

RESEAU FRANCAIS DE MECANOSYNTHESE

Lettre N°28

Juillet 1997

75 Groupes de Recherches (16 Etrangers) - 105 Correspondants

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

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ANNONCE DE CONGRES ET / OU ECOLES

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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 - Coso 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

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

2ème Symposium Normand de la Forge Européenne

De la Paléométaballurgie à l'Innovation

(Métallurgie des Poudres et Frittage)

NOUVEAU

Notre Dame de Gravenchon - 4 - 12 Octobre 1997

Contact : Elementa - Gare d'Etainhus - 76430 Etainhus

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)

Oxydes Magnétiques

Journées d'Etudes - Dijon 24 - 26 Mars 1998

Contact : J.C. Niepce - L2RS CNRS UPR5613

NOUVEAU

Faculté des Sciences de Mirande - 9 Avenue A. Savary - BP 400 - 21011 Dijon Cedex

E-Mail : JOXMAG@U-BOURGOGNE.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)

MECHANOCHEMICAL PROCESSING OF CELESTINE

Zhang QW. Saito F. - Chemical Engineering Journal. 66(1):79-82, 1997

Room temperature milling of celestine mineral and dry sodium hydroxide powder mixture was conducted in a planetary ball mill. Strontium sulfate, which is a main component in the celestine sample reacts spontaneously with sodium hydroxide powder during milling, forming strontium hydroxide. As a result of carbonation in a final product of milling, strontium hydroxide is transformed into strontium carbonate. The milling operation enables to accelerate the reaction rate. The strontium carbonate in the product can be concentrated up to over 90% by washing sodium sulfate out with water.

EFFECT OF MILLING CONDITIONS ON THE STRENGTH OF ALUMINA SILICON CARBIDE NANOCOMPOSITES

Jeong YK. Nakahira A. Morgan PED. Niihara K. - Journal American Ceramic Society. 80(5):1307-1309, 1997

Processing effects of wet ball-milling on the microstructure and fracture strength of Al₂O₃/5 vol% SiC nanocomposites were investigated. Homogeneous microstructure and a high fracture strength of 1200 MPa could be achieved under the milling condition of relatively low wear contents of Al₂O₃ grinding media, maintaining the homogeneity of the ball-milled powders. The fracture strength decreased with increasing wear content. Degradation of fracture strength was caused by abnormal grain growth related to wear particles from the Al₂O₃ balls.

EFFECT OF BALL MILLING TIME ON Ti-AL AND Ni-AL POWDER MIXTURES

Benhaddad S. Bhan S. Rahmat A. - Journal of Materials Science Letters. 16(10):855-857, 1997

MICROSTRUCTURE AND MAGNETIC HYSTERESIS OF CO₈₀CU₂₀ MECHANICALLY ALLOYED POWDERS

Elkalkouli R. Morel R. Dinhut JF. - Nanostructured Materials. 8(3):313-320, 1997

Nanostructured Cu₈₀Co₂₀ powders are synthesized by mechanical alloying. From results obtained mainly by X-ray diffraction and transmission electron microscopy observations, it appears that CuCo metastable alloyed powders are homogeneous, and that a solid solution of fcc CuCo is realized. However, magnetic measurements show that small Co-rich clusters are still present, even after long milling times. Results are compared with previous studies on CuCo films and more recent suggestions of a possible spinodal decomposition in these alloys

CHANGE OF THE STRUCTURE OF THE FE_{73.5}CU₁NB₃SI_{13.5}B₉ ALLOY BY MECHANICAL ALLOYING

Feng W. He KY. Cheng LZ. Xu H. Dong XQ. Zhang YM. - Nanostructured Materials. 8(3):367-371, 1997

The change of the structure by mechanical alloying on the Fe_{73.5}Cu₁Nb₃Si_{13.5}B₉ nanostructured alloy was studied experimentally. It was found that the nanostructured ribbons obtained from the crystallization of amorphous state by a proper annealing treatment can be changed into amorphous powder via short time milling. By increasing the milling time, the milled powder returns to crystallization and exhibits smaller crystal size. A still smaller crystal size can be formed if the milled powder is annealed again.

MODELLING OF RECRYSTALLISATION IN MECHANICALLY ALLOYED MATERIALS

Sha W. Bhadeshia HKDH. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 223(1-2):91-98, 1997

The exceptionally high recrystallisation temperatures of ultra-fine-grained mechanically alloyed metals cannot be adequately explained in terms of gamma' dissolution, dispersoid coarsening, or ordinary grain growth theory. Such alloys have an unusually fine grain structure before recrystallisation, the grain size typically being a fraction of a

micrometre. It is demonstrated here that in these circumstances, the fine grains cannot be treated as being topologically independent, because the grain-boundary junctions behave like strong pinning points, preventing the easy nucleation of recrystallisation. A model based on this idea, to new and published experimental data, are found to explain all of the known features of recrystallisation in mechanically alloyed oxide dispersion strengthened metals.

