

RESEAU FRANCAIS DE MECANOSYNTHESE

Lettre N°36

Mars 1998

110 (+ 8) Groupes de Recherches (dont 47 (+ 8) à l'étranger)

147 (+ 10) Correspondants

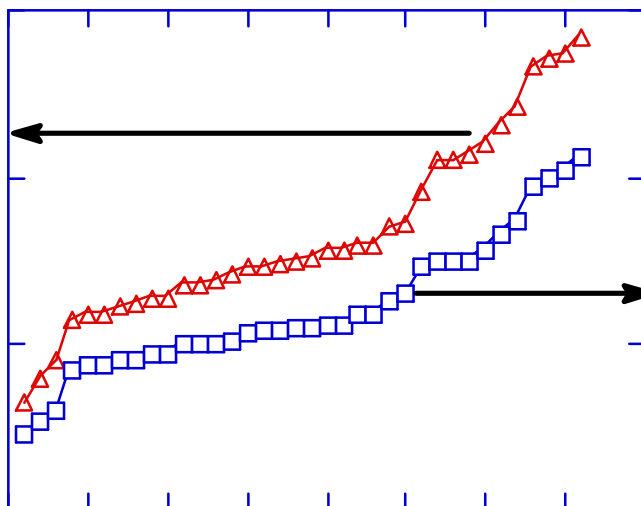
Bureau : E. Gaffet (Président), G. Le Caër (Secrétaire Général), A.R. Yavari (Trésorier)

10 NOUVELLES ADHESIONS

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3 ANS Déjà !!

Le Réseau Français de Mécanosynthèse existe depuis 3 ans



!!!!

Prière de mettre à jour vos cotisations pour 1997 et 1998

!!

JRFM98

Le Mans - 12 & 13 Mai 1998

*Prière de vous inscrire rapidement afin de faciliter le travail
du Comité d'Organisation*

Comité d'organisation : N. Randrianantoandro - J.-M. Grenèche- Y. Labaye- G. Silly - E. Gaffet

Cher(e) Collègue,

Le Laboratoire de Physique de l'Etat Condensé (UPRES A 6087 CNRS), UFR Sciences, Université du Maine, organise cette année **JRFM98**, c'est à dire les 3èmes Journées du Réseau Français de Mécanosynthèse.

Ces journées auront lieu les **12-13 mai 1998 à l'Université du Maine au Mans**

Cette année, le comité d'organisation a choisi comme thème

Propriétés Physiques et Chimiques des matériaux nanostructurés

Vous trouverez ci-joint une fiche de pré-inscription contenant un appel à communication à retourner avant le 28 février 1998. Le **programme** avec une fiche d'inscription définitive sera publié en **mars dans le journal du RFM** et la fiche d'**inscription définitive** sera à **renvoyer avant le 30 avril 1998** au comité d'organisation (Le Mans) des JRFM98. L'inscription à ces Journées est gratuite pour les doctorants accompagnés par un membre du RFM. Une liste des hôtels est donnée ci-contre pour faciliter vos recherches d'hébergement, les hôtels proposés se situent à proximité de l'Université du Maine ou de de la gare TGV.

Liste des Conférenciers Invités (liste provisoire)

Mesures Granulométriques

J. Dodds - Ecole des Mines d'Albi)

Microstructures et Susceptibilité en hyperfréquences de Matériaux Magnétique

M. Bertin - ONERA Chatillon

Utilisation des Techniques de Diffusion des Neutrons et des Rayons X pour étudier les Propriétés Structurales d'Alliages obtenus par Mécanosynthèse

N. Cowlam - Université de Sheffield

Amélioration des Propriétés Mécaniques par nanodispersion d'Al₂O₃ dans l'Intermétallique FeAl

F. Thévenot - Ecole des Mines de St Etienne

Fiche d'inscription

3èmes Journées du Réseau Français de Mécanosynthèse

Le Mans les 12 13 mai 1998

à renvoyer avant le 28 février 1998 à

Nirina Randrianantoandro (Journées RFM98), Laboratoire de Physique de l'Etat Condensé (UPRES A 6087 CNRS), UFR Sciences, Université du Maine, Avenue Olivier Messiaen , 72085 Le Mans Cedex 09

Tél : 02.43.83.35.11, Fax: 02.43.83.35.18,

E-mail : Nirina@lola.univ-lemans.fr

Nom : Prénom :

Adresse :

Téléphone : / Fax :

E-Mail :

Souhaite présenter une communication lors des JRFM98 sous forme de
dune communication orale dun poster

