RESEAU FRANCAIS DE MECANOSYNTHESE

Lettre N°30

Septembre 1997

78 Groupes de Recherches (19 Etrangers) - 110 (+3) Correspondants

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

3 Nouvelles Adhésions

J.Z. Jiang - Dept Phys. - Tech. Univ. Denmark - Danemark
A. Van Neste - Mines et Métallurgie - Université de Laval - Canada
A.N. Streletskii - Inst. Chem. Phys. - Dept Kinetics & Catalysis - Moscou - Russie

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RAPPEL

Assises des Matériaux - CNRS

Au cours des Assises des Matériaux (Lyon - 15 et 16 Décembre 1997), seront présentées et discutées les conclusions de l'analyse stratégique et présenté le nouveau programme du CNRS.

Une intervention au nom des 32 Sociétés Savantes et Associations concernées par la recherche sur les matériaux est prévue au cours de cette manifestion. Y. Farge, Président de la Société Française de Métallurgie et de Matériaux, sollicité par G. Beck, a accepté de présenter cette contribution.

Afin de construire cette intervention, Y. Farge nous sollicite afin de connaitre l'avis du RFM sur le bilan et les propositions permettant d'améliorer la recherche sur les matériaux menée en France.La réponse au nom du RFM doit lui parvenir avant le 1er Octobre 1997.

Tout membre du RFM est donc invité à me faire parvenir tout document qu'il jugera utile avant le 15 Septembre 1997, afin que nous puissions rédiger une note présentant la contribution du RFM à cette réflexion sur l'avenir de la recherche sur les matériaux. Cette note de synthèse sera diffusée prochainement dans la lettre du RFM.

E. Gaffet

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ANNONCE DE CONGRES ET / OU ECOLES

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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

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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

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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 Paléométallurgie à l'Innovation
(Métallurgie des Poudres et Frittage)
Notre Dame de Gravenchon - 4 - 12 Octobre 1997
Contact : Elementa - Gare d'Etainhus - 76430 Etainhus

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

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Réunion J2IM - Joints Intergranulaires et Interphases

NOUVEAU

8 - 10 Octobre 1997
Contacts : http://sivet1.glvt-cnrs.fr/J2IM/index.htmlx

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

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JA97

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

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

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Surface - Controlled Nanoscale Materials for High Added Value Applications

NOUVEAU

MRS Fall Meeting
Boston - 1 - 5 December 1997
Org. : M.-I. Baraton, K.E. Gonsalves, J.X. Chen, J.A. Akkara
Contact : M.-I. Baraton E_Mail : Baraton@unilim.fr

The 7th Joint MMM-Intermag Conference

San Francisco - 6 - 9 Janvier 1998
Web Site : http://www.aip.org/edops/mmmconf.html

NANO 98

NOUVEAU

Odeillo - 5 - 7 Janvier 1998
Contact : C. Monty - E_Mail: Monty@imp-odeillo.fr

Society of Automotive Engineers Congress

Cobo Center - Detroit - Michigan - USA - 24 Fevrier 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
Faculté des Sciences de Mirande - 9 Avenue A. Savary - BP 400 - 21011 Dijon Cedex
E_Mail : JOXMAG@U-BOURGOGNE.Fr

European Workshop on Nanocrystalline Materials

NOUVEAU

Grenoble - Avril 1998
Contact : A.R. Yavari

Colloque National de Métallurgie des Poudres

Efets de la microstructure et de la porosié sur les propriétés mécaniques et physiques des matériaux MdP
Grenoble - 6 - 8 Avril 1998 - Org. SF2M

NANO 98

NOUVEAU Stockholm - Suède - 14 - 19 Juin 1998
Secrétariat Cof. : nano98@kth.se

CIMTEC'98 - World Ceramics Congress and Forum on New Materials
Florence - 14 - 19 Juin 1998
Web Site : http://www.dinamica.it/cimtec98/

3rd International Symposium on Metallic Multilayers (MML'98)
NOUVEAU **Vancouver** - 14 - 19 Juin 1998
Contact : E_MAil : Conference_Service@SFU.CA

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/

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Annonces de Soutenance de Thèses

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"Contribution à l'Etude de la Transformation Tribologique Surperficielle en Fretting"

E. Sauger - Ecole Centrale de Lyon - Génie des Matériaux - 26 Septembre 1997
Jury : L. Mora - Ponsonnet, P. Blanchard, K. Dang Van, C. Esnouf, E. Gaffet, E. Rosset, A.B. Vannes, L. Vincent

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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)

SYNTHESIS CONDITIONS FOR OBTAINING FINE POWDERS OF PURE AND ALLOYED TITANIUM ALUMINIDE WITHOUT MILLING
Sinelnikova VS. Prilutskii EV. Markovskaya VY. Smirnov VP. - Powder Metallurgy & Metal Ceramics. 35(9-10):501-504, 1996
We have studied processes not involving milling for obtaining fine powders of either pure titanium aluminide or titanium aluminide alloyed with chromium and manganese. We consider the effect of heat treatment, pressing pressure, and also the degree of dilution of the starting mixture with titanium aluminide. We show that manganese and chromium form solid solutions based on AlTi that cause an elevation in microhardness by 50% and 80%

respectively. We have investigated treatment of titanium aluminide powder particles with an Al2O3 coating.

SYNTHESIS, MAGNETIC PROPERTIES AND FORMALISM OF MAGNETIC PROPERTIES OF HIGH-QUALITY REFINED ND2FE14B POWDERS FOR PERMANENT MAGNET DEVICES

Ram S. - Journal of Materials Science. 32(15):4133-4148, 1997

Stable Nd2Fe14B powders of refined grain size of 0.1-1.0 mu m were prepared using a combination of the rapid quenching (of the melt into thin ribbons), mechanical attrition and grain-surface passivation (or surface hardening) and coating by a thermally rigid, adhesive and corrosion-proof material in air. The ribbons (of 15-30 mu m thickness) were cut, crushed and milled under H-2 gas at approximately 1 bar and room temperature to give hydrided Nd2Fe14BHx less than or similar to 5, flakes of 1-5 mu m sizes, which are brittle and easily obtained in powder form by high-energy ball milling. The interstitial H atoms in the hydride sample were desorbed by slowly heating (5 degrees C min(-1)) the sample between 25 and 600 degrees C in N-2 gas (which helps the desorption of the H atoms without decomposition of the sample) in a reactor and then pumping off the total gas at 600 degrees C. The H-desorbed specimen, when annealed at 600-800 degrees C under a dynamic vacuum, results in a refined powder, showing a characteristically high remanence, J(r) of 9-12 kG, together with a high intrinsic coercivity, H-ci, of 10-28.3 kOe, depending on the size and surface structure of the grains. This powder is highly pyrophoric and catches fire in open air but can be stabilized by passivating and coating the grain surfaces with a mixture of carbon, AlN and Nd2O3 by milling the mixture in a suitable organic liquid (to allow the additives to adhere the sample without excess oxidation) followed by annealing at an elevated temperature in N-2 gas at approximately 1 bar. In this process, the separated Nd2Fe14B grains acquire a thin nitride-carbide (probably amorphous) stabilized surface passivation layer which prevents further oxidation of the sample in air at room temperature. The passivation layer, in combination with a thin film of the Nd-rich intergranular phases, if any, peculiarly appears to be non-magnetic compared with the main ferromagnetic Nd2Fe14B phase. It keeps the ferromagnetic Nd2Fe14B grains separated and thus inhibits mixing between the local magnetic lines of forces confined to them. As a result, they behave like ideal single-domain particles and therefore exhibit a reasonably improved H-ci value, without a significant decrease in the high J(r) or the high saturation magnetization M-s which are useful for the high-energy-density magnets and related devices and components. The results are modelled and discussed with microstructures, magnetic properties, thermal stability and loss, if any, in the mass of the specimens during exposure to ambient atmosphere.

