

RESEAU FRANCAIS DE MECANOSYNTHESE

Lettre N°27

Juin 1997

75 (+4) Groupes de Recherches (16 Etrangers) - 105 (+10) Correspondants

Bureau : E. Gaffet (Président), G. Le Caër (Secrétaire Général), A.R. Yavari (Trésorier)

10 Nouvelles Adhésions

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M. Sarfati - ETCA - France
H. Souma - L2RS - France
R. Souza de Figueiredo - Lab. Magnetismo et Materiais Magneticos - Brésil

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Le RFM a entamé sa Troisième Année d'existence,
Merci de penser à vos adhésions au titre de l'Année 1997.
Les membres du RFM ayant déjà réglé leur cotisation sont indiqués sur l'annuaire par (1997)

Bulletin d'adhésion **1997** (à retourner à l'adresse suivante) :

Eric GAFFET

UPR CNRS 423 - Groupe "Elaboration et Transitions de Phases Hors Equilibre" - IPSé - F90010 - Belfort Cedex

Nom : Prénom :

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Renseignements complémentaires :

Téléphone : Télécopie :

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désire adhérer au Réseau Français de Mécanosynthèse

(Joindre un chèque de 100 F, à l'ordre du Réseau Français de Mécanosynthèse, représentant la cotisation pour l'année 1997)

à

le

1997

(Signature)

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ANNONCE DE CONGRES ET / OU ECOLES

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Congrès Société Européenne de Céramique

Versailles - Juin 1997 - Contact : Groupe Français de la Céramique - Fax : 05 55 79 09 98 (Limoges)

International Conference on Fatigue of Composites

3 - 5 Juin 1997 - Paris - Org. SF2M

Développement Récents en Fatigue des Matériaux et des Structures

Ecole d'Eté - St Pierre d'Oléron - 11 18 Juin 1997

Contact : D. Francois - Ecole Centrale de Paris - E-Mail : francois@mssmat.ecp.fr

ICAM'97 et E-MRS'97

Strasbourg 16 - 20 Juin 1997

Cont. : P. Siffert - E-MRS1997 Spring Meet. - BP 20, 67037 Strasbourg Cedex 2 - Fax : 03 88 10 63 43 -
EMRS@FRCPN11.IN2P3.FR

V International Workshop on Non - Crystalline Solids

2 - 5 Juillet 1997 - Santiago - Espagne

Secretariat : Paula Achermann - Viajes Atlantico, S.A. - Pl. Fuenterrabia, 3 - APratada 136 - E - 15702 - Santiago de
Compostela-Espagne : Fax : +34 - 81 57 28 67 - E-Mail : fajesua@uscmail.usc.es

Thermal spray processing of Nanoscale Materials

Davos - 3 - 8 Août 1997

Contact : E. Lavernia (Irvine - USA) E-Mail : E.Lavernia@uci.edu

2nd International Conference on Mechanochemistry and Mechanical Activation (INCOME - 2)

Novosibirsk - Russie - 12 - 16 Août 1997

Contact : Prof. N. Lyakhov - Institute of Solid State Chemistry - Kutateladze 18 - Novosibirsk 630128 - Russie
E-Mail : Conf@solid.nsk.su - Fax : 7 (383) - 2) 32 28 47 - Tel. 7 (383 - 2)32 86 83

ISMAM97

Sitjes (Barcelone) - 31 Août - 5 Sept. 1997

Contact : M.D. Baro Fax : (+34) - 3 - 581 - 2155 - E-Mail : Ismanam97@cc.uab.es

Mechanical Behavior of Bulk Nanocrystalline Solids"

Indianapolis - TMS Fall Meeting - 14 - 18 Septembre 1997

Contact : Naresh.Thadhani@msi.gatech.edu

EUROSOLID 4

European Conference on Transformation Kinetics and Reactivity of Solids

St Vincent - Vallée d'Aoste - Italie 15 - 16 Septembre 1997

Contact : L. Montanaro - General Secretary of EUROSOLID 4 - Dipartimento di Scienza dei Materiali ed Ingegneria
Chimica - Politecnico - Corso Duca degli Abruzzi, 24-I-10129 Torino - Italie
E-Mail : Negro@athena.polito.it

**Colloque National sur les Contraintes Résiduelles dans les procédés d'élaboration des
Céramiques et des Traitements de Surface**

NOUVEAU

Limoges - France 23 - 24 Septembre 1997

Contact : Prof. F. Nardou - LMCTS / CNRS ESA 6016 - Faculté des Sciences - 123 Avenue
A. Thomas - 87060 Limoges Cedex - France - Tél : 05 55 45 74 87 - Fax 05 55 45 75 86 -
E_MAIl : Nardou@unilim.fr

2nd Int. Symposium on Structural Intermetallics

Champion (PA - USA) - 21 - 25 Septembre 1997

Contact : B. Kamperman , T.M.S., 420 Commonwealth, Warrendale, PA 15086 - E-Mail : Kamperman@tms.org

2nd European Meeting on Integrated Ferroelectrics

Jouy en Josas - France - 29 - 30 Septembre 1997

Contact : P.Abelard@ensci.fr

4th Int.Symp. on Self Propagating High Temperature Synthesis

Toledo - 6 - 10 Octobre 1997

Contact : Institut de Céramique et du Verre - Fax (+34) 1 - 870 - 05 - 50

**4th Int. Symposium on Electrochemical / Chemical Reactivity
of Amorphous and Nanocrystalline Alloys**

Dresden - 8 - 10 Octobre 1997

Contact : L. Shultz / A. Gebert - Institute of Solid State and Materials Research Dresden - P.O. Box 270016 - 01171
Dresden - Allemagne : Fax : +(49) 351 - 4659 - 541 - e-Mail : Schultz ou gebert @ifw-dresden.de

European Conference on Advances in Structural PM Component Production - PM97

Munich - 15 - 17 Octobre 1997

Contact : Euro PM97 Conference Secrétariat - European Powder Metallurgy Association
OLD Bank Buildings, Bellstone, Shrewsbury SY1 1HU, UK-Fax : +44 1743 362968 - E-Mail :
epma@dial.pipex.com

JA97

Paris - Maison de la Chimie - 25 - 27 Novembre 1997

Coordinateur SF2M: ean - Marie DUBOIS (LSG2M - Nancy)

1A Les Matériaux de l'Automobile

Animateurs P. Antona (Fiat - Turin) et G. Maeder (Renault)

1B - Mise en forme des polymères : incidence sur les structures et propriétés

Animateurs : B. Escaig et J.M. Lefebvre de l'Université de Lille et J. Pabiot de l'Ecole des Mines de Douai.

1C - Aciers spéciaux : présent et futur

Animateurs : G. Béranger, UTC, G. Labbe et P. Mazars, Usinor-Sacilor

2A Nanomatériaux et Nanostructures : architectures ultim, phénom. fondam. fonct. & appl.