Titre

(joindre un résumé dune page maximum, avec le titre et l'adresse complète
(tél, e-mail), caractère : Times 12 et marges 25 mm)

Frais d'inscription(+ 2 déjeuners et un dîner)

Etudiant(e) en thèse : gratuit autre : 300 FF

Règlement de l'inscription 1998 au RFM100 FF

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Bon de commande Chèque

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Laboratoire de Physique de l'Etat Condensé (UPRES A 6087 CNRS), UFR Sciences, Université du Maine, Avenue

Olivier Messiaen , 72085 Le Mans Cedex 09 - Fax : 02.43.83.35.18

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**ANNONCE DE CONGRES ET / OU ECOLES
CONGRESS AND SCHOOL ANNOUNCEMENTS**

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All the details may be obtained by E-Mail to E. Gaffet

**First ESAFORM Conference on
Material Forming**

Sophia Antipolis - France 17 - 20 Mars 1998
Contact : Noëlle.Billon@CEMEF.CMA.Fr ou <http://esaform.cma.fr>

Oxydes Magnétiques

Journées d'Etudes - Dijon 24 - 26 Mars 1998
Contact : J.C. Niepce - L2RS CNRS UPR5613
Faculté des Sciences de Mirande - 9 Avenue A. Savary - BP 400 - 21011 Dijon Cedex
E-Mail : JOXMAG@U-BOURGOGNE.Fr

**Journées d'Etudes des Equilibres entre Phases
XXIV JEEP**

Nancy - France - 2 et 3 Avril
Contact : JEEP98@lrm.u-nancy.fr

Colloque National de Métallurgie des Poudres

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

**"Recent Advances in Granular Nanostructures and Nanocrystalline Materials
II. - Crystallisation of Amorphous Solids**

Grenoble - 22 - 24 Avril 1998
Contact : A.R. Yavari - E-Mail : Yavari@ltpcm.inpg.fr

JRFM 98

Le Mans - France - 12 - 13 Mai 1998
Comité d'organisation : J.-M. Grenèche, Y. Labaye, N. Randrianantoandro, G. Silly, E. Gaffet

PM2Tec 98

Int. Conf on Powder Metallurgy and Particulate Materials
Las Vegas - USA - 31 Mai 4 Juin 1998
E-Mail : info@mpif.org - WebSite : www.mpif.org

**NOUVEAU
NEW**

**3rd Pacific RIM International Conference on
Advanced Materials and Processing**

Honolulu - Hawaï - 12 - 16 Juin 1998
Contact : M. Imam - Naval Research Lab. Washington - E-Mail Imam@anvil.nrl.navy.mil
et R. DeNale NSWC - E-Mail : DeNale@Oasys.dt.Navy.mil

NANO'98

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

**NOUVEAU
NEW**

Advanced Nanomaterials from Vapor (ANfV'98)

Uppsala - Suède (Satellite Meeting to NANO'98) - 17 Juin 1998
Contact : L. Kiss - Uppsala University / E-Mail : Laszlo.Kiss@Material.uu.se

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

Florence - 14 - 19 Juin 1998
Web Site : <http://www.dinamica.it/cimtec98/>

3rd International Symposium on Metallic Multilayers (MML'98)

Vancouver - 14 - 19 Juin 1998
Contact : E-MAIL : Conference_Service@SFU.CA

E_MRS 1998 - Spring Meeting

Strasbourg - France - 16 - 19 Juin 1998
E-Mail : emrs@phase.c-strasbourg.fr

**NOUVEAU
NEW**

**"High Temperature Nanostructured Materials" Session
High Temperature Materials, Processing and Diagnostics Gordon Conference**

New Hampshire - 19 - 24 Juin 1998
Contact : J. Gole - Georgia Institute of Technology E-Mail : PH294jg@prism.gatech.edu
et N. Jacobson E-Mail Nathan.S.Jacobson@lerc.nasa.gov

41ème Colloque de Métallurgie de l'INSTN

"Ségrégation Interfaciale dans les Solides

Saclay - France - 23 - 25 Juin 1997

Contact : J. Pugnetti - Secrétariat 41 ème Colloque INSTN

CEA Saclay - INSTN - 91191 - Gif sur Yvette Cedex - Fax : +33 - (0) 1 - 69 08 97 77

Journées Francophones des Jeunes Physico-Chimistes

7-9 juillet 1998 - Montpellier

La 4e édition de ces journées multidisciplinaires propose aux étudiants et jeunes chercheurs un lieu de rencontre et d'échange autour de la Physico-Chimie. Trois grands thèmes seront abordés: - Biologie, Santé, Environnement;

- Matière, Matériaux;

- Réactivité, Surfaces, Interfaces.

