RESEAU FRANCAIS DE MECANOSYNTHESE

Lettre N°22/23

Janvier / Février 1997

59 Groupes de Recherches - 80 Correspondants

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

3 Nouvelles Adhesions !!
Ressources en Innovation - N. Lecomte - Lyon
Lab. Physicochimie Matière Condensée - Corr. D. Ravot : Univ. de Montpellier II
Dpt Electronic Mat. & Eng. - Corr. Y. Chen - Nat. Univ. Canberra - Australie

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! JRFM97 !

29 - 30 Avril 1997 à l'**Ecole des Mines de Nancy**
Contacts: S. Begin - Colin, G. Le Caër, J.C. Gachon, E. Gaffet, R. Martin -- Lopez, R. Welter
(Pour les coordonnées, se reporter à l'annuaire en fin de LRFM)

!!! Pré - Inscription : 31 Décembre 1996 !!!
!!!! IL EST ENCORE TEMPS de s'inscrire!!!!

(à l'attention de V. Domingues - Secrétariat Journées RFM - LSG2M - URA CNRS 159
Ecole des Mines de Nancy - Parc de Saurupt - 54042 - Nancy Cedex
E_Mail : Domingue@mines.u-nancy.fr

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ANNONCE DE CONGRES ET / OU ECOLES

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1997 TMS Annual Meeting

Chemistry and Physics of Nanostructures and Related Non - Equilibrium Materials
Orlando - 9 13 Février 1997
Contact : B. Fultz - Caltech, Mail Code 138 - 78 - Pasadena, CA 91125 - Fax : 818 - 795 - 6132

Micro Mat 97

Berlin - 16 - 18 Avril 1997
Contact : B. Michel - Fax +49 - 30 46403200 E_Mail : Michel@izm.fhg.de

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Euromat97

Maastricht - 21 - 23 Avril 1997 - Contact : Euromat97

28ème Journées d'Etudes sur la Cinétique Hétérogène

Lille - Avril ou Mai 1997 - Contact : J.P. Bonnelle - Université des Sciences et Technologies de Lille

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Powders and Grain 97

Durham - Caroline du Nord - USA -**Contact :** R. Behringer, Duke University Fax : 1 919 660 2525

Congrès Société Européenne de Céramique

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

ICAM'97 et E-MRS'97

Strasbourg 16 - 20 Juin 1997
Cont. : P. Siffert - E-MRS1997 Spring Meet. - BP 20, 67037 Strasbourg Cedex 2 - Fax : 03 88 10 63 43 - EMRS@FRCPN11.IN2P3.FR

V International Worhshop on Non - Crystalline Solids

2 - 5 Juillet 1997 - Santiago - Espagne
Secretariat : Viajes Atlantico, S.A. - Pl. Fuenterrabia, 3 - APratada 136 - E - 15702 - Santiago de Compostela-Espagne

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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

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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

ISMANAM97

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

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

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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

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2nd Int. Symposium on Structural Intermetallics
Champion (PA - USA) - 21 - 26 Septembre 1997

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

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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

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 - 25 - 27 Novembre 1997

1) Les Matériaux de l'Automobile

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

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

Animateurs : L. Schultz (Dresden), P. Panissod (Strasbourg), E. Gaffet (Belfort)

Contact : SF2M

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

Comité d'organisation : J. Dodds (Pdt) , C. Frances(Secrét. Scient.) , N. Le Bollay (Secrét. Scient.), Benhassaine, Bousquet, Chamaillou, Cristil, Forssberg, Guigon, Mamourian, Morizot, Redecker, Soustelle, Tondeur, Yvon

Le RFM sera associé à l'organisation de la session "Mécanosynthèse"- Les autres thèmes prévus sont :
Mechanical activation and mechano - chemical reaction / Classification in wet and dry systems
Machine design and practical experience / Control and instrumentation
Operation of comminution circuits and system/ Wear in crushers and mills.

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AVIS DE SOUTENANCE DE THESES

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"Etude Expérimentale et Modélisation des Changements de Phases sous Broyage à Haute Energie"

P. Pochet

Université des Sciences et Technologies de Lille I / Sciences des Matériaux **6 Janvier 1997**

Jury : J. Foct (Pdt), Y. Brechet (Rapp.), G. Le Caer (Rapp.), P. Bellon, G. Martin (Dir.)

Le but de ce travail était d'étudier quelques changements de phases sous broyage, plus précisément, de déterminer les conditions de broyage permettant de stabiliser préférentiellement une phase plutôt qu'une autre et de voir dans quelle mesure, les résultats pouvaient se rationaliser dans le cadre de la théorie des alliages forcés. Pour cela, nous avons choisi deux transformations de phases : ordre - désordre (FeAl) et précipitation dissolution (NiGe). On trouve que le degrè d'ordre à grande distance d'un alliage Fe50Al50 atteint sous broyage une valeur stationnaire qui dépend de la température et de l'intensité de broyage. La réversibilité de la transformation en fonction de la température a été démontrée. Il existe un domaine de températures et d'intensités à l'intérieur duquel l'état stationnaire désordonné est atteint en passant par un état métastable de degré d'ordre intermédiaire. Ce comportement rappelle celui d'une transition du premier ordre alors que la transition hors broyage est du second ordre. Ces comportements sont bien reproduits par un modèle simple d'alliages forcés", développé pour les alliages sous irradiation. Le degré d'ordre

résulte de la compétition entre deux mécanismes de saut des atomes : les déplacements forcés par le cisaillement des cristallites et les sauts activés thermiquement provoqués par la migration des lacunes. Pour essayer de comprendre comment un modèle aussi grossier peut décrire un phénomène aussi complexe, nous avons eu recours à des simulations à l'échelle atomique. Nous avons développé un algorithme Monte Carlo cinétique où deux mécanismes agissent en parallèle : la diffusion thermiquement activée par mécanisme lacunaire et la création de désordre par cisaillement. Plusieurs variants sont explorés : bandes de cisaillement formées par un dipôle de dislocations coplanaires ou non, lacunes conservatrices ou non. Toutes les variantes du modèle reproduisent bien la dépendance du degré d'ordre stationnaire avec la température et une intensité réduite, définie comme le nombre d'atomes déplacés par atome, par unité de temps et par lacune. De plus, nous montrons que le système présente, dans certaines conditions de sollicitations, deux états d'ordre localement stables et que suivant l'amplitude du bruit externe (imposé par le cisaillement), soit le système oscille entre ces deux états, soit il s'accroche temporairement à l'état partiellement ordonné avant de se stabiliser dans l'état désordonné. Ce dernier cas reproduit assez bien les principales caractéristiques des états métastables observés expérimentalement. Par ailleurs, nous montrons que les mécanismes de production et d'élimination spécifique des défauts sont actifs dans une solution solide NiGe diluée ; ceci permet d'envisager la possibilité d'un phénomène de précipitation induite par broyage dans une solution solide sous - saturée.