Animateurs : E. Gaffet (Belfort), P. Panissod (Strasbourg), H.-E. Schaeffer (Stuttgart), L. Schultz (Dresden)

2B - Utilisation des techniques spectroscopiques pour l'étude d'interfaces, de défauts et de ségrégation dans les matériaux.

Animateurs : C. Senemaud, Université P. et M. Curie et A. Traverse, LURE

3A - Thermodynamiques numérique et expérimentale.

Animateurs : C. Bichara, CTM Marseille et C. Chatillon, LTPCM Grenoble

3B - Transferts thermiques dans les matériaux : des puits de chaleur aux barrières thermiques.

Animateurs : R. Mévrel, ONERA et P. Pigeat, Centre d'Ingénierie des Matériaux, Nancy

MRS Fall Meeting 97

Symposium B : Phase Transformations and Systems Driven Far From Equilibrium"

Boston - 1 - 5 December 1997

Org. : M. Atzmon, P. Bellon, E. Ma, R. Trivedi **Contact :** MRS Website

The 7th Joint MMM-Intermag Conference

San Francisco - 6 - 9 Janvier 1998

Web Site : <http://www.aip.org/edops/mmmconf.html>

Society of Automotive Engineers Congress

Cobo Center - Detroit - Michigan - USA - 24 Février 1998

E-Mail : SAE@SAE.Org

29èmes Journées de Cinétique Hétérogène

Université Paris XI - Orsay - Printemps

Org. : A.M. Huntz **Contact :** G. Thomas (Thomas@emse.fr)

Colloque National de Métallurgie des Poudres

Efets de la microstructure et de la porosité sur les propriétés mécaniques et physiques des matériaux MdP

Grenoble - 6 - 8 Avril 1998 - Org. SF2M

CIMTEC'98 - World Ceramics Congress and Forum on New Materials

Florence - 14 - 19 Juin 1998

Web Site : <http://www.dinamica.it/cimtec98/>

Congrès Européen sur le Broyage

Albi - 8 - 10 Septembre 1998 - sous l'égide de l'European Federation of Chemical Engineering

Contacts : P. Guigon et J. Dodds

Powder Metallurgy 98

Granada - Espagne - 18 - 22 Octobre 1998

Site Web : <http://www.epma.com/congress/>

Bibliographie Récente

N.B. : En cas de difficultés à vous procurer une copie des articles suivants,
n'hésitez pas à contacter E. Gaffet (CNRS / IPSé - Belfort)

Livres ou "Special Issues"

"Chemical MechanoSynthesis of Nanomaterials"

The International Journal of Non - Equilibrium Processing - Guest Editor : E. Gaffet - disponible 1998

Editeur : A.L. Greer, Editeurs Associés : M. Atzmon, L. Battezzati, M. Umemoto

"Mécanosynthèse"

Les Annales de Chimie - Science des Matériaux - Coordinateur G. Le Caër (1997) - disponible Juillet 97

Les Matériaux à Grains Ultrafins produits par Hypercorroyage"

Les Annales de Chimie - Science des Matériaux - Coordinateur R.Z. Valiev (1997)

Proceedings International conference on Nano Clusters and Granular Materials Sendai (1995)

Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 217:R 11, 1996

Proceeding du Congrès "Mechanically Alloyed and Nanocrystalline Materials" - Rome (1996)

Editor : D. Firoani, M. Magini - Materials Science Forum - Volumes 235 - 238 (1997)

Proceeding du Congrès "Mechanically Alloyed and Nanocrystalline Materials" - Québec (1995)

Editor : R. Schulz - Materials Science Forum - Volumes 225 - 227 (1996)

"Mechanical Properties & Deformation Behavior of Materials having Ultra-fine Microstructures"

Ed. M. Nastasi, D.M. Parkin, H. Gleiter - Nato ASI Series. Ser. E : Appl. Sci. Vol. 233 (1993)

Proceeding du Congrès "Mechanically Alloyed and Nanocrystalline Materials" - Grenoble (1994)

Editor : A.R. Yavari - Materials Science Forum Volumes 179 - 181 (1995)

"Mechanochemistry of Solid Surfaces"

E.M. Gutman (Ben - Gurion University of the Negev) - World Sci. Pub Co. Pte. Ltd (1994) - ISBN 981-02-1781-1

Thèses Etrangères (disponible sur demande auprès de E. Gaffet)

"Microstructure and phase transformation in mechanically alloyed materials" (En Anglais)

J.Y. - Huang - Lab. Atomic Imaging of Solids - Shenyang - Chine - Systèmes Etudiés : Cu, Co, Fe - Cu, Ti - Ni - C

Périodiques (Rubrique assurée en partie grâce au concours de Mme TAUZIN - FIN BiPSé)

THE MECHANOCHEMICAL SELF-PROPAGATING REACTION BETWEEN HEXACHLOROBENZENE AND CALCIUM HYDRIDE

Mulas G. Loiselle S. Schiffini L. Cocco G. - Journal of Solid State Chemistry. 129(2):263-270, 1997

We report on studies of the solid state reaction between hexachlorobenzene and calcium hydride carried out by high-energy ball milling. The transformation behavior depends on the intensity of the mechanical energy transferred to the reactants at the impact. At lower energy regimes, chemical conversions increase gradually as a function of the milling time and a large excess of calcium hydride was found to favor the reaction rate. Calcium hydride-chloride and benzene are formed as end products. Beyond an impact energy threshold, self-sustaining transformations were observed leading to an instantaneous transformation to hydrogen, graphite, and, depending upon the reactant molar ratio, to calcium hydride-chloride or calcium chloride. The sudden increase of the reactor-vial temperature was proportional to the hexachlorobenzene content in the reacting mixture and the total heat evolved was found to be in good agreement with the forecasted reaction enthalpies. The ignition time, i.e., the milling time at which the combustion-like event occurs, was followed as a function of the reactant composition. The incubation period rapidly decreases by increasing the calcium hydride to hexachlorobenzene molar ratio, that is, moving away from the stoichiometric composition at which calcium chloride forms predominantly. Some suggestions concerning the activation energy of the two competing end products were inferred from the mechanochemical yield which has been calculated as the ratio between the moles of reacted hexachlorobenzene divided by the total injected energy dose. Keeping the molar composition constant and modulating the shock power intensity, the self-sustaining reaction takes place only when the same dose of mechanical energy has been supplied to the reacting system, irrespective of the single impact energy.

GRAIN GROWTH IN NANOCRYSTALLINE IRON PREPARED BY MECHANICAL ATTRITION

Malow TR. Koch CC. - Acta Materialia. 45(5):2177-2186, 1997 May.

The grain growth in nanocrystalline Fe produced by high energy ball milling is investigated. Grain growth data are analysed using two different models of grain growth, one of which takes pinning forces on the grain boundaries into account. The grain growth exponents n from $D-l/n - D-o(l/n) = kt$ for nanocrystalline and conventional polycrystalline Fe are compared. The use of the above mentioned equation yields an activation energy of 125 kJ/mol, while the second model gives 248 kJ/mol. These Values are compared to those for grain boundary and lattice diffusion in Fe. Some evidence for two different sets of mechanisms governing the grain growth in nanocrystalline Fe are discussed.