**NOUVEAU
NEW**

Chaque thème sera introduit par une personnalité scientifique, développé par une série de communications orales et complété par une séance de communications par affiche.

Responsables: Josette Olivier-Fourcade, Jean-Claude Jumas, Pierre-Emmanuel Lippens

Renseignements: Pierre-Emmanuel Lippens

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Internet : <http://ubik.crbm.cnrs-mop.fr/jfjpc98/jfjpc-98.html>

High Temperature Nanostructured Materials

Gordon Conference - New Hampshire - USA - 19 - 24 Juillet 1998

Contact : Nanthan.S.Jacobson@lerc.nasa.gov

EuroMAT'98

Lisbonne - Portugal - 22 - 24 Juillet 1998

E-Mail : lfspm@lemac.ist.utl.pt

Intelligent Processing of Nanostructured Ceramics

Materials Science Summer Institute - New Brunswick - 20 - 29 Août 1998

Contact : L.C. Klein Rutgers University - E-Mail ; Licklein@RCI.Rutgers.Edu

**NOUVEAU
NEW**

6èmes Journées de la Matière Condensée "JMC6" et 17th General Conference of the Condensed Matter Division of the European Physical Society "CMD17"

Grenoble - France - 25 - 29 Août 1998

Org : Société Française de Physique et European Physical Society

Website : <http://www.polycnrs-gre.fr/eps.html>

Fatigue Damage of Structural Materials II

Engineering Foundation Conference

Cape Cod - Massachusetts - USA 31 Août - 4 Septembre 1998

Org. A.K. Vasudevan, J.C. Cammett, T. Nicholas, K. Jata

E-Mail : engfnd@aol.com

ESTAC 7 et EUROSOLID - 5

Baltonfüred - 30 Août - 4 Septembre 1998

Contact : Prof. G. Liptay - Hungarian Chemical Society - Fö u. 68, Budapest - H - 1027 Hongrie

5th International Conference on Nanometer scale Science and Technology (NANO 5)

Birmingham - UK - 31 Aout - 4 Septembre 1998

Site : <http://www.iop.org/IOP/Confs/IVC>

Ninth International Symposium on Small Particles and Inorganic Clusters (SSPIC 9)

Lausanne - Suisse - 1 - 5 Septembre 1998

Website : <http://ipent.epfl.ch/isspic9>

Magnetism of Nanostructured Phases - MNP Conference

EMMA Satellite Meeting

San Sebastian (Espagne) - 4 / 6 Septembre 1998

E-Mail : wupdocal@sp.ehu.es

9th European Symposium on Comminution and Classification

Albi (France) - 8 - 10 Septembre 1998

sous l'égide de l'European Federation of Chemical Engineering

Contacts : J. Dodds - Chairman of the Organizing Committee

First Joint ESF - NSF Symposium on Aerosols for Nanostructured Materials and Device

Edinburgh - Ecosse - 12 Septembre 1998

Contact : h. Fissan@uni-duisburg.de ou dyhpui@tc.umn.edu

First International Conference on Inorganic Materials

Synthesis, Characterisation, Properties and Applications of Inorganic Materials

Versailles - 16 / 19 Septembre 1998 - France

Website : <http://www.elsevier.nl/locate/materials98>

Solid State Chemistry : Novel Syntheses and New Materials

Bordeaux - France - 24/26 Septembre 1998

Website : <http://chemistry.rsc.org/rsc/confs.htm>

**The Reh binder Memorial International Conference
on Colloid Chemistry and Physical Chemical Mechanics**

Moscou (Russie) - 4 - 8 Octobre 1998

Contact : Prof. N.B. Uriev - Institute of Physical Chemistry - Leninsky Prospect 31 -
11795 Moscow - Russie

