RESEAU FRANCAIS DE MECANOSYNTHESE

Lettre N°24

Mars 1997

63 Groupes de Recherches (8 Etrangers) - 85 Correspondants

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

2 ANS DEJA

6 Nouvelles Adhésions !!
University of Western Australia : Dr. J. Harrowfield
Korea Institute of Science and Technology (Corée du Sud)**: Prf. S. H. Hong**
Andong National University (Corée du Sud) : Prf. J. H. Ahn
SINTERTECH - Le Pont de Claix - Corresp. : Dr. A. Venot
CENIM - CSIC - Madrid (Espagne) **: Dr. P. Crespo**
CENG - Grenoble : Dr. S. Revol

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Afin de réduire les frais d'émission de la Lettre du RFM et permettre la mise en place d'un serveur RFM
prière de transmettre vos coordonnées électroniques (E_Mail)

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! JRFM97 !

29 - 30 Avril 1997 à l'**Ecole des Mines de Nancy**
PRIERE DE S'INSCRIRE Rapidement
afin de faciliter la tâche du Comité d'Organisation

Contacts: S. Begin - Colin, G. Le Caër, J.C. Gachon, E. Gaffet, R. Martin - Lopez, R. Welter
(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

Propositions de COMMUNICATIONS ORALES ET POSTERS AU 24/02/97

CONFERENCES INVITEES
Gaffet (Belfort), Barbara (Grenoble), Yavary (Grenoble), Mazerolles (Vitry), Yvon (Nancy)
COMMUNICATIONS ORALES
"Elaboration d'intermétalliques Fe-Al nanométriques par MASHS. Suivi en temps réel de leurs formations"
F. Charlot et al. - Dijon / Belfort
"Etudes des propriétés d'absorption de l'hydrogène par des alliages de type AB2 élaborés par mécanosynthèse et caractérisés par MET, diffraction des RX et microsonde électronique"
D. Cracco et al. - Meudon
"Mesures de fréquences de chocs dans un broyeur planétaire type P7"
Y. Labaye et al. - Le Mans
"Transitions de phases et synthèses chimiques par macrobillage : Traitements d'oxydes en poudre"
H. Szwarc et al. - Orsay
"Comparaison de ferrites nanométriques synthétisés par deux techniques"
N. Millot et al. - Dijon.
POSTERS
"Suivi en temps réel des transitions de phase dans le système Fe/Si lors d'un recuit mécaniquement activé"
C. Gras et al. Dijon/Belfort
Etude spectroscopique de nanofluorures obtenus par broyage mécanique.
H. Guerault et al. - Le Mans
Titre à confirmer
O. Held et F.A. Kunast, Vandoeuvre les Nancy
Elaboration par mécanosynthèse et caractérisation de poudres de brasage destinées à la réparation des pièces chaudes de moteurs d'avions et de turbines industrielles.
F. Jacquot et al. - Futuroscope Poitiers
Influence du broyage et de la mécanosynthèse sur la conductivité électronique de la poudre de nickelate de lithium

(LiNiO2) utilisée dans les batteries au Li-ion
P. Perrot et al. - Futuroscope Poitiers

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ANNONCE DE CONGRES ET / OU ECOLES

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

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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

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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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
Coordinateur SF2M Jean Marie DUBOIS (LSG2M - Nancy)

1A Les Matériaux de l'Automobile
Animateurs P. Antona (Fiat - Turin) et G. Maeder (Renault)
1B - Mise en forme des polymères : incidence sur les structures et propriétés
organisé avec le patronage du Groupe Français des Polymères et de la Société des Ingénieurs des Plastiques (sous réserve d'accord définitif)
Animateurs : B. Escaig et J.M. Lefebvre de l'Université de Lille et J. Pabiot de l'Ecole des Mines de Douai.
1C - Aciers spéciaux : présent et futur
Animateurs : G. Béranger, UTC, G. Labbe et P. Mazars, Usinor-Sacilor
2A Nanomatériaux et Nanostructures : architectures ultim, phénom. fondam. fonct. & appl.
Animateurs : E. Gaffet (Belfort), P. Panissod (Strasbourg), H.-E. Schaeffer (Stuttgart), L. Schultz (Dresden)
2B - Utilisation des techniques spectroscopiques pour létude d'interfaces, de défauts et de ségrégation dans les matériaux.
Animateurs : C. Senemaud, Université P. et M. Curie et A. Traverse, LURE
3A - Thermodynamiques numérique et expérimentale.
Animateurs : C. Bichara, CTM Marseille et C. Chatillon, LTPCM Grenoble
3B - Transferts thermiques dans les matériaux : des puits de chaleur aux barrières thermiques.
Animateurs : R. Mévrel, ONERA et P. Pigeat, Centre d'Ingénierie des Matériaux, Nancy

2A - Nanomatériaux et nanostructures.
Architectures ultimes, phénomènes fondamentaux, fonctions et applications.

Steering Committee: E. Gaffet (Belfort), P. Panissod (Strasbourg), H.-E. Schaeffer (Stuttgart), L. Schultz (Dresden)
Objet : Les nanomatériaux et les nanostructures forment un vaste ensemble de matériaux allant des alignements atomiques (1D) et des multicouches (2D) aux nanocristaux et aux matériaux granulaires ultrafins. Ils présentent des propriétés qui peuvent être ajustées pour satisfaire un grand nombre d'applications technologiques.
Le symposium s'intéressera aussi bien à l'état de l'art de ces matériaux dans les secteurs de développement industriel qu'aux phénomènes fondamentaux qui naissent de la réduction des dimensions. L'intention est ainsi de faciliter l'interaction entre les travaux de recherche de base et les études technologiques visant des applications industrielles.
Les principaux sujets qui seront abordés sont :

- Synthèse et procédés de mise en oeuvre des nanomatériaux
- Multicouches
- Nanomanipulation d'atomes et de molécules
- Transport atomique
- Thermodynamique des nanomatériaux
- Propriétés mécaniques
- Propriétés magnétiques et magnéto-transport
- Propriétés optiques

L'essentiel du symposium portera sur les métaux et alliages, les intermétalliques, les céramiques et les nanocomposites.
Le symposium sera organisé conjointement par la SF2M et la Fédération Européenne des Sociétés de Matériaux. Il devrait rassembler des scientifiques, surtout européens, venant autant de l'industrie que des laboratoires universitaires. Les conférences plénières, dont la liste suit, seront données en anglais. Les participants sont invités à proposer des communications pour les séances poster mais un petit nombre d'entre elles seront sélectionnées ultérieurement pour une présentation orale.
Conférenciers :
R. Birringer : Aspects of nanomaterials *(sous réserve de confirmation)*
R. Coehoorn : Giant Magneto-resistance
H. Davies : Enhanced Properties Through Nanostructural Refinement for Bulk Hard and Soft Magnetic Alloys
R. Wurschum : Atomic transport
L. Greer : Thermodynamics of nanomaterials *(sous réserve de confirmation)*
A. Inoue : Mechanical properties *(sous réserve de confirmation)*
J.M. Gérard : Nanomanipulations
M. Backhaus Ricoult: Nanoceramics, Nanocermets : Structures and Properties
J. Perez : Nanophases in polymers
J. Eckert : Solid state processing of nanomaterials

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.