CHARACTERIZATION OF HARD MAGNETIC TWO-PHASE MECHANICALLY ALLOYED SM₂FE₁₇N₃/ALPHA-FE NANOCOMPOSITES

Odonnell K. Coey JMD. - Journal of Applied Physics. 81(9):6310-6321, 1997

A range of exchange-coupled two-phase nanocomposites composed of hard magnetic Sm₂Fe₁₇N₃ and soft magnetic alpha-Fe was prepared by mechanical alloying with a view to optimizing the hysteresis loop shape. The main variables were the crystallization conditions, the nitriding treatment, and the chemical additives. A model of the diffusion of nitrogen in the two-phase nanocomposite is proposed that explains how the presence of Fe permits the nitrogenation of samples at lower temperatures than in single phase Sm₂Fe₁₇ materials. Studies of samples both resin bonded and cold compacted measured in open and closed circuits revealed that the correct choice of demagnetizing factor used to correct demagnetizing fields depends critically on the sample density. Transmission electron microscopy (TEM) studies of the materials prepared revealed grain sizes in the range 10-50 nm. The shape of the magnetic hysteresis loop and resulting magnetic properties reflects the grain size of both phases. Image analysis of high resolution scanning electron microscopy micrographs of etched samples showed that in general two to three soft grains cluster together and are surrounded by hard grains, but the grain sizes of both phases were found to be the same. The crystallization of the hard phase from the mainly amorphous precursor is the primary factor determining grain size. Zr and Ta were the most successful additives in controlling the grain growth during crystallization,

reducing the grain size from 20-30 to 10-20 nm. High resolution TEM indicated the presence of a grain boundary phase between the crystallites of two phases. This phase was confirmed in Mossbauer studies of samples where it seems to constitute 15 vol % of the samples and has a significant effect on the coupling between the two phases. Susceptibility measurements are an effective indicator of the degree of coupling between the hard and soft magnetic phases

EFFECT OF BALL-MILLING ON T-C VALUES OF BI-BASED BULK SUPERCONDUCTORS

Yavuz M. Mikheenko PN. Dou SX. Vance ER. - Czechoslovak Journal of Physics. 47(4):481-484, 1997

Superconducting powders were synthesised by solid-state reaction. They were ball-milled, and pressed into pellets. It was found that particle size prior to sintering has a substantial influence on the properties of Bi-based bulk superconductors, such as reactivity, phase homogeneity and T-c values. Powders of fine particle size were found to show low T-c values.

EFFECT OF IMPURITIES FROM BALL MILL ON DENSIFICATION BEHAVIOR OF ALPHA-Fe₂O₃ POWDERS

Nippon Seramikkusu Kyokai Gakujutsu Ronbunshi-Journal of the Ceramic Society of Japan. 105(4):361-365, 1997

Alpha-Fe₂O₃ powder was ground by ball-mill with alumina and zirconia balls. Densification behavior with respect to both specific surface area and the amount of impurities from the balls was investigated. The onset temperature of shrinkage for the powders milled with alumina balls decreased with increasing specific surface area and decreasing impurity contents. On the other hand, the onset temperatures for the powder milled with zirconia balls increased, although the specific surface area of the powder was increased by milling. Density and microstructure of the samples sintered at 1100 degrees C for 10h were evaluated. Relative densities of sintered samples by the powders with 3.5 mol% and 1.1 mol% Al₂O₃ were 99.4% and 99.3%, respectively. Average grain sizes of these samples were 5.4 μm and 10.1 μm, respectively. The relative density of sintered sample by the powder with 0.7 mol% ZrO₂ was 97.8%. This value was lower than those by the powders milled with alumina balls, although there was no difference in specific surface area in these powders. This result indicates that Al₂O₃ does not influence the densification of alpha-Fe₂O₃, however ZrO₂ inhibits densification. The cause of densification inhibition by ZrO₂ was considered that the diffusion rate of oxygen ion is decreased by the decrease of oxygen ion vacancies, which is caused by the dissolution of ZrO₂ in alpha-Fe₂O₃.

FORMATION OF TiAl/Ti₅Si₃ NANOCOMPOSITE BY MECHANICAL ALLOYING AND SUBSEQUENT ANNEALING

Liu KW. Zhang JS. Wang JG. Chen GL. - Scripta Materialia. 36(10):1113-1117, 1997 May 15.

MOSSBAUER ANALYSIS OF THE PHASE DISTRIBUTION OF Fe_{64.5}Co₁₈B₁₆SiC_{0.5} SOFT MAGNETIC SAMPLES IN POWDER FORM

Partiti CSM. Rechenberg HR. Gonzalez J. Tello PG. Gonzalez JM. - Journal of Applied Physics. 81(8 Part 2A):4655-4657, 1997

We report on data, obtained from the analysis of Mossbauer spectra, on the phase distribution present in FeCoBSiC samples which were prepared by milling a melt spun amorphous precursor in a high energy ball mill. The milling process reduced the material to a powdered form, induced the precipitation of FeCo and Fe₃C crystallites and resulted in relatively high coercive force values. In contrast with this, short time thermal treatments, carried out in the as-milled samples at temperatures in the range from 575 up to 650 K, produced a very remarkable softening which, according to our phase distribution results, should be associated to the partial dissolution of a certain percentage of the atoms involved in the grain boundary structures. This resulted in improved coupling between the crystalline and amorphous phases forming the sample and consequently, in an easier displacement of the domain walls.

STRUCTURE AND MAGNETISM OF MULTIPHASE Sm_{0.080}Co_{0.645}Fe_{0.276} POWDERS

Harris VG. Liou M. Das BN. Browning VM. Snyder JE. Rubinstein M. Lawrence SH. Littleton R. Pappas DP. - Journal of Applied Physics. 81(8 Part 2B):5121-5123, 1997

A Sm-poor mixture of Fe-substituted Sm₂Co₁₇, having the nominal stoichiometry of Sm_{0.080}Co_{0.645}Fe_{0.276}, was ball-milled to explore the possibility of enhancing its remanence through direct microstructural refinement. With milling, the Sm-2(Co_{0.7}Fe_{0.3})(17) compound disassociates to a body-centered-cubic supersaturated SmCoFe solid solution and a residual SmCoFe amorphous phase. Correspondingly, the coercive field values first increase, peaking at 0.83 kOe after 180 min of milling, then decrease with continued milling to <0.1 kOe after 1200 min. The remanence, as M_r/M_s, is measured to track closely the coercive field behavior, experiencing modest increases to 0.26 after just 120 min of milling. Magnetization values are found to increase first with the dissociation of the 2:17 phase, and again with the ejection of Sm from the bcc component.