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RAPPEL DES SOUTENANCES DE THESES PRECEDENTES (à partir de Fin 95)

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E. LUCAS- Ecole des Mines de Paris -**14 Octobre 1996**
"Les Assemblages céramique / céramique par frittage réactif : Application au carbure de silicium infiltré silicium"
(Directeur de Thèse : Y. Bienvenu)

KIENTZ M.O. - Université de Nancy -**12 Septembre 1996**
"Mécanosynthèse et caractérisation de solutions solides nanocristallines Fe - X (X = Sn, Sb). Transformations de phases induites par broyage dans les composés intermétalliques"
Jury : J. Foct (P^{dt}), B. Malaman (Rapp.), , E. Gaffet (Rapp.), L. Fournes, G. Le Caër (Dir.), A. R. Yavari

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S. RENAUD - Ecole Centrale de Paris -**13 Décembre 1995**
"Elaboration de poudres Ti_2Be_{17}, Al - Be et Al - Be - Ti par mécanosynthèse et étude préliminaire de leur consolidation"
Jury : Y. Barbaux, D. François, J.B. Guillot, G. Le Caër, F. Moret, F. Ravel

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

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

Nom : .....**Prénom :** .....
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 **1996**

(Signature)

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Bibliographie Récente

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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

Proceeding du Congrès "Mechanically Alloyed and Nanocrystalline Materials" - Rome (1996)
Editor : D. Firoani, M. Magini - Materials Science Forum - Volumes 235 - 238 (1997)
Les textes publiés dans les Actes d'ISMANAM97 par les membres du RFM sont repris dans la liste ci - dessous.

Proceeding du Congrès "Mechanically Alloyed and Nanocrystalline Materials" - Québec (1995)
Editor : R. Schulz - Materials Science Forum - Volumes 225 - 227 (1996))

"Mechanical Properties and 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) - ISBN 0-7923-2195-2

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 Universtity 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)

"BN / Al composite formation by high energy ball milling"
Du YJ. Li SY. Zhang K. Lu K. - Scripta Materialia. 36(1):7-14, 1997

"Nano - experts, no more"
Physics World. 9(12):10, 1996

"Mechanochemical reaction of DDT with calcium oxide"
Hall AK. Harrowfield JM. Hart RJ. Mccormick PG. - Environmental Sci. & Technology. 30(12):3401-3407, 1996
Evidence is presented that, in the mechanochemical destruction of DDT [2,2-bis(4-chlorophenyl)-1,1,1-trichloroethane] by ball milling in the presence of calcium oxide, a complex series of reactions occurs along the pathway to a product that appears to be essentially graphitic, though aromatic chloro and hydroxy substituents are retained to some degree. The production of the various intermediates can be understood in terms of processes initiated at both CaO and steel (of the milling device) surfaces. With the exception of DDE [2,2-bis(4-chlorophenyl)-1,1-dichloroethene], most of these intermediates attain maximum concentrations corresponding to < 1 mol % of the original DDT and have been characterized only by their mass spectra. In the case of dichlorotolane [bis(4-chlorophenyl)ethyne], however, yields are sufficient for it td be isolated chromatographically as a pure, crystalline solid and characterized further by NMR spectroscopy. After 12 h of milling, no organic materials volatile enough to be detected by conventional GC/MS procedures are present, but the black, graphitic residue does retain some chlorine that is only slowly removed by extended milling.

"Trends in solids process engineering 'Review" (allemand)
Peukert W. - Chemie Ingenieur Technik. 68(10):1254-1263, 1996
This paper describes trends in the field of particle technology. The properties of dispersed matter which has to be handled in the chemical engineering processes or which has to be produced are of decisive importance. Future trends can be characterized by improvement of existing technologies, by a better understanding of fundamentals, combination of principles, extension of process parameters, process control and simulation, and new processes. An important trend is the production and handling of finer particles. Traditional methods of comminution and classification reach their limit around 1 mu m. New processes for synthesis of particles by physical and chemical methods appear to becoming more popular. Nano-sized particles may become of future relevance in various fields of technology such as material science, micro electronics or medicine.

"Synthesis of Mg_2Si powder by mechanical alloying and its consolidation"
Munozpalos JM. Cristina MD. Adeva P. - Materials Transactions Jim. 37(10):1602-1606, 1996
In this work, bulk Mg2Si has been processed using the PM route. The Mg2Si powders were synthesized by the mechanical alloying technique and were characterized microstructurally and thermally. The mechanical alloying was attained al room temperature by the use of a high ball-to-powder ratio of 33:1. An X-ray diffraction technique was used to follow the alloying process. Powders obtained from an Al-Mg2Si two-phase aluminium ternary alloy by dissolving the aluminium matrix were also obtained as reference intermetallic compounds. Although both kinds of powders exhibited a good compaction behavior, the compact fabricated from the mechanically alloyed powders showed the highest green density. The thermal characterization of the intermetallic by differential scanning calorimetry and thermogravimetry showed a low thermal stability. The intermetallic Mg2Si exhibited a mass gain due to its oxidation at temperatures above 773 K even under the pressure of 1.33 x 10(-3) Pa. This phenomenon limits its processing capability during the sintering step, since the experiment must be conducted at intermediate temperatures below 773 K.