RAPPEL COMPLET DES SOUTENANCES DE THESES PRECEDENTES

P. Pochet - Université des Sciences et Technologies de Lille I / Sciences des Matériaux **6 Janvier 1997**
Etude Expérimentale et Modélisation des Changements de Phases sous Broyage à Haute Energie
Jury : J. Foct, Y. Brechet, G. Le Caer, P. Bellon, G. Martin

M.O. Kientz - 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, B. Malaman , , E. Gaffet, 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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R. Elkalkouli - Poitiers - Science des Matériaux - **9 Novembre 1995**
"Microstructures et propriétés physiques d'alliages magnétiques obtenus par mécanosynthèse"
Jury : J. Rabier, E. Gaffet, F. Nardou, A. Walder, J. Mimault, M. Grosbras, J.F. Dinhut

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O. Drbolhlav - INP Grenoble - **19 Octobre 1995**
"Matériaux magnétiques nanocristallins métastables à base de Fer et de Cuivre"
Jury : J.C. Joubert, J.C. Perron, G. Le Caër, J. Kulda, A.R. Yavari

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A. Malchere - Paris XI - Métallurgie Spéciale et Matériaux - **3 Octobre 1995**
"Elaboration par mécanosynthèse, puis études structurales, microstructurales et mécanique d'un composite à base Al et à renforts nanométriques SiC"
Jury : L. Priester, Y. Bienvenu, G. Le Caer, M. Grosbras, E. Gaffet

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M. Souza de Rigueiredo - Lille I - Science des Matériaux - **17 Juillet 1995**
"Mécanosynthèse d'alliages binaires et ternaires Fe - Me - N caractérisées et discutées à partir des résultats de spectrométrie Mössbauer"
Jury : J. Foct, G. Le Caër, E. Gaffet, J.P. Morniroli, J.P. Ganne

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F. Wolf - INP Lorraine - Science et Génie des Matériaux **9 Juin 1995**
"Etude & Modélisation du broyage ultrafin d'une poudre de zircone. Mécanosynthèse d'alliages à base de zircone. Matériaux denses à nanograins"
Jury : A. Mocellin, G. Cizeron, E. Gaffet, C. Bert, G. Braichotte, G. Le Caer

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D. Osso - INP Lorraine - Science et Génie des Matériau **19 Mai 1995**
"Elaboration de nanocomposites Alumine-Métal (Fe,Cr,Ni) par mécanosynthèse"
Jury : J. Bigot, E. Gaffet, P. Matteazzi, T. Lindback, G. Le Caër, A. Mocellin

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D. Galy - Université Paris XI - 11 Janvier 1995
Contribution à l'étude des mécanismes d'amorphisation par sollicitation mécanique de composés intermétalliques"
Jury : A. Revcolevski, P. Guyot, J.P. Poirier, P. Donnadieu, L. Boulanger, G. Martin

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C. Chausse - Université de Limoges - **16 Décembre 1994**
"Etude du frittage en phase solide d'un alliage base tungstène. Influence du broyage des poudres"
Jury : P. Goursat, C. Allibert, G. Le Caër, Y. Bienvenu, G. Braichotte, E. Gaffet, F. Nardou

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P. Pierrat - INP Lorraine - 12 Décembre 1994
"Elaboration de poudres thermoélectriques du type Bu - Sb - Te par mécanosynthèse. Mise en forme et caractérisation"
Jury : G. Le Caër, G. Borchardt, A. Thomy, A. Pianelli, S. Scherrer, H. Scherrer, C. Tête

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L. Aymard - Université de Picardie Jules Verne - **26 Octobre 1994**
"Préparation par broyage réactif d'alliages Ag-Pd, Ni-Co, La-Ni, Pd-Rh et étude des mécanismes de réaction dans le cas du système Ag-Pd"
Jury : P. Boch, J.-M. Tarascon, G. Le Caër, A. Percheron - Guegan, M. Touboul, A. Delahaye - Duval

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K. Wolski - INPG et ENS des Mines de Saint Etienne - **14 Octobre 1994**
"Influence de la dispersion de phases céramiq. sur la résistance au fluage de l'intermétal. FeAl"
Jury : P. Desré, G. Le Caër, F. Moret, D. Morris, J. Le Coze, F. Thévenot, T. Khan

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M. Abdellaoui - Université Paris VI - Chimie des Matériaux - **4 Juillet 1994**
"Transitions de Phases sous chocs mécaniques : Mécanosynthèse du système magnétique Fe -Si
Jury : R. Portier, C. Djega - Mariadassou, J. Degauque, Ph. Fluzin, E. Gaffet, G. Le Caër

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M. Grardel - Institut National Polytechnique de Grenoble - **17 Juin 1994**
"Mécanosynthèse d'acier ferritique O.D.S.:Paramètres du procédé et microstructure du matériau"
Jury : G. Le Caër, G. Martin, J.L. Bernard, D. Francois, F. Louchet, F. Moret

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L. Yousfi - Université Paris VI - **19 Mai 1994**
"Transitions de phases sous sollicitations mécaniques. Elaboration de poudre Ni_3AL par broyage de poudres élémentaires (Al et Ni) ou de rubans de Ni_3Al"
Jury : R. Portier, C. Djega-Mariadassou, G. Bouquet, O. Dimitrov, E. Gaffet

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Jacques Secondi - INPG - Science et Génie des Matériaux - **15 Avril 1994**
"Composites nanocristallins céramique - métal préparés par broyage réactif d'alliages précurseurs"
Jury : C. Bernard, M.D. Baro, A.L. Greer, M. Suery, P.J. Desré, A.R. Yavari

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N. Malhouroux - Gaffet - Université Paris VII - **9 Avril 1993**
"Synthèse de siliciures : Etude des transitions de phases dans le système Fe - Si"
Jury : S. Rigo, E. Gaffet, P. Gas, G. Le Caër, G.M. Martin

Y. Chen - Université Paris XI - **22 Octobre 1992**
"Contribution à la physique du procédé de mécanosynthèse"
Jury : J. Philibert, J.P. Poirier, G. Le Caër, G. Martin, R. Le Hazif

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F. Bordeaux - INP Grenoble - **13 Octobre 1989**
"Mélange atomique exothermique et amorphisation en phase solide dans des composites de multicouches fines métal - métal préparées par co - laminage"

Jury : J. Philibert, M. Gerl, P. Desre, A.R. Yavari, J.L. Castagne, J. Muniesa

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

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 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)

"Oxide dispersed materials prepared by reaction milling of Al - Li and Al - Li - Mg alloys with various oxides" (Japonais)
Higuchi H. Sugamata M. Kaneko J. - Journal of the Japan Institute of Metals. 60(12):1207-1214, 1996
With a purpose of attesting selective oxidation of solute Li and Mg in solid Al, mechanical alloying was carried out for AI-Li and Al-Li-Mg alloys with or without addition of various metal oxides, and subsequently P/M materials were prepared. Constituent phases were determined by XRD for the mechanically alloyed powders and the as-extruded and heat-treated P/M materials. Hardnesses and microstructures were examined for the P/M materials. It has been confirmed that Li is selectively oxidized and LiAlO2 is generally formed in the AI-Li alloys. In the Al-Li-Mg alloys, Mg is oxidized in preference to Li. After hot extrusion, hardness increases were not observed with progressing preferential oxidation and reduction of the added metal oxide. Coarsening of LiAlO2 particles was observed in the P/M materials after heating at 873 K. As-extruded P/M materials showed relatively high hardness and hence dispersion hardened materials of high mechanical strength can be expected by applying preferential oxidation of the solute in aluminum.