EFFECT OF MILLING CONDITIONS ON THE STRENGTH OF ALUMINA SILICON CARBIDE NANOCOMPOSITES

Jeong YK. Nakahira A. Morgan PED. Niihara K. - Journal of the American Ceramic Society. 80(5):1307-1309, 1997

Processing effects of wet ball-milling on the microstructure and fracture strength of Al₂O₃/5 vol% SiC nanocomposites were investigated. Homogeneous microstructure and a high fracture strength of 1200 MPa could be achieved under the milling condition of relatively low wear contents of Al₂O₃ grinding media, maintaining the homogeneity of the ball-milled powders, The fracture strength decreased with increasing wear content, Degradation of fracture strength was caused by abnormal grain growth related to wear particles from the Al₂O₃ balls.

GAS-SENSITIVE PROPERTIES AND STRUCTURE OF NANOSTRUCTURED (ALPHA-FE₂O₃)(X)-(SNO₂)(1-X) MATERIALS PREPARED BY MECHANICAL ALLOYING

Jiang JZ. Lin R. Lin W. Nielsen K. Morup S. Damjohansen K. Clasen R. - Journal of Physics D-Applied Physics. 30(10):1459-1467, 1997

The gas-sensitive properties of nanostructured 94, 85, 71 and 7 mol% alpha-Fe₂O₃SnO₂ materials prepared by high-energy ball milling have been investigated in atmospheres containing alcohol, CO and CH₄ gases. It has been found that the materials show a high sensitivity with a short response time to the alcohol gas and almost no sensitivity to the other two gases, CO and CH₄ in the concentration ranges studied. Furthermore, the gas-sensitive behaviour of these materials in response to the alcohol gas has been discussed on the basis of the microstructure of the materials. It is found that the tin ion content in alpha-Fe₂O₃ and tin ions on the surface of alpha-Fe₂O₃ particles may play an important role in the enhancement of the gas sensitivity.

INFLUENCE OF AUSTENITISING TEMPERATURE ON AUSTEMPERING OF AN MN-MO-CU ALLOYED DUCTILE IRON .2. MECHANICAL PROPERTIES

Ali ASH. Elliott R. - Materials Science & Technology. 13(1):24-30, 1997

Measurements of ultimate tensile strength, 0.2% proof strength, elongation, and impact energy are presented as a function of austempering time in the range 1 to 4320 min for austempering temperatures of 350, 375 and 400 degrees C and austenitising temperatures of 870 and 920 degrees C in a ductile iron of composition Fe-3.49C-2.33Si-0.42Mn-0.25Cu-0.23Mo-0.035Mg. With an austenitising temperature of 920 degrees C, the mechanical properties satisfy the ASTM A897M:1990 austempered ductile iron (ADI) standard at an austempering temperature of 350 degrees C but not at 375 and 400 degrees C. This is consistent with the predictions from the austempering kinetic measurements of an open processing window at 350 degrees C and a closed window at 375 and 400 degrees C. The mechanical properties measured with an austenitising temperature of 870 degrees C satisfy the standard at 350 and 375 degrees C but not at 400 degrees C. This is consistent with the predictions from austempering kinetic measurements of an open window at 350 and 375 degrees C and a closed window at 400 degrees C. Decreasing the austenitising temperature increases the closure temperature of the processing window and moves it to earlier austempering times and reduces the width of the processing window. Decreasing the austenitising temperature is shown to be a factor to be considered in obtaining the higher ductility grades of the ADI standard.

EFFECT OF MECHANOCHEMICAL DEGRADATION ON GELATION AND MECHANICAL PROPERTIES OF PVC

Xu X. Guo SY. Wang ZQ. - Journal of Applied Polymer Science. 64(12):2273-2281, 1997

The effect of vibromilling or jet milling on gelation and mechanical properties of poly(vinyl chloride) (PVC) was studied through SEM, FTIR, DSC, and mechanical properties tests. The experimental results show that the size of the grain and apparent density of PVC are decreased. The grains become much more loosely aggregated and the crystallinity of PVC is decreased during milling. The extensional fracture of degraded PVC is obviously different from that of undegraded PVC, the tensile strength and degree of gelation of degraded PVC are increased as compared with undegraded PVC. The mechanical properties of PVC are improved quite a lot after blending it with a small amount of mechanochemically degraded PVC.

X-RAY DIFFRACTION STUDY OF SOLID-STATE FORMATION OF METASTABLE MO₅Si₂ AND TiSi₂ DURING MECHANICAL ALLOYING

Yen BK. - Journal of Applied Physics. 81(10):7061-7063, 1997

The formation of metastable C40-MoSi₂ and C49-TiSi₂ during the mechanical alloying of metal/silicon powder mixtures has been investigated by x-ray diffraction. The mechanical alloying of a Si-rich Mo/Si powder mixture results in the formation of both C11(b)-MoSi₂ and C40-MoSi₂. The C40-MoSi₂ phase is metastable and transforms to C11(b)-MoSi₂ after annealing at 850 degrees C. The mechanical alloying of a stoichiometric Ti/Si powder mixture leads to the incipient formation of C49-TiSi₂ and the subsequent polymorphic transformation to C54-TiSi₂. The lattice parameters of C49-TiSi₂, as determined from a mechanically alloyed Ti/Si powder sample that has been annealed at 700 degrees C, are as follows: a(0) = 3.56 Angstrom, b(0) = 13.57 Angstrom, and c(0) = 3.55 Angstrom.