"Ultrafine alumina particles prepared by mechanochemical / thermal processing"
Ding J. Tsuzuki T. Mccormick PG. - J. American Ceramic Society. 79(11):2956-2958, 1996
Ultrafine alumina particles have been prepared by the mechanical milling and subsequent heat treatment of a mixture

of AlCl3 and CaO. Heat treatment of the as-milled powder at temperatures above 350 degrees C and washing with water resulted in gamma-Al2O3 particles 10-20 nm in size. Single-phase alpha-Al2O3 was formed in the sample after heat treatment at 1250 degrees C. This study demonstrates a novel process for synthesizing nanoscale alumina particles.

"Nanostructural materials formation by mechanical alloying - Morphologic analysis based on transmission and scanning electron microscopic observations"

Gaffet E., Tachikart M., ElKedim O., Rahouadj R. - Materials Characterization, 36 (4 - 5) : 185 - 190, 1996

The development of nanostructured materials offers nex scientific and technological perspectives due to the specific interesting physical properties of these materials. These properties derive either from their reduced grain size of from the structure and properties of the grain boundaries, which constitute a significant volume fraction. Mechanical alloying, widely used to produce dispersion - strengthened and amorphous alloys, has been employed in recent years to synthesize nanocrystalline metallic, semiconductors, and covalent component based materials. Based on statistical analysis of transmission and scanning electron microscopic images, the distribution and spatial repartition of the nanostructural materials prepared by mechanical alloying and / or attrition are presented for some specific cases.

"Structure and magnetic properties of ultrafine Fe powders by mechanochemical processing"

Ding J. Tsuzuki T. Mccormick PG. Street R. - J. Magn. & Magn. Mat.. 162(2-3):271-276, 1996

Ultrafine Fe powders have been synthesized by mechanochemical processing. The kinetics of the formation of metallic Fe by the reduction of FeCl2 during mechanical milling has been studied. The influence of milling conditions and reductant on structure and magnetic properties was investigated. It is shown that the production of uniform nano-sized powders by mechanochemical processing requires a high particle-to-by-product ratio and avoidance of combustion by the use of low energy milling conditions or by the addition of diluents.

"Attrition milling of cordierite powders (espagnol)"

Camerucci MA. Cavalieri AL. - Anales de la Asociacion Quimica Argentina. 83(6):365-368, 1995

Attrition milling is a conventional method used to mill powder to submicron sizes with minimal contamination and high efficiency. The purpose of this work is to establish the best attrition milling conditions to produce cordierite particles finer than 1 mu m to enhance their sinterability.

"Increase of oxygen content in Si_3N_4 powder during ball milling using alcohol as solvent (japonais)"

Wada S. - Nippon Seramikkusu Kyokai Gakujutsu Ronbunshi-Journal of the Ceramic Society of Japan. 104(11):1085-1087, 1996

Si3N4 powder is usually mixed with sintering additives in a ball mill using not water but alcohol as a solvent to avoid the increase of oxygen produced by chemical reaction of Si3N4 with water. However, after the ball milling for a few hundreds of hours using alcohol which dissolves effectively little amount of water, oxygen content in the mixed Si3N4 powder increases in percent order. The oxygen was thought to come from the alcohol mechanochemically disintegrated during ball-milling.

"Deformation enhanced thermal stability of an amorphous $Fe_{80}B_{20}$ alloy"

Fan GJ. Quan MX. Hu ZQ. - Journal of Applied Physics. 80(10):6055-6057, 1996

By means of differential scanning calorimeter (DSC) measurements, the thermal stability of an amorphous Fe80B20 alloy after various periods of low-energy ball milling has been studied. The results indicate that the thermal stability of the amorphous Fe80B20 ribbons can be enhanced upon mechanical deformation with a low milling intensity. The crystallization temperature T-p, the crystallization enthalpy Delta H, and the crystallization activation energy E(x) increase with milling time. The above observations will be compared with our previous findings that extensive mechanical deformation with a high milling intensity can otherwise induce a structural relaxation in an amorphous Fe80B20 alloy. Based on conventional thermodynamic and kinetic arguments, a reasonable interpretation will be made to explain the enhanced thermal stability of the amorphous Fe80B20 alloy after mechanical deformation.

"Synthesis of nanocrystalline M50 steel powders by cryomilling"

Lau ML. Jiang HG. Perez RJ. Juarezislas J. Lavernia EJ. - Nanostructured Materials. 7(8):847-856, 1996

The present paper reports on a study of the synthesis of nanocrystalline high speed tool steel M50 powders (4.5% Mo, 4.0% Cr, 1.0% V, 0.8% C, balance Fe) by cryogenic high energy ball milling (cryomilling). Pre-alloyed M50 steel is spray atomized, and subsequently cryomilled in liquid nitrogen for 25 hours. Elemental Al powder is added prior to cryomilling to promote the formation of nanoscale Al2O3 and AlN dispersoids to improve the thermal stability of the nanocrystalline M50 steel. High resolution transmission electron microscopy (HRTEM) reveals the formation of various carbides (V8C7, Fe3C, and FeC), oxides (Al2O3, MoO3, and V3O7), and a nitride phase (AlN) during cryomilling. Following one hour of heat treatment at 1373 K (0.77 T-m), an average grain size of 70 nm was retained for the M50 steel powders.

"Synthesis of nanocrystalline SiC at ambient temperature through high energy reaction milling"

Yang ZG. Shaw LL. - Nanostructured Materials. 7(8):873-886, 1996

This study investigated the in-situ synthesis of nanosized crystalline SiC powders at room temperature through high energy ball milling of elemental silicon and carbon mixtures. Milling conditions including the mill design, the milling speed, the milling time and the ball-to-powder weight ratio (i.e. the charge ratio) necessary for the in-situ synthesis were studied. It was found that uniform formation of nanosized crystalline SiC powders within the powder charge could be achieved with a correctly designed attritor and the contamination could be minimized with proper selections of milling conditions. The crystalline beta-SiC powders synthesized were themselves in nanosize scale, quite different from many previous studies which have shown that it is the internal grain structure of milled powders that is the "nanocrystalline" component of the powders (typically 5 - 20 nm), while the powders are themselves typically 0.1 mu m to > 1 mu m in size. Furthermore, it was found that the product structures generated by high energy reaction milling depended strongly on the milling speed, the charge ratio and the milling time.

