal stability than the short time milled alloys.

[26] EFFECT OF EXTENDED BALL MILLING ON GRAPHITE

Welham NJ. Berbenni V. Chapman PG. - Journal of Alloys & Compounds. 349(1-2):255-263, 2003

Graphite has been milled for up to 1000 It in a laboratory scale tumbling ball mill under vacuum. Raman spectroscopy of the powders indicated the increasing dominance of D-type graphitic sp(2) bonding over G-type bonding with increasing milling time. Diamond-like sp(3) bonding and possibly fullerene-like bonding also became evident after milling. TEM of the 100 It sample showed the presence of ribbons which were composed of sheets showing defects, delamination, translation, warping and curvature. Interplanar spacings of 0.40-0.50 nm were measured with the spacing increasing towards the edge of the ribbons where delamination was evident. Thermogravimetric analysis in argon of the powder after exposure to air showed an increasing mass loss with milling time indicating the presence of chemisorbed gas. Using TG-FTIR the gas was found to be a mixture of CO₂ and an unidentified gas (probably oxygen). BET surface area measurements showed a maximum in the surface area; however, this was shown to be massively in error for the longer milling times due to the presence of the chemisorbed gas.

[25] THE EFFECT OF GRAIN REFINING ON THE DISCHARGE CAPACITY OF MG₂NI/MMNI(5-X)(COALMN)(X) COMPOSITE PREPARED BY MECHANICAL ALLOYING

Zhu M. Wang ZM. Peng CH. ZEng MQ. Gao Y. - Journal of Alloys & Compounds. 349(1-2):284-289, 2003

Hydrogen storage composites were prepared by ball-milling the powder mixtures of Mg₂Ni and MmNi(5-x)(CoAlMn)(x) (denoted as MmM(5)) alloys. X-ray diffraction (XRD) and scanning electron microscopy (SEM) analysis show that the particles of Mg₂Ni/MmM(5) nanophase composite are aggregates of constituent phases. The electrode properties of Mg₂Ni alloys, MmM(5) alloy and nanophase composites with different grain sizes were measured by simulation battery test. It was found that the discharge capacity of Mg₂Ni alloy was enhanced with increased milling time, but decreased with milling time for MmM(5) alloy. As for the nanophase composite, its discharge capacity was not simply the linear sum of the capacities of components constituting the composite. An enhancement effect on capacity has been observed in the composite when the grain size of the phase inside the composite is less than about 100 nm

[24] ELECTROCHEMICAL PROPERTIES OF NIS AS A CATHODE MATERIAL FOR RECHARGEABLE LITHIUM BATTERIES PREPARED BY MECHANICAL ALLOYING

Han SC. Kim HS. Song MS. Lee PS. Lee JY. Ahn HJ. - Journal of Alloys & Compounds. 349(1-2):290-296, 2003

Nickel sulfide (NiS) as a cathode material for a lithium rechargeable battery is charged and discharged at room temperature (30 degreesC). In order to synthesize a homogeneous NiS phase, mechanical alloying (MA) was adopted. The homogeneous NiS phase is easily formed after ball milling for 12 h under Ar atmosphere. The ball-milled NiS particles are relatively larger than those of the starting materials and have a nanocrystalline structure. The initial discharge capacity of the NiS positive electrode is 580 mAh/g-NiS, at 1.4 V versus Li/Li+. The NiS powders synthesized by MA show proper cycling properties, by retaining 65% of the initial discharge capacity even after 100 cycles at 30 degreesC. Also, NiS has a good rate capability. It has 87% of its theoretical capacity at a current rate of 2 C, comparable with that of 1/6 C