THE BAUSCHINGER EFFECT IN FINE-GRAINED AL-TI ALLOYS PREPARED BY MECHANICAL ALLOYING

Liao SH. Kao PW. Chang CP. - Scripta Materialia. 36(11):1227-1232, 1997

IN SITU OBSERVATION OF COMBUSTION TO FORM TIN DURING BALL MILLING Ti IN NITROGEN

Chin ZH. Perng TP. - Applied Physics Letters. 70(18):2380-2382, 1997

Mechanical milling of Ti powder in nitrogen by a high-energy ball mill was performed. The pressure was monitored in situ. Initially, nitrogen was dissolved in Ti to form a solid solution. Under suitable conditions, when the nitrogen content had reached 32 at.%, the nitrogen pressure drastically dropped and instant formation of TiN took place. The

TiN was formed by means of self-catalytic combustion, which was triggered by the high impact energy and the large clean surface of the fine Ti particles.

EFFECT OF ADDITIVE ELEMENTS ON AMORPHIZATION IN M75C25 (M=FE, CO OR NI) AND FE80N20 SYSTEMS BY MECHANICAL ALLOYING

Omuro K.- Jap. J Appl. Phys. Part 1 - 36(4A):2236-2241, 1997

Mechanical alloying (MA) of elemental powder mixtures of the $M(75-x)A(x)C(25)$ system (at.%) (M=Fe, Ca or Ni; A=Ti, V, Cr, Mn, Fe, Co or Ni) with $x = 0$ and 20 is performed using a planetary ball mill, in the MA processing, the A additives with a negative value of the interaction parameter $W-AC(M)$, such as Ti and V: markedly promote solid-state reactions in the MA products, leading to their amorphization, in contrast to the additives with a positive $W-AC(M)$ value such as Co and Ni in Fe-C systems; the parameter $W-AC(M)$ represents the difference in bonding energy of A-C atomic pairs (E-AC) and M-C pairs (E-MC) in the ternary M-A-C solution, i.e. $W-AC(M)-E-AC-E-MC$. Such effects of additives seen for M-C materials are also true of experimental results of MA of $Fe(64)A(16)N(20)$ (A=Cr or Nb) materials with a negative interaction parameter $W-AN(Fe)$.

MECHANICALLY ACTIVATED CARBOTHERMIC REDUCTION OF ILMENITE

Chen Y. Hwang T. Marsh M. William JS. - Metallurgical & Materials Trans. A-28(5):1115-1121, 1997

A systematic study of the effect of milling conditions on the low-temperature carbothermic reduction of the mineral ilmenite has been carried out. It was found that after ball milling of an ilmenite-carbon mixture at room temperature, the ilmenite was reduced to rutile and metallic iron during subsequent low-temperature annealing (760 degrees C for 30 minutes). A longer milling time results in a lower reduction temperature and a higher reduction rate. Higher milling intensity also leads to a lower reduction temperature. This enhanced reduction reaction induced by ball milling mainly results from the intimate mixing and large contact area between milled ilmenite and carbon particles.

BALL-MILLING-INDUCED CRYSTALLIZATION AND BALL-MILLING EFFECT ON THERMAL CRYSTALLIZATION KINETICS IN AN AMORPHOUS FEMOSIB ALLOY

Guo FQ. Lu K.- Metallurgical & Materials Transactions A, 28(5):1123-1131, 1997

Microstructure evolution in a melt-spun amorphous $Fe_{77.2}Mo_{0.8}Si_{9}B_{13}$ alloy subjected to high-energy ball milling was investigated by means of X-ray diffraction (XRD), a transmission electron microscope (TEM), and a differential scanning calorimeter (DSC). It was found that during ball milling, crystallization occurs in the amorphous ribbon sample with precipitation of an alpha-Fe solid solution, and the amorphous sample crystallizes completely into a single alpha-Fe nanostructure (rather than alpha-Fe and borides as in the usual thermal crystallization products) when the milling time exceeds 135 hours. The volume fraction of material crystallized was found to be approximately proportional to the milling time. The fully crystallized sample with a single alpha-Fe nanophase exhibits an intrinsic thermal stability against phase separation upon annealing at high temperatures. The ball-milling effect on the subsequent thermal crystallization of the amorphous phase in an as-milled sample was studied by comparison of the crystallization products and kinetic parameters between the as-quenched amorphous sample and the as-milled partially crystallized samples. The crystallization temperatures and activation energies for the crystallization processes of the residual amorphous phase were considerably decreased due to ball milling, indicating that ball milling has a significant effect on the depression of thermal stability of the residual amorphous phase.

AMORPHIZATION OF SELENIUM INDUCED BY HIGH-ENERGY BALL MILLING

Fan GJ. Guo FQ. Hu ZQ. Quan MX. Lu K. - Physical Review B, 55(17):11010-11013, 1997

Structural changes occurring in the crystalline element selenium upon high-energy ball milling has been studied. Milling experiments have been performed at the ambient temperature and at a cryogenic temperature of -100 degrees C, respectively. The final milling products under both conditions were found to be a fully amorphous phase after several hours of mechanical milling. Calorimetric evidences indicate that during cryogenic-temperature milling amorphization procedure is faster than that at ambient temperature. Our experimental results suggest that amorphization of the crystalline element selenium is driven by defects created by frequent mechanical deformation.