"On some physical properties of nanstructured Cu - Pb alloy prepared by mechanical alloying"

Kim JC. Ko BH. Moon IH. - Nanostructured Materials. 7(8):887-903, 1996

Cu-Pb alloys have no solubility in the whole solid state and their physical properties are very different from each

other. In the present study, nanostructured Cu-Pb alloy powders were synthesized by the mechanical alloying process, and their nanostructural characteristics were evaluated in order to elucidate the relationship between structure and properties. By appropriate control of mechanical alloying process variables, the Pb solid solubility in Cu matrix was increased up to 10 wt.%. The monotectic temperature of Cu-Pb alloy was also decreased by decreasing the crystalline size. The relation between the structure and properties of this nanostructured Cu-Pb alloy is discussed on the basis of the experimental results.

"Microstructural changes in a mechanically alloyed Al6.2 Zn2.5Mg1.7 Cu alloyed (7010) with and without particulate SiC reinforcement"

Bhaduri A. Gopinathan V. Ramakrishnan P. Miodownik AP.- Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 27(11):3718-3726, 1996

Elemental powders of Al, Zn, Mg, and Cu (corresponding to the composition of 7010 aluminium alloy) were milled in a high-energy attritor with and without additions of SiC particulates. The microstructural changes taking place in the milled powders (which eventually lead to mechanical alloying) are found to be retarded by SiC additions. High-resolution techniques such as electron probe microanalysis (EPMA) and transmission electron microscopy/energy-dispersive X-ray analysis (TEM/EDX) revealed the presence of localized. solute-rich regions long after the diffraction line from these solutes had ceased to appear in the X-ray diffractograms, Zinc appears to be more difficult to be mechanically alloyed into aluminum than either Cu or Mg in spite of its comparatively larger diffusivity in aluminum.

"Microstructural evolution of 304 stainless steel during mechanical milling"

Huang H. Ding J. Mccormick PG. - Materials Science & Engineering A - 216(1-2):178-184, 1996

The microstructural development in 304 stainless steel pou der during mechanical milling has been investigated. X-ray diffraction, transmission electron microscopy (TEM) and saturation magnetisation measurements show that a strain-induced martensitic transformation occurs during milling. Measurements of the saturation magnetisation have been used to study the effect of collision energy on transformation kinetics and average particle strain. The results illustrate the important effect of collision energy on microstructural evolution during milling.

"Degradation of dispersant during milling"

Chartier T. Souchard S. Baumard JF. Vesteghem H. - J. European Ceramic Society. 16(12):1283-1291, 1996

Deterioration of the ammonium salt of poly(methacrylic acid) (PMMAA-NH3), used as a dispersant during wet ball-milling of an alumina, has been investigated. Rheological behaviour, sedimentation tests, adsorption isotherms, pH and isoelectric point measurements, and infra-red analysis have allowed us to conclude that degradation of the dispersant takes place in two stages: (i) decrease of the charge by dehydration and (ii) complete neutralization of the dispersant by the formation of monodentate of the dispersant by the formation of monodentate COOX groups, which can lead to desorption of the polymer from the alumina surface. This second phenomenon involves a strong increase of viscosity. A low viscosity can be recovered by a subsequent addition of PMAA-NH3 at the end of milling.

"Thermal decomposition of mechanically alloyed nanocrystalline FCC $Fe_{60}Cu_{40}$"

Huang JY. Yu YD. Wu YK. Ye HQ. Dong ZF. - Journal of Materials Research. 11(11):2717-2724, 1996

A ferromagnetic and supersaturated fee Fe60Cu40 solid solution was prepared by mechanical alloying (MA). The phase transformations of the as-milled Fe60Cu40 powder upon heating to 1400 degrees C and subsequently cooling to room temperature were characterized by differential thermal analysis (DTA) and thermal magnetic measurement. The fee Fe60Cu40 solid solution decomposes into alpha-Fe(Cu) + gamma-Fe(Cu) + Cu(Fe) upon heating from 300 to 460 degrees C, and on further heating, alpha-Fe(Cu) transforms to gamma-Fe(Cu) at 640 --> 760 degrees C; during cooling, the reverse transformation occurs from 800 --> 640 degrees C (obtained from thermomagnetic measurement) or from 700 --> 622 degrees C (obtained from DTA). The gamma reversible arrow alpha transformation in mechanically alloyed Fe60Cu40 nanocrystalline occurs in a wide temperature range; the transformation temperature is higher than that of the martensite transformation in as-cast Fe-Cu alloys, but is much lower than that of the allotropic transformation of pure Fe. These differences may be caused by the different fabrication process, the nonequilibrium microstructure of MA, as well as the inhomogeneous grain size in alpha-Fe(Cu). High resolution transmission electron microscope (HRTEM) observations carried out in the specimen after the DTA run show that N-W or K-S orientation relationships exist between alpha-Fe(Cu) and Cu(Fe), which also represent the orientation relationship between alpha-Fe(Cu) and gamma-Fe(Cu) due to excellent coherency between gamma-Fe(Cu) and Cu(Fe). The grain size of the alpha-Fe(Cu) is inhomogeneous and varies from 50-600 mm. Energy dispersive x-ray spectroscopy (EDXS) result shows that the Cu content in these alpha-Fe(Cu) grains reaches as high as 9.5 at. % even after DTA heating to 1400 degrees C, which is even higher than the maximum solubility of Cu in gamma-Fe above 1094 degrees C. This may be caused by the small grain size of alpha-Fe(Cu).

"Synthesis of submicrometer grained ultrahigh carbon on steel containing 10 percent aluminum by ball milling of powders"

Taleff EM. Nagao M. Ashida Y. Sherby OD. - Journal of Materials Research. 11(11):2725-2730, 1996

An ultrahigh-carbon (1.25 wt. %) steel alloy containing 10 wt. % aluminum (UHCS-10Al) was processed by a powder metallurgy technique. Gas-atomized powders were subjected to ball-milling in an attritor in order to obtain a submicrometer grain size. Powder material was consolidated by both hot isostatic pressing (HIP) and by hot isopressure extrusion (HIE). Bulk material with submicrometer grain sizes was produced from attrited powders. The chemical composition and microstructure of this material are characterized at each processing step, from atomization through consolidation. Tensile tests show that a high strength results from the submicrometer grain size produced in the bulk material.