E-Mail : Reh binder98rehb.chem.msu.su ou <http://www.chem.msu.su>

Powder Metallurgy 98

Granada - Espagne - 18 - 22 Octobre 1998

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

Symposium on Advanced Technologies for Particle Production

AIChE Annual Meeting

15 - 20 November - Miami Beach - Fl - USA

Technical Sessions and ChairPersons

- 1/ Particle Synthesis in Dispersions and Supercritical Fluids - R. Davis/MT Harris/D. Tomasko
 - 2/ Sol - Gel Synthesis of Particles - A McCormick/PN Kumta/T. Okubo
 - 3/ Chemical Kinetics during Particle Formation - J. Floess, K. Higashitani, S. E. Pratsinis
 - 4/ In-Situ Diagnostics during Particle Formation - Ph. W. Morrison, R.M. Carangelo, D.T. Spicer
 - 5/ Agglomerate Particle Dynamics - G. Fotou, SK Friedlander, Takahashi
 - 6/ Computational Fluid Dynamics during Particle Formation and Growth - L. Collins, K. Kontomaris
 - 7/ Aerosol Reactors - A.W. Weimer, M. Kamal Akhtar
 - 8/ Particle Charging - T. Matsoukas
 - 9/ Film synthesis by Particle Technologies - G. Grader, S. Bhandarkar
- 10/ Nanoparticles - M. Senna, T.J Mountziaris, H. Glicksmn**
- 11/ Particulate deposits : Transport mechanisms, microstructure and properties : D. Rosner
- 12/ Posters on Advanced Technologies for Particles Production : G. Beaucage, H. Riemenschneider
- Web Site : www.aiche.org

ISMANAM98

International Symposium on Metastable, Mechanically Alloyed and Nanocrystalline Materials

Wollongong (Sydney) - Australie - 7 - 12 Décembre 1998

International Advisory Committee :

V.V. Boldyrev, R.W. Cahn, S. Enzo, H. Fecht, E. Gaffet, A. Garcia - Escorial, A.L. Greer, E.Y. Gutmanas,
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R. Bormann, G. Cocco, A. Hernando, C.C Koch, M. Magini, R. Schulz, A.R. Yavari

Contact : A. Calka E-Mail : Andrzej_Calka@uow.edu.au et

Website : <http://www.uow.edu.au/conferences/ismanam98>

Satellite Symposium on Mechanochemistry / ISMANAM98

(Mechanochemical Synthesis and Mechanochemistry)

Wollongong - Australie 7 /12 Decembre 1998

International Advisory Committee :

E. Ivanov (Chairman), A. Calka, V. Bodyrev, P. Butyagin,
E. Gaffet, E. Gutman, M. Senna, C. Suryanaryana, R. Schwarz

Website : <http://www.uow.edu.au/conferences/ismanam98>

Nanostructured Hybrid Materials

Symposium TMS Annual Meeting - San Diego CA - USA - 28 Février 4 Mars 1999

Contact : gmchow@anvil.nrl.navy.mil

Nanostructured Materials Symposium at the 5th IUMRS International Conference on Advanced Materials (ICAM'99)

Beijing - Chine - 31 Mai - 5 Juin 1999
Contact : Kelu@imr.ac.cn

10th International Conference on Rapidly Quenched and Metastable Materials (RQ10)

Bangalore - Inde - 23 - 27 Août 1999
Website : <http://www.metalrg.iisc.ernet.in/rqten/>

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Annonces de Soutenance de Thèses
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Effects of the mechanical milling on carbons : negative electrode materials of Li - ion batteries"

F. Salver Disma - Université de Picardie Jules Verne - 4 Février 98

Jury : Aymard L., Beguin F., Coulon M., Furdin G., Lassegues JC, Percheron Guegan A., Rouzard JN, Tarascon JM.

Depuis ces huit dernières années, le graphite est utilisé comme électrode négative dans les accumulateurs au lithium. Il permet d'intercaler rapidement et réversiblement un lithium pour six carbones (correspondant à une capacité de 372 mAh/g) à un potentiel inférieur à 0.5 V vs Li. Récemment, des matériaux carbonés désordonnés ont présenté des capacités électrochimiques de 700 mAh/g et tendent à remplacer les carbones graphitiques. Le mode de préparation de la plupart de ces nouveaux matériaux est limité à la décomposition thermique de diverses substances organiques. Dans ce travail, nous montrons que le broyage mécanique est un autre moyen pour obtenir des matériaux carbonés dits "matériaux sur mesure".

L'essentiel de notre travail a consisté à établir puis à comprendre les facteurs régissant les mécanismes impliqués lors d'une expérience de broyage mécanique sur les poudres de carbone. Pour ce faire, deux modes de broyage mécanique ont été étudiés. Ils génèrent des contraintes différentes (choc et roulement) qui modifient la texture et la structure des matériaux. Les performances électrochimiques des matériaux broyés dépendent essentiellement de leurs caractéristiques physico-chimiques. A l'aide d'une interaction de type choc, des matériaux carbonés désordonnés ont pu être préparés et intercalent réversiblement deux lithiums pour six carbones. Grâce à un choix judicieux de différentes techniques de caractérisations (surface spécifique BET, diffraction des rayons X, DSC, microscopie à balayage, ...) un schéma réactionnel mettant en jeu deux types de surfaces a été proposé en ce concerne les capacités réversibles et irréversibles. Cette approche innovatrice ouvre de nouvelles voies de recherche.