METASTABLE PHASES OF AL-FE SYSTEM BY MECHANICAL ALLOYING

Huang B. Ishihara KN. Shingu PH. - Materials Science & Engineering A - 231(1-2):72-79, 1997

The preparation and characterization of metastable phases of the Al-Fe alloy system by mechanical alloying are reported. In Al-rich (up to 10 at.% Fe) alloys, the supersaturated f.c.c, solid solution of Fe in Al (up to 1 at.% Fe) is formed. Almost complete amorphization is confirmed in the composition range 17-33 at.% Fe. The metastable disordered b.c.c. solid solution of about 10 nm in grain size has also been formed by ball-milling for over 180 h in Fe-rich (above 50 at.% Fe) alloys. Examination of lattice parameter and magnetization have shown that the composition range and degree of disorder are comparable to those formed by crushing and spatter deposition.

MECHANICAL ALLOYING AND THERMAL STABILITY OF AL67TI25M8 (M=CR, ZR, CU)

Materials Science & Engineering A - 231(1-2):111-116, 1997

By means of X-ray diffraction (XRD), transmission electron microscopy (TEM) and differential scanning calorimetry (DSC) measurements, the sequence of the metastable phase formation and thermal stability of the mechanically alloyed Al67Ti25M8 (M = Cr, Zr, Cu) have been studied systematically. It was found that with the addition of Cr and Zr, nanocrystalline ordered Ll(2)-Al3TiM compounds were obtained after 40 h of milling. Subsequent annealing in the differential scanning calorimeter causes a grain growth process of the nanocrystallites. With the addition of Cu, the final product was nanocrystalline disordered Al(Ti,Cu) solid solution. Two types of instability upon thermal annealing for the nanocrystalline disordered Al(Ti,Cu) solid solution were observed: an instability against the grain growth; and an instability against L1(2) chemical ordering. The grain growth and L1(2) chemical ordering seem to occur simultaneously at temperatures in the range 400-700 degrees C. Our experimental results suggest that Cr and Zr may be promising alloying additions to stabilize the L1(2) structured Al3Ti compound for further application.

NANOSTRUCTURE CHARACTERIZATION OF MECHANICAL ALLOYED AND CONSOLIDATED IRON ALLOYS

Rawers J. Krabbe R. Duttlinger N. - Materials Science & Engineering A - 230(1-2):139-145, 1997

High-energy ball milling (which is readily adoptable to commercial application) was used to develop sufficient quantities of nanostructured material to produce compacts capable of being measured for macroscopic properties. Characterization of the ball-milled powders show that grain boundary properties play a significant role in the overall properties of the milled powder. Nearly full-dense compacts were produced by hot-pressing. Characterization of the strength properties of these compacts show that there was little influence of hardness, density, or alloy composition on the failure properties. The range of failure stress was large and when fitted to a Weibull distribution suggest that failure was the result of flaws or cracks resulting from the hot-pressing. Hardness data, commonly used to evaluate the strength of nanostructured materials: showed no correlation to tensile strength, but correlated highly to compression maximum stress.

MECHANOMAKING OF FE/AL2O3 AND FECR/AL2O3 NANOCOMPOSITES POWDERS FABRICATION

Matteazzi P. Alcala M. - Materials Science & Engineering A- - 230(1-2):161-170, 1997

Nanocomposite powders (Fe or Fe-Cr alloy)/alpha-Al2O3 (75 and 85 vol.%) were obtained by room-temperature high-energy milling powder mixtures of hematite (and chromium oxide) with aluminum and alumina in a high-capacity mill for 8-10 h. The composition of iron and iron alloys was followed by Mossbauer spectroscopy, while the appearance of other phases was revealed by X-ray diffraction. The powder particles produced are assemblies of grains (10-20 nm in size) with a wide size distribution (from well below 1 mu m up to several hundreds) and low porosity (fully dense particles). Both the metallic and ceramic phases have crystallite sizes below 15 nm for all the compositions investigated. Nano-nano type ceramic nanocomposites were, therefore, obtained.

PROPERTIES OF MECHANICALLY ALLOYED AL75-XTI25CRX

Lee KY. Ahn JH. - Materials Science & Engineering A - 229(1-2):63-67, 1997
Mechanical alloying and hot-consolidation of Al7(5-x)Ti(25)Cr(x) (x = 6, 8, 10, 12, 14) have been investigated to see the alloying effect of Cr on the phase changes and the properties of AI(3)Ti. Mechanical alloying resulted in the formation of a metastable f.c.c. phase. The phase was transformed into L1(2) and second phases during hot-consolidations and subsequent annealing. For the alloy with the composition of x = 6 (Al69Ti25Cr6), the DO22 phase coexisted with these phases. Such alloy exhibited higher strength but lower ductility than the other alloys exempt DO22 structure. Room temperature ductility was markedly improved by subsequent annealing of hot-pressed samples. A good combination of mechanical strength and ductility was observed for the alloys with the composition of x = 8-14 (Al75-xTi25Crx).

PREPARATION OF THE BI8SB32TE60 SOLID SOLUTION BY MECHANICAL ALLOYING
Pierrat P. Dauscher A. Lenoir B. Martinlopez R. Scherrer H. - J. Mat. Science. 32(14):3653-3657, 1997
Bi2Te3, Sb2Te3 and Bi8Sb32Te60 thermoelectric materials have been prepared by mechanical alloying using a high energy planetary ball milt. The alloy formation was followed by X-ray diffraction (XRD), the morphology by scanning electron microscopy (SEM) and the composition by electron microprobe. The samples of Bi8Sb32Te60 were prepared in a reasonable milling time (less than 8 h) by mechanical alloying of binary alloys (Bi2Te3 and Sb2Te3). The single phase Bi8Sb32Te60 solid solution obtained presents convenient stoichiometry and good homogeneity in composition.