"Melting process of nanometer-sized in particles embedded in an Al matrix synthesized by ball milling"

Sheng HW. Xu J. Yu LG. Sun XK. Hu ZQ. Lu K. - J. Materials Research. 11(11):2841-2851, 1996

Dispersions of nanometer-sized In particles embedded in an Al matrix (10 wt. % In) have been synthesized by ball milling of a mixture of Al and In powders. The as-milled product was characterized by using x-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive x-ray spectrometer (EDX), transmission electron microscopy (TEM), and high resolution transmission electron microscopy (HREM), respectively. It was found that In and Al are

pure components immiscible with each other, with nanometer-sized In particles dispersively embedded in the Al matrix. The melting behavior of In particles was investigated by means of differential scanning calorimeter (DSC). The calorimetric measurements indicate that both the melting point and the melting enthalpy of the In nanoparticles decrease with increasing milling time, or refinement of the In particles. Compared to its bulk melting temperature, a melting point depression of 13.4 K was observed when the mean grain size of In is 15 nm, and the melting point depression of In nanoparticles is proportional to the reciprocal of the mean grain size. The melting enthalpy depression was interpreted according to the two-state concept for the nanoparticles. Melting of the interface was deduced to be an exothermal process due to its large excess energy/volume.

"Mossbauer investigation of intermixing during ball milling of Fe 0.3 Cr07 and Fe0.5 W0.5 powder mixtures"

Lecaer G. Delcroix P. Shen TD. Malaman B. - Physical Review B, 54(18):12775-12786, 1996

The intermixing of Fe and T (T=Cr,W) during ball milling of elemental powder mixtures Fe1-xTx, with x=0.70 for T=Cr and x=0.50 for T=W, has been followed by Fe-57 Mossbauer spectroscopy at room temperature (RT) and by magnetization measurements for T=W. The chemical compositions have been chosen to yield final alloys or compounds which are nonmagnetic at RT to better follow the evolution of magnetic phases with milling times. For a long period of milling time t(m) before reaching the final stationary state, the hyperfine magnetic field distributions remain stationary in shape for both T=Cr and T=W. Only the relative weight of the magnetic contribution decreases with t(m). For T=W, the average moment of magnetic Fe atoms is further shown to remain constant with t(m). Stationary hyperfine field distribution shapes are found to be similar not only for T=Cr and W but also for T=Si (x=0.50) while published spectra suggest to add T=Al, Ti, V, Ta, Re to the latter nonexhaustive list. The stationary shape is characterized by a narrow peak located at a field close to the field of alpha iron at RT (330 kG) and by a broad, almost featureless, band from similar to 50-100 kG to similar to 300-320 kG. The broad band represents about 2/3 of the normalized field distribution. We deduce that the interpretation which consists in attributing the x-ray diffraction peaks of Fe-based bcc solid solutions to a single Fe-rich homogeneous solid solution must be done with care for intermediate milling times. We cannot infer from such hyperfine measurements a detailed description of the regions of the powders which are responsible for such magnetic features. We argue however that irregular interfaces between nanometer-sized Fe-rich zones and T-rich zones may play a role to explain the observed shape of the hyperfine field distributions. The general conditions (process and materials) in which such phenomena may occur remain to be clarified.

"Physical and mechanical properties of sintered Nd - Fe - B type permanent magnets"

Rabinovich YM. Sergeev VV. Maystrenko AD. Kulakovsky V. Szymura S. Bala H. -Intermet., 4(8):641-645, 1996

Effective application of the Nd-Fe-B type permanent magnets in electrotechnical products, instruments, aviation and space devices demands knowledge of their proper service parameters. In this connection, a comprehensive study of a wide spectrum of sintered Nd-Fe-B type permanent magnets with various additions of rare earth (RE) and 3-d metals (14 compositions, including so-called commercial and laboratory permanent magnets) was carried out. The mechanical (elastic and strength properties) and physical (magnetic and thermal properties, and electrical resistivity) properties of the studied magnets are tabulated, and the data are discussed. The results obtained allow selection of the optimal permanent magnet composition, depending on required properties and the application. Moreover, the results obtained for various permanent magnets are discussed, taking into account the influence of the factors which are sensitive or insensitive to structure and/or composition changes of the magnets. (C) 1996 Elsevier Science Limited.

"Interactions between titanium oxide and polystyrene during the PAN milling process"

Wang Q. Cao JZ. Li GJ. Xu X. - Polymer International. 41(3):245-249, 1996

The interactions between titanium oxide (TiO2) and polystyrene (PS) during the pan-milling process were studied. The results show that TiO2 contributes to the crushing of PS when they are ground together. Compared with material prepared by the traditional method, the PS/TiO2 prepared by pan-milling exhibits much better properties, such as impact strength, rheological behaviour and thermostability. It is a new method for preparing polymer material with high performance.

"A neutron depolarisation study of the magnetic correlations in a particulate dispersion during milling"

PT. Kraan WH. Mayo PI. Rekveldt MT. Ogrady K. - J. Magn. & Magn. Mat.. 162(1):139-146, 1996

This paper presents the results of a neutron depolarisation study of the magnetic correlations relations in a particulate dispersion during milling. It appears that clusters or aggregates of particles break up in the first 90 min of the milling process, and in the final stages of milling, when dispersion is diluted, the dispersion does not flocculate. Moreover, the magnetic correlation length within the dispersion remains stable over a period of two weeks after preparation. The results of a numerical approach to predict the microstructure of a non-magnetised dispersion agree well with the experimental results.

"Mechanosynthesis of Nd - Fe - B alloys"

Oleszak D. Kaszuwara W. Wojciechowski S. - Journal of Materials Science. 31(21):5725-5729, 1996

A mechanical alloying technique has been applied for Nd-Fe-B alloy synthesis from the mixture of neodymium, iron and Fe-B powders. The direct formation of Nd2Fe14B phase (phi phase) was not observed, but an Nd-Fe multilayer structure was formed during the milling process. Annealing of milled powders at 1023 K for 1 h resulted in magnet formation. The dependence of the magnetic properties on milling time was observed. For the applied milling device and parameters, the optimum milling time proved to be 4 h and the coercive force reached a value of about 1000 kA m(-1).