[23] STRUCTURE AND MAGNETIC PROPERTIES OF ND-2(Fe,Co,Al,Cr)(14)B/ALPHA-Fe NANOCOMPOSITE MAGNETS



Jakubowicz J. Giersig M. - Journal of Alloys & Compounds. 349(1-2):311-315, 2003

The structure, magnetic properties and corrosion behaviour of two-phase nanocomposite Nd-2(Fe,Co,Al,Cr)(14)B/ α -Fe-type magnets, which have tetragonal/cubic Nd₂Fe₁₄B/ α -Fe structure, have been investigated. The magnetic hardening was achieved by high-energy ball-milling (HEBM) of the Nd₂Fe₁₄B-type hard magnetic phase with different vol.% of α -Fe as soft magnetic phase, followed by annealing. Fully dense Nd-2(Fe,Co,Al,Cr)(14)B/ α -Fe type magnets have been produced by hot pressing. Magnets with good corrosion resistance as well as high temperature stability have been produced. The corrosion resistance is improved in the case of Co-, Al-, Cr-doped nanocomposite magnets with a volume fraction of the soft magnetic phase of 37.5 vol.%. Effective protection against corrosion was realised also by surface coating with Zn metal.

[22] AN IN SITU NEUTRON TIME-OF-FLIGHT DIFFRACTION STUDY OF LAMM(NICOALMN)(5-X) BATTERY ELECTRODE MATERIALS AND THEIR DEUTERIDES, FOR X=0 AND X=0.2

Georgiev PA. Liu J. Ross DK. Andersen KH. Otto A. - Journal of Alloys & Compounds. 349(1-2):325-333, 2003

The structural properties of two mechanically activated commercial LaNi₅ type battery materials and their deuterides have been investigated by means of in situ neutron powder diffraction analysis. A discussion of the hysteresis between the absorption and desorption isotherms is given. Using standard Rietveld refinement procedures, information on the variation of the deuterium site occupancy, the lattice symmetry and the cell volume are also presented. In particular, we report on the variation of line-broadening with concentration as well as on the complex time-dependent changes in the lattice parameters and the line-broadening following a step change in the deuterium pressure. We then show that these relatively novel data provide valuable information on the rather different phase transition processes observed for the two materials and offer an explanation for the superior kinetics of the AB(4.8) alloy.

[21] PREPARATION AND CHARACTERIZATION OF ACRYLIC/NANO-TiO₂ COMPOSITE LATEX

He QY. Wu LM. Gu GX. You B. - High Performance Polymers. 14(4):383-396, 2002

Acrylic/nano-TiO₂ composite latex samples were prepared via high-shear stiffing and mixing (SM), ball milling and mixing (BM) and in situ polymerization (IS) methods, and investigated by transmission electron microscopy (TEM), testing with an Instron machine, dynamic mechanical analysis (DMA) and ultraviolet-visible (UV-VIS) spectrophotometry. It was shown that the IS method led to the best dispersion of nano-TiO₂ particles in acrylic copolymer films, the SM method was the second best and the BM method gave the least good result. Addition of nano-TiO₂ by SM and BM methods led to higher tensile strength and T_g for polymers than introduction with micro-TiO₂ and without any fillers. UV-VIS spectra indicated that addition of nano-TiO₂ into acrylic copolymers could increase the absorbance and decrease the transmittance in the UV region, whereas the absorbance or transmittance in the UV region basically remained unchanged for the composites filled with micro-TiO₂

[20] INFRARED STUDY OF MAGNESIUM-NICKEL HYDROXIDE SOLID SOLUTIONS

de Oliveira EF. Hase Y. - Vibrational Spectroscopy. 31(1):19-24, 2003

The IR spectra of the co-precipitated solid solutions Mg_xNi_{1-x}(OH)₂ were studied in the 4000-400 cm⁻¹ region. The spectra as a whole resemble those of Mg(OH)₂ and beta-Ni(OH)₂, while certain differences are noted when compared with the spectra of the mechanically mixed samples Mg(OH)₂ + beta-Ni(OH)₂. Such a behavior may imply formations of mono-phase solid solutions which have a brucite-like crystal structure. The composition-dependent band shifts were observed for the fundamentals and this tendency is discussed in terms of polarization of the O-H bond and partial covalency of the M-O bonds. The gradual changes in band position of Mg_xNi_(1-x)(OH)₂ (1.00 greater than or equal to x greater than or equal to 0.00) were used to assign the IR active lattice modes of the solid solutions and to review the assignment of beta-Ni(OH)₂.