INFLUENCE OF RAW MATERIALS AND MILLING TECHNOLOGICAL PROCESS ON MAGNETIC PROPERTIES OF SINTERED ANISOTROPIC Sr-FERRITE MAGNET

Teng YM. Lu BS. Yao JZ. - Journal of Applied Physics. 81(8 Part 2B):5134-5136, 1997

It is necessary to reduce the Sr-ferrite grain size below 1 μm for high coercivity, besides a high degree of density and orientation for high residual induction. However, submicron Sr-ferrite particles are difficult to orient in a

magnetic field at pressing stage because of their cohesion. For preventing this cohesion, treatments with dispersants and reducing coercive force of S-ferrite particles are very effective. In this research, a study was done correlating purity and aggregation state of raw materials with intrinsic coercive force and microstructure of presintered Sr-ferrite sample, and a presintered sample with uniform grain size distribution and lower coercivity was gained. The classical pulverizing process is composed of coarse dry milling and fine wet milling. The results confirm that compared with wet milling, a long dry milling time promotes the development of crystal deformation and reduces the coercive force of the powders effectively; hence the viscosity is also lowered. By enhancing dry-milling time to control average particle size under 1.8 μm and fine wet milling up to 0.75 μm with optimum amount of dispersant, a well dispersed Sr-ferrite slurry is prepared. By these methods, a remarkable improvement of magnetic properties was achieved.

COERCIVITY AND TRANSMISSION ELECTRON MICROSCOPY STUDY OF NANOCOMPOSITE SM-CO POWDERS BY MECHANICAL ALLOYING

Chen SK. Tsai JL. Chin TS. - Journal of Applied Physics. 81(8 Part 2B):5631-5633, 1997

A nanocomposite $\text{Sm}_2\text{Co}_{17}/\text{Co}$ magnet with a composition of Sm_7Co_9 was prepared by mechanical alloying. A domain-wall pinning mechanism was found to be dominant in the magnet, as investigated using Kronmuller's micromagnetic theory. Transmission electron microscopy micrographs indicate that the half width between neighboring Co grains is about 40 nm. This value is two times larger than the range factor, r_0 , obtained from Kronmuller's model. It appears that crystallographic defects other than the cobalt particles also play an important role in coercivity.

VARIATION OF MAGNETISM AND STRUCTURE ON MECHANICALLY ALLOYED $\text{Fe}_{40}\text{Co}_{10}\text{Cu}_{50}$ POWDER WITH PROCESS TIME AND HEAT TREATMENT

Yu SC. Yoo YG. Kim WT. Anderson C. Dickson D. Zeiske T. - Journal of Applied Physics. 81(8 Part 2B):5799-5801, 1997

The effects of milling time and heat treatment on local structural changes during mechanical alloying $\text{Fe}_{40}\text{Co}_{10}\text{Cu}_{50}$ have been investigated by means of x-ray diffraction (XRD), neutron diffraction technique, vibrating sample magnetometer, and Mossbauer spectroscopy. Mossbauer spectroscopy showed that two kinds of magnetic phases coexist in the mechanically alloyed powder, which is in agreement with XRD and neutron diffraction study. One is corresponding to $\alpha\text{-Fe}$ and the other is a new magnetic phase formed during mechanical alloying. With increasing processing time, the new magnetic phase becomes more ordered and dominant. Mossbauer spectrum from the annealed sample consists of ferromagnetic component with a hyperfine field of 36.2 T and a paramagnetic component.

STUDY OF MECHANICAL ALLOYING OF SM AND FE

Seifu D. Oliver FW. Hoffman E. Aning A. Babu VS. Seehra MS. Catchings RM.- Journal of Applied Physics. 81(8 Part 2B):5805-5807, 1997

Mechanical alloying of Sm and Fe with the composition of SmFe_3 was studied using x-ray-diffraction (XRD), Mossbauer, and magnetization measurements. Data taken as a function of milling time for up to 20 h show significant changes occurring during ball milling. The XRD studies show that the initial crystalline Bragg reflections changed to a broad maximum, which is attributed to the formation of an amorphous phase. The initial six-line pattern in the Mossbauer spectrum, characteristic of magnetic ordering, changed to a broad singlet, characteristic of a nonmagnetic material. Magnetization measurements revealed that the coercive field was at its maximum after 5 h of milling and decreased sharply as the milling time increased. The remanent magnetization was at its maximum between 5 and 10 h of milling. The final product of the ball milling, which exhibited the characteristics of an amorphous paramagnetic material in its XRD and Mossbauer spectrum, was studied after heat treatment. The XRD and the Mossbauer spectra of the heat treated alloy show that substantial changes occurred during heat treatment in that sharp Bragg reflections, characteristic of crystalline materials, reappear and the alloy changed from a paramagnetic to a ferromagnetic state.

INVESTIGATION OF THE TERNARY PHASE DIAGRAM OF MECHANICALLY ALLOYED FeCuAg

Cohen NS. Ahlswede E. Wicks JD. Pankhurst QA.- Journal of Physics-Condensed Matter. 9(15):3259-3276, 1997

The structural and magnetic properties of mechanically alloyed Fe-Cu-Ag at room temperature have been investigated using Fe-57 Mossbauer spectroscopy, x-ray diffraction and differential scanning calorimetry. The elements are naturally immiscible, but through prolonged and energetic ball milling (70 hours at 600 rpm) one can make metastable alloys, the structure of which depends on the elemental composition. In the binary Cu-Ag and Fe-Cu systems, crystalline single-phase solid solutions result, whereas in Fe-Ag the alloying is limited, with the product a mixture of elemental particles. In the ternary system it is possible to produce copper- and silver-rich single-phase Fe alloys, but not the equivalent bcc iron-based structure. As the proportions of the three elements become more equal, the resulting structure becomes highly disordered or amorphous. The composition range of this amorphous phase is different to that observed in sputtered Fe-Cu-Ag systems.

BALL MILLING ASSISTED LOW TEMPERATURE FORMATION OF IRON-TIC COMPOSITE

Chen Y.- Scripta Materialia. 36(9):989-993, 1997

OBSERVATION OF NITROGEN INDUCED AMORPHIZATION OF THE V-CU SYSTEM BY EXAFS, X-RAY AND NEUTRON DIFFRACTIONS

Fukunaga T. Mizutani U. - Science Reports of the Research Institutes Tohoku University Series A-Physics Chemistry & Metallurgy. 43(2):129-134, 1997

Mechanical alloying (MA) of V70Cu30 powders which is characterized by a positive heat of mixing was carried out under the N₂ gas atmosphere. The structure factor S-X(Q) observed by X-ray diffraction gradually changes into a structure characteristic of an amorphous state with increasing milling time. This indicates the amorphization induced by nitrogen. The S-N(Q) by neutron diffraction after 200 hours of milling also confirmed the formation of an amorphous phase with the residual pure Cu. Since the coherent scattering length of the V atom in the neutron diffraction is negligibly small, crystalline peaks can be easily identified as the fee Cu. A drastic change of the EXAFS profile around a V atom was more clearly observed than that around a Cu atom. These results allow us to conclude that the bcc solid solution is formed at the early stage of milling and then that the amorphization gradually proceeds in the subsequent stage of milling by incorporating N atoms into the bcc solid solution.