"Amorphous formation processess of Al - 20 Mol percent Cr alloys by mechanical grinding of equilibrium intermetallic compounds"

Kobayashi KF. Kawaguchi H. - Journal of Materials Science. 31(21):5821-5828, 1996

The amorphization of Al-20 mol % Cr alloys were investigated when different starting powders were mechanically ground using a vibration-mill. Mechanical grinding (MG) of two types of starting powders were examined: (1) single delta (Al-20 mol % Cr) phase powders and (2) a mixture of gamma (Al-15 mol % Cr) and epsilon (Al-30 mol % Cr) phases powders. Amorphous Al-Cr alloys could not be obtained by MG of single intermetallic compound (delta phase)

as the starting powders. But in the case of MG using the mixture of intermetallic com pounds (gamma and epsilon phases), some parts of the MG powders changed phase into an amorphous phase with a Cr content of about 30 mol % at the initial stage of the milling. On further milling, the formed amorphous phase reacted with residual crystalline gamma phase (Al-15 mol % Cr) to produce an amorphous phase with a Cr content of 20 mol %. The former amorphous phase crystallized into the delta and epsilon phases, and the latter crystallized into an equilibrium delta phase by subsequent heat treatment.

"Effects of high energy ball milling on ceramic oxides"

E. Gaffet, D. Michel, L. Mazerolles, P. Berthet - Mater. Sci. For. , 235 - 238 (1997) 103 - 108

High energy milling using a planetary apparatus was applied to various ceramic materials. Mechanical alloying allows in particular to obtain nanocrystalline and amorphous oxides. For instance, the room temperature sysnthesis of stabilized zirconia and the preparatin of amorphous phases in the Al2O3 - ZrO2 - SiO2 system were recently reported. Nanocrystalline metastable phases (hig pressure and/or high temperature phases) induced by phase transition are also obtained. The structure of these various nanocrystalline and amorphous oxides prepared by hig energy ball milling is compared to that of the crystalline oxides. Studies by transmission electron microscopy and EXAFS reveal disorder effects induced by the mechanical process

"Ni - Zn ferrites nanaparticles prepared by ball milling"

G. Nicoara, D. Fratiloiu, M. Nogues, J.L. Dorman, F. Vasiliu - Mater. Sci. For., 235 - 238 (1997) 145 - 150

Samples consisting of Ni0.8Zn0.2Fe2O4 nanoparticles,, both interconnected or dispersed in a matrix, were prepared by high energy ball milling, starting from bulk materail previously synthesized by the conventional ceramic method at 1250°C under O2 flux. Samples with different grain size were performed. The as - milled samples were characterized by chemical analysis, X - ray diffraction and transmission electron microscopy (TEM). The nondispersed samples consist of grains with a mean sized of 100 nm and a large size distribution. The dispersed powder in silica shows individual particles almost spherical with size ranging from 8 up to 50 nm. A good agreement was obtained between X - ray and TEM results. Because the as - milled powder showed after 200 hours of milling a high impurity degree, due to the stainless steel vial and balls, an original method was used to separate the phases. Magnetic measurements and Mossbauer spectra were performed.

"Processing of ultra fine dispersion of TiB2 in SiC ceramic matrix"

C. Blanc, F. Thevenot - Mater . Sci. For., 235 - 238 (1997) 249 - 254

SiC/TiB2 particulates reinforced composites have been prepared by in situ reaction : 5, 10 and 15 vol. % TiB2 have been in situ formed through the reaction between TiO2, B4C and SiC. Pressureless sintering process at temperature about 2190°C has been used to densify composites. The influence of composition, of sintering and soaking temperature on the microstructure and the density are investigated. The density decrease with increasing TiB2 content. But dense composites (98% of theretical density for 5 vol. %, 97% for 10vol.% and 96% for 15 vol. %) can be obtained. The open porosity is about 0.1 % for 5 vol.ù, 0.2 % for 10 vol.% and 2 % for 15 vol.%. Size distribution of TiB2 is investigated by images analysis. It shows that 75% of the particulates for 5 vol.% TiB2, 60% for 10 vol.% and 55 % for 15 vol. % had a size below µm. Mechanical properties (microhardness, toughnesss) are investigated.

"Mechanically activated state of nanograins during and just after in situ deformation using synchrotron radiation"

A.R. Yavari, W. Botta Filho, A. Le Moulec, H. Graafsma, A. Kvick - Mater. Sci. For.? 235 - 238 (1997) 507 - 516

Use of a monchromatic high energy synchrotron x - ray beam allowed the acquisition of x - ray diffraction spectra for nanocrystalline Ni and Cu powders during deformation inside steel jackets in transmission geometry across 2 mm of stell. Good statistics were obtained during 0.2 to 0.5 seconds and led to two major findings : Firstly, a large elastic (reversible) component contributing 0.2 degrees to FWHM indicates the existence of a mechanically activated state of the nanograins during which dislocations, stacking faults and probably locally increased concentrations of vacancies occur under stress. It is show, that with stress release, these defects are expected to relax to the grain boundaries in times much shorter than the acquisition time per frame of 0.5 s here. This mechanically activated state in nano - single crystals should be representative of the structure of nanograin powders under impact in ball milling. Secondly, in the 10 seconds following the application of stress, a monotonous decrease in FWHM of nanocrystalline copper is observed. The magnitude of FWHM loss is about 0.17 degrees or about 20% of the maximum value and 30% of the initial FWHM and corresponds to thermally activated grain growth immediatly after deformation induced grain - size reduction. The very rapid acquisition times seem to have detected these phenomena for the first time.