[19] SOLVENT-FREE REACTIONS OF FULLERENES AND N-ALKYLGLYCINES WITH AND WITHOUT ALDEHYDES UNDER HIGH-SPEED VIBRATION MILLING

Wang GW. Zhang TH. Hao EH. Jiao LJ. Murata Y. Komatsu K. - Tetrahedron. 59(1):55-60, 2003

The solvent-free reactions of fullerenes and N-alkylglycines with and without aldehydes (RCHO) 2a-e under high-speed vibration milling (HSVM) conditions have been investigated. Fulleropyrrolidines 4a-e (C-60(CH₂N(CH₃)CHR), R=H (4a), C₆H₅ (4b), pNO₂-C₆H₅ (4c), p-CH₃O-C₆H₄ (4d), p-(CH₃)₂N-C₆H₄ (4e)) were obtained in moderate yields from reactions of C-60 with aldehydes 2a-e and N-methylglycine (Prato reaction). In all these solvent-free reactions, 4a was found to be formed besides 4b-e, indicating that fullerenes can react with N-substituted glycines in the absence of aldehyde to give fulleropyrrolidines. For this novel reaction, a possible reaction mechanism involving an electron transfer process has been proposed. Intrigued by this observation, the dependence of the yield on the reagent ratio for the reaction of C-60 with paraformaldehyde and/or N-methylglycine was examined to search the optimal conditions. The reaction of C-70 with paraformaldehyde and/or N-methylglycine under HSVM conditions was also studied and was found to give the positional isomers of [70]fulleropyrrolidines

[18] SOLID-PHASE SYNTHESIS OF ZINC(II) BETA-DIKETONATES UPON MECHANICAL ACTIVATION

Petrova LA. Borisov AP. Makhaev VD. - Russian Journal of Inorganic Chemistry. 47(12):1827-1832, 2002

Mechanically activated solid-phase reactions between zinc chloride and sodium beta-diketonates are studied. The course of the reaction, the product yield, and some properties of the activated mixtures are studied in relation to mechanical treatment conditions and the nature of beta-diketone. Zinc beta-diketonates are separated by sublimation from the activated mixtures in >70% yield. Unsolvated zinc beta-diketonates are characterized by physicochemical methods.

[17] CHARACTERISTICS OF POLYVINYLPIRROLIDONE-LAYERED SILICATE NANOCOMPOSITES PREPARED BY ATTRITION BALL MILLING



Koo CM. Ham HT. Choi MH. Kim SO. Chung IJ. - Polymer. 44(3):681-689, 2003

Polyvinylpyrrolidone (PVP)/sodium montmorillonite (MMT) nanocomposites prepared via the solution intercalation method were investigated by UV/vis, SEM, X-ray diffraction, TEM, FT-IR and PLM (polarized light microscopy). PVP/MMT nanocomposites show exfoliation below 20 wt% MMT and intercalation above this concentration. Nanocomposites retain good optical clarity and increased thermal resistance with MMT content. The compatibility between PVP and MMT and their enhanced properties may be explained by hydrogen bonding interactions. In addition, the nanocomposites prepared under more rigorous mixing conditions show better transparency because the smaller particle sizes are induced. In addition, the study on optically clear PVP/MMT suspensions helps one to understand how optical anisotropy of MMT is affected by the existence of polymer in aqueous solution

[16] EVOLUTION OF VACANCY DENSITIES IN POWDER PARTICLES DURING MECHANICAL MILLING

Zhang BQ. Lu L. Lai MO. - Physica B: Condensed Matter. 325(1-4):120-129, 2003

This paper investigates the change in the density of vacancies during ball milling. A model for the evolution of vacancies has been proposed in this paper. Results from the simulation show that the density of vacancies increases asymptotically with collision times and the rate of increase becomes slower with further ball milling. The accumulation of vacancies in the powder particles is dramatically accelerated at lower milling temperature. Vacancies are shown to accumulate more easily in powder materials with higher vacancy migration energies. The present simulation indicates that higher collision frequency results in a faster accumulation of vacancies and therefore higher density of vacancies can be obtained. In addition, higher impact energy introduces higher density of vacancies per collision. Low milling temperature, high collision frequency and high collision energy may be advantage for the formation of new alloys, but low migration rate of vacancies at low temperature may reduce the rate of the formation of new alloys if the milling temperature is too low.