MAGNETIC PROPERTIES OF BCC FE-PD EXTENDED SOLID SOLUTIONS

Birsan M. Fultz B. Anthony L. - Physical Review B-Condensed Matter. 55(17):11502-11506, 1997

Supersaturated Fe-Pd solid solutions were prepared by mechanical alloying. X-ray diffractometry showed that the as-milled alloys were bcc from 0-26 at. % Pd. Saturation magnetization measurements and Mossbauer spectrometry measurements were performed on these powders. Consistent interpretations of both sets of measurements were possible if the magnetic moment at the Pd atom was 0.35 μ_B and constant, and if the magnetic moments at Fe atoms were enhanced by neighboring Pd atoms in a way similar to the effects of Ni.

SiC NANOPARTICLE-REINFORCED AL2O3 MATRIX COMPOSITES - ROLE OF INTRA- AND INTERGRANULAR PARTICLES

Xu YR. Zangvil A. Kerber A. - Journal of the European Ceramic Society. 17(7):921-928, 1997.

Submicron (or 'nanocrystalline') SiC-reinforced Al_2O_3 matrix composites have been prepared by co-milling followed by hot-pressing at 1650 degrees C. Both intra- and intergranular SiC particles were present. The volume ratio for intra- to intergranular particles was approximately 30 : 70 for 6 vol% SiC composites and 20 : 80 for 12 vol% SiC composites. The intragranular SiC (50-200 nm) formed mainly by growth of the Al_2O_3 matrix grains, while Zener pinning, mainly by the larger (200-300 nm) particles thought to be effectively limited; the Al_2O_3 size. Approximately 15% toughening was achieved by incorporating 12 vol% SiC nanoparticles into the matrix. The intergranular particles provided most of the toughening, mainly through a SiC particle-attracted crack deflection mechanism and crack impediment. No evidence was found of toughening by the intragranular particles, although they did cause some grain refinement by dislocation sub-boundary formation. A mixed fracture mode was observed, changing to a mostly intergranular mode with increasing volume of SiC particles.

MAGNETIC PROPERTIES OF FE-AL2O3 GEL GRANULAR SOLIDS PREPARED BY BALL MILLING

Giri AK. - Materials Research Bulletin. 32(5):523-529, 1997

Granular materials in the system $Fe-x-(Al_2O_3)(1-x)$ With different volume fractions of Fe have been prepared by ball milling pure Fe and sol-gel derived Al_2O_3 gel powders up to 80 h. The final metal average particle size obtained

ranged from 7-18 nm in different samples. We studied the variation of the magnetization and coercivity with the milling time. Coercivities as high as 550×10^{-4} T and 990×10^{-4} T at room temperature and at 5 K, respectively, were obtained for the $x = 0.4$ sample milled for 42 h. Presence of single domain and superparamagnetic particles may result in such high values. Low-temperature magnetization studies confirm the presence of superparamagnetic particles in the samples.

HYDROGEN STORAGE CAPACITY OF SUBMICRON MAGNESIUM-NICKEL ALLOYS

Holtz RL. Imam MA. - Journal of Materials Science. 32(9):2267-2274, 1997

Magnesium-nickel alloys with nickel concentrations from 0 to 60 at % were prepared by three methods: inert gas condensation of sputtered nanocrystalline powder, cosputtering of amorphous thin films and ball milling. Of the three methods, ball milling yields the best hydrogen storage properties in terms of hydrogen capacity, hydriding/dehydriding rates, and activation requirements. In addition, these characteristics are achieved in magnesium with only very small nickel concentrations, on the order of a few atomic per cent.

AN XAFS STUDY ON RECONSTRUCTION OF SHORT-RANGE ORDER IN MECHANICALLY ALLOYED AL-TI AND AL-TI-O COMPLEXES

Kojima Y. Isobe T. Senna M. Sakurai M. Sumiyama K. Suzuki K. - J. All.Comp. 248(1-2):52-58, 1997

Mechanically alloyed (MA) Al-Ti and Al-O-Ti complexes have been studied by X-ray absorption fine structure (XAFS) at the Ti K-edge. In the MA mixture of Al and Ti, the single pre-edge peak due to the electron transition $1s \rightarrow 3d$ disappears, and the intensity of the first nearest neighbor peak in the EXAFS structure function $\chi(r)$ decreases. In the MA mixture of Al and TiO₂ hydrogel, the number of pre-edge peaks apparently decreases from three to one. At the same time, the intensity of the $\chi(r)$ peak due to the first nearest neighbor increases, and its position approaches that of Al₂TiO₅. Similar phenomena are observed in the MA mixture of Al₂O₃ and TiO₂ hydrogels. These results suggest that mechanical stressing causes characteristic electronic interaction and the reconstruction of short-range order through intimate mixing at an atomic level. This accords with our previous NMR studies.