"Contrast modulation during decomposition of supersaturated fcc Fe - Cu solid and liquid solutions"

A. Garcia Escorial, W. Botta Filho, O. Drbohlav, P. Crespo, M. Urchulutegui, M. Vittori Antisari, A. hernando, A.R. Yavari - Mater. Sci. For., 235 - 238 (1997) 559 - 564

Clear microstructural evidence is obtained from transmission electron microscopy of the occurence of a modulated structure, as occuring in spinodal decomposition, during the decomposition of metastable mechanically alloyed Fe50Cu50 solid solutions and liquid quenched (Fe20Cu80)99B1

"Rapid solidification and mechanical grinding of Cu - Zn alloys"

S. Gialanella, M.D. Baro, X. Amils, S. Surinach, A.R. Yavari - Mater. Sci. For., 235 - 238 (1997) 571 - 576

Rapid solidification was used to prepare metastable Cu - Zn alloys having a composition close to the b - brass interval. In the as spun condition ribbons had a body centred cubi (bcc) crystal structure typical of b - brass. After isothermal heat treaments at 400°C such a structure was turned into a mixture of the initial bcc one plus the a phase of brass, having a face centred cubin (fcc) structure. A similar transformation could be also induced by ball milling. Indeed the mentioned mixture was evoved from the same initail cubic phase by milling the as supun ribbons in a high energy planetary mill. From X - ray diffraction data, it was not only possible to folow the phase evolution, but also to evaluatre the composition of the two Cu - Zn phases from lattice parameters data. The a phase obtained by ball - milling was estimated to have off equilibrium compositons. Equilibrium conditions were established by heat treating the milled powders at selected temperatures. The stability of the different structures obtained in the course of the present investigations was checked using differential scanning calorimetry (DSC) measurements. In the milled saples,

when heated up in the calorimeter, it was noticed the occurence of exothermic irreversible transformations, to be ascribed to recrystalisation and recovery.

"Comparison of disorder induced thermally and by ball milling in Ni2MnSn"

G. Le Caër, P. Delcroix, B. Malaman, R. Welter, B. Fultz, E. Ressouche - Mater.Sci.For., 235 - 238 (1997) 583 - 588

Neutron diffraction, magnetic measurements and 119Sn Mossbauer spectrometry have been used to study the disorder induced thermally and by ball milling in a L21 Heusler alloy Ni2MnSn. High temperature neutron diffraction measurements show that the structure of Ni2MnSn is till of the L21 type above 98O K and not of the B2 type as proposed in the literatue. The magnetic properties and the hyperfine field distributions af as milled samples differ strongly from those of saples quenched from high temperatures. The magnetic structure of the as milled saples is disordered while the as quenched saples remain ferromagnetic. Magnetic measurements, Mossbauer and neutron results suggest that the chemical disorder induced by miling differs from chemical disorder induced by temperature.

"Interfaces and defects in nanocrystalline oxides"

D. Michel, L. Mazerolles, E. Gaffet - Mater. Sci. For., 235 - 238 (1997) 601 - 606

Nanocrystalline oxides prepared by ball milling were studied by high resolution transmissin electron microscopy (HRTEM). The image contrast and diffraction of these nanoparticles are not significantly different from that of crystalline materials. Well defined grain boundaries are observed and only rare crystal defects are found. However, disordered areas and amorphized regions are frequently see around and between crystalline particles.

"On the antiferromagnetism of Fe - Rh"

A. Hernando, J.M. Rojo, A.R. Yavari, E. Navarro, J.M. Barabdiaran, M.R. Ibarra - Mater.Sci.For, 235-238 (1997) 675

Structural and magnetic properties of ordered a' CsCl like and nanocrystalline disordered gamma fcc FeRh are reported and discussed. In particular it is remarked the spin glass behavior exhibited at low temperature by the metastable fcc phase. The transformation from antiferromagnetci to ferromagnetic configuration shown by the ordered CsCl like phase and that from spin glass to paramagnetic exhibited by the fcc phase are analysed within the framework of a new description of interactions.

"Electrocatalytic bahavior of metastable alloys prepared by ball milling"

T. Benameur, B. Rezgui, A.R Yavari, R. Durand - Mater. Sci. For., 235 - 238 (1997) 917 - 922

The effect of ball millin gof melt spun Ti2Ni ribbons and Al65Ni35 elemental powder mixtures on their electrocatalytic activity for the hydrogen evolution reaction is reported. Transmission electron microscopy and x - ray diffraction results of Ti2Ni alloy show the emergence of nanstructure with increasing milling time and transformation to the amorphous state after further milling. We have found that the nanostructured and amorphous metastable states of Ti2Ni are more active that the crystallized alloy. The transformation from the equilibrium state to the nanocrystalline induces a gain of 60 mV of the potential to be applied at 300 mA/Cm2. This gain becomes much higher for the amorphous state. Moreover, a major difference between the amorphous, nanocrystalline and crystalline alloys is the existence of two Tafel slopes for both the nanocrystalline and the crystalline alloys. The increase of activtiy is not evident for the metastable bcc Ni obtaine after leaching Al65Ni32 mechanically alloyed. However, the Nernst potential of Ni/Ni2+ was found 23 mV lower on the bcc than on the fcc electrode, which was correlated to the Gibb's free energy difference between the two crystalline states.

"Electrochemical investigatin of nanocrystalline Ni obtained by different preparations"

M. Schneider, W. Zeiger, U. Birth, K. Pischang, E. Gaffet, O. El Kedim - Mater. Sci. For., 235-238 (1997) 961 - 966

The electrochemical behavior of nanocrystalline Ni obtained by different preparations (coating and cold pressed) in different electrolytes has been investigated by potentiodynamic test methods, compared with polycrystalline Ni. The results are discussed and the passivation mechanism are proposed.

"Application of high energy ball miling in mineral materials : extraction of TiO_2 from minceral FeTiO3"

Y. Chen, J.S. Williams - Mater. Sci. For., 235 - 238 (1997) 985 - 992

A systematic study of extraction of rutile (TiO_2) from ilmenite ($FeTiO_3$) by using a high energy ball milling technique has been carried out. It was found that rutile can be obtained by either ball milling of ilmenite with elemental sulphur of other additives during which mechanochemical reactions occur at room temperature, or by ball milling of a mixture of ilmenite with carbon whereby carbothermmic reduction takes place during a subsequent low temperature annealing treatment. In the latter case, the enhancing effect of ball milling results from the intimate mixing and large contact area between ilmenite and carbon particles.