[15] ON THE INFLUENCE OF N ON RESIDUAL MICROSTRAIN IN CRYOMILLED NI

Chung KH. Lavernia EJ. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 33(12):3795-3801, 2002

The factors that influence the development of residual microstrain during milling in a liquid nitrogen atmosphere, defined hereafter as cryomilling, are investigated. The residual microstrains in cryomilled Ni, processed under various cryomilling conditions, were examined by X-ray diffraction (XRD) and analyzed through the single line approximation (SLA) method. The average residual microstrains are determined to be in the range of 2×10^{-3} to 6×10^{-3} . The residual microstrain on the (200) plane is higher than those on the other planes by 33 pct. The residual microstrain and its anisotropy in Ni are reduced after heat treatment at 800 degreesC for 1 hour. The measured microstrain is proposed to evolve from the presence of N and O as impurity atoms in the Ni lattice. Both N and O are introduced from the environment and then their solubility in Ni is enriched via the generation of defects that occurs during cryomilling. The stable site for N and O atoms in Ni is the octahedral site, and the sizes of N and O atoms exceed those of the octahedral site of Ni by 48 and 16 pct respectively. Accordingly, a lattice strain field is expected around interstitial N atoms that are located at octahedral sites. By comparing the crystal structure around the octahedral site, the stable site for impurity N atoms, in the Ni lattice with that of Ni₃N structure, the lattice strains are estimated to be in the range of 5 to 15 pct. The result shows that the (200) plane has strains that are 2 times higher than those in other planes, and this is argued to be the reason for the measured anisotropy of residual strain in Ni after cryomilling

[14] MICROSTRUCTURE AND MAGNETIC PROPERTIES OF NANOSIZED FE-CO ALLOY POWDERS SYNTHESIZED BY MECHANOCHEMICAL AND MECHANICAL ALLOYING PROCESS

Lee BH. Ahn BS. Kim DG. Oh ST. Jeon H. Ahn J. Kim YD. - Materials Letters. 57(5-6):1103-1107, 2003

An optimum route to synthesize nanosized Fe-Co alloy powder with enhanced magnetic properties was investigated. Two methods of mechanical alloying (MA) and mechanochemical alloying (MCA) for developing a nanosized alloy powder were compared on the basis of the resulting microstructural characteristics and magnetic properties. The alloy powder, synthesized by MCA process with ball milling and hydrogen reduction using Fe₂O₃ and Co₃O₄ powders, showed ordered BCC structure with the grain size of 40 nm. Also, this powder exhibited low coercivity of 43 Oe and good permeability compared with MA powder. Enhanced magnetic properties of the MCA powder were explained by the formation of ordered structure and relaxation of internal strain

[13] MECHANOCHEMICAL REACTIONS BETWEEN Ag₂O AND V₂O₅ TO FORM CRYSTALLINE SILVER VANADATES

Kittaka S. Nishida S. Ohtani T. - Journal of Solid State Chemistry. 169(1):139-142, 2002

Solid-state reactions between Ag₂O and V₂O₅ were studied under ball-milling conditions. Single-phase crystalline Ag₄V₂O₇ was formed from the mixture of Ag₂O and V₂O₅ of corresponding (2:1) composition. The main component in the product when the Ag₂O mole fraction is less than V₂O₅ is amorphous AgVO₃, which is crystallized into needle-like alpha-AgVO₃ in the presence of water. Excess V₂O₅ was hydrated into V₂O₅ · nH₂O intercalated with Ag⁺ ions. The mixtures with more than two parts of Ag₂O relative to V₂O₅ are composite materials of Ag₄V₂O₇ and Ag₃VO₄, together with Ag₂O. The crystalline phases in these systems resist attack by water.