SOLID STATE AMORPHIZATION REACTION BY ROD-MILLING ALXTA1-X POWDERS AND THE EFFECT OF ANNEALING

Eleskandarany MS. Aoki K. Sumiyama K. Suzuki K.- Science Reports of the Research Institutes Tohoku University Series A-Physics Chemistry & Metallurgy. 43(2):135-143, 1997

High thermal stable amorphous Al_xTa_{1-x} alloy powders with wide amorphization range (10 less than or equal to x less than or equal to 90) have been synthesized by rod-milling technique using a mechanical alloying (MA) method. During the first few kiloseconds (11-173 ks) of the MA time, the layered-composite particles of Al and Ta are intermixed and form an amorphous phase upon heating at about 680 K in a differential thermal analyzer by thermally assisted solid state amorphization (TASSA). The heat formation of an amorphous Al_xTa_{1-x} alloy via the TASSA process, Delta H-aT has been measured as a function of the MA time. The crystallization characteristics indexed by the crystallization temperature, T-xT and the enthalpy of crystallization, Delta H-xT of the amorphous phase formed via the TASSA process are also investigated as a function of the MA time. Comparable with the TASSA process, a homogeneous amorphous Al_xTa_{1-x} alloy is formed after longer MA time (1080 ks). The amorphization process in this case is attributed to a mechanical solid state amorphization (MDSSA). At the end of the MA time (1080-1440 ks), the maximum heat of formation of an amorphous Al_xTa_{1-x} alloy via the MDSSA process, Delta H-aM, has been calculated. Moreover, the thermal stability characterized by the crystallization temperature, T-xM and the enthalpy of crystallization, Delta H-xM, are also estimated. The role of amorphization via each process has been discussed.

MECHANICALLY INDUCED CARBONIZATION FOR FORMATION OF NANOCRYSTALLINE TIC ALLOY

Eleskandarany MS. Omori M. Kamiyama T. Konno TJ. Sumiyama K. Hirai T. Suzuki K. - Science Reports of the Research Institutes Tohoku University Series A-Physics Chemistry & Metallurgy. 43(2):181-193, 1997

A single phase of NaCl-type structure of Ti₄₄C₅₆ alloy powder has been synthesized by ball-milling elemental Ti and graphite powders at room temperature. The end-product of Ti₄₄C₅₆ that is obtained after 720 ks of milling consists of fine grains of about 3 nm in diameter and possesses homogeneous powder with an average particle diameter of less than 0.4 mu m. The milled powder has been consolidated into bulk samples, using a plasma activated sintering method. This consolidation step leads to the formation of fully-dense TiC compacts with nano-structure grains. The as-milled powder and the as-consolidated bulk samples have been characterized after selected milling times by means of X-ray diffraction, transmission electron microscope, scanning electron microscope and chemical analyses. Some of the compacted samples were investigated by small-angle X-ray scattering and high-resolution transmission electron microscope. The hardness and some mechanical properties of the end-product are reported. On the basis of the results of the present study, the ball-milling technique accompanied with plasma activated sintering can provide powerful tools for fabrication of nanocrystalline TiC bulk alloys.

CONSOLIDATION OF NANOSTRUCTURED METAL POWDERS BY RAPID FORGING - PROCESSING, MODELING, AND SUBSEQUENT MECHANICAL BEHAVIOR

Shaik GR. Milligan WW.- Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 28(3A):895-904, 1997

Fe-10Cu powders containing 20-nm grains were produced by attritor milling of elemental powders in argon. A rapid powder forging technique was developed to consolidate the powders into fully dense compacts while maintaining nanoscale grain sizes. Grain growth during the consolidation was controlled by reducing the time of exposure at elevated temperature to a few minutes or less, a technique which is applicable to all materials and does not necessitate the addition of dispersoids. This was achieved by heating green compacts quickly using an induction heater, and then forging and rapidly cooling them back to room-temperature; Forging was conducted in a protective argon atmosphere to limit contamination. Fully dense compacts were produced at relatively low temperatures, mainly due to the accelerated creep rates exhibited by the nanostructures. Transmission electron microscopy and X-ray diffraction analysis found an average grain size of 45 nm in the fully dense samples forged at 530 degrees C. Indications are that

finer grain sizes should be attainable by using slightly lower temperatures and higher pressures. The success of the technique (compared to hot-isostatic pressing ("hipping")) is due to both reducing time at elevated temperatures and applying relatively high pressures. Microhardness tests revealed a significant strengthening effect due to grain size refinement, following a Hall-Petch relation. Compression testing at room temperature showed no strain hardening during plastic deformation, which occurred by shear banding. High strengths, up to 1800 MPa, were obtained at room temperature. Compression testing at 575 degrees C revealed a significant strain rate dependence of mechanical behavior and also the possibility of superplastic behavior. Power-law creep was observed at 575 degrees C, with very high steady-state creep rates on the order of 50 pct/s at 230 MPa. The consolidation process was successfully modeled by slightly modifying and applying the Arzt, Ashby, and Easterling (AAE) hot-isostatic press (HIP) model. The experiments and modeling indicated that creep was the dominant densification mechanism in these materials, even at relatively low temperatures and high loading rates. The results of this investigation suggest the possibility of a commercially viable nanostructured metal, which is easily processed to large strains at moderate temperatures, yet maintains high strength at room temperature without the necessity of heat treatment or mechanical working.

CYCLIC CRYSTALLINE-AMORPHOUS TRANSFORMATIONS OF MECHANICALLY ALLOYED CO₇₅Ti₂₅

Eleskandarany MS. Akoi K. Sumiyama K. Suzuki K. - Applied Physics Letters. 70(13):1679-1681, 1997

We have found that a cyclic crystalline-amorphous phase transformation can occur in Co₇₅Ti₂₅ alloy powder when subjected to ball milling. The results have shown that a single amorphous phase of Co₇₅Ti₂₅ is obtained after 11 ks of mechanical alloying (MA) time. This amorphous phase transforms into a new metastable phase of bcc-Co₃Ti upon milling for 86 ks. The bcc-Co₃Ti is thermally stable and does not transform to any other phase(s) upon heating up to 1300 K. It however returns to the same amorphous phase of Co₇₅Ti₂₅ upon milling for 360 ks. Further milling leads to the formation of crystalline and/or amorphous phases depending on the MA time.

HARD MAGNETIC PROPERTIES OF THE NOVEL COMPOUND SM-3(Fe,Cr)₍₂₉₎N-Y

Wang YZ. Hu BP. Liu GC. Li HS. Han XF. Yang CP. - Journal of Physics-Condensed Matter. 9(13):2787-2791, 1997

The novel nitride Sm₃Fe₂₄Cr₅Ny with monoclinic Nd-3(Fe, Ti)₍₂₉₎ structure is synthesized by gas-solid reaction. Its hard magnetic properties have been investigated by ball milling and magnetic measurements. The intrinsic coercivity $\mu(0i)H(c)$ increases with decreasing average particle size d and, after reaching a maximum at $d = 0.4 \mu m$, then decreases slowly. $\mu(0i)H(c)$ increases also with increasing magnetizing field. A value of $\mu(0i)H(c) = 0.79 T$ has been attained. The magnetizing field for obtaining coercivity saturation is about 1.5 T. The hard magnetic properties of remanence $B-r = 0.87 T$ and energy product $(BH)(max) = 104.8 kJ m(-3)$ for Sm₃Fe₂₄Cr₅Ny are achieved at 293 K.