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"Elaboration et Caractérisations de Cermets Alumine - Métal à partir de poudres obtenues par Mécanosynthèse"

J.-L. Guichard - INPL - Nancy - 23 Janvier 1998

Jury : A. Simon, C. Carry, F. Thévenot, G. Le Caër, A. Mocellin

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"Spinelles nanométriques à valence mixte et à fort taux de lacunes cationiques : Transfert électroniques dans un ferrite de molybdène Fe_{2.47}Mo_{0.53}O₄, de la synthèse aux propriétés magnétiques dans le système fer - vanadium Fe_{3-x}V_xO₄ (0²x<2).

V. Nivoix - Université de Bourgogne - 17 Décembre 1997

Jury : M. Lenglet, H. Pascard, G. Bertrand, E. Gaffet, M. Guyot, M. Lallemand, A. Rousset, B. Gillot

=====
"Suivi par Diffraction X en Temps Réel de la Formation par Combustion des intermétalliques des systèmes Al - Ni, Al - Ti, Al - Ni - Ti"

J. F. Javel - Université de Nancy I - 3 Octobre 1997

Jury : J.F. Berar, F. Bernard, M. Bessiere, M. Dirand, J.C. Gachon, P. Galez, J.C. Jorda

=====
"Contribution à l'Etude de la Transformation - Tribologique Superficielle en Fretting"

E. Sauger - Ecole Centrale de Lyon - Génie des Matériaux - 26 Septembre 1997

Jury : L. Mora - Ponsonnet, P. Blanchard, K. Dang Van, C. Esnouf, E. Gaffet, E. Rosset, A.B. Vannes, L. Vincent

=====
"Mechanically induced order-disorder transitions in AlFe"

Thesis by **M. Meyer**, December 1996

presented at Universidad Nacional de La Plata, Argentina - directed by L. Mendoza-Zéllis

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Bibliographie Récente

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

Livres ou "Special Issues"

"Mechanical Behaviour of Nanostructured Materials prepared by Mechanical Alloying"

par D.G. Morris Ed. : Materials Science Foundation Series - Trans Tech Publ. (http://www.ttp.ch/ttp_pps.htm)

"Aluminium -Based Metallic Glasses and Nanocrystalline Alloys (Formation, Thermal Stability and Mechanical Properties) - par U. Köster

Ed. : Materials Science Foundation Series - Trans Tech Publications (http://www.ttp.ch/ttp_pps.htm)

"High Strength Bulk Amorphous Alloys with Good Viscous Flowability" - par A. Inoue

Ed. : Materials Science Foundation Series - Trans Tech Publications (http://www.ttp.ch/ttp_pps.htm)

"Miedema' Semi - Empirical Model for estimating Enthalpies in Alloys - par H. Bakker

Ed. : Materials Science Foundation Series - Trans Tech Publications (http://www.ttp.ch/ttp_pps.htm)

"Chemical MechanoSynthesis of Nanomaterials"

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

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

"Mécanosynthèse"

Les Annales de Chimie - Science des Matériaux - Coordinateur G. Le Caër (1997) -22(6) (1997) 341 - 433

Les Matériaux à Grains Ultrafins produits par Hypercorroyage"

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

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

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

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

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

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

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

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

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

"Mechanochemistry of Solid Surfaces"

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

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

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

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

[62] PHONONS IN NANOCRYSTALLINE Ni3FE

Frase H. Fultz B. Robertson JL. - Physical Review B-Condensed Matter. 57(2):898-905, 1998

inelastic neutron-scattering spectra were measured to obtain the phonon density of states (DOS) of nanocrystalline FeNi₃. The materials were prepared by mechanical alloying, and were also subjected to heat treatments to alter their crystallite sizes and internal strains. In comparison to material with large crystallites, the nanocrystalline material shows two distinct differences in its phonon DOS. The nanocrystalline DOS was more than twice as large at energies below 15 meV. This increase was approximately proportional to the density of grain boundaries in the material. Second, features in the nanocrystalline DOS are broadened substantially. This broadening did not depend in a simple way on the crystallite size of the sample, suggesting that it has a different physical origin than the enhancement in phonon DOS at energies below 15 meV. A damped harmonic oscillator model for the phonons provides a quality factor Q(u), as low as 7 for phonons in the nanocrystalline material. The difference in vibrational entropy of the bulk and nanocrystalline Ni₃Fe was small, owing to competing changes in the nanocrystalline phonon DOS at low and high energies.