PROPERTIES OF Zr_{0.5}Ti_{0.5}(V_{0.25}Mn_{0.15}Ni_{0.6})₂ ALLOY BALL-MILLED WITH NICKEL POWDER

Chen J. Dou SX. Liu HK. - Journal of Alloys & Compounds. 248(1-2):146-150, 1997

As-cast Zr_{0.5}Ti_{0.5}(V_{0.25}Mn_{0.15}Ni_{0.6})₂ alloy with C14 Laves phase was ball-milled with nickel powders. Structure characterization and surface properties of the ball-milled powders were examined by using X-ray diffraction analysis and scanning electron microscopy. When the ball-milled alloy is used in a metal hydride electrode, it exhibits rapid activation, good high-rate discharge capacity and long cycle life, owing to the catalysis of the nickel-rich subsurface layer of the ball-milled alloy powders.

PREPARATION OF THE HYDRIDES Mg₂FeH₆ AND Mg₂CoH₅ BY MECHANICAL ALLOYING FOLLOWED BY SINTERING

Huot J. Hayakawa H. Akiba E. - Journal of Alloys & Compounds. 248(1-2):164-167, 1997

Mg₂CoH₅ and Mg₂FeH₆ were prepared by mechanical alloying (MA) and followed by sintering under mild conditions. The effect of milling atmosphere on the end products was also studied. The mixtures 2Mg+Fe and 2Mg+Co were milled in a planetary ball mill under hydrogen and argon atmospheres. After milling, the mixtures were sintered at 623 K for 1 day under 50 bars of hydrogen. X-ray powder diffraction analysis was used not only for identification of phases formed but also for crystal structural refinement and mass fraction calculation using the Rietveld method. We found that, under the above sintering conditions, the hydrides Mg₂FeH₆ and Mg₂CoH₅ were produced only in samples that were milled prior to sintering. For 2Mg+Fe mixture, milling under hydrogen prior to sintering produced 84% Mg₂FeH₆ in the product excluding oxide. The mixture 2Mg+Co was milled and sintered producing MgH₂ and Mg₂CoH₅, but the mass fraction of Mg₂CoH₅ was independent of the milling atmosphere.

STRUCTURAL AND MAGNETIC INVESTIGATIONS ON THE METASTABLE PHASES OF THE MECHANICALLY ALLOYED FE-CU SYSTEM

Majumdar B. Raja MM. Narayanasamy A. Chattopadhyay K. - J. All. Comp. 248(1-2):192-200, 1997

Attempts have been made to produce a solid solution of Fe_{100-x}Cu_x ($x = 10$ to 50) by the mechanical alloying technique at room temperature. A combination of X-ray diffractometry, electron microscopy, Mossbauer spectroscopy and magnetisation measurements has been used to characterise the alloys. Our results indicate that formation of the b.c.c. Fe-Cu solid solution is difficult up to a copper concentration of 20%. For higher copper concentrations, Fe-Cu solid solutions are obtained. The magnetisation and Mossbauer data confirm that excess milling leads to the precipitation of b.c.c. Fe from the f.c.c. solid solution. The Mossbauer studies also show the coexistence of a small amount of gamma-Fe for all compositions.

NEW BALL MILLING INDUCED SOLID SOLUTION AND AMORPHOUS PHASES IN THE ZN-V SYSTEM

Zhang H. Kisi EH. - Journal of Alloys & Compounds. 248(1-2):201-205, 1997

The solid phase transition induced by ball milling in the Zn-V system has been investigated by X-ray diffraction, differential scanning calorimetry and thermodynamic analysis approaches. Milling for a short period in the Zn-V system leads to the formation of an extensive solid solution, despite these elements being virtually immiscible in the solid state, continuous milling results in complete amorphization, finally transforming to a vanadium-gas compound. A strong case can be made that the driving force for amorphization is not provided by the enthalpy of mixing, but by stored deformation energy in the system.

STRUCTURAL EVOLUTION OF BALL-MILLED AMORPHOUS Fe₂Sc POWDERS DURING HEAT TREATMENT

Xia SK. Assuncao FCR. Avillez RR. Doyle S. Wroblewski T. Baggiosaitovitch E. - J.All. Comp., 248(1-2):246-250, 1997

Structural evolution during heat treatment of amorphous Fe₂Sc powder obtained by mechanical milling of Fe₂Sc Laves phase has been studied by in situ X-ray diffraction using synchrotron radiation, in situ Mossbauer spectroscopy and differential scanning calorimetry (DSC). The results indicate that low temperature heating leads to formation of Fe clusters. Upon further heating, the alpha-Fe phase precipitates within a large temperature range. A granular alpha-

Fe system with controlled grain size can be obtained. At high temperatures, several crystalline phases appear. Two exothermic reactions are observed in the DSC curves.

THE SYNTHESIS OF NBSI₂ BY MECHANICAL ALLOYING

Leu TP. Fan GJ. Ding BZ. Hu ZQ - Journal of Materials Research. 12(5):1172-1175, 1997

The stoichiometric intermetallic compound NbSi₂ has been synthesized by mechanical alloying (MA) elemental Nb and Si powders. The alloying process has been investigated by means of x-ray diffraction (XRD) and differential scanning calorimetry (DSC). It was found that the formation of the Nb₂Si intermetallic compound occurs abruptly after 65 min of milling without any interruptions during the alloying process. However, short interruptions at a 5 min interval during ball milling result in a gradual reaction for the formation of the NbSi₂ compound as well as a new metastable bcc structured solid solution. We conclude that the temperature rise during mechanical alloying plays an important role in initiating the abrupt reaction after an incubation milling time.