SOLID-STATE REACTION IN NANOCRYSTALLINE FE/SIC COMPOSITES PREPARED BY MECHANICAL ALLOYING
Shen TD. Koch CC. Wang KY. Quan MX. Wang JT. - J. Materials Science. 32(14):3835-3839, 1997
Different solid-state reactions, i.e. Fe + SiC --> Fe3C + Fe(Si), and Fe + SiC --> Fe3Si + Fe2Si + C, were found in mechanically alloyed nanocrystalline Fe/SiC composites induced by prolonged milling or heat treatment, respectively. The solid-state reaction between nanocrystalline iron and SIC upon heating is greatly enhanced when compared with that between bulk iron and SiC. It is believed that the prolonged milling-induced reaction is related to the changed thermodynamics and kinetics while the heat-treatment-induced reaction, completed during a short time, is attributable to the changed reaction kinetics.

ALUMINIUM-LITHIUM/SICP COMPOSITES PRODUCED BY MECHANICALLY MILLED POWDERS
Hanada K. Khor KA. Tan MJ. Murakoshi Y. Negishi H. Sano T. - J. Mat. Proc. Techn.. 67(1-3):8-12, 1997
Aluminium-lithium/SiCp composites have attracted special interest because of their low density and high modulus. However, the major problem is the difficulty in securing uniform mixing of the Al-Li matrix powders and the SiC particles. Conventional mixing cannot prevent the reinforcements from agglomerating at prior particle boundaries. This paper reports the application of a mechanical milling method for mixing Al-Li powders with SiC particles. The SiC particulates as reinforcements are dispersed homogeneously in this method. Al-Li/SiCp composite powders mechanically milled at the high speed of 500 rpm are extruded at 773 K. The microstructure and mechanical properties are discussed. The optimal milling time for Al-Li/10vf%SiCp is 2 h; the: resulting Young's modulus being 86 GPa. The microstructures are studied by transmission electron microscope, The composites appeared to have good interfaces and fine grain structures.

DIFFUSION IN MECHANICAL ALLOYING
Lu L. Lai MO. Zhang S. - J. Materials Processing Technology. 67(1-3):100-104, 1997
This paper studies the roles of two key factors in the mechanical alloying process, these factors bring: activation energy, which is related to the formation of defects during the collision of powder particles; and crystalline size, which is related to the formation of nanometer crystalline during mechanical alloying. According to thermodynamic theory, the decrease in activation energy can result in an increase in diffusivity at constant temperature. Therefore, a decrease in activation energy is equivalent to an increase in temperature. High diffusivity can be obtained by creating a large number of defects through mechanical alloying. In addition, by creating nanometer size crystalline particles through the repeated fracturing and cold-welding of the powder particles, diffusion can take place easily through the grain boundaries. Consequently, elements which are difficult to diffuse may be alloyed using this technique.

MECHANICAL ALLOYING OF A TIC-TIN CERAMIC SYSTEM
Zhang S. Tam SC. - Journal of Materials Processing Technology. 67(1-3):112-116, 1997
Reported here is a preliminary study of the mechanical alloying (MA) process of an all-ceramic-phases component, i.e., TiC + TiN. The respective ceramic powders were mixed in weight proportions of 50:50 and 70:30 and milled in a planetary ball mill at a ball-to-powder weight ratio of 20:1. High angle XRD peaks were used to calculate the lattice parameters before and after milling for different milling times. The lattice parameter measurements suggested that Ti(C,N) solid solution was formed during the MA process. Inter-particle necking was observed. The rate of the solid solution reaction seems to be independent of the compositional change in the TiC-TiN mixture. Particle size refinement is achieved mostly during the first few hours of milling.

NANOPHASE FLUORITE-STRUCTURED CEO2-ZRO2 CATALYSTS PREPARED BY HIGH-ENERGY MECHANICAL MILLING - ANALYSIS OF LOW-TEMPERATURE REDOX ACTIVITY AND OXYGEN STORAGE CAPACITY
Trovarelli A. Zamar F. Llorca J. Deleitenburg C. Dolcetti G. Kiss JT. - J. Catal.. 169(2):490-502, 1997
The utilization of mechanical milling for the preparation of catalysts based on ceria structurally modified with zirconia is presented. It is shown that room-temperature high-energy hall milling is an effective tool for the synthesis of nanophase CeO2-ZrO2 solid solution in a wide composition range, The use of combined X-ray diffraction analysis, Raman spectroscopy, and electron microscopy indicate that the milling process induces the formation of true solid solutions with a contraction of the cell parameter for cubic ceria following the introduction of Zr into the lattice, This, in turn, remarkably enhances the oxygen storage/transport and redox capacity compared to pure ceria and zirconia or to a simple mixture thereof. An unusual resistance to high-temperature cycling was also evidenced, These features were analyzed by the study of the reduction profile of doped ceria carried out by temperature-programmed reduction at different milling times, The oxygen storage capacity (OSC) of the catalysts was also evaluated; both the total and the kinetic accessible OSC indicated that the best composition is CexZr1-xO2 with x > 0.5. This was correlated to the

structural features and to the presence of a high concentration of ions with redox character (i.e., Ce4+ ions) which favor oxygen mobility.

AMORPHIZATION REACTION OF NI-TA POWDERS DURING MECHANICAL ALLOYING

Lee PY. Yang JL. Lin CK. Lin HM. - Metallurgical & Materials Transactions A - 28(7):1429-1435, 1997

This study examined the amorphization behavior of NixTa100-x alloy powders synthesized by mechanically alloying (MA) mixtures of pure crystalline Ni and Ta powders with a SPEX high energy ball mill. According to the results, after 20 hours of milling, the mechanically alloyed powders were amorphous for the composition range between Ni10Ta90 and Ni80Ta20. A supersaturated nickel solid solution formed for Ni90Ta10 as well. X-ray diffraction analysis reveals two different types of amorphization reactions. Through an intermediate solid solution and by direct formation of amorphous phase. The thermal stability of the amorphous powders was also investigated by differential thermal analysis. As the results demonstrated, the crystallization temperature of amorphous Ni-Ta powders increased with increasing Ta content. In addition, the activation energy of amorphous Ni-Ta powders reached a maximum near the eutectic composition.

EFFECT OF MILLING TEMPERATURE ON MECHANICAL ALLOYING IN THE IMMISCIBLE CU-TA SYSTEM

Xu J. He JH. Ma E. - Metallurgical & Materials Transactions A - 28(7):1569-1580, 1997

Elemental powder blends with atomic composition of Cu100-xTax (x = 10, 30, 50, 70, and 90) were ball milled in a SPEX mill at several temperatures (room temperature (RT), liquid nitrogen temperature (LN2T), -80 degrees C, and 95 degrees C) to examine the effect of milling temperature on the extent of alloying and microstructural refinement. For the Cu-rich powders ($10 < x < 50$), high-energy ball milling to steady state at all temperatures produced a mixture of nanocrystalline Cu and Ta with no observable extension of mutual solid solubility. Compared with milling at RT, cryomilling (LN2T) caused further refinement of Cu crystallites, while the same steady-state grain size was reached for Ta crystallites. On the Ta-rich side ($50 < x < 90$), ball milling at all temperatures led to refined Cu and Ta grain sizes. Partial amorphization seemed to be present, which apparently increased in extent with increasing contamination from the milling media upon extended milling. Very similar results were obtained for milling at RT and LN2T. It was concluded that high-energy ball milling at LN2T did not drastically enhance the amorphization reaction between Cu and Ta nor extend their mutual solubility. The limited power of cryomilling to alloy immiscible elements such as Cu-Ta is explained as a consequence of the inability to fully suppress, during energetic collisions, the atomic mobility responsible for phase separation even when the milling is conducted at the nominal LN2T. The temperature dependence of milling-induced microstructural refinement and alloying is analyzed in terms of the dynamics of the generation and annihilation of the nonequilibrium vacancies in an externally driven system. It is predicted that externally forced mixing as well as diffusion assisted by high-energy ball milling can be merely weakly temperature dependent between RT and LN2T. As a result, the extension of solubility by using cryomilling is feasible only in limited systems, and this process cannot be expected to alloy all immiscible elements.