[61] MECHANOCHEMICAL SYNTHESIS OF CATALYSTS AND THEIR COMPONENTS

Shirokov YG. - Russian Journal of Applied Chemistry. 70(6):919-934, 1997

The work reviewed was performed in recent years at the Ivanovo State Academy for Chemistry and Technology in the field of mechanochemical synthesis of catalysts and their components. The effect of the mechanochemical activation of solid chemical compounds on their reaction with the liquid phase components is considered. Examples are given of mechanochemical reactions between solid components (oxides, hydroxides, metals) in power-intensive mills with the formation of the active catalyst phase. Ways of mechanochemical synthesis of catalysts, precluding formation of waste waters and gases, are outlined.

[60] CHEMICAL PROCESSES FOLLOWING MECHANICAL PULSE ACTION ON POLYEPOXIDE RESINS DOPED WITH COBALT AND NICKEL ACETATES

Aleksandrov AI. Metlenkova IY. Zelenetskii AN. Prokofev AI. Solodovnikov SP. Bubnov NN. - Russian Chemical Bulletin. 46(9):1546-1548, 1997

The action of elastic wave pulses from a rheological explosion on solid glassy polyepoxides, including those doped with Co²⁺ and Ni²⁺ complexes, leads to the generation of stable dimethylbenzyl radicals. The structures of the polyepoxides and the concentration of Co²⁺ and Ni²⁺ complexes in them have an effect on the mechanochemical yield of these radicals and on the pressure at which the rheological explosion occurs.

[59] MECHANOCHEMICAL APPROACHES TO FULLERENE CHEMISTRY [Review]

Braun T. - Fullerene Science & Technology. 5(7):1291-1311, 1997.

The efficiency of the utilization of mechanical activation, a non-conventional form of energy, is discussed in the framework of the development of the fields of mechanochemistry and fullerene chemistry. It is applied to processes taking place within solid phases as a consequence of the influence of mechanical energy. Various types of mechanochemical syntheses of fullerene compounds are described.

[58] ENHANCEMENT EFFECT OF THE COERCIVE FORCE OF BARIUM FERRITE BY GRINDING AND ANNEALING TREATMENTS

Uchida H. Tokunaga H. Ogura K. - Nippon Kagaku Kaishi. (12):857-861, 1997

Coercive force of barium ferrite is much influenced by a grinding process with a planetary ball mill or an automatic glass mortar. Influential factors on this occasion have been analyzed by measuring the normal magnetic hysteresis loop curve, remainder magnetization curve, and X-ray diffraction pattern of various samples prepared by calcining, milling, and annealing barium ferrite. The coercive force of barium ferrite that was ground with a planetary ball mill increased or decreased, but the pretreatment with the automatic glass mortar led only to the increase of the coercive force. It was disclosed that the enhancement of the coercive force is mainly caused by the orientation of crystal plate and the reduction of the coercive force by the random distortion of the crystal lattice

[57] THE EFFECTS OF HIGH-ENERGY MILLING ON GDFE2

Biondo A. Larica C. Alves KMB. Guimaraes AP. Baggiosaitovitch E. - Journal of Magnetism & Magnetic Materials. 176(2-3):272-278, 1997

With the aim of better understanding, the effects of severe milling on the magnetic and structural properties of crystalline compounds. samples of the intermetallic compound GdFe₂ wire milled up to 276 h under inert atmosphere and characterized by X-ray diffractometry (XRD) and Mossbauer spectroscopy (MS). On milling GdFe₂ a strong segregation of the elements Gd and Fe initially occurred, and the characteristic peaks of the initial compound GdFe₂ in the XRD pattern are absent after 1 h of milling. MS and XRD results also show that the milled samples contain a mixture of three main components: the segregated elements, the initial compound and an amorphous alloy, with relative proportions that vary with milling time. At the longer milling times the process induces a solid-state reaction between these components leading to the formation of an amorphous solid solution of Gd and Fe. In these cases the Mossbauer spectra taken at 300, 84 and 4.2 K were fitted with a combination of a set of peaks typical of metallic alpha-Fe and a continuous distribution of hyperfine magnetic fields that are ascribed to the amorphous Gd-Fe phase.