"Metastable Ti - Ru - Fe - O nanocrystalline alloys for the hydrogen evolution reaction in the chlorate industry"

M. Blouin, L. Roue, S.-H. Yip, D.Guay, J. Huot, S. Boily, A. Van Neste, R. Schulz - Mater.Sci.For., 235-238 (1997) 979 - 984

The high energy mechanical alloying of a Ti - RU - Fe - O powder mixture has been performed by extensive ball - milling. The structural evolution of the milled powders has been studied by X - ray powder diffraction analysis. The metallic ratios were first varied to find the best alloy composition (Ti_2RuFe). In the first stage of the structural transforamtion, TU or Fe atoms dissolved into Ti to yield to the formation of beta Ti. Upon further ball milling, a new simple cubic Ti2uFe phase is formed, with crystallite size as small as 8 nm. The electrochemical properties of these materials have been tested in a typical chlorate electrolyte by cold pressing the powders into a disk electrodes.

"Characterization of Co - Cu mechanical alloys by linear sweep voltammetry"

Lopezhirata VM. Arceestrada EM. - Electrochimica Acta. 42(1):61-65, 1997.

The mechanical alloying process has shown that it is possible to obtain supersaturated solid solutions at any composition in immiscible metallic systems by ball milling of elemental powder mixtures. The mechanically alloyed Co-Cu powders are chemically homogeneous supersaturated solid solutions and exhibit crystallite sizes of a nanometer order. In the present work, the mechanically alloyed Co-Cu powders have been characterized by linear sweep voltammetry in 0.5 M NaOH and 0.15 M $Na_2B_4O_7$ aqueous solutions. The electrochemical behavior has been related to the characteristics of enhanced solubility found in Co-Cu mechanical alloys. In order to perform a systematic study of this system, the voltammograms of pure Cu, pure Co, Co-Cu original powder mixtures and Co-Cu

alloys, obtained by melting and casting, were also analyzed for comparison. The results showed that the voltammograms of mechanically alloyed Co-Cu powders presented peaks of dissolution at potentials intermediate between those exhibited in pure Cu and Co samples. Moreover, a third dissolution peak occurred at more noble potentials in mechanical alloys, but not in as-cast alloys. This peak is associated with the formation of the solid solution, in mechanical alloys. This suggests that both elements are dissolved simultaneously, which confirms that Co-Cu mechanical alloys are true supersaturated solid solutions. Additionally, it was found that the MA Co-Cu alloys showed corrosion rates higher than those in as-cast alloys, which is attributed to the high value of stored energy presented in MA alloys as a result of the severe plastic deformation during ball milling.

"Fe3O4/Fe magnetic composite synthesized by mechanical alloying"

Ding J. Miao WF. Street R. Mccormick PG. - Scripta Materialia. 35(11):1307-1310, 1996

"Effect of HIP temperatures on the microstructure and mechanical properties of carbide dispersed Ti-48Al-1Mn mechanically alloyed compacts"

Ameyama K. Hashii M. Imai N. Fujii T. Sasaki N. - J. Japan Institute of Metals. 60(10):944-951, 1996

The effect of hot isostatic pressing (HIP) temperature on the microstructure and mechanical properties of Ti-48 mol%Al-1 mol%Mn compacts fabricated by mechanical alloying was investigated. N-heptane was used as a process control agent for the mechanical alloying. The compacts HIP treated at 1173, 1373 or 1573 K showed an ultra-fine equiaxed grain structure, i.e., a microduplex structure, consisting of TiAl (gamma) and Ti2AlC phases, and their average grain sizes were 185 nm, 510 nm and 1.5 mu m, respectively. The gamma phase was considered to be formed by an alpha-->gamma massive transformation during heating. On the other hand, the compacts HIP treated at 1623 or 1673 K showed quite different microstructures from the above HIP compacts. The 1623 K-HIP compact was composed of equiaxed gamma grains, whose size was approximately 11.5 mu m, rectangular shaped Ti_2AlC particles, and a small amount of the grain boundary nucleated alpha phase. Although the 1673 K-HIP compact showed a microstructure similar to the 1623 K-HIP compact, the gamma grains were coarsened to be approximately 27.8 mu m in diameter and the Ti2AlC particles were more elongated rectangles. Furthermore, the amount of the grain boundary nucleated ct phase was increased and the lamella alpha phase nucleated at gamma twin boundaries was observed in the 1673 K-HIP compact. Mechanical properties determined by compressive testing at various temperatures made clear that the compacts HIP treated at 1173, 1373 or 1573 K have good workability at elevated temperatures and those HIP treated at 1623 or 1673 K have good high temperature strength. These mechanical properties were influenced significantly by the microstructure, especially by the grain size and morphology of the Ti_2AlC phase.

"Mechanical alloying of TI-24Al-11Nb (at percent) using the simoloyer"

Zoz H. Ernst D. Weiss H. Magini M. Powell C. - Metall. 50(9):575-579, 1996 Sep.

The production of large quantities of contamination free mechanically alloyed powders from titanium based materials has proven to be major challenge. Feasibility of such a goal can be carried out at laboratory level by any milling device like the very common planetary ball mill. In this case however; the possibility of a subsequent scaling up for larger production is hindered by the intrinsic limits of a planetary ball mill design. On the contrary the horizontal Simoloyer can be experimented at laboratory level using small volume chamber-units (0.25, 0.5, and 2 I) and for industrial production, using the large volume units (up to 400 I) based on the some conceptual design. Therefore, experiments have been conducted on blended elemental Ti-24Al-11 Nb (at%) powder using a simoloyer with a small unit-chamber (0.5 I). Due to the inherent ductility of the powder the material has the tendency to adhere to the grinding unit and the steel balls. Further in order to avoid high contamination and to make the process realistic from an economical point of view. the milling time has to be reduced to a minimum. The above points identify a Critical Milling Behaviour (CMB) of the system under investigation that must be kept under control to achieve the wanted goal. It will be shown that by adopting a suitable milling and discharging procedure low contamination and good yield have been substantially achieved with respect to preliminary not unambiguous trials carried out at Idaho university.

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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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