[12] FRACTURE ANALYSIS OF ALUMINUM MATRIX COMPOSITE MATERIALS REINFORCED WITH (Ni₃Al)_p

Velasco F. Da Costa CE. Candela N. Torralba JM. - Journal of Materials Science. 38(3):521-525, 2003



This paper studies the influence of Ni₃Al intermetallic particles on the fracture behaviour of aluminium matrix (2014) composite materials. Intermetallics were obtained by mechanical alloying and by atomisation. The composite materials were manufactured by mixing, uniaxial compacting of a preform, and subsequent extrusion without canning or degassing. The study considered materials in extruded state and after T6 heat treatment. Assessments were made from the viewpoint of microstructure (by means of optical and scanning electron microscopy), and studying the reactions between the matrix and the reinforcement. These reactions produce a highly copper-enriched interphase. The influence of the reinforcement and state of the alloy on the fracture behaviour of the composite materials was studied through scanning electron microscopy

[11] MICROSTRUCTURES AND MECHANICAL PROPERTIES OF ZrO₂/NiAl MATRIX COMPOSITES ELABORATED FROM MECHANOFUSION-PROCESSED POWDERS

Ouchetto M. Grosbras M. Chouiyakh A. - Journal of Materials Science. 38(3):589-595, 2003

ZrO₂-coated NiAl powders have been elaborated using an innovative mill process, the so-called mechanofusion process. Following processing conditions, different types of particles (size, morphology, degree of coating) can be obtained. These powders were consolidated to full density by HIPping and mechanical tests were carried out on the various elaborated products at temperatures ranging between 20-800degreesC. Results are compared to those previously obtained for ZrO₂/NiAl composites elaborated by conventional methods. It is shown that mechanical strength of ZrO₂/NiAl composites can be considerably improved by using the mechanofusion process.

[10] GRAIN GROWTH AND RECRYSTALLIZATION OF NANOCRYSTALLINE Al₃Ti PREPARED BY MECHANICAL ALLOYING

Zhang F. Lu L. Lai MO. Froes FH. - Journal of Materials Science. 38(3):613-619, 2003

The grain sizes and lattice strains during mechanical alloying of Ti-75 at.% Al powder mixtures were studied using X-ray diffraction methods. Nanocrystalline L1(2)-Al₃Ti was obtained after a certain time period of ball milling. Minimum grain sizes of 17 nm for Al and 28 nm for Ti have been determined using XRD. During subsequent thermal annealing processing, an obvious recrystallization resulting in significant reduction of grain size was observed. The recrystallization in nanocrystalline Al₃Ti was affected by both the temperature and the degree of order. The incubation period for recrystallization at 400degreesC was about 6 hours while those at 510 and 700degreesC were about 2 hours. The completion time of recrystallization in Al₃Ti at 400 and 700degreesC was about 15 hours and 8 hours at 510degreesC. It is clear that the recrystallization at 700degreesC was retarded as a result of the higher degree of order structure which limited the mobility of the boundaries. Phase transformation occurring within the recrystallization temperature range was observed to have little influence on the recrystallization itself. However, transformation products do have significant effects on it which is originated from the degree of order in the products. The recrystallization in this alloy system provides an excellent means to maintain the nanocrystalline microstructure during the necessary consolidation thermal cycle by decreasing the processing temperature and increasing the hold time considerably

[9] SYNTHESIS AND CONSOLIDATION OF TiAl BY MA-PDS PROCESS FROM SPONGE-Ti AND CHIP-AL

Sun ZM. Wang Q. Hashimoto H. Tada S. Abe T. - Intermetallics. 11(1):63-69, 2003

Low cost sponge-Ti and recycled chip-Al were used as starting materials for the synthesis and consolidation of TiAl alloy via a mechanical alloying (MA) and pulse discharge sintering (PDS) process. Amorphous structured TiAl powder was obtained by MA process from sponge-Ti/chip-Al and was sintered and concurrently consolidated with the PDS process. The obtained TiAl alloy consists of gamma-TiAl and alpha(2)-Ti₃Al and additional Ti₂AlC phase. The microstructure of the alloy can be controlled by the sintering temperature. It was found that fine equiaxed grains were obtained by low temperature sintering while fine duplex structures formed at high temperature sintering. Compared with alloys fabricated from high cost elemental Ti/Al powder with a similar process, the alloys sintered at high temperature with this process possess much higher strength at both room temperature and elevated temperatures.