STRUCTURE AND PROPERTIES OF ATTRITION-MILLED ALUMINIUM POWDER

Rodriguez JA. Gallardo JM. Herrera EJ. - Journal of Materials Science. 32(13):3535-3539, 1997

Aluminium powder has been attrition milled in the presence of 1.5 wt% of a wax. The aim was to achieve a mechanically alloyed powder amenable to powder metallurgy processing. Changes in particle size and form, microstructure, hardness and other properties of powders with milling time has been studied. Under the experimental conditions employed, a process time of 10 h was selected for the mechanical alloying of Al powder. The powder milled in this way shows a Vickers microhardness (127 HV) more than six times higher than the starting powder (20 HV), a coarser particle size (mean particle size is doubled) and a better flowability.

INFLUENCE OF THE REINFORCEMENT ADDITIONS ON THE CREEP STRENGTH OF DISPERSION STRENGTHENED ALUMINIUM ALLOYS

Elmagd E. Ismail Y. - Metall. 51(6):311-317, 1997

A comparitive study of creep behaviour was conducted on eight different aluminium alloys which are strengthened by carbide and oxide dispersoids with three different volume fractions (C002, C101, C102). The matrix materials are AISi20, AlSi20Fe5, AlMgSi1, AlSi12Fe5, AlMg1, AlMg2, AlMg4 and AlCuMg1. The dispersoids were added to the matrix material powder and processed by mechanical alloying. Creep tests were carried out on cylindrical specimens at 300 degrees C and 400 degrees C up to fracture. These products were fabricated by cold isostatic pressing (CIP) followed by hot extrusion to improve its mechanical properties. The applied stress was chosen to have a fracture life ranging from 0.1 to 3000 hours. It was concluded that the adding of 1% carbon and 2% oxygen as a dispersioid materials in the aluminium alloy matrix has a great effect on the creep strength in comparison with the addition of the alloying elements such as Si, Mg, Cu and Fe in high percentages up to 20%.

HIGH-ENERGY BALL-MILLING-INDUCED NON-EQUILIBRIUM PHASE TRANSFORMATIONS

Chen Y. Williams JS. - Materials Science & Engineering A - 226:38-42, 1997

alpha-Fe powder has been ball milled in an anhydrous atmosphere at two different temperatures (room temperature and 200 degrees C). The composition and structure of milled samples were investigated using X-ray diffraction, Mossbauer spectroscopy, combustion analysis. and thermal analysis. A series of non-equilibrium phase transformations was observed during milling. The nitriding reaction sequence at room temperature established as a function of milling time (an increase of nitrogen in the powder) corresponds to a high-temperature reaction sequence in the equilibrium phase diagram. In contrast, hot milling at 200 degrees C leads to a lower average nitrogen content in milled powders. It seems that formation of metastable nitride phases with high N content induced by ball impacts is in competition with thermally activated decomposition of these phases to equilibrium states with low N content during milling at 200 degrees

SOLID-STATE AMORPHIZATION IN TA-BASED ALLOY SYSTEM BY MECHANICAL ALLOYING TECHNIQUE

Lee PY. Yang JL. - Materials Science & Engineering A - 226:43-47, 1997

Amorphization of mechanically alloyed TM-Ta (TM: Fe, Co, Ni) powders was performed in this study. The results

indicated that complete amorphization was possible for the composition ranges TM10Ta90-TM80Ta20 (TM: Ni, Co) and Fe10Ta90-Fe70Ta30. The range is larger than for amorphous Ni-Ta alloys prepared by the splat-quenching process and compatible with those of amorphous alloys of Co-Ta and Fe-Ta systems produced by vapor quenching methods. A supersaturated solid solution formed for Co- and Fe-rich compositions. The thermal stability was investigated by differential thermal analysis. The crystallization temperature of amorphous Co-Ta and Ni-Ta powders was found to be proportional to Ta content and higher than those of amorphous alloys produced by rapid cooling techniques.

MECHANICAL ALLOYING OF AL70PD20MN10 ELEMENTAL POWDER MIXTURE

Asahi N. - Materials Science & Engineering A

Pure crystalline powders of Al, Pd and Mn were mechanically alloyed using a planetary ball mill (Fritsch P-7) with an intensity setting of 7 in an Ar atmosphere. Formation of an icosahedral phase (i-phase) quasicrystal in mechanically alloyed Al70Pd20Mn10 powder and its structure were examined by X-ray diffraction analysis and transmission electron microscopy. The i-phase quasicrystal with a face-centered 6-dimensional hypercubic lattice was formed in Al70Pd20Mn10 powder milled for 30 h. However, it was found from deconvolution of the X-ray diffraction profile that the i-phase contained other phases, such as Al3Pd2 intermetallic compound and unidentified phases. Electron microscopic observation shows that a number of Al3Pd2 intermetallic compound in the shape of a spot with a diameter of about 10 nm are distributed irregularly throughout the quasicrystalline matrix.

BALL MILLING OF FE-75-C-25 - FORMATION OF FE3C AND FE7C3

Campbell SJ. Wang GM. Calka A. Kaczmarek WA. - Materials Science & Eng. A - 226:75-79, 1997

Powder mixtures of Fe-75-C-25 (both graphite and activated carbon) have been ball-milled in vacuum for periods of up to 285 h. X-ray diffraction, Mossbauer spectroscopy and thermal analysis measurements indicate that an amorphous Fe3C-type phase is produced on short-term milling (less than 70 h), with a crystalline Fe3C being obtained on further milling to 140 h. This Fe3C-type phase was found to undergo partial carbon oxidation between 500 and 1000 degrees C during thermogravimetric measurement, indicating the metastable state of this phase. The carbon-rich Fe7C3 phase was observed on extended milling of Fe75C25 (graphite) to 285 h, in agreement with earlier findings.