"Structural evolution of the NM(O.52)Sb(0.48) compound during high energy ball milling"
Yin FX. Gu NJ. - Journal of Materials Science & Technology. 12(6):434-438, 1996
The structural transitions of the NiAs-type Mn0.52Sb0.48 magneto-ordered compound, ball milled to different periods, have been characterized by X-ray diffraction and DSC analysis. On the basis of lattice parameter results a structural evolution mode with three stages is proposed. In the first stage lattice parameters keep nearly unchanged with the refinement of grains and increase of lattice strain. In the second stage, microstrain shows a lowering tendency accompanying the successive decreases of grain size. The X-ray revealed internal strain is found to be strains inside the lattice, which can be relaxed with new grain formation. The change of T-c is shown to be affected by the dimension of c axis, however the overall magnetization is continuously decreased with milling, due to the disordering process occurred in milling. Correspondent disordering mechanisms have been tentatively postulated and discussed according to: the changes of lattice parameters.

"Prediction of impact forces in a vibratory ball mill using an inverse technique"
Huang H. Pan J. Mccormick PG. - International Journal of Impact Engineering. 19(2):117-126, 1997
To understand the dynamic mechanisms of the mechanical milling process in a vibratory mill, it is necessary to determine the characteristics of the impact forces associated with the collision events. However, it is difficult to directly measure the impact force in an operating mill. This paper describes an inverse technique for the prediction of impact forces from acceleration measurements on a vibratory ball mill. The characteristics of the vibratory mill have been investigated by the modal testing technique, and its system modes have been identified. In the modelling of the system vibration response to the impact forces, two modal equations have been used to describe the modal responses. The superposition of the modal responses gives rise to the total response of the system. A method based on an optimisation approach has been developed to predict the impact forces by minimising the difference between the measured acceleration of the vibratory ball mill and the predicted acceleration from the solution of the modal equations. The predicted and measured impact forces are in good agreement.

"Selective mechanochemical dehalogenation of chlorobenzenes over calcium hydride"
Loiselle S. Branca M. Mulas G. Cocco G. - Environmental Science & Technology. 31(1):261-265, 1997
We have shown that in the presence of a reactive substrate, chlorinated organic compounds can be dehalogenated by mechanical treatment, and a specific reaction product can be obtained. We have used a ball milling process at low temperature and atmospheric pressure to produce a dechlorination of up to 100% for both liquid and solid chlorinated compounds. The products of the completed reaction for trials with hexa chlorobenzene and chlorobenzene were both principally restricted to benzene and chloride salts. The use of CaH2 as a source of active hydrogen produces a much more specific reaction in significantly less time in comparison with CaO and MgO substrates reacted under hydrogen atmosphere. The process was found to depend an the injected mechanical energy and the collision frequency. In the

case of hexachlorobenzene, an explosive-type reaction was observed to occur at specific milling times as a function of the kinetic energy employed.

"Solid state reaction in Al based composites made by mechanofusion"

Csanady A. Csordaspinter A. Varga L. Toth L. Vincze G. - Mikrochimica Acta. 125(1-4):53-62, 1997.

Mixtures of aluminium and other metal powders were milled in a Hosokawa AM-15F mechanofusion system in order to produce composite materials with coated or layered microstructure. These composites were then annealed or used in plasma spraying experiments. The intermetallic phases produced in the consecutive steps of the treatment were investigated by different methods. In the case of the Al-Ni powder system the presence of intermetallic phases confirmed that phase forming solid state reactions start during the first, mechanofusion step. After milling the powder mixture in the Hosokawa equipment, crystalline Al3Ni could be detected by TEM. A second intermetallic phase, Al3Ni2 was also observed after a heat treatment at 750 K. In Al-Cu-Fe and Al-Cu-Co powder composites, produced by milling, binary and ternary phases could be found only after plasma spraying. That means that in these cases the thermodynamic and kinetic requirements of the reactions could not be fulfilled by this mild milling. Nevertheless, the considerably large specific surface of the metal-metal interfaces, formed during the milling process in the ternary composites, makes it possible to produce multi-phase coatings From these composites. It means that only two technological steps are required (milling - plasma spraying). All of the other known technologies consist of three steps: alloy preparation - milling - plasma spraying or alloy preparation atomisation - plasma spraying. Similarities and differences between the reactions taking place in thin films and thin and/or small milled particles are discussed.

"Mechanical alloying of an immiscible alpha Fe2O3 -SnO2 ceramic"

Jiang JZ. Lin R. Morup S. Nielsen K. Poulsen FW. Berry FJ. Clasen R. - Phys. Rev. B,. 55(1):11-14, 1997

A solid solution of about 6 mol% SnO2 in alpha-Fe2O3 has been prepared by mechanical alloying of alpha-Fe2O3 and SnO2 powder blends. This result demonstrates that high energy ball milling can be used to prepare metastable oxide solid solutions with an extended range of compositions in the immiscible ceramic oxide system. X-ray diffraction and Mossbauer spectroscopy investigations show that mechanical milling of alpha-Fe2O3 and SnO2 involves alloying on an atomic scale and that true solid solution formation occurs. We suggest that the high defect concentration and the chemical enthalpy of Fe3+-O2--Sn4+ interfaces between nanostructured alpha-Fe2O3 and SnO2 regions may serve as a driving force for the formation of a solid solution in the immiscible ceramic system.

"Zeolite ball milling as a means of enhancing the selectivity for base catalyzed reactions"

Xie JH. Kaliaguine S. - Applied Catalysis A-General. 148(2):415-423, 1997 Jan 2.

High energy ball milled KNaX zeolites were characterized using X-ray diffraction, BET, and FTIR techniques. The alkylation of toluene with methanol was chosen as a probe reaction for catalytic testing. Ball milling results in the collapse of the zeolite crystalline structure and its transformation into an XRD amorphous phase. Proper ball milling was shown to enhance the catalytic selectivity towards the formation of ethylbenzene + styrene during the alkylation. It was concluded that proper milling can moderately decrease both the Lewis base and Lewis acid sites concentration in alkali exchanged faujasite zeolites while deeply decreasing the strong Bronsted acid site density. The formation of xylenes is mainly dependent on Bronsted acid but not on Lewis acid site centers.

"High temperature deformation of a mechanically alloyed Niobium Yttria alloy"

Chou I. Koss DA. Howell PR. Ramani AS. - Materials Science & Engineering A- 222(1):14-20, 1997

Mechanical alloying (MA) and hot isostatic pressing have been used to process two Nb alloys containing yttria particles, Nb-2vol.%Y2O3 and Nb-10vol.%Y2O3. Similar to some thermomechanically processed nickel-based alloys, both alloys exhibit partially recrystallized microstructures, consisting of a 'necklace' of small recrystallized grains surrounding much larger but isolated, unrecrystallized, cold-worked grains. Hot compression tests from 1049 to 1347 degrees C (0.5-0.6T(MP)) of the 10% Y2O3 alloy show that MA material possesses a much higher yield and creep strength than its powder-blended, fully recrystallized counterpart. In fact, the density-compensated specific yield strength of the MA Nb-10Y(2)O(3) exceeds that of currently available commercial Nb alloys.

"Preparation of nanocrystalline Fe - Si alloys and their magnetic properties"

Zhou TJ. Zhang JR. Xu JF. Yu ZQ. Gu G. Wang DH. Huang H. Du YW. Wang JH. Jiang YM. - Journal of Magnetism & Magnetic Materials. 164(1-2):219-224, 1996

Nanocrystalline Fe-Si alloys have been prepared using the mechanical alloying method. The structure of the obtained Fe-Si alloys is the same as that of bcc alpha-Fe. The final powders obtained mainly consist of particles with a grain size of from a few hundred nanometers to several microns, and the particles are composed of subgrains with a microcrystalline size of about 15-20 nm and an almost completely random orientation with respect to each other. The magnetic properties have also been investigated. The specific saturation magnetization (sigma(s)) values are slightly less than those of the corresponding single-crystalline and polycrystalline Fe-Si alloys, while the coercive force (H-c) values are much higher than those of the corresponding bulk Fe-Si alloys, The magnetic spectra are all relaxation-type except for Fe75Si25; the values of mu' and mu " are comparable to those of ferrite composite materials and domain wall displacements are predominant in the magnetization process.