HARD MAGNETIC PROPERTIES OF THE NOVEL COMPOUND SM-3(Fe,Cr)₍₂₉₎C-Y

Authors Wang YZ. Hu BP. Liu GC. Li HS. Han XF. Yang CP. - Journal of Physics-Condensed Matter 9(13):2793-2798, 1997

The novel carbide Sm₃Fe₂₄Cr₅Cy with the monoclinic Nd-3(Fe, Ti)₍₂₉₎ structure is synthesized by gas-solid reaction. Its hard magnetic properties, temperature stability, and corrosion resistance have been investigated by conventional methods. The hard magnetic properties were developed by the ball-milling technique. An intrinsic coercivity $\mu(0i) H-c$ of 0.80 T at 293 K has been obtained. The temperature coefficient of the remanence is worse than that of Nd-Fe-B, while the temperature coefficient of the coercivity is better. The carbide powder has good corrosion resistance in comparison with Nd-Fe-B powder.

STRUCTURE AND CHEMICAL REACTIVITY OF BALL-MILLED GRAPHITE

Hermann H. Schubert T. Gruner W. Mattern N. - Nanostructured Materials. 8(2):215-229, 1997

Nanostructured carbon powders are prepared from graphite powder in a planetary mill using WC, stainless steel, and agate milling tools. The evolution of microstructure and chemical reactivity is studied by X-ray diffraction and combustion methods, respectively. The specific surface area of milled samples is determined by BET measurement and the impurity content is estimated by spark source mass spectroscopy. There is no monotonous relationship between reactivity and specific surface area of particles and agglomerations of particles. In contrast to impurity contents, distortion of atomic short- and medium-range order is shown to be essential for the observed enhancement of chemical reactivity. A statistical model for the milling process is formulated and used to discuss the experimental data. As a result, an expression for the dependence of combustion temperature on milling time is proposed.

MICROSTRUCTURE AND HOMOGENEITY OF NANOCRYSTALLINE CO-CU SUPERSATURATED SOLID SOLUTIONS PREPARED BY MECHANICAL ALLOYING

Huang JY. Yu YD. Wu YK. Li DX. Ye HQ. - Journal of Materials Research. 12(4):936-946, 1997

Mechanical alloying (MA) has been performed in the Co,Cu-x((100-x)) (x = 10, 25, 50, 60, 75, and 90) system. High resolution electron microscopy (HREM) and field emission gun transmission electron microscopy (FEG TEM) were used to characterize the microstructure and homogeneity of the nanocrystalline Co₂₅Cu₇₅ solid solution. After 20 h of MA, all the mixtures show an entirely face-centered cubic (fcc) phase. HREM shows that the ultrafine-grained

(UFG) materials prepared by MA contain a high density of defects. Two kinds of typical defects in UFG Co₂₅Cu₇₅ are deformation twins and dislocations. The dislocations are mostly 60 degrees type, and in many cases they dissociate into 30 degrees and 90 degrees partials. The grain boundaries are ordered in structure, curved, and slightly strained, which is similar to that observed in NC-Pd. Nanoscale energy dispersive x-ray spectroscopy (EDXS) shows that the Co concentration in both the interior of grains and the GB's is close to the global composition, which proves that supersaturated solid solutions are indeed formed. In the meantime EDXS revealed that the mixing of Co and Cu in the solid solutions is homogeneous at nanometer scale. MA in the Co-Cu system is suggested to be a diffusion-controlled process, and stress-stimulated diffusion is proposed to be the reason for the formation of supersaturated solid solutions in this immiscible system.

SIMILARITIES AND DIFFERENCES IN THE MICROSTRUCTURE OF ATTRITOR-MILLED FE-AL-N COMPOSITIONS

Rawers JC. Cook D. - *Journal of Materials Research*. 12(4):947-952, 1997

Although numerous studies of high-energy, ball-milled metal powders have been conducted, to date few studies have characterized the mechanical processing of identical elemental compositions of prealloyed powders and of powder blends. This study reports on the mechanical processing (attritor ball milling) in argon and nitrogen gas environments of (a) iron powder and prealloyed iron-2 wt. % aluminum powder, and (b) iron-aluminum, iron-aluminum nitride, and iron-iron nitride powder blends. When nitrogen was milled into iron particles either from nitride powder or by gas infusion, the nitrogen dissolved interstitially in bcc-Fe (principally at the grain boundaries) or was present as bcc-Fe nanoparticles at the bcc-Fe nanograin boundaries. The resulting nitrogen distribution was independent of how the nitrogen was added. Milled blends of iron and aluminum powder and prealloyed iron-aluminum powder resulted in similar microstructures: micrometer size particles with similar nanograin size. The aluminum in the blended powder mixture developed an ultrafine distribution on the grain boundaries, but it did not become uniformly distributed within the bcc-Fe grains. In contrast, the aluminum in prealloyed Fe-Al powder remained in solid solution during the mechanical milling.

SiC PARTICLE REINFORCED OXYNITRIDE GLASS - PROCESSING AND MECHANICAL PROPERTIES

Baron B. Chartier T. Rouxel T. Verdier P. Laurent Y. - *Journal of the European Ceramic Society*. 17(6):773-780, 1997.

After optimization of the processing route, the mechanical properties of the composites were evaluated with varying particle sizes and volume fractions of reinforcement. The best dispersion of the particles in the composite was obtained by using attrition milling followed by spray-drying, nevertheless, ball-milling led to satisfactory results for particle sizes higher than 3 μ m. Elastic moduli, hardness and fracture toughness increase with the volume fraction of SiC. fracture strength increases with both decreasing particle size and increasing volume fraction to reach 400 MPa for a glass matrix composite containing 47 vol% of SiC with 1 μ m average particle size. A further improvement is achieved by crystallizing the matrix.