[8] SYNTHESIS OF NANOCRYSTALLINE MOSe₂ BY SONOCHEMICAL REACTION OF SE WITH MO(CO)₆

Kristl M. Drogenik M. - Inorganic Chemistry Communications. 6(1):68-70, 2003

Nanocrystalline MoSe₂ was prepared by a sonochemical reaction between Mo(CO)₆ and Se in decalin at 273 K in nitrogen atmosphere. The products were characterized by X-ray powder diffraction (XRD), transmission electron microscope (TEM), energy-dispersive X-ray spectroscopy (EDXS) and thermal analysis (TG and DSC). The XRD patterns showed that the product is amorphous, while annealing at 330degreesC yields nanocrystalline MoSe₂. The influence of ultrasound and temperature is discussed

[7] EFFECTS OF MECHANICAL TREATMENT ON PHASE TRANSFORMATION AND SINTERING OF NANO-SIZED GAMMA-Fe₂O₃ POWDER

Hsiang HI. Yen FS. - Ceramics International. 29(1):1-6, 2003.

The effects of mechanical treatment on the phase transformation and sintering of nano-sized gamma-Fe₂O₃ powder were studied. gamma-Fe₂O₃ powder was obtained by calcining iron tartrates at 300 degreesC. The mechanical treatment increased the contact areas in the gamma-Fe₂O₃ powder, which acted as nucleation sites for the gamma->alpha-Fe₂O₃ phase transformation, and resulted in lowering the transformation temperature. The greater surface area and fine equiaxed particle of the milled powder thus obtained were due to the vermicular microstructure development being inhibited. Consequently, the samples with mechanical treatment after sintering developed a uniform fine-grained microstructure



[6] RE-CO/AL₂O₃ BIMETALLIC CATALYSTS PREPARED BY MECHANICAL TREATMENT: CO HYDROGENATION AND CH₄ CONVERSION

Guczi L. Takacs L. Stefler G. Koppány Z. Borko L. - *Catalysis Today*. 77(3):237-243, 2002

Re-Co/Al₂O₃ (M) bimetallic catalysts were prepared by ball milling in a high-energy shaker mill using a WC container and four WC balls. The structure and activity of the Re-Co/Al₂O₃ (M) are compared to a Re-Co/Al₂O₃ (IM) sample prepared by the incipient wetness technique. The mechanically treated sample contains disordered, probably partly amorphous metal particles. Heat treatment of the Re-Co/Al₂O₃ (M) sample was carried out in hydrogen at 450 and 650 degreesC. It affects the crystallinity and the catalytic properties of the metal particles. The rate measured for the CO hydrogenation over Re-Co/Al₂O₃ (IM) catalyst is significantly higher than that measured over the Re-Co/Al₂O₃ (M) sample. On the other hand, in the CH₄ conversion to higher hydrocarbons, the Re-Co/Al₂O₃ (M) showed higher activity after treatment at 650 degreesC. The opposite behavior of the M and IM samples is explained by the changing morphology of the active sites responsible for the two different reactions.

[5] PREPARATION OF FUNCTIONAL MATERIALS VIA NON-CONVENTIONAL ROUTES

Senna M. - *Annales de Chimie-Science des Materiaux*. 27(6):3-14, 2002

An overview is given on the non-conventional solid-state processes for the preparation of functional materials. Emphasis is laid on the formation of heterogeneous bridging bonds (BBB) across the boundary between two dissimilar solids under mechanical stressing. Loss of the coordination number for the near surface atoms and/or disturbance of the symmetry of the crystal field or the ligand field are discussed in conjunction with the BBB formation. Case studies on complex oxides, ferrous coordination compounds and some organic - inorganic composites are given