"Phase formation during mechanical alloying of Palladium, Silicon and copper powder mixtures"

Zhang DL. - Journal of Materials Science Letters. 16(1):12-15, 1997 Jan 1.

"Study of structural transformations in potassium exchanged zeolite induced by thermed and mechanochemical treatments"

Kosanovic C. Subotic B. Smit I. Cizmek A. Stubicar M. Tonejc A. - Journal of Materials Sci.. 32(1):73-78, 1997

Thermal transformations of potassium-exchanged zeolite A and the X-ray amorphous material obtained by ball milling the potassium-exchanged zeolite A were investigated by different methods, such as differential thermogravimetric analysis (DTA), differential scanning calorimetry (DSC), X-ray diffraction (XRD) and Fourier transform infrared (FTIR) spectroscopy. Controlled heating of crystalline, potassium-exchanged zeolite A causes a phase transformation in the sequence: [0.22 Na2O, 0.78 K2O]. Al2O3 . 2SiO(2) . 3.48 H2O double right arrow amorphous double right arrow kalsilite + kaliophilite, while the heating of mechanochemically amorphous potassium-exchanged zeolite A results in its transformation into a mixture of kalsilite and kaliophilite. The differences in the

pathways of the transformation processes are discussed in terms of the structural properties of the starting materials.

"Magnetic properties of Ba and Sr hexaferrite prepared by mechanical alloying"

Ding J. Street R. Nishio H. - Journal of Magnetism & Magnetic Materials. 164(3):385-389, 1996

Samples of Ba- and Sr-hexaferrite were prepared by mechanical alloying and subsequent heat treatment were found to consist of single domain particles of the single hexaferrite phase. The particles had a wide distribution of anisotropy fields. Study of irreversible magnetisation suggested, that the demagnetisation process is mainly controlled by the Wohlfarth rotation. It was deduced from the results of measurements of magnetic viscosity, that the activation volume was of same order of magnitude as the cube of the domain wall thickness.

"Microstructure and nanoscale composition analysis of the mechanical alloying of FexCu100-x (x = 16, 60)"

Huang JY. Yu YD. Wu YK. Li DX. Ye HQ. - Acta Materialia. 45(1):113-124, 1997

The microstructures of Fe16Cu84 and Fe60Cu40 (atomic percent) during mechanical alloying (MA) were studied by high resolution electron microscopy (HREM). Nanoscale composition distribution in Fe16Cu84 was determined using a HF 2000 FEG TEM. In the Fe16Cu84 specimen, a number of deformation twins were observed. In the Fe60Cu40 specimen, shear band and generation of nanocrystals in the shear band were observed, which is shown to be a typical mechanism for grain size reduction during MA. In both specimens, the b.c.c. grains tend to be very small (<5 nm) before alloying, which is shown to be a prerequisite condition for the dissolution of Fe in Cu and is also direct evidence to support the thermodynamical model proposed by Yavari et al. Nanoscale composition analysis in Fe16Cu84 specimen shows that the average Fe contents in both the interior of grains and the grain boundaries (GBs) are close to the designed composition, thus proving that a supersaturated solid solution has really formed. However, the Fe contents in both cases are rather inhomogeneous, indicating that the mixing of Fe and Cu during MA is inhomogeneous. The process of MA is suggested to be divided into two stages: at the early stage, the grain sizes reduce quickly to a steady value due to the mobility of dislocations; further deformation can be fully accommodated by GBs. As a result, very fast volume diffusion and GB diffusion are achieved. NC-structure and greatly enhanced diffusion coefficients allow the formation of supersaturated solid solutions in immiscible systems with positive enthalpy of mixing.

"Microstructural development in dispersion strengthened NiAl produced by mechanical alloying and secondary recrystallization"

Grahle P. Arzt E. - Acta Materialia. 45(1):201-211, 1997

In order to improve the creep strength of NiAl, we have developed oxide-dispersion strengthened (ODS) NiAl alloys through a powder metallurgical processing route involving Mechanical Alloying (MA) and secondary recrystallization. The powders and the consolidated material are analysed by means of TEM, quantitative metallography and X-ray diffraction. Particular attention is paid to achieving, by thermomechanical processing, a favourable microstructure in terms of creep strength, i.e. a large grain size and finely distributed dispersoids. It is found that undesirable coarsening of dispersoids during recrystallization can be reduced by encouraging abnormal grain growth (secondary recrystallization) through predeformation. The connection between grain growth and dispersoid coarsening is analysed quantitatively. Overall, it is demonstrated that the conflicting requirements of large grains and relatively fine dispersoids can be satisfied by well-defined processing steps.

"Novel spin glasses by mechanical milling"

Zhou GF. Bakker H. - Science in China Series E-Technological Sciences. 39(4):354-368, 1996

Novel spin-glass alloys were synthesized by milling intermetallic compounds and also by milling mixtures of crystalline elemental powder in a high-energy ball mill. Spin glass behaviour was found in amorphous Co2Ge, which was amorphised by milling in mechanically disordered crystalline GdAl2 in ball-milled crystalline and amorphous CoZr, and in mechanically alloyed Co-Cu, which formed a supersaturated f.c.c. solid solution. All these materials are binary alloys and the concentration of the magnetic element is high, which makes them novel types of spin glasses. It is shown that ball milling may not only lead to structural metallic glasses, but can also generate the magnetic pendant of a structural glass, namely the spin glass.

"Mechanical alloying process of 93W-5.6Ni-1.4Fe tungsten heavy alloy"

Ryu IJ. Hong SH. Baek WH. - Journal of Materials Processing Technology. 63(1-3):292-297, 1997

The mechanical alloying process of 93W-5.6Ni-1.4Fe tungsten heavy alloy from the elemental powders of W, Ni and Fe by a high energy ball mill in argon atmosphere was investigated. The mechanical alloying process parameters such as milling speed, milling time, ball-to-powder ratio and ball filling ratio were varied in order to investigate their influence on the microstructural evolution of mechanically alloyed powders. The mechanical alloying process proceeded following five distinct stages such as flattening stage, welding dominant stage, equiaxed particle forming stage, random lamellar forming stage and steady state stage with increasing the milling time. The steady state stage of mechanical alloying was reached after milling for 48 hours with milling speed of 75 rpm, ball-to-powder ratio of 20:1 and ball filling ratio of 15%. Nanocrystalline grain size of 16 nm was obtained at the steady state stage of mechanical alloying. Mechanically alloyed powders were consolidated by cold isostatic pressing and followed by sintering at temperature ranged 1300-1485 degrees C for 1 hour in hydrogen atmosphere. When liquid phase sintered at 1485 degrees C, tungsten heavy alloy from mechanically alloyed powders showed finer tungsten particles about 27 mu m than that from conventionally blended powders. The density of liquid phase sintered tungsten heavy alloy decreased with increasing the milling time due to the swelling during sintering. When solid state sintered below 1430 degrees C, tungsten heavy alloy from mechanically alloyed powders showed ultra-fine tungsten particles about 3 mu m and showed high relative density above 97% insensitive to the milling time.