MECHANICAL ALLOYING EFFECT ON THE STRUCTURE OF FE-AL-(SI) ALLOY

Zhang JQ. Wu BY. Hu Z. Wang GQ. - Journal of Materials Science & Technology. 13(3):219-222, 1997

The mechanical alloying effects on the structures of Fe_{71.3}Al_{28.4}(CeO₂)_(0.3) and Fe_{73.3}Al_{19.8}-Si-16.6(CeO₂)_(0.3) samples have been investigated mainly by Mossbauer spectroscopy and X-ray diffraction. It is found that with the increase of milling time, a substituting disordered alpha-Fe solution is formed gradually and almost completed after milled for 72 h. Further increasing milling time causes the atomic rearrangement within the powders. Correspondingly, the average lattice parameter and average crystalline size change until 72 h and remain constant after 72 h. The addition of Si leads to the higher disordered degree reflected in the smaller average hyperfine field and larger mean square deviation.

MECHANICAL ALLOYING OF FE100-XBX

Cheng LZ. Feng W. Dong XQ. Bai KC. He KY. - J. Materials Science & Technology. 13(3):230-232, 1997

The formation of nonequilibrium phase by mechanical alloying (MA) of Fe and B powders in a high energy vibration ball mill has been made for Fe_{100-x}B_x system with x=15, 30, 35, 45, 55 and 70. By using the X-ray diffraction, magnetic measurement and Mossbauer spectrum methods, it was revealed that the structure of the MA product varies with milling time and B contents.

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

INDENTATION CREEP IN NANOCRYSTALLINE FE-TIN AND NI-TIN ALLOYS PREPARED BY MECHANICAL ALLOYING

Ogino Y. Yamasaki T. Shen BL. - Metallurgical & Materials Transactions B - 28(2):299-306, 1997

Mechanical properties of nanocrystalline Fe-TiN and Ni-TiN alloys with various TiN contents between 17 and 64 vol pct, which are prepared by dynamically consolidating mechanically alloyed powders, have been investigated by means of hardness measurements and indentation creep tests at intermediate temperatures. The hardness increases with decreasing grain size to about 10 nm. The indentation creep curves conform well to an equation derived from a transient creep rate equation. The analysis of creep curves indicates that the deformation occurs by a dislocation mechanism controlled by grain boundary diffusion.

FABRICATION AND SINTERING OF FINE YTTRIA-DOPED CERIA POWDER

Vanherle J. Horita T. Kawada T. Sakai N. Yokokawa H. Dokiya M. - J. Amer. Cer. Society. 80(4):933-940, 1997

Yttria-doped ceria powder was prepared from oxalate precursors. The oxalate coprecipitation bath parameters were closely monitored and found to influence the sintering behavior of the subsequently obtained oxide powders strongly. The use of concentrated (Ce,Y) metal nitrate solutions and dilute neutralized oxalic acid for coprecipitation were identified as the most-important parameters. Following calcination at 700 degrees C, compacts of such powders were sintered to high density (98%) at 1400 degrees C (4 h). Ball milling of the powder further reduced the sintering temperature. Dry milling, for tape-casting applications of the powder in particular, was more effective than wet milling. Tape-cast membranes were fired at 1400 degrees C (2 h), with resulting densities of 98%.

INFLUENCE OF AUSTENITISING TEMPERATURE ON AUSTEMPERING OF AN MN-MO-CU ALLOYED DUCTILE IRON .1. AUSTEMPERING KINETICS AND THE PROCESSING WINDOW

Ali ASH. Elliott R. - Materials Science & Technology. 12(12):1021-1031, 1996

X-ray diffraction, optical microscopy, and hardness measurements were used to determine the austempering kinetics of an alloyed ductile iron of composition (wt-%) Fe-3.49C-2.33Si-0.42Mn-0.25Cu-0.23Mo-0.035 Mg at austempering temperatures of 300, 350, 375, and 400 degrees C and austenitising temperatures of 870 and 920 degrees C. The stage I reaction during austempering occurs in two steps, the first in the eutectic cell and the second in the intercellular area. Decreasing the austenitising temperature is shown to increase the driving force for the stage I reaction but to have a lesser effect on the stage II reaction. Decreasing the austenitising temperature produces a more uniform austempered microstructure and reduces the amount of martensite in this structure. These changes move the processing window to shorter austempering times and increase the temperature at which the processing window closes.

MECHANICALLY ALLOYED, FERRITIC OXIDE DISPERSION STRENGTHENED ALLOYS - STRUCTURE AND PROPERTIES

Czyrskafilonowicz A. Dubiel B. - Journal of Materials Processing Technology. 64(1-3):53-64, 1997

The paper presents information concerning the production of oxide dispersion strengthened (ODS) alloys by mechanical alloying process. The macro- and microstructures of the ferritic ODS alloys, INCOLOY MA 956 and PM2000 are discussed in detail. The basic engineering properties of these alloys, namely oxidation resistance and mechanical properties, especially tensile, compression and high temperature creep, are outlined.