[4] MECHANICALLY ACTIVATED POWDER METALLURGY PROCESSING: A VERSATILE WAY TOWARDS NANOMATERIALS SYNTHESIS

Gaffet E. Bernard F. - *Annales de Chimie-Science des Materiaux*. 27(6):47-59, 2002

The mechanical activation of powder metallurgy processing has been shown to lead to the synthesis of nanostructured micropowders (ERAM / M2AP process), homogeneous but porous nanomaterials (MASHS process). Recently, such an activation step before self heat sustaining reaction has been found to lead to the synthesis of fully dense bulk nanostructured materials (patented process, so called MAFAPAS

[3] NANOCRYSTALLINE MATERIALS PREPARED BY HOMOGENEOUS AND HETEROGENEOUS MECHANOCHEMICAL REACTIONS [REVIEW]

Sepelak V. - *Annales de Chimie-Science des Materiaux*. 27(6):61-76, 2002

At present, the high-energy milling method becomes widely used for the preparation of nanocrystalline materials due to its relative simplicity and availability. In this overview, selected examples are presented of the preparation of nanoscale materials by homogeneous and heterogeneous mechanochemical reactions, in spinel ferrites. Despite numerous efforts, the understanding of the nonequilibrium mechanochemical processes is considered to be far from complete, leaving large scope for further research in this exciting field.

[2] SOFT MECHANOCHEMICAL SYNTHESIS: PREPARATION OF CATHODE MATERIALS FOR RECHARGEABLE LITHIUM BATTERIES

Kosova N. Devyatkina E. - *Annales de Chimie-Science des Materiaux*. 27(6):77-90, 2002

To prepare intercalation lithium - transition metal oxide cathode materials for rechargeable lithium batteries, the reactions in the mixtures of the correspondent hydroxides in highly energetic planetary activators, so called 'soft mechanochemical synthesis', were studied. The method can be used for direct preparation of final products in a high dispersed and disordered state, as well as for obtaining high reactive precursors yielding final products by the subsequent brief heating, at considerably lower temperatures as compared to conventional ceramic method. The as prepared products were analyzed using X-ray diffraction, TG, IR, XPS, Li-7 NMR, EPR, diffuse reflectance spectroscopy, electron microscopy, BET, and electrochemical measurements. The peculiarities of crystal structure, electronic state of transition metal ions and cycling behaviour of materials are discussed. The method as proposed is concluded to be economically effective and ecologically clean.

[1] ROLE OF MOLECULAR STRAIN ON THE SOLID-STATE SYNTHESIS OF COORDINATION COMPOUNDS FROM IRON(II) CHLORIDE TETRAHYDRATE AND 1,10-PHENANTHROLINE UNDER MECHANICAL STRESS

Ohshita T. Nakajima D. Tsukamoto A. Tsuchiya N. Isobe T. Senna M. Yoshioka N. Inoue H. - *Annales de Chimie-Science des Materiaux*. 27(6):91-101, 2002

A powdered crystalline mixture of iron(II) chloride tetrahydrate (FeCl₂·4H₂O) and 1,10-phenanthroline (phen) was subjected to mechanical stressing in Ar by a planetary ball-mill. By milling for 3h, the mixture turned completely non-crystalline. New IR absorption bands appeared simultaneously at 358cm⁻¹ due to Fe-N(phen) stretching and at 207cm⁻¹ due to N-Fe-N(phen) bending for [Fe(phen)₃]²⁺. Only a doublet peak due to [Fe(phen)₃]Cl₂·nH₂O was detected in the Mossbauer spectrum. All these results indicate consistently the formation of [Fe(phen)₃]Cl₂·nH₂O by milling with an almost quantitative yield. Disproportionation of the hydrated water molecules was observed after milling FeCl₂·4H₂O alone for 3h. The molecular strain triggers a solid-state exchange reaction between H₂O and phen at the contact point of two dissimilar solid particles under mechanical stressing, and promotes formation of [Fe(phen)₃]Cl₂·nH₂O in the solid state.