"Preparation of Cu - Al - Ni shape memory alloys by mechanical alloying and powder metallurgy method"

Tang SM. Chung CY. Liu WG. - Journal of Materials Processing Technology. 63(1-3):307-312, 1997

The conventional cast shape memory alloys (SMA) billets can be hot worked into components with uniform cross sections. However, near-net shape components can be made more easily by the powder metallurgy method which can save a lot of hot working procedures and thus simplify the fabrication process. The elemental powders are pre-alloyed by high energy ball milling, this mechanical allowing process significantly modifying the characteristics of the

ponders and shortening the sintering time. SEM, X-ray Diffraction analysis and DSC thermal analysis have been used to characterize the pre-alloyed powders, the green compacts and the solution-treated compacts. After suitable heat treatment, the shape memory property is produced. However, it is found that when the ball milling time is too long, the amount of martensite produced by solid solution treatment and subsequent quenching will be lowered, and reflected in the endothermic peak during phase transformation in DSC analysis. This investigation is focused on the necessary conditions for fabricating Cu-Al-Ni-based(CAN) shape memory ally by means of powder metallurgy.

"Microstructures and mechanical properties of Al - Li / SiCp composite produced by extrusion processing"

Hanada K. Murakoshi Y. Negishi H. Sano T. - J. Materials Proc Techn.. 63(1-3):405-410, 1997

This paper describes the influence of extrusion parameters on the microstructures and the mechanical properties of SiC particles reinforced AI-Li composite. Al-Li/SiCp composite powders were mechanically milled at 500 rpm. The composite powders were consolidated at 573K by hot pressing, and then extruded at 773K with different reduction ratios. The specimens extruded at the reduction ratio of 10 had difference of SiCp distribution between the core and the outer layer, while the difference was not almost observed for specimens extruded at over 20. The increase of reduction ratio improved the distribution of SiC particles and gave recrystallization of Al-Li matrix, and the consequently tensile properties was enhanced. But higher reduction ratio such as 40:1 decreased the mechanical properties. The reduction ratio of 20:1 was superior in the mechanical properties of extruded Al-Li/10vol%SiCp composite under all of conditions.

"Milling induced microstructural change in furnace cooled eutectoid Zn - Al alloy"

Zhu YH. Hirata VML. Saucedomunoz M. - J. Materials Processing Technology. 63(1-3):624-627, 1997

During mechanical milling, the microstructure of the furnace cooled eutectoid Zn-Al based alloy filings changed from lamellar structure to fine-grain structure and the decomposition of the Zn rich metastable eta'(FC) phase and the four-phase transformation were fastened. which were essentially the same results as with the tensile and creep induced microstructural change and phase transformations.

"Synthesis of nanostructured materials by mechanical alloying"

Matteazzi P. Lecaer G. Mocellin A. - Ceramics International. 23(1):39-44, 1997.

Materials can be processed by grinding not only for comminution, but also to obtain a variety of structures (amorphous, nanophased), fine mixing of phases, alloys and to directly synthesize compounds such as carbides. Nanocomposites can also be synthesized by reduction, exchange and mixing reactions driven by grinding, as well as by combining the above processes. Large scale economical production of such materials is feasible by grinding in mills designed for that purpose. The mechanical properties of materials are enhanced by the nanophase structure.

"Processing and properties of SiC particulate reinforced Al - 6.2Zn - 2.5 Mg - 1.7 Cu alloy (7010) matrix composites prepared by mechanical alloying"

Bhaduri A. Gopinathan V. Ramakrishnan P. Miodownik AP. - Mat. Sci. & Eng. A-221(1-2):94-101, 1996 SIC particulate reinforced aluminium alloy (7010) matrix composites have been processed by mechanical alloying route starting from elemental powders. Proper post-consolidation by hot pressing or hot extrusion results in a fully dense composite compact that retains the fine microstructure developed during mechanical alloying. Addition of SIC was found to result in lower strengths at room temperature but higher strength at temperatures above 200 degrees C. There was nearly a 64% increase in the yield strength at 350 degrees C, brought about by addition of 20 wt.% SiC. The beneficial effects of SiC additions on mechanical strength are best realized at elevated temperatures. Fine precipitates of various intermetallics (CuAl2, Mg2Zn11, etc.) are found to be present in the solutionised and aged samples in addition to fine dispersoids of Al2O3, MgAl2O4, etc. inherited from the as-milled powders and/or formed during subsequent degassing and consolidation

"Nanostructured WC - Co alloy prepared by mechanical alloying"

Xueming MA. Gang JI. - Journal of Alloys & Compounds. 245:L 30-L 32, 1996

Nanocrystalline cemented carbide powder of WC-Co was directly synthesized by mechanical alloying. The structure evolution of the powders was monitored by X-ray diffration, scanning electron microscope and thermal analysis. Results show that the formation of the compounds is controlled by an inter-diffusion reaction between elements. Powders of WC-Co milled for 100 h were compacted and sintered to a cylinder with the size circle divide 6 mmx8 mm. The hardness and sintered density were measured.

"Production of rutile from ilmenite by room temperature ball milling induced sulphurisation reaction"

Chen Y. Marsh M. Williams JS. Ninham B. - Journal of Alloys & Compounds. 245:54-58, 1996

Rutile (TiO2) was produced at room temperature from mineral ilmenite (FeTiO3) by high energy ball milling of an ilmenite-sulphur mixture. A mechanochemical reaction sulphurisation of iron in ilmenite producing TiO2 and FeS2, occurred during the milling process. During the subsequent annealing, the nanocrystalline TiO2 obtained from milling recrystallised and FeS2 decomposed at 600 degrees C. Pure TiO2 powder was obtained by a simple selective chemical leaching with HCl acid solution. The results indicate that mechanochemistry is a useful room temperature process of material production.

"Formation of amorphous alloys with significant supercooled liquid region by mechanical alloying"

Eckert J. Seidel M. Schultz L. - Journal of Non-Crystalline Solids. 207(Part 2):500-503, 1996

Amorphization and undercooling of Zr-, Mg-, and Al-based transition metal alloys prepared by mechanical alloying of elemental powders are investigated. The progress of alloying and the resulting phases are characterized by X-ray diffraction and differential scanning calorimetry. Zr- and Mg-based alloys were found to exhibit a large glass-forming ability and an extended supercooled liquid region. In contrast, the mechanically alloyed Al-based alloys consist of two-phase mixtures of a small amount of amorphous material and nanocrystalline phases and show no extended supercooled liquid region. The results are compared with data for quenched bulk amorphous alloys. First results on bulk samples obtained by consolidation of as-milled powders above glass transition temperature are presented.

"Mg based amorphous alloys with extended supercooled liquid region produced by mechanical alloying"

Seidel M. Eckert J. Zuecorodrigo E. Schultz L. - J. Non-Crystalline Solids. 207(Part 2):514-517, 1996

Mg-Y-TM (TM = Cu, Ni) amorphous alloys have been prepared by mechanical alloying exhibiting an extended

supercooled liquid region before crystallization. The powders were investigated by X-ray diffraction and differential scanning calorimetry (DSC). The thermal stability of these materials, their crystallization kinetics and their grain growth were investigated by means of DSC heated at 40 K/min and isothermal annealing. The results indicate that the crystallization behavior of the mechanically alloyed powders is nearly identical with that of rapidly quenched samples.