PHOTOLUMINESCENCE FROM MECHANICALLY MILLED Si AND SiO₂ POWDERS

Shen TD. Shmagin I. Koch CC. Kolbas RM. Fahmy Y. Bergman L. Nemanich RJ. McClure MT. Sitar Z. Quan MX. - *Physical Review B-Condensed Matter*. 55(12):7615-7623, 1997

The photoluminescence (PL) in as-received and milled Si and SiO₂ powder is reported. The Si and SiO₂ powder is characterized by chemical analysis, Raman scattering, x-ray photoelectron spectra, infrared absorption, x-ray diffraction, and differential thermal analysis. The results indicate that the Si powder has amorphous Si oxide and suboxide surface layers. The milling of Si powder results in the formation of nanocrystalline/amorphous Si components. An amorphous SiO₂ component is formed by milling crystalline SiO₂. The PL spectra for as-received Si, milled Si, and SiO₂ powder exhibit similar peak shapes, peak maxima, and full width at half maximum values. For both the as-received and the milled Si powder, experimental results appear to exclude mechanisms for PL related to an amorphous Si component or Si-H or Si-OH bonds, or the quantum confinement effect. Similarly, for milled SiO₂ powder mechanisms for PL do not appear related to Si-H or Si-OH bonds. Instead the greatly increased intensity of PL for milled SiO₂ can be related to both the increased volume fraction of the amorphous SiO₂ component and the increased density of defects introduced in the amorphous SiO₂ upon milling. It is suggested that the PL for as-received Si, milling-induced nanocrystalline/amorphous Si, and milled SiO₂ results from defects, such as the nonbridging oxygen hole center, in the amorphous Si suboxide and/or SiO₂ components existing in these powder samples. The PL measurement for milled SiO₂ is dependent on air pressure whereas that for as-received SiO₂ is not, suggesting that new emitting centers are formed by milling.

INFLUENCE OF MIXING ENTHALPY OF SYSTEM TO FORMATION OF THE SOLID SOLUTIONS DURING MECHANICAL ALLOYING

Grigoreva TF. Barinova AP. Ivanov EY. Boldyrev VV. - *Doklady Akademii Nauk*. 350(1):59-60, 1996

NANOCRYSTALLINE OXIDES SYNTHESIZED BY MECHANICAL ALLOYING

Begincolin S. Wolf F. Lecaer G. - *Journal de Physique III*. 7(3):473-482, 1997

Mechanical alloying is a synthesis technique which is used to produce nanostructured powders. Nevertheless this high energy ball-milling process is not used only for refining of microstructure, but also for inducing chemical reactions between two powders or between powders and the atmosphere of grinding or for inducing chemical or phase transformations... Polymorphic transformations induced by ball-milling in SnO₂, TiO₂, Y₂O₃ and WO₃ and the influence of the nature of milling media on these transformations are described as well as the formation mechanisms of nanocrystalline based zirconia alloys. The nanometric structure of so obtained powders is characterized by transmission electron microscopy and a hyperfine technique: the Mossbauer spectroscopy.

MECHANOCHEMICAL SYNTHESIS OF ULTRAFINE ZrO₂ POWDER

Ding J. Tsuzuki T. McCormick PG. - *Nanostructured Materials*. 8(1):75-81, 1997

The synthesis of ultrafine zirconia powders by mechanochemical reaction of ZrCl₄ with CaO has been investigated using x-ray diffraction, TEM and DSC measurements. Mechanical milling resulted in a nanoscale mixture of CaO and amorphous ZrCl₄, with no evidence of any reaction having occurred. Subsequent heat treatment at temperatures above 300 degrees C resulted in the formation of separated particles of cubic ZrO₂, 5-10 nm in diameter, within an CaCl₂ matrix. Measurements of the effect of particle size on the crystal structure of ZrO₂ are also reported.

NANOCRYSTALLINE Ag FORMED BY LOW-TEMPERATURE HIGH-ENERGY MECHANICAL ATTRITION

Xu J. Yin JS. Ma E. - *Nanostructured Materials*. 8(1):91-100, 1997

High-energy mechanical attrition of Ag powders was conducted at the liquid nitrogen temperature without using process control agents. This process improves the milling efficiency and yields powders with truly nanocrystalline grain structure. The microhardness and grain growth behavior at different storage temperatures, as well as the excess enthalpy introduced by milling, have been studied and compared with the results for nanophase Ag prepared using other methods.

FORMATION OF TITANIUM HYDRIDE AT ROOM TEMPERATURE BY BALL MILLING

Zhang H. Kisi EH. - *Journal of Physics-Condensed Matter*. 9(11):L 185-L 190, 1997

Titanium hydride with a body centred tetragonal structure was rapidly synthesized at room temperature by ball milling titanium powders under a hydrogen atmosphere. The structure evolution was monitored by x-ray diffraction and Rietveld analysis. Unit cell volume dilation suggests the hydrogenation process begins with the formation of a titanium-hydrogen solid solution followed by the formation of titanium hydride. Dehydrogenation of the hydride produced by milling and titanium hydride prepared by standard techniques was investigated by thermogravimetric analysis (TGA). The hydride formed by the milling process has lower initial and final dehydrogenation temperatures and a slightly narrower dehydrogenation temperature range compared to hydride prepared by traditional methods. The extremely fine particle size, large surface area and lattice defects introduced by milling are believed to be responsible for modification of the hydrogen storage behaviour.

SYNTHESIS AND STRUCTURAL EVOLUTION OF TUNGSTEN CARBIDE PREPARED BY BALL MILLING

Wang GM. Campbell SJ. Calka A. Kaczmarek WA. - *Journal of Materials Science*. 32(6):1461-1467, 1997

Tungsten carbide has been synthesized directly by ball-milling tungsten powder and activated carbon in vacuum. The structural development of the WC phase with milling times up to 310 h has been followed using X-ray, neutron diffraction and scanning electron microscopy. Subsequent annealing (at 1000 degrees C for 1 and 20 h) of material milled for 90 h or longer, results in samples comprising almost entirely crystalline WC. The production of WC itself during milling results in enhanced iron contamination from the steel mill and balls on extended milling which were monitored by energy-dispersive X-ray and Mossbauer spectroscopies.

INVESTIGATION OF MICROSTRUCTURE OF PRODUCTS OF MECHANICAL ALLOYING IN SYSTEM CU-CR

Gerasimov KB. Mytnichenko SV. Pavlov SV. - *Doklady Akademii Nauk*. 351(3):349-351, 1996

MECHANICAL ALLOYING OF IMMISCIBLE ELEMENTS - Ag-Fe CONTRASTED WITH Cu-Fe

Ma E. He JH. Schilling PJ. - *Physical Review B-Condensed Matter*. 55(9):5542-5545, 1997

Extended x-ray-absorption fine-structure analysis demonstrates that the Cu-Fe and Ag-Fe systems, both immiscible in equilibrium, exhibit contrasting alloying behavior when subjected to mechanical milling. While Cu-Fe undergoes atomic-level alloying forming solid solutions, Ag-Fe remains largely unreacted. The alloying behavior observed is explained based on a model for a dynamic system, combining both the effects of externally driven mixing and thermal evolution under the thermodynamic driving force prevailing in a ball-milled mixture.

MECHANOCHEMISTRY OF SOME HYDROCARBONS

Field LD. Sternhell S. Wilton HV. - *Tetrahedron*. 53(11):4051-4062, 1997

Aromatic hydrocarbons (biphenyl, naphthalene, anthracene and phenanthrene) were subjected to ball milling (SPEX(R) 8000) with approximately ten-fold weight of inorganic materials (alumina or silica). After about 24 h all of the hydrocarbons were converted largely to carbon (graphite), but at intermediate stages disproportionation products (tetralin, phenylcyclohexane, bicyclohexyl, 9,10-dihydroanthracene, 1,2,3,4-tetrahydroanthracene, 1,2,3,4,4a,9,9a,10-

octahydroanthracene, 1,2,3,4,5,6,7,8-octahydroanthracene, 9,10-dihydrophenanthrene, 1,2,3,4-tetrahydrophenanthrene, 1,2,3,4,4a,9,9a, 10-octahydrophenanthrene, 1,2,3,4,5,6,7,8-octahydrophenanthrene) were also obtained in significant yields.