MECHANOCHEMICAL NITRIDATION BY BALL MILLING IRON WITH PYRAZOLE - A STRUCTURAL INVESTIGATION

Wang GM. Campbell SJ. Kaczmarek WA. - Materials Science & Engineering A - 226:80-83, 1997

Nitrides of iron have been obtained by mechanochemical treatment of Fe powder with the amine compound, pyrazole (C3H4N2). The structural developments which occur during the milling of the Fe and pyrazole powder mixture have been monitored by x-ray diffraction. The hexagonal epsilon-Fe3Nx phase with x similar to 1.1-1.2 was found to be the main milling product. The decomposition of the nitride and the formation of iron carbide Fe3C have been observed after transient heating at 550 degrees C. Analysis of x-ray diffraction data for the as-milled and thermally treated powders has led to information on the locations of the carbon and the hydrogen stemming from the pyrazole compound.

A STUDY OF AMORPHOUS FE58TA42 ALLOYS PRODUCED BY MECHANICAL ALLOYING

Cooper RJ. Randrianantroandro N. Cowlam N. Greneche JM.- Materials Sci. & Eng. A- 226:84-89, 1997

The solid state 'amorphisation' reaction in Fe58Ta42 samples produced by mechanical alloying (MA) has been studied using X-ray and neutron diffraction; neutron small-angle-scattering (SAS) and Mossbauer spectroscopy. The diffraction and Mossbauer measurements have shown that the reaction is rapid and that the parental elements are transformed into a genuinely amorphous alloy after 6 h of MA in a conventional Spex 8000 mill. The total structure factor S(Q) of the amorphous alloy has been obtained and the reduced radial distribution G(r) derived from its Fourier transform. In addition, the neutron SAS has been used to study the changes in the large scale structures of the samples in the initial stages of the reaction, which were measured in the as-received and contrast-matched states. The results are consistent with the presence of a duplex, filamentary, composite whose interfaces are fractal, in those samples with the shortest times of MA.

STUDY ON MECHANISM OF MECHANICAL ACTIVATION

Chen Y. Hwang T. Marsh M. Williams JS.- Materials Science & Engineering A - 226:95-98, 1997

A mixture of ilmenite and graphite with a mass ratio (1:1) was ball milled at room temperature and subsequently annealed under Ar flow. The enhanced carbothermic reactions were monitored by using thermogravimetric analysis and X-ray diffraction analysis. A series of reduction reactions occurs with a higher reaction rate in a temperature range lower than that without pre-milling. A longer milling time leads to a lower reaction temperature. This effect of mechanical activation induced by ball milling results from a composite structure formed during the milling process.

EXPLOSIVE CONSOLIDATION OF MECHANICALLY ALLOYED TI-AL ALLOYS

Szewczak E. Paszula J. Leonov AV. Matyja H. - Materials Science & Engineering A - 226:115-118, 1997

Ti-Al intermetallics are of great interest because of their attractive mechanical properties, but they require advanced processing methods, due to their low ductility at room temperature. In this work, the following processes were employed in order to obtain bulk TiAl and Ti3Al intermetallics: (1) mechanical alloying: ball milling of elemental Ti and Al powders caused formation of supersaturated solid solutions; (2) explosive consolidation: in this operation no phase transitions were observed and sample densities after consolidation reached over 90% of theoretical values; (3) annealing: resulted in additional increase of material density and caused phase transitions leading to formation of TiAl and Ti3Al compounds.

STRUCTURE CHANGES OF MELT-SPUN AND MECHANICALLY ALLOYED AGCUCD EUTECTIC ALLOY

Dutkiewicz J. Litynska L. Maziarz W. - Materials Science & Engineering A - 226:285-289, 1997

Structural changes of melt-spun ribbons and mechanically alloyed AgCuCd alloy powders of ternary eutectic composition 17 wt.% Ag, 29.5 wt.% Cu and 53.5 wt.% Cd were studied using X-ray diffraction, and transmission and scanning electron microscopy. The equilibrium eutectic consists of alpha, beta and gamma phases. Rapidly quenched

ribbon consists of metastable beta(AgCd) phase showing B2 ordering with modulations following the [100] direction originating from spinodal decomposition. A small fraction of a fine mixture of randomly oriented alpha and gamma crystallites (about 40 nm in size) with large beta inclusions was found in very thin areas transparent for the transmission electron microscope observations. Fine globular clusters of gamma(Ag, Cu)(5)Cd-8 precipitates of the same crystallographic orientation as the beta matrix were observed in thicker areas. The mechanical alloying of mixed powders caused both formation of supersaturated a solid solutions after 32 h of milling and secondary crystallisation of gamma(Ag, Cu)(5)Cd-8 and beta(2)(Cu2Cd) phases after 64 h of milling. A decrease of the amount of initial elements is accompanied by refinement of its grain size. Transmission electron microscopy observations performed using the samples milled for 32 and 64 h revealed the nanocrystalline structure of alpha solid solution and gamma and beta(2) particles of about 15 nm size.

STRUCTURAL RELAXATION IN AMORPHOUS NI-TI ALLOYS PREPARED BY MECHANICAL ALLOYING

Makifuchi Y. Terunuma Y. Nagumo M. - Materials Science & Engineering A - 226:312-316, 1997

Structural changes in amorphous states during prolonged milling after the completion of amorphization have been investigated through the crystallization behaviors with Ni-Ti alloys of compositions corresponding to the intermetallic compounds. In 33Ni-67Ti, the crystallization to NiTi2 takes place in two steps, a higher crystallization temperature dominating after prolonged milling. In 50Ni-50Ti, structural relaxation leading to the crystallization to NiTi2 and Ni3Ti takes place. In 30Ni-70Ti, which is close to the border of the amorphization range, the initially formed amorphous phase turns into a supersaturated solid solution of Ti in Ni.

MECHANICAL ALLOYING OF HIGHLY PROCESSABLE GLASSY ALLOYS

Eckert J. - Materials Science & Engineering A-- 226:364-373, 1997

Glasses are generally produced from the undercooled liquid state by rapid quenching methods or quasi-statically at slow cooling by the effective control of heterogeneous nucleation sites. For metallic systems the latter method recently has led to the development of multicomponent metallic glasses with large glass forming ability and a wide supercooled liquid region before crystallization. Large-scale bulk samples of such alloys can be produced by conventional casting techniques. Alternatively, glass formation can be achieved by solid-state processing without passing through the liquid state. This crystal-to-glass transition is observed when a sufficiently high energy level is reached and kinetic conditions prevent the establishment of equilibrium. Hence, mechanical alloying as a special form of solid-state reaction technique and subsequent consolidation of the resulting powders above the glass transition temperature can be used to prepare bulk metallic glasses via the powder metallurgy route. The glass formation and the thermal stability of mechanically alloyed glassy alloys are compared with data for melt quenched samples showing that basically the same glassy state can be reached approaching it from the liquid or the solid state. Special emphasis is given to the glass forming ranges achievable by the different techniques and to the influence of impurities. Results for consolidated bulk samples are presented and compared with data for cast bulk specimens.