"EXAFS study on mechanically alloyed Fe - B powder mixtures"

Nasu T. Sakurai M. Suzuki K. Koch CC. Edwards AM. Sayers DE. - J. Non-Cryst. Sol. 207(Part 2):527-530, 1996

The main purpose of this work is to utilize EXAFS to observe the atomic-scale structural changes during the MA (mechanical alloying) process of two Fe-B powder mixtures: one is Fe80B20 (which is amorphized by rapid quenching (RQ but not by MA) and the second is Fe50B50 (which is amorphized by MA, but not by RQ). In the MA process of Fe50B50, the dissolution of B atoms into the alpha-Fe crystal was observed. In contrast to this, in the MA process of Fe80B20, the B atom dissolution into the alpha-Fe crystal was not observed.

"Structure of amorphous Se prepared by milling"

Fukunaga T. Utsumi M. Akatsuka H. Misawa M. Mizutani U. - J. Non-Cryst. Sol.. 207(Part 2):531-535, 1996

The amorphization of trigonal Se can proceed by mechanical milling. During the amorphization, the second and third nearest neighbours of the trigonal crystal structure were observed to be drastically changed in the RDF(r). The number of atoms located at the second distance (r = 3.4 Angstrom) is reduced from four to zero as a result of a shift of atoms towards a higher r value. After the amorphization we found that the number of atoms located in the vicinity of 3.7 Angstrom increased to six. The result leads to the conclusion that the bonding between chain-molecules in the trigonal Se is destroyed by the strain and defects induced by the milling.

"The atomic structure of aluminum based metallic glasses and universal criterion for glass formation"

Egami T. - Journal of Non-Crystalline Solids. 207(Part 2):575-582, 1996 Oct.

It recently became recognized that several phenomena related to the formation of glass or liquid for metallic alloy systems can be explained in terms of a universal criterion for glass formation. These include the composition limit for glass formation by rapid cooling of a liquid alloy, that for solid state amorphization by radiation, thermal or mechanical alloying, and the conditions for glass transition and melting of a crystalline alloy. The aluminum-based metallic glasses, however, appear to be an exception for this rule. It is shown that, by modifying the rule to allow for the interstitial site occupation, the composition range of glass formation for aluminum-based glasses can also be explained by the same principle of local topological instability.

"Mechanically alloyed Fe - Zr (B, Cu) alloys - Effect of composition and heat treatment on the microstructure and the magnetic properties"

Stiller C. Eckert J. Roth S. Schafer R. Klement U. Schultz L. - J Non-Cryst. Sol.. 207(Part 2):620-623, 1996

Nanocrystalline Fe86Zr7(B,Cu)(7) alloys were prepared by mechanical alloying of elemental powders, and investigated by X-ray diffraction, differential scanning calorimetry and electron microscopy. The as-milled powders consist of supersaturated bcc solid solutions with grain sizes of typically 5-10 nm and a small amount of amorphous phase, which depends on the overall composition of the alloy. The refinement of the crystal size to the nanometer range is accompanied by an increase in atomic-level strain. Heat treatment at elevated temperatures leads to chemical redistribution, strain release and grain growth for the bcc solid solutions prior to the crystallization of the amorphous phase. Both grain growth and strain release depend on the volume fraction of amorphous phase and the particular annealing treatment of the powders. The magnetic properties of the material are discussed with respect to the composition of the material, the deformation and recovery processes during milling, and the strain release and the phase transitions upon subsequent heating. The results are compared with data for rapidly quenched materials.

"Amorphous and metastable Fe1-xSix alloys and compounds prepared by solid state synthesis"

Ruuskanen P. - Journal of Non-Crystalline Solids. 207(Part 2):629-632, 1996

Metastable amorphous and crystalline Fe1-xSix alloys and compounds were synthesized in an argon atmosphere with and without hexane, using a solid-state alloying method in a composition range of $0.1 < x < 0.4$. Systems alloyed with hexane formed a single-phase amorphous structure with a composition range of $0.1 < x < 0.2$. The Curie temperature of the amorphous phase decreased from 376 to 173 degrees C, and that of the crystalline phase from 722 to 517 degrees C as a function of x. The carbon content of the powder alloyed with hexane increased during the alloying time, reaching a maximum value of 8 wt% after 350 h of milling. It is concluded that carbon increases the tendency for the formation of amorphous phase. The Fe1-xSix system synthesized without hexane includes an amorphous phase together with crystalline FeSi and Fe2Si, with the composition range of $0.3 < x < 0.4$. Solid solution exists in the composition range of $0.1 < x < 0.2$. Curie temperatures decreased from 716 to 544 degrees C in crystalline phases and from 330 to 115 degrees C in the amorphous phases as a function of x.

"Effect of non - stoichiometry on the ordering behaviour of nanocrystalline NiAl produced by mechanical alloying"

Murty BS. Joardar J. Pabi SK. - Journal of Materials Science Letters. 15(24):2171-2172, 1996

"Investigation of grinding effects in binary mixtures from the TiO2 - SnO2 - V2O5 system"

Begincolin S. Lecaer G. Mocellin A. Jurenka C. Zandona M. - J. Solid State Chemistry. 127(1):98-108, 1996

Solid solution formation induced by dry ball-milling under an argon atmosphere has been investigated in binary mixtures from the TiO2-SnO2-V2O5 system mainly by X-ray diffraction and differential scanning calorimetry, Grinding experiments followed by heat treatments result in substantial solid solubilities in all three binaries, Under our experimental conditions, the solubility after grinding depends solely on the nature of grinding media particularly in V2O5 based systems, when reduction reactions take place between grinding tools and oxide particles. The results demonstrate that the interaction between TiO2 or SnO2 and V2O5 follows from the fact that SnO2 and TiO2 are first slightly reduced during milling and subsequently reoxidized by V2O5. These interactions promote solid solution formation and the development of a reduced disordered vanadium oxide layer at the surface of SnO, grains. The final state of powders, where the interface density between both oxides is very high, could be of interest in catalysis.

"Quantitative sonochemistry"

Reisse J. Caulier T. Deckerkheer C. Fabre O. Vandercammen J. Delplancke JL. Winand R. - Ultrason.

Sonochemistry. 3(3):S 147-S 151, 1996
The measurement of a sonochemical effect requires a comparison of the reaction rates with and without ultrasound when all other parameters remain constant. Various artefacts make this last condition difficult to fulfil. Some of them are discussed in the case of the Diels-Alder reaction. To evidence a frequency effect in sonochemistry, the reaction rates have to be compared by changing the frequency alone and maintaining all the other parameters constant. The major difficulty in fulfilling this condition led us to use a relative method where a couple of reactions were studied at 20 kHz and 1.7 MHz. The third topic concerns the effect of ultrasound on various electrochemical reactions for the purpose of producing nano- to microparticles of metals, oxides and semiconductors.

"Electrical behaviour of attritor processed Al/PMMA composites"
Singh V. Tiwari AN. Kulkarni AR. - Materials Science & Engineering B - 41(3):310-313, 1996
Al/PMMA composites containing different volume fractions of aluminium were prepared by attritor milling followed by hot pressing and their thermal expansion and electrical behaviour were studied. Room temperature resistivity oi these composites dropped by several orders of magnitude when the volume percent of aluminium ranged between 20 and 40 vol.%. Microstructural examination revealed the presence of continuous chains of aluminium in highly conducting composites. The dielectric constant asci dissipation factor of the composites increased with increase in aluminium content. This has been attributed to the interfacial polarization associated with aluminium particles.