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

Fukunaga T. Mizutani U. - Sci. Rep. Research Institutes Tohoku University Series A - 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.

MICROSTRUCTURE AND STRENGTH OF NBCR2/CR IN-SITU COMPOSITES

Aoyama N. Hanada S. - Materials Transactions Jim. 38(2):155-162, 1997

To assess the potential of intermetallic-matrix composites as high temperature structural materials, the microstructures of the cast-annealed, the hand milled-HIPed and the ball milled-HIPed NbCr₂/Cr in-situ composites were observed by optical, scanning and transmission electron microscopy. The as-cast Cr-24.5 mol%Nb is composed of primary NbCr₂ Laves plates with C15 structure and lamellar eutectics consisting of NbCr₂ and a Cr solid solution (Cr-ss). The NbCr₂ phase has a lot. of planar faults and Cr-ss contains a high density of dislocations. Fine NbCr₂ precipitates form in Cr-ss of the cast and annealed Cr-24.5 mol%Nb, although planar faults remain unchanged in the NbCr₂ phase. Microstructural observation indicates that the eutectic composition exists between Cr-17.2 mol%Nb and Cr-18.3 mol%Nb. In the as-cast and the cast and annealed Cr-5.5 mol%Nb and Cr-12.2 mol%Nb, primary Cr solid solution particles are delineated by networks based on the lamellar eutectic consisting of Cr-ss and NbCr₂. Primary Laves plates in the cast and annealed Cr-24.5 mol%Nb were fragmented by hand-milling and pulverized into nearly round shapes by ball-milling. By hot isostatically pressing (HIPing) the hand-milled or ball-milled powder, porosity-free compacts were fabricated. At a high volume fraction of the NbCr₂ phase the NbCr₂/Cr in-situ composites possess extremely high yield stresses at elevated temperatures, although their ductile-brittle transition temperature is considerably high. Ductility is increased by refining the microstructure via hand- or ball-milling followed by HIPing and by increasing the volume fraction of Cr-ss.

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

Shaik GR. Milligan WW. - Metallurgical & Materials Transactions A - 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-Fetch 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.

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 Co₂₅Cu_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.

THE EFFECT OF EXTENDED MILLING ON MINERALS

Welham NJ. - CIM Bulletin. 90(1007):64-68, 1997

The effect of extended ball milling has been studied for two industrial processes: (1) the processing of metal sulphide concentrates; and (2) the carbothermic reduction of ilmenite to synthetic rutile. The plant concentrates were a gold ore with 128 g/t Au of which 8% was non-refractory and a zinc concentrate with copper impurities. The gold concentrate showed complete dissolution of the gold bearing minerals after milling giving almost complete gold liberation. The zinc concentrate showed almost all of the sulphides had solubilized leaving the gangue and elemental sulphur. A study of pure pyrite and arsenopyrite shows that selective dissolution should be possible by a simple leaching process directly after milling. The milling of ilmenite and coal together results in a temperature decrease of 150 degrees C in the subsequent carbothermic reduction of ilmenite, in addition to improving both the rate of reaction and the selectivity of reduction to rutile.

SiC PARTICLE REINFORCED OXYNITRIDE GLASS - PROCESSING AND MECHANICAL PROPERTIES

Baron B. Chartier T. Rouxel T. Verdier P. Laurent Y. - J. Europ; 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 µm. 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 µm average particle size. A further improvement is achieved by crystallizing the matrix.

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.

DETERMINATION OF LATTICE PARAMETERS, DIFFRACTING CRYSTALLITE SIZE, AND THERMAL STABILITY IN MECHANICALLY ALLOYED CU-ZN SOLID SOLUTIONS

Bayer AE. - Journal of Materials Engineering & Performance. 6(2):149-152, 1997

Four copper-zinc (Cu-Zn) alloy powders are produced by mechanical alloying to investigate the effects of zinc composition on the lattice parameter, diffracting crystallite size (DCS), and thermal stability. The DCS versus either milling time or composition data indicates that the substitutional zinc solutes alter the dislocation generation, organization, and recovery processes. Lattice parameter determinations agree well with published values at small compositions, but are slightly less than the expected values for larger compositions. Annealing the powders at 823 and 1023 K for 6 h resulted in a substantial decrease of root-mean-square (rms) atomic-level strain and slight increases in the lattice parameters. In spite of the high annealing temperatures, the DCS remains less than 30 nm for all

compositions, Furthermore, this high degree of thermal stability was inversely related to the concentration of zinc in the alloy.

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.

A METHOD FOR SIMULATING THE THREE-DIMENSIONAL MOTION OF BALLS UNDER THE PRESENCE OF A POWDER SAMPLE IN A TUMBLING BALL MILL

Kano J. Chujo N. Saito F. - Advanced Powder Technology. 8(1):39-51, 1997.