PROGRESS OF SOLID-STATE REACTION DURING MECHANICAL ALLOYING OF ZR-AL-CU-NI BULK METALLIC GLASS-FORMING ALLOYS

Seidel M. Reibold M. Bacher I. Bauer HD. Eckert J. Schultz L. - Mater. Sci. & Eng. A - 226:383-387, 1997

The progress of amorphization in mechanically alloyed Zr65Al7.5Cu17.5Ni10 powders has been investigated by X-ray diffractometry, differential scanning calorimetry, scanning and transmission electron microscopy and wavelength-dispersive X-ray spectroscopy. For this, mixtures of elemental powders were used as starting materials. Detailed investigations of the microstructural evolution during milling indicate that the amorphization proceeds by solid-state reaction, similar to what is known for mechanical alloying of binary alloy systems and other multicomponent alloys. The results are compared with interdiffusion experiments on diffusion couples of Zr-Al and Cu-Ni solid solutions isothermally annealed at elevated temperatures.

SYNTHESIS OF MULTICOMPONENT FE-BASED AMORPHOUS ALLOYS WITH SIGNIFICANT SUPERCOOLED LIQUID REGION BY MECHANICAL ALLOYING

Schlorke N. Eckert J. Schultz L. - Materials Science & Engineering A - 226:425-428, 1997

Mechanical alloying was utilized to synthesize multicomponent Fe-Al-P-B-C alloys. The resulting phases were characterized by X-ray diffraction, differential scanning calorimetry, and isothermal annealing experiments. Coexistence of an amorphous and a nanoscale b.c.c. phase with an average grain size of about IO MI was found after milling. The amorphous phase exhibits a rather wide supercooled liquid region before crystallization. Some characteristic thermal properties of the different alloys are presented and the composition dependence of the glass transition temperature, the crystallization temperature and the crystallization products are compared with data for rapidly quenched samples.

CALORIMETRIC AND STRUCTURAL ANALYSES OF MECHANICALLY ALLOYED AND RAPIDLY QUENCHED ZR-NI-AL ALLOYS

Kuhnast FA. Held O. Regnier F. Illekova E. - Materials Science & Engineering A - 226:463-467, 1997

Mechanical alloying and rapid quenching from the melt are used to form Zr-Ni-Al amorphous alloys. An experimental investigation of the amorphous to crystalline transformation is carried out using differential scanning calorimetry, X-ray diffraction and microprobe analysis. The experiments are managed to analyse and correlate the results both for mechanically alloyed powders and rapidly quenched ribbons: (i) a preliminary analysis of the kinetics of relaxation and crystallization, (ii) a connection between the final crystallized phases and the equilibrium ternary phase diagram.

NANOSTRUCTURE FORMATION AND STEADY-STATE GRAIN SIZE OF BALL-MILLED IRON POWDERS

Borner I. Eckert J. - Materials Science & Engineering A - 226:541-545, 1997

Nanocrystalline iron with grain sizes of 13-30 nm has been prepared by mechanical attrition using several different types of mills with different milling intensity. The grain size and the strain of the powders as well as the milling time for achieving the ultimate grain size strongly depend on the milling intensity and the milling temperature. The results

obtained under different experimental conditions are discussed in the framework of previously proposed models for the evolution of nanocrystalline grain sizes during mechanical deformation.

STRUCTURAL AND MAGNETIC PROPERTIES OF NANOCRYSTALLINE (FE-CU)(93)ZR-7 ALLOYS PREPARED BY MECHANICAL ALLOYING

Stiller C. Eckert J. Crespo P. Roth S. Schultz L. - Mater. Science & Engineering A - 226:577-580, 1997

Nanocrystalline ferromagnetic (FexCu100-x)(93)Zr-7 alloys (x = 50, 70) were prepared by mechanical alloying of elemental powders, For x = 70 a supersaturated bcc solid solution forms upon milling, whereas a supersaturated fee solid solution is observed for x = 50. The deformation during milling reduces the grain size of the alloys to about 5 nm. Heating to elevated temperatures leads to structural relaxation, phase separation and grain growth of the metastable solid solutions. Upon heating to 670-970 K the as-milled Fe-rich bcc phase decomposes into alpha-Fe and a Cu-rich solid solution, whereas the initially Cu-rich fee solid solution transforms into a mixture of pure Cu and to pure alpha-Fe. The magnetic properties of as-milled and annealed powders will be discussed with respect to the composition and the microstructure of the material, the plastic deformation during milling and the strain release and decomposition upon subsequent heating.

RECENT ADVANCES IN THE CATALYTIC PROPERTIES OF METASTABLE MATERIALS

Hashimoto K. - Materials Science & Engineering A - 226:891-899, 1997

New catalysts and electrocatalysts which are amorphous alloys themselves or prepared from amorphous and other metastable alloys are reviewed in this paper. The most important characteristic of metastable materials including amorphous and nanocrystalline alloys from the chemical point of view is the homogeneous single phase nature consisting of a variety of elements, whose concentrations sometimes exceed the solubility limits at equilibrium. For the enhancement of the activity various treatments are carried out before catalytic reaction, such as oxidation-reduction and selective dissolution of alloy constituents. For example, after immersion in HF, amorphous Cu-Zr alloys show higher activity than amorphous Cu-Ti alloys for dehydrogenation of 2-propanol and hydration of acrylonitrile. This is due to dissolution of smaller amounts of titanium from Cu-Ti alloys than dissolution of zirconium from Cu-Zr alloys in HF, since the corrosion resistance of titanium in HF is higher than zirconium. The catalysts prepared from some amorphous nickel alloys are most effective for methanation of CO2 at atmospheric pressure. Amorphous nickel-refractory metal alloys are the best cathode materials for electrolytic hydrogen evolution. The catalyst for CO2 methanation and the electrode for electrolytic hydrogen evolution are used for building a CO2 recycling plant to avoid global warming and to supply abundant energy.

METASTABLE TI-RU-FE-O NANOCRYSTALLINE ALLOYS FOR APPLICATIONS IN THE CHLORATE INDUSTRY

Schulz R. Blouin M. Yip SH. Huot J. Boily S. Roue L. Baazi T. Caron P. Guay D. - Mater. Sci. & Engineering A - 226:915-919, 1997

Metastable Ti2FeRuOx alloys have been produced using three different methods: rapid melt-quenching, vapor phase condensation and high energy ball milling. Depending on the technique used and on the oxygen composition, metastable amorphous or nanocrystalline structures with various degrees of chemical order are produced. The structural properties have been studied using X-ray diffraction, scanning Auger microscopy and thermal analyses. The electrocatalytic properties for the hydrogen evolution reaction in conditions identical to that of the sodium chlorate industry are presented.