"Solid solubility and transformation in mechanically alloyed Ti - Mg powders"
Hida M. Asai K. Takemoto Y. Sakakibara A. - Materials Transactions Jim. 37(11):1679-1683, 1996
Mechanical alloying (MA) was examined by ball-milling the powder mixtures of various starling compositions, Ti-10, 20, 30, 40, 50, 60, 70, 80, 95 at%Mg plus 2 mass%NaCl powder. NaCl powder was an excellent anti-sticking agent for MA. The process of MA was mainly followed by X-ray diffraction and TEM methods. Super-saturated quasi-solid solution was formed for the samples up to 60 at%Mg. A single hcp structure was newly created up to 30 at%Mg samples, and over this composition range hcp peaks were present in addition to the diffraction pattern which will lead to bcc like nano-crystallite. In the Mg-rich side, Ti-70 similar to 95 at%MA samples showed the coexistence of hcp phases, Ti-based and Mg-based dilute solid solution. The solubility was estimated from the change of lattice constant. But it was somewhat difficult to detect the alloying effect of MA on Mg and Ti in Ti-95 at%Mg sample. MA samples, Ti-20 and -50 at%Mg, showed the starting of phase separation after one hour annealing at 673 and 473 K, respectively.

"Effect of milling atmosphere on the preparation of Sm2Fe17Nx powder by mechanical grinding"
Ito M. Majima K. Katsuyama S. Nagai H. - Materials Transactions Jim. 37(11):1704-1709, 1996
The preparation of Sm2Fe17Nx permanent magnet powders has been tried by mechanical grinding (MG) for a short time without crystallization heat treatment which was usually needed to obtain a Th2Zn17 structure in the conventional method by MG or MA. MG was carried out in three kinds of atmospheres; i.e., Ar, N-2 and NH3 gas. Untreated and hydrogenated Sm2.5Fe17 alloy powders were used as the starting materials. The Y-Fe precipitation was detected in the powder after MG for over 14.4 ks. The grain size of powder obtained by MG for 10.8 ks was around 10 nm which was much finer than the single magnetic domain size of about 300 nm. The existence of hydrogen promoted not only the nitriding of powders but the oxidation. The hydrogenated powder after MG in NH3 and nitriding had a high oxygen content of about 13000 ppm. This oxidation prevented the absorption of nitrogen during the nitriding process. The untreated powders after MG in NH3, however, were nitrided efficiently up to 38000 ppm at a low temperature of 623 K, and the oxygen content was about 7000 ppm. The maximum coercivity of 0.93 MA . m(-1) (11.7 kOe) was obtained with the untreated powder after MG in NH3 and nitriding at 623 K for 10.8 ks in N-2.

"Crystalline Mg2Ni obtained by mechanical alloying"
Chen J. Dou SX. Liu HK. Journal of Alloys & Compounds. 244(1-2):184-189, 1996
A crystalline Mg2Ni alloy has been prepared by mechanical alloying. Details of the ball milling conditions, such as milling time and milling speed of the equipment, have been investigated. Structure characterization, and the determination of particle distribution and surface properties of the alloyed powders were performed by using X-ray diffraction analysis, the Malvern particle analyser and scanning electron microscopy. It has been found that the crystalline alloy obtained by mechanical alloying has excellent surface properties compared with that prepared by a normal metallurgical method.

"Synthesis and formation mechanisms of molybdenum silicides by mechanical alloying"
Yen BK. Aizawa T. Kihara J. - Mater. Sci. Eng. A, 220(1-2):8-14, 1996
The synthesis and formation of MoSi2, Mo5Si3, and Mo3Si compounds by the mechanical alloying of Mo-Si powder mixtures has been investigated. Ball-milling experiments were conducted for the composition range of 10-80 at.% Si. The formation of molybdenum silicides, especially MoSi2, during mechanical alloying and the relevant reaction rates markedly depended on the powder composition. The spontaneous formation of alpha MoSi2 during mechanical alloying at 67 at.% Si (MoSi2 stoichiometry) proceeded by a mechanically-induced self-propagating reaction (MSR), the mechanism of which is analogous to that of the self-propagating high-temperature synthesis (SHS). At the compositions of 54 and 80 at.% Si, however, the formation of MoSi, proceeded by the gradual formation of both the alpha and beta phases instead of the MSR mode. The formation of Mo5Si3 during mechanical alloying was characterized by a slow reaction rate as the reactants and product coexisted over a long period. The milling of Mo-rich powder mixtures up to 150 h did not lead to the direct formation of Mo3Si. The Mo3Si phase appeared only after brief annealing at temperatures of 800 degrees C and above.

"Nitrogen addtion to iron powder by mechanical alloying"
Rawers JC. Govier D. Doan R. - Mat. Sci. & Eng. A - 220(1-2):162-167, 1996
Nitrogen was alloyed into iron (a) by mechanical processing in a nitrogen gas environment, and (b) by mechanically alloying with iron-nitride powders to characterize resulting nano-structure and nitrogen distribution. Although the infused nitrogen concentration was significantly greater than the thermodynamic equilibrium solubility of iron, no nitrides formed, even for nitrogen concentrations as high as 4.1 wt.% However, a bct-Fe phase did form. Lattice

expansion calculations indicate that the sum of the interstitial bcc-Fe and bct-Fe nitrogen concentrations was significantly less than the total measured nitrogen concentration. A considerable portion of the mechanically infused nitrogen was determined to be associated with nanograin boundaries.

"Effect of mechanical grinding on the lithium intercalation process in graphites and soft carbons"

Disma F. Aymard L. Dupont L. Tarascon JM. - J. Electrochemical Soc.. 143(12):3959-3972, 1996

The effects of mechanical grinding on morphology and electrochemical performance of graphite and soft carbon powders with respect to lithium insertion were studied. The morphology of the milled graphitic powders was found to depend strongly upon the nature of the interactions (e.g., impact or shear) generated by the two kinds of mixer mills used. For the same milling time, crystallite size was smallest and the density of defects highest for graphitic powders that were ball-milled using impact interactions. The specific surface area of the milled samples does not increase indefinitely with increased milling time, but there is a critical milling time (m(c)) beyond which the specific surface area goes through a maximum (graphite) or levels off for cokes. By controlling milling conditions, graphite and soft carbon powders with well-defined morphology, d-spacings, surface area, and crystallite size can be made. The reversible (reversible amount of inserted Li) vs. irreversible capacity (irreversible Lithium loss between the first discharge and charge) was measured for various C/Li cells using various tailor-made graphite and soft carbon powders. A direct correlation between the irreversible capacity of the milled samples and their specific surface area was observed, consistent with catalytically induced reduction of the electrolyte. For milling times greater than m(c), the irreversible capacity remains constant or even decreases while the reversible capacity still increases. With mechanical grinding, both graphite and coke samples having irreversible capacity of 328 mAh/g for a reversible capacity of 708 mAh/g (similar to Li2C6) were obtained.

"Nd - Fe - (C, B) permanent magnets made by mechanical alloying and subsequent annealing"

Sui YC.Zhang ZD.Xiao QF.Liu W.Zhao XG. Zhao T. Chuang YC. - J.Phys.-Cond. Matter. 8(50):11231-11242, 1996

The structure, phase transformation and magnetic properties of mechanically alloyed (MA) alloys Nd16Fe76-mCm (7 less than or equal to m less than or equal to 11) and Nd16Fe84-mCm-yBy (0 less than or equal to y less than or equal to m, m = 7, 8, 9) have been studied systematically. The Nd2Fe14C compound can be formed within a wide window for the range of composition and annealing temperature. More carbon than the stoichiometric content for Nd2Fe14C is necessary to stabilize the tetragonal structure. Substitution of boron for carbon can accelerate the phase transformation from Nd2Fe17Cx to Nd2Fe14(C, B) and increase the magnetic properties drastically. There is an fee structure Nd-rich phase in the mechanically alloyed Nd-FeC alloy. The lattice constant of the Nd-rich phase decreases as the boron content increases. Nd2Fe14(C, B) compounds formed in the alloys with same boron content and different carbon contents have different Curie temperatures. The Curie temperatures also vary with the variation of annealing temperature. The best magnetic properties achieved in Nd16Fe76B5C3 alloy are H-i(c) = 1480 kA m(-1), M(r) = 0.71 T and(BH)(max) = 91.5 kJ m(-3).