[56] STUDIES OF LITHIUM INSERTION IN BALLMILLED SUGAR CARBONS

Xing WB. Dunlap RA. Dahn JR. - Journal of the Electrochemical Society. 145(1):62-70, 1998

Hard carbons were prepared by pyrolyzing sugar precursors at 1000 degrees C. The sugar carbons have a microporous structure and large specific capacity (greater than or equal to 550 mAh/g) for lithium insertion in carbon/Li electrochemical test cells. Powders of sugar carbon were then treated by high-impact ballmilling either in argon or air. These carbon samples were characterized by x-ray diffraction, small-angle x-ray scattering, thermogravimetric analysis, chemical analysis, and Brunauer-Emmett-Teller surface area measurements. The structure of the ballmilled powders was different from that of the original sugar carbons. As milling proceeds in argon or in air, the graphene layers initially become more stacked (as indicated by changes in the 002 diffraction peak), the nanoscopic or microscopic pores are rapidly eliminated, and the number of macropores or mesopores increases. Upon further milling, the 002 diffraction peak weakens again, as the carbon structure becomes more disordered. We explain these trends with a qualitative model. Thermogravimetric analysis and chemical composition analysis on the air-milled samples confirm that the materials contain substantial oxygen, suggest that oxygen-containing surface functional groups are formed and show that the amount of the functional groups increases with milling time. Carbons ballmilled in argon atmosphere needed to be slowly exposed to air and kept cool or they burst into flames when brought into contact with air. This implies that the milling created broken carbon-carbon bonds, which are highly reactive, in the material. Studies of ballmilled carbon/Li coin cells showed that ballmilled carbons have large reversible specific capacities of more than 600 mAh/g for lithium insertion. However, the cells demonstrated large hysteresis compared to that of unmilled sugar carbon/Li cells. We propose that the mechanism for quasi-reversible lithium insertion in ballmilled carbons may involve (i) reactions of Li atoms at the edge of small graphene sheets, (ii) intercalation in cases where stacked layers remain, and (iii) reactions with surface functional groups where they exist. It was found that hysteresis in the ballmilled carbons is only weakly dependent on temperature and cycling rate.

[55] PREPARATION OF SM₂FE₁₇N_x POWDERS AND THEIR BONDED MAGNETS WITH HIGH-PERFORMANCE PERMANENT MAGNETIC CHARACTERISTICS

Izumi H. Machida K. Shiomi A. Iguchi M. Noguchi K. Adachi GY. - Chem. Materials. 9(12):2759-2767, 1997

Uniformly ground Sm₂Fe₁₇N_x fine powders with high-performance permanent magnetic property and oxidation resistance characteristics were obtained from the coarse powders by ball milling in an organic solution containing a surfactant and surface modification for them, followed by fabrication of their bonded magnets. Their remanence (B-r) and coercivity (H-cj) values were well-balanced and considerably higher than those for the samples prepared by conventional methods, and a huge maximum energy product (BH)(max) around 330 kJ m⁻³ was consequently attained as a reproducible average. The Sm₂Fe₁₇N_x powder stabilized by surface coating with the zinc metal produced via photodecomposition of diethylzinc (Zn-(C₂H₅)₂) showed the better oxidation resistivity than the uncoated samples, so that the B-r, H-cj, and (BH)(max) values were maintained at high levels even after the heat treatment at 353-423 K for several hours as required to cure epoxy resin bonded magnets. The bonded magnets prepared from the Zn/Sm₂Fe₁₇N_x powders provided the highest (BH)(max) value (similar to 176 kJ m⁻³) among the values reported up to date, even after exposure to air, and their irreversible flux loss was also smaller than that of the uncoated one.

[54] MECHANICALLY ATTRITED SILICON FOR HIGH REFRACTIVE INDEX NANOCOMPOSITES

Papadimitrakopoulos F. Wisniecki P. Bhagwagar DE. - Chemistry of Materials. 9(12):2928-2933, 1997

High-energy milling provides an effective and environmentally conscious method for nanosizing silicon. Colloidal suspensions of nanosized silicon are presently demonstrated and utilized for the fabrication of high refractive index nanocomposites. Si nanoparticles with average sizes of 20-40 nm and size distributions of about 25% have been separated from milled powder via sonication and centrifugation. These nanoparticles were analyzed using transmission electron microscopy, dynamic light scattering, X-ray diffraction and UV-vis/FTIR spectroscopy. Formation of stable colloids is in part attributed to a thin surface-oxide layer. The decrease in the average particle size causes a blue shift in their absorption spectrum, thus increasing transparency in the red part of the visible region. These Si nanoparticles were used to fabricate high refractive index nanocomposites, with refractive indexes up to 3.2, when dispersed in gelatin.