A method for simulating the motion of balls under the presence of a powder sample in a tumbling mill is proposed in this paper. The method is based on the particle element method and a coefficient of friction of colliding balls is the key to simulate the motion under powder filling. Four kinds of samples, i.e. glass beads, silica, aluminum hydroxide and kaolinite powders, were used for the milling. The movements of balls with and without a sample were observed visually by a video recorder and compared with the simulation results. The actual movement of balls in the mill is dependent on the kind of sample and the movement calculated by the present method can be simulated by choosing a suitable coefficient of friction for a given sample. The suitable coefficient of friction for the kaolinite sample is the highest value of all of the samples and is more than four times larger than that in grinding without sample. Suitable coefficients of friction for the samples are correlated with their angles of repose.

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.

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

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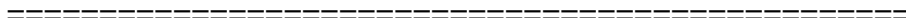
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**Le RFM a entamé sa Troisième Année d'existence,
 Merci de penser à vos adhésions au titre de l'Année 1997.
 Les membres du RFM ayant déjà réglé leur cotisation sont indiqués sur l'annuaire par (1997)**

Bulletin d'adhésion 1997 (à retourner à l'adresse suivante) :
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UPR CNRS 423 - Groupe "Elaboration et Transitions de Phases Hors Equilibre" - IPSé - F90010 - Belfort Cedex

Nom : **Prénom :**

Adresse complète :

.....

Renseignements complémentaires :

Téléphone : **Télécopie :**

e-Mail :

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)

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

Jean Yves BUZARE

Laboratoire de Physique de l'Etat Condensé - CNRS - UPRES-A 60877

Université du Maine - Avenue Olivier Messiaen - 72085 LE MANS CEDEX 9

tél : 02 43 83 32 90 - fax : 02 43 83 35 18 - email : buzare@aviion.univ-lemans.fr

=====

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

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

JOURNEES D'ETUDES

24 -26 MARS 1998 Organisées par :

Laboratoire de Recherches sur la Réactivité des Solides - UMR 5613 à DIJON

OXYDES MAGNETIQUES

Poudres, céramiques, films, process et applications

Première circulaire - Programme prévisionnel

Organisation :

-Club 11 de la SEE "Etudes générales" Président : M. PAYS

-Cercle thématique 11.14 "Céramiques pour l'électronique et l'électrotechnique" Animateur : P. ABELARD

- Groupe Français de la Céramique : Président : J.F. BAUMARD

-Laboratoire de Recherches sur la Réactivité des Solides : J.C NIEPCE

Comité du programme technique :

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Comité scientifique local : J.C. NIEPCE, B. GILLOT, P. PERRIAT, D. AYMES et F. BERNARD.

Lieu : Domaine Sainte-Anne à MESSIGNY ET VANTOUX (21380)

(à une dizaine de kilomètres de DIJON)

Thèmes principaux :

Ces deux journées feront le point sur les innovations et avancées technologiques et scientifiques dans le domaine des matériaux à base d'oxydes pour applications magnétiques. Ces mises au point pourront concerner aussi bien des aspects physico-chimiques tels que la composition, la structure et la microstructure que des aspects relatifs aux technologies d'élaboration ou de mise en oeuvre; cependant, il est souhaité que toutes les contributions se placent dans la préoccupation d'un progrès en termes de performances ou d'économie. Ces journées réuniront les partenaires des milieux industriels et des équipes de recherche intéressés par ces sujets.

e.mail : joxmag@u-bourgogne.fr

La première matinée sera articulée autour du thème "Matières premières /Process"

Y seront présentés des travaux portant sur les techniques de synthèse et frittage des poudres et les moyens de contrôle chimique et morphologique de celles-ci.

Session I Matières premières, process - Contrôles chimique et morphologique

Animateurs S. BEGIN (Ecole des Mines de Nancy)

P. BEUZELIN (SIEMENS)

Les deux demi-journées suivantes seront consacrées aux différentes applications des oxydes magnétiques (durs et doux). Sera abordé l'examen des différentes techniques de mises en oeuvre, envisageables suivant le type d'applications

Session II Couches minces, matériaux pour enregistrement magnétique et magnétooptique

Animateurs A. ROUSSET (LCMS CNRS/Univ. P. Sabatier - Toulouse) M. X (GIGADISC)

Session III Matériaux de puissance et à haute perméabilité - Ferrites doux

Animateurs M. GUYOT (LMOV CNRS/Univ.- VERSAILLES) R. KORMANN (THOMSON PC)

Session IV Matériaux pour applications hyperfréquences, absorbant

Animateurs H. PASCARD (LSI POLYTECHNIQUE) G. FORTERRE (TEKELEC)

Session V Ferrites durs pour aimants permanents

Animateurs M. VINCENT (ENSPG)- M. TENAUD (UGIMAG)

La dernière demi-journée sera dévolue aux applications nouvelles des ferrites, en particulier dans le domaine de la biologie, des nanomatériaux, des ferrofluides, etc...

Session VI Applications nouvelles

Animateurs J. ETOURNEAU (CNRS-Univ Bordeaux) D. AUTISSIER (CEA)

Contacts :

Prof. J.C. NIEPCE

Journées "Oxydes Magnétiques"

Laboratoire Réactivité des solides - Faculté des Sciences Mirande

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