"A parametric study of the mechanically activated carbothermic reduction of ilmenite"

Welham NJ. - Minerals Engineering. 9(12):1189-1200, 1996

The Becher process has been the commercial way to upgrade ilmenite for over twenty years and the basic process has changed surprisingly little. It has recently been shown that ball milling ilmenite and coal together prior to thermal processing leads to a decrease in both temperature and time of reaction compared with powders milled separately then mixed. In this paper, the effect of milling time and annealing conditions on the extent of reduction and leachability of products are examined to give an overview of potential improvements in the process. Reduction of ilmenite to rutile can be attained within an hour of annealing at temperatures below that used industrially. Leaching indicates that it may be possible to produce a higher grade of TiO2 than the current processing technique. However, over-reduction leads to the formation of phases which cause the fraction of titanium leached to increase and the iron solubility to decrease.

"Fullerite additions as a phonon scattering mechanisms in P type Si 20 % Ge"

Cook BA. Harringa JL. Loughin S. - Materials Science & Engineering B - 41(2):280-288, 1996

In a series of experiments to evaluate the possibility of reducing the lattice thermal conductivity in silicon-germanium alloys through the formation of an inert, intragranular nanophase, a number of p-type Si-20 at.% Ge alloys, nominally doped with 0.5 at.% boron, were prepared with varying amounts of fullerite, a mixture of 90% C-60 + 10% C-70. The fullerite, in the form of a fine black powder with a particle size of 0.7 nm, was shown by X-ray diffraction (XRD) to be nearly amorphous. The alloys were synthesized by mechanical alloying (MA) and the fullerite was added during various stages of preparation. Following consolidation by hot pressing, the compacts were found to be fully dense and homogeneous. Slices of each compact were characterized by the Hall effect at room temperature and also by electrical resistivity, the Seebeck coefficient, and thermal diffusivity measurements to 1000 degrees C. Addition of 0.2-0.8 wt.% fullerite by a simple mechanical blending operation resulted in an overall decrease in the carrier concentration of up to 35% in alloys hot pressed at 1200 degrees C, compared with 3.0 x 10(20) cm(-3) for baseline alloys without fullerite additions. Higher hot-pressing temperatures resulted in bath an increase in the carrier concentration and carrier mobility. A reduction in thermal conductivity of up to 22% compared with standard p-type alloys was observed in samples containing 0.8 wt.% additions. In this study, a maximum integrated average figure of merit, Z, between 300 and 1000 degrees C of 0.65 x 10(-3) degrees C-1 was obtained, corresponding to 0.4 wt.% addition of fullerite, which is 30-35% higher than that of 'standard' p-type Si-Ge alloys doped with 0.25 at.% boron prepared by conventional methods. Larger amounts of fullerite caused a decrease in Z. Observation of selected samples by transmission electron microscopy (TEM) revealed that the fullerite reacted with silicon to form nanophase SiC inclusions.

"Morphological and structural studies of mechanically alloyed Ti44 C 56 powders"

Eleskandarany MS. Konno TJ. Sumiyama K. Suzuki K. Materials Science & Engineering A- 217:265-268, 1996

The mechanical alloying process has been employed successfully for preparing nanocrystalline titanium carbide (TIG) alloy powders. This process was performed in a high-energy ball mill under an argon atmosphere at room temperature. The mechanically reacted powders have been characterized as a function of the milling time by means of

X-ray diffraction, scanning electron microscopy and transmission electron microscopy. A single phase of NaCl-type Ti44C56 alloy powders is formed after very short milling time (20 ks). The end product of the milled powders are uniform in size (less than 0.5 mu m in diameter) and homogeneous in shape (almost spherical). Moreover, the fabricated Ti44C56 alloy powders have extremely fine cell-like structure, being of about of 3 nm. The presence of free Ti and/or C (reactant materials) in the end product could not be detected. These results demonstrate that the mechanical alloying process can provide a powerful tool for the fabrication of TIC alloy powders al room temperature.

"Formation of nanocrystalline Cu(70Ti(20)Ni(10) powders by mechanical alloying"

Kim HG. Park JY. Yamamuro S. Sumiyama K. Suzuki K. Materials Science & Engineering A 217:269-272, 1996

The formation and thermal stability of nanocrystalline Cu70Ti20Ni10 powders by mechanical alloying has been studied by X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and differential scanning calorimetry (DSC). After milling, the material has a disordered fee structure, with nanocrystalline grain sizes around 4.7 nm determined from the fee (111) diffraction peak. The DSC signal monotonically decreases with isothermal transformation owing to grain growth. The crystal grains do not grow by heating until the intermetallic TiCu3 and NiTi phases nucleate at 450 degrees C.

"Reaction of nanocrystalline Cu - Sn alloy with Ga - In - Sn eutectic"

Ivanov E. Patton V. Grigorieva TF. Materials Science & Engineering 217:277-280, 1996

An extended solid solution of Cu-20%wtSn with a lattice parameter of 0.3715 nm and a crystalline grain sizes D ranging from 30-7 nm was obtained by mechanical alloying. The reaction rate for the formation of CuGa2 due to the interaction of Cu-20%wtSn with a Ga-In-Sn liquid at room temperature increased significantly when D decreased to 7 nm. A mechanical alloying process was used to control the reactivity of the nanocrystalline solid solutions, used as components in the low temperature solder pastes.

"Magnetoresistance and magnetism in Fe - Cu alloys produced by electrodeposition and mechanical alloying methods"

Ueda Y. Ikeda S. Mori Y. Zaman H. Materials Science & Engineering A- 217:371-375, 1996

We have attempted to produce metastable Fe-Cu alloy by the electrodeposition (ED) and the mechanical alloying (MA) methods. The ED films exhibit a wider composition range for the bce phase in comparison with the results for the MA samples. The composition dependence of the magnetizations show a similar tendency for the ED films and the MA samples. The magnetization curves show hysteresis for the ED films, but the hysteresis was not observed for the MA samples. The value of the maximum MR ratio and the Cu concentration showing the maximum MR are different for the samples prepared by two different methods. The maximum MR ratio is 0.4% at about 50 at.% Cu for the ED films, and is 1.7% at about 70 at.% Cu for the MA samples.

"Magnetoresistance in (CoxFe1-x)(20)Cu80 granular alloys produced by mechanical alloying"

Ikeda S. Houga T. Takakura W. Ueda Y. - Materials Science & Engineering A - 217:376-380, 1996

The supersaturated solid solution (CoxFe1-x)(20)Cu-80 alloys (x=0-1) were prepared by mechanical alloying. The magnetoresistance (MR), magnetic properties and crystal structure of these alloys were investigated. The MR ratio for the (CoxFe1-x)(20)Cu-80 alloys shows a maximum of 6.4% in the Co20Cu80 alloy (x=1), and decreases with decreasing Co concentration. The b.c.c.-Fe and h.c.p.-Co phases disappear after milling for 50 h, and the f.c.c. phase only is observed. The b.c.c. phase is observed for the alloys with a small MR ratio (x less than or equal to 0.8), and is the alloy phase of Fe and Co. The b.c.c. phase is not observed for the alloys with a high MR ratio (x greater than or equal to 0.9).

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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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*(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)