ACTIVATION OF AMORPHOUS CU-M (M=TI, ZR OR HF) ALLOY POWDERS MADE BY MECHANICAL ALLOYING

Molnar A. Domokos L. Katona T. Martinek T. Mulas G. Cocco G. Bertoti I. Szepvolgyi J. - Materials Science & Engineering A - 226:1074-1078, 1997

Amorphous Cu-M alloy powders (Cu40Ti60, Cu50Zr50 and Cu65Hf35) produced by mechanical alloying are activated by being applied as catalyst in the transformation of various alcohols at elevated temperature (523-573 K). During the dehydrogenation of 2-propanol to acetone and the transformations of allyl alcohol to form propanal and 1-propanol active, stable and selective catalysts are generated from Cu-Zr and Cu-Hf. DSC, XRD and XPS data indicate bulk crystallization and copper segregation to the surface. Due to surface titanium enrichment Cu-Ti proved to be inferior to the other two alloys.

MULTIRANGE FRACTALS IN MATERIALS - APPLICATIONS TO FRACTURE AND MECHANICAL ALLOYING UNDER BALL MILLING

Lung CW. - Journal of Materials Science & Technology. 13(4):255-259, 1997

A new model of multirange fractals is proposed to explain the experimental results observed on the fractal dimensions of the fractured surfaces in materials. A new explanation to the Williford's multifractal curve on the relationship of fractal dimension with fracture properties in materials has been given. It shows the importance of fractorizing out the effect of fractal structure from other physical causes and separating the appropriate range of scale from multirange fractals. Mechanical alloying process under ball milling as a non-equilibrium dynamical system has been also analyzed.

FORMATION OF A FCC AL54TI23C23 METASTABLE PHASE BY BALL MILLING

Wu NQ. Li ZZ. Wang GX. Mu JM. - Journal of Materials Science & Technology. 13(4):345-347, 1997

The powder mixture of Al, Ti and graphite has been mechanically alloyed in a planetary ball mill. The structural evolution of as-milled powder sample has been characterized by XRD, DTA. The results show that the amorphous phase is formed first at an early milling stage, then crystallization occurs during further milling, leading to formation of a nanocrystalline fee metastable phase. In contrast, during annealing the amorphous phase is crystallized to the equilibrium phase instead of the fee phase. This indicates that crystallization during ball milling is different from that induced by annealing.

MECHANICALLY DRIVEN ALLOYING AND STRUCTURAL EVOLUTION OF NANOCRYSTALLINE FE60CU40 POWDER

Dong YD. Ma XM. Yang YZ. Liu FX. Wang GM. - J. Mat. Sci. & Technology. 13(4):354-358, 1997

Highly supersaturated nanocrystalline fee Fe60Cu40 alloy has been prepared by mechanical alloying of elemental powders. The phase transformation is monitored by X-ray diffraction (XRD), Mossbauer spectroscopy and extended X-ray absorption fine structure (EXAFS). The powder obtained after milling is of single fee structure with grain size of nanometer order. The Mossbauer spectra of the milled powder can be fitted by two subspectra whose hyperfine magnetic fields are 16 MA/m and 20 MA/m while that of pure Fe disappeared. EXAFS results show that the radial structure function (RSF) of Fe K-edge changed drastically and finally became similar to that of reference Cu K-edge, while that of Cu K-edge nearly keeps unchanged in the process of milling. These,imply that bcc Fe really transforms to fee structure and alloying between Fe and Cu occurs truly on an atomic scale. EXAFS results indicate that iron atoms tend to segregate at the boundaries and Cu atoms are rich in the fee lattice. Annealing experiments show that the Fe atoms at the interfaces are easy to cluster to alpha-Fe at a lower temperature, whereas the iron atoms in the lattice will form gamma-Fe first at temperature above 350 degrees C, and then transform to bcc Fe.

THE EFFECT OF ADDITIVES ON STIRRED MEDIA MILLING OF LIMESTONE

Zheng J. Harris CC. Somasundaran P. - Powder Technology. 91(3):173-179, 1997

The effects of additives (sodium hydroxide, sodium carbonate, sodium oleate, oleic acid and poly(acrylic) acid), on stirred media milling of limestone have been investigated. The results are evaluated in terms of specific surface area, specific energy and energy efficiency as a function of additive dosage, polymer molecular weight, solids concentration and relevant operating variables. Use of additives generally results in changes in specific surface area and energy efficiency, while under certain conditions a more than 100% increase in these parameters can occur. Relevant interfacial properties have been measured and the milling mechanisms involved are explored, particularly in terms of the effect of additives on the flow patterns at higher solids concentration.

BALL MILLING OF SYSTEMS WITH POSITIVE HEAT OF MIXING - EFFECT OF TEMPERATURE IN AG-CU

Klassen T. Herr U. Averback RS. - Acta Materialia. 45(7):2921-2930, 1997

Ag50Cu50 alloys prepared by ball milling were subsequently milled at different temperatures ranging from about 85 to 473 K and investigated by X-ray diffraction and differential scanning calorimetry. Milling at subambient temperatures led to a homogeneous f.c.c. solid solution, while milling at 473 K led to a fully decomposed, two-phase mixture. Partially decomposed systems were obtained during milling at intermediate temperatures with three co-existing phases all present. The milling-induced decomposition process had a very low apparent activation enthalpy, approximate to 0.11 eV, which is attributed to a first-order kinetic process involving non-equilibrium vacancies. The results are discussed in terms of competing mixing and de-mixing reaction rates.

MICROSTRUCTURE AND MECHANICAL PROPERTIES OF AL-4.5 WT-PERCENT-CU/L5 WT-PERCENT-SIC COMPOSITE PREPARED USING BALL MILL

Lu L. Lai MO. Yap SH. -Materials Science & Technology. 13(3):192-202, 1997

Aluminium based metal matrix composite (MMC) material was prepared using ball milling (BM). Elemental powders of aluminium and copper, and SiC particle reinforcement were milled in a horizontal ball mill. Two different speeds of rotation of 63 and 113 rev min(-1) were employed to investigate the influence of collision pressure. It was observed that SiC particles began to be incorporated into the metal matrix (Al-Cu) after 3.6 ks of BM. After 36 ks of BM, almost no individual SiC particles could be observed. Energy dispersive X-ray analysis showed that copper and SiC particles had already been well distributed in the powder particles after 54 ks. However, aluminium and copper could not be mechanically alloyed even after 360 ks of BM. Because the BM process was carried out in a low energy ball mill, the size of the the SiC particle remained unchanged. Irregularly shaped SiC particles were, however, seen to have been reshaped into a more spherical form thus reducing the stress concentration near the particles. Microstructural observation revealed that the advantage of using a low energy ball mill to produce composite powder particles was that it provided well distributed SiC particles and good bonding between the metal matrix and the SiC particles. The effects of the BM duration and the addition of aluminium on the fracture toughness of the MMC have also been evaluated in the present study.

THE INFLUENCE OF COLLISION ENERGY AND STRAIN ACCUMULATION ON THE KINETICS OF MECHANICAL ALLOYING

Schaffer GB. Forrester JS. - Journal of Materials Science. 32(12):3157-3162, 1997

The kinetics of mechanical alloying have been investigated by examining the effect that ball mass has on the rate at which titanium carbide forms from the elements. By varying the ball density while keeping the ball diameter and the charge ratio constant, the collision energy was independently controlled. Grinding media with a density from 3.8 g cm(-3) (agate) to 16.4 g cm(-3) (tungsten carbide) were used. The reaction rate increases exponentially with ball mass until a critical level is reached, which is determined by the induced temperature rise. Above this level, collisions of higher energy have no advantage. It is also shown that the reaction rate increases exponentially with the rate at which strain accumulates in the reactants. It is suggested that the strain accumulation rate in mechanically induced reactions is analogous to temperature in thermally induced chemical reactions.