THERMOCHEMISTRY OF COMBUSTION REACTION IN AL-TI-C SYSTEM DURING MECHANICAL ALLOYING

Ye LL. Liu ZG. Li SD. Quan MX. Hu ZQ. - Journal of Materials Research. 12(3):616-618, 1997

The combustion reaction while mechanical alloying (MA) the Al-Ti-C system has been detected by in situ thermal analysis and the results of x-ray diffraction (XRD), Based on the information provided by in situ thermal analysis, the reaction temperature is estimated to be 1677 K, which is in good agreement with the value of the adiabatic temperature of 1700 K. It is considered that the formation reaction of Ti-C, which ignited by the heavy collisions of milling balls, induced the following reaction between Ti and Al at high temperature.

CHARACTERIZATION OF MECHANICAL NANOCRYSTALLIZATION PROCESS OF AMORPHOUS FE-MO-SI-B ALLOY BY TRANSMISSION MOSSBAUER SPECTROSCOPY

Liu XD. Guo FQ. Lu K. Umemoto M. - Journal of Materials Research. 12(3):681-687, 1997

The nanocrystallization process of the amorphous Fe-Mo-Si-B alloy under ball milling is characterized by means of transmission Mossbauer emission spectroscopy (TMES) in the present paper. It was found that a single alpha-Fe phase with the bcc structure is formed under ball-milling the amorphous Fe-Mo-Si-B alloy. A significant increase in the relative area of the subspectra of 8Fenn and 7Fenn and a remarkable decrease in isomer shift and half linewidth of the subspectra of various Fe configurations, especially in the case of 6Fenn, were observed during the ball-milling process. The diffusion of metalloid atoms from the bcc alpha-Fe phase to the remaining amorphous phase and alpha-Fe/alpha-Fe grain boundaries is suggested to occur during the mechanical crystallization of the current amorphous alloy based on the above TMES investigations.

AMORPHIZATION MECHANISMS OF NiZr2 BY BALL-MILLING

Galy D. Chaffron L. Martin G.- Journal of Materials Research. 12(3):688-696, 1997

The microstructure of NiZr2 in the course of amorphization by ball-milling is studied by transmission electron microscopy (TEM) and x-ray diffraction (XRD). The evolution from the initial fully crystalline alloy to a fully amorphized material is described. It is shown that prior to amorphization, the powder aggregates achieve a 100% nanocrystalline structure; the amorphous phase then appears and develops to the expense of the nanocrystalline phase. No massive chemical disordering is observed, but a small amount cannot be ruled out. It is proposed that amorphization occurs by chemical disordering at interfaces, induced by the scattering of shear waves.

MECHANICAL ALLOYING OF FE+32 AT.PERCENT NI POWDERS [Russian]

Tsurin VA. Barinov VA. Pupyshev SB.- Fizika Metallov i Metallovedenie. 81(5):108-117, 1996

MAGNETIC PROPERTIES OF MILLED AND THERMAL RELAXED YBa2(Cu1-xFex)(3)O-y

Timko M. Kovac J. Sargankova I. Zentkova M. Mihalik M. Matas S. Ciasnohova A. - Acta Physica Polonica A. 91(2):355-358, 1997

The influence of the mechanical milling and subsequent thermal relaxation on magnetic and superconducting behaviour of YBa2(Cu1-xFex)(3)O-y system has been studied. Two methods of heat treatment were used: Set I - slow cooling from 980 degrees C in flowing O-2 and Set II - reducing at 770 degrees C in flowing Ar followed by reoxidation in flowing O-2 below 400 degrees C. The transition to superconductivity, diamagnetic response, critical current density and the effective magnetic moment in the normal state have been estimated. Our measurements indicate that the reducing atmosphere preparation is less detrimental on superconducting properties. The results are discussed in terms of occupancy Cu sites by Fe and redistribution of oxygen atoms.

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à la demande de G. Silly (Université du Maine - Le Mans)
FORMATION PERMANENTE CNRS / DR 17
Laboratoire de Physique de l'Etat Condensé
UPRES-A CNRS n_ 6087, Université du Maine, LE MANS
INITIATION A LA RMN HAUTE RESOLUTION 1D APPLIQUEE A L'ETUDE DES MATERIAUX

La RMN est un outil indispensable dans le domaine de la physique et de la chimie des matériaux. Si son utilisation dans le domaine des liquides est ancienne et fondamentale, c'est seulement depuis une dizaine d'années qu'elle connaît un essor important dans le domaine des matériaux solides grâce aux techniques dites de haute résolution.

Nous disposons au Mans d'un spectromètre RMN à transformée de Fourier (BRUKER MSL 300) dédié à l'étude des matériaux solides. Nous utilisons cette opportunité pour proposer une formation sous forme de stage d'initiation à la pratique de ces techniques.

PUBLIC :

Chercheurs, enseignants-chercheurs, doctorants, physiciens et chimistes des matériaux, ingénieurs des secteurs publics et privés désirant s'initier à la technique. 15 personnes (nombre limité du fait des T.P.)

OBJECTIFS :

Donner une description de différentes expériences et techniques utilisées en RMN 1D pour l'étude des matériaux en phase solide, et les mettre en œuvre sur le spectromètre du laboratoire.

On s'intéressera essentiellement aux informations de nature physico-chimiques que les différentes techniques permettent d'obtenir.

PROGRAMME :

La formation se fera sous forme de cours/travaux dirigés et de travaux pratiques en petits groupes (5) sur le spectromètre BRUKER MSL 300 de l'Université du Mans. Elle sera assurée par des enseignants chercheurs du laboratoire de Physique de l'Etat Condensé.

Cours :

Rappels sur le magnétisme macroscopique et microscopique

Expérience de RMN pulsée

Les interactions de la RMN

La RMN haute résolution dans les solides ; La mesure des temps de relaxation

Etudes de cas

Travaux Pratiques :

Présentation du spectromètre ; Mise en œuvre d'une expérience simple ; Mesures de temps de relaxation

Rotation à l'angle magique (MAS) ; Découplage haute puissance ; Expérience CP/MAS

Utilisation de logiciels

ORGANISATION :

DATES : 4 jours du 20 au 23 octobre 1997

LIEU : Université du Mans - Faculté des Sciences

RENSEIGNEMENTS et préinscription

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à la demande de F. Nardou - LCN Limoges
Colloque National sur les Contraintes Résiduelles dans les procédés d'élaboration des Céramiques et des Traitements de Surface.

Limoges - France 23 - 24 Septembre 1997

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