PREPARATION AND STRUCTURE OF BULK NANOSTRUCTURED WC-CO ALLOY BY

Ma XM. Ling Z. Gang J. Dong YD. - Journal of Materials Science Letters. 16(12):968-970, 1997

NON-EQUILIBRIUM W-CU SYSTEM ALLOY POWDER AND BULK BODY PREPARED BY MECHANICAL ALLOYING AND SHOCK COMPRESSION

Mashimo T. Huang XS. Tashiro S. -Journal of Materials Science Letters. 16(12):1051-1054, 1997

MICROSTRUCTURAL REFINEMENT AND STRENGTHENING OF CU-4 CR-2 NB ALLOY BY MECHANICAL MILLING

Anderson KR. Groza JR. Ulmer DG. - Scripta Materialia. 37(2):179-185, 1997

BI-SB SEMICONDUCTOR ALLOY SYNTHESIZED BY MECHANICAL ALLOYING

Martinlopez R. Lenoir B. Dauscher A. Devaux X. Dummler W. Scherrer H. Zandona M. Remy JF. - Scripta Materialia. 37(2):219-226, 1997

SYNTHESIS AND PROPERTIES OF MECHANICALLY ALLOYED Y-NI-B-C

Eckert J. Jost K. Schultz L. - Materials Letters. 31(3-6):329-333, 1997
The formation and stability of YNi2B2C compounds by mechanical alloying of elemental powders has been investigated by X-ray diffraction and thermal analysis. The superconducting YNi2B2C phase forms after about 16 h of milling. Extended processing leads to the formation of an amorphous phase which shows indications for a glass transition before crystallization. Crystallization reproduces the stoichiometric YNi2B2C compound. The thermal stability and the superconducting properties of the powders are compared with data for samples prepared by other preparation techniques and are discussed with respect to the influence of impurities introduced during milling.

EXTRACTION AND COMPARATIVE CHARACTERIZATION OF BALL-MILLED LIGNIN (LM), ENZYME LIGNIN (LE) AND ALKALI LIGNIN (LA) FROM WHEAT STRAW

Lawther JM. Sun RC. Banks WB. - Cellulose Chemistry & Technology. 30(5-6):395-410, 1996
Ball-milled straw lignin LM and enzyme lignin LE were extracted with 95% dioxane, following with 50% (v/v) dioxane-water from ball-milled, for 1, 2, 4, 6, 8, 10 and 15 days wheat straw in a porcelain rotary ball-mill, and from the following hydrolyzed by a cellulase for 3 days straw residues, respectively. Alkali lignin LA was obtained by treatment of wheat straw with 0.5 M NaOH at 75 degrees for 2 h, and isolated by two steps of precipitation. The effect of ball-milling time (BMT) on lignin yield and molecular weight was examined in this study. Optimal BMT was 6 days. A comparative study of the three lignin preparations, LM, LE and LA, using UV-, GPC-, IR-, C-13-NMR-spectroscopy and nitrobenzene oxidation, is reported. Alkali lignin LA, which is relatively free of polysaccharides and appeared to have high molecular weight, was found to be the greatest potential for further investigation.

SELECTIVE OXIDATION OF N-BUTANE TO MALEIC ANHYDRIDE WITH VANADIUM PHOSPHORUS CATALYSTS PREPARED BY COMMINUTION IN THE PRESENCE OF DISPERSANTS

Hutchings GJ. Higgins R. - Applied Catalysis A-General. 154(1-2):103-115, 1997
A method of preparation of high surface area vanadium phosphorus catalysts for the partial oxidation of n-butane is described that is based on a comminution procedure. The ball milling of catalysts precursors in the presence of dispersants is shown to decrease significantly the mean crystallite size of the VOHPO4.0.5H(2)O from >5 x 10(-6)m to ca. 3.5 x 10(-8) m. A range of dispersants and solvents is described and the use of cyclohexane as solvent and the use of a dispersant based on poly-12-hydroxystearic acid is discussed in detail. These catalyst precursors give final catalysts with high surface area following activation (ca. 40 m(2) g(-1) versus ca. 10 m(2) g(-1) in the absence of the comminution). These catalysts are found to be particularly active for use under fuel rich reaction conditions {[butane] > the higher explosion limit} when high maleic anhydride yields can be obtained. The effect of the addition of promoters is also discussed. In general addition of low levels of La, Ce, Cu and Mo all increase the activity of maleic anhydride formation without any significant effect on selectivity when fuel lean conditions are used {[butane] > lower explosion limit}. However, the effect on activity is much less pronounced when fuel rich conditions are used and in this case the addition of these compounds leads to lower maleic anhydride selectivity

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N.B. : Pour la rédaction du prochain N° de la Lettre du Réseau Français de Mécanosynthèse, tout(e) article, annonce, thèse ... peut être envoyé(e) à Eric Gaffet - CNRS UPR A0423 - Groupe "Elaboration et Transitions de Phases Hors Equilibre" - IPSé - F90010 Belfort Cedex - Tél. : 84 - 58 - 31 - 02 / Fax : 84 - 58 - 30 - 27 ou par E-mail

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DEMANDES

- **Pascal Pochet :** 28 Ans - Docteur en Science des Matériaux (soutenance le 6/01/97) Ingénieur Chimiste - Domaine : Métallurgie Physique, Changements de phases sous sollicitation extérieure, mécanosynthèse, Génie des Procédés - Techniques : Caractérisation des matériaux (DRX, ATD-TG, MEB, MET), techniques de métallurgie des poudres, simulations numériques (Monte Carlo) sur stations de travail - Résultats : Identification des paramètres de broyage qui controlent l'état final du produtio (ordre - désordre dans FeAl, précipitation dans NiGe) Modélisation du processus à l'échelle atomique, découverte de microstructures inhabituelles - Publications : Phys Rev. B, Mater. Sci. For., MRS Symp. ISMANAM95 (conf. invité), J. Phys. - Divers, Anglais, Unix, Fortran 77.
- **Dr. F. Guo - Jiang** recherche Post Doc en France : **s**trongly recommended by Prof. H. Zhuang - Qi - Academician, Director of State Key Laboratory of NonEquilibrium Alloys.
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Characterization of Fe - base nanoparticles, including iron, iron nitride and carbide as well as carbonitride ultrafine particles, Phase transformation and transformation thermodynamics of the naocrystalline particles, Microstructure and magnetic properties of Fe - base nanoparticles.(13 Articles à Comité de Lecture) E_Mail : zjz-dms@mail.tsinghua.edu.cn

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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)

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*(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)