[53] X-RAY DIFFRACTION CHARACTERIZATION OF HEAVILY DEFORMED METALLIC SPECIMENS

Lutterotti L. Gialanella S. - *Acta Materialia*. 46(1):101-110, 1997

Ordered intermetallic compound powders can be heavily deformed and disordered by ball-milling. This highly energetic deformation process introduces several kinds of defects into the powder and changes the aspect of the grains. By a novel analysis of the diffraction spectra we can analyse most of such defects and characterize the correlation between the shape of the powder and its crystallographic orientation. In this way we can also recognize how deformation may occur along specific crystallographic planes. The quantitative evaluation of dislocations, twins, stacking and deformation faultings allows us to correlate the disordering process to the introduction and development of defects under the effect of milling. It is shown how a full pattern fitting of diffraction spectra can be successfully applied to a comprehensive investigation of a material modification process, if appropriate physical models are used to describe the scattering event instead of semiempirical functions.

[52] STRUCTURAL EVOLUTION IN MECHANICALLY MILLED EUTECTOID ZN-AL BASED ALLOY

Zhu YH. Hirata VML. Munoz MS. - *Zeitschrift für Metallkunde*. 88(12):934-937, 1997

Phase transformation and microstructural changes in a furnace-cooled eutectoid Zn-Al based alloy were investigated during mechanical milling. A four-phase transformation and decomposition of a metastable η (FC) phase occurred during milling. The lamellar structure was destroyed into a fine grain structure by decomposition of the η (FC) phase. The mechanism of the structural change was different from that induced by tensile-creep stress.

[51] MECHANICAL PROPERTIES OF FLO-DEFLOCCULATED AND MILLED POWDERS IN THE AL₂O₃/ZrO₂(CeO₂) SYSTEM

Burelli M. Maschio S. Meriani S. - *Journal of Materials Science*. 33(2):441-444, 1998

Samples of alumina, zirconia and their mixtures were prepared using powders produced by two different processes. The first process only involved the flo-deflocculation of diluted suspensions whereas the second required two steps, first flo-deflocculation and then ball milling of the slurries. Materials comprising a matrix containing a relatively small quantity of the dispersed phase (20 vol% and 80 vol% ZrO₂) show better properties when they are produced from flo-deflocculated powders. When samples contain a large quantity of the dispersed phase, milling after flo-deflocculation is beneficial because it provides better homogeneity, which provides greater density and strength although toughness is not significantly improved.

[50] ELECTROCHEMICAL PROPERTIES OF AB(5)-TYPE HYDRIDE-FORMING COMPOUNDS PREPARED BY MECHANICAL ALLOYING

Lenain C. Aymard L. Salverdisma F. Leriche JB. Chabre Y. Tarascon JM. - *Sol. St. Ion.* 104(3-4):237-248, 1997

Two types of intermetallic compounds, LaNi₄M with M=Mn, Co, Cu and Al and polysubstituted alloys Mm(NiAlMnCo)₅ with Mm=Mischmetal were synthesized by mechanical alloying. In most cases, 10 h grinding was sufficient to obtain single phase alloys with crystallites of about 6 nm and particles ranging between 5 and 80 nm in diameter. The polysubstituted MmNi_{3.6}Al_{0.35}Mn_{0.25}Co_{0.8} alloys, once removed from the grinding container, were found to present an electrochemical capacity of 159 mA h/g. Such a capacity was increased to 216.3, 241.7 and 273.5 mA h/g by a post-anneal step under vacuum to temperatures of 673, 873 and 1073 K, respectively. The various electrochemical capacity depends on both surface states and internal strains in the sample so that the heat treatments release these strains and enhance the capacity.

[49] ABSENCE OF EVIDENCE OF DECOMPOSITION OF Fe₂B DURING MECHANICAL GRINDING

Balogh J. Horvath ZE. Pusztai T. Kemeny T. Vincze I. - *Physical Review B-Condensed Matter*. 57(1):29-32, 1998

The appearance of bcc Fe after grinding Fe₂B for a long time has been formerly interpreted as being due to the decomposition of the thermodynamically stable intermetallic compound when the crystallite size is reduced to a few nanometers. The results of our control experiments performed by tungsten-carbide milling tools show that the appearance of bcc Fe has no relation to the grain size but should be connected to contamination when tools made of steel are used to pulverize this material. Mossbauer spectroscopy was applied to search for the appearance of bcc Fe while changes in the grain size were checked by x-ray diffraction and transmission electron microscopy.

[48] EARLY STAGES OF SOLID-STATE AMORPHIZATION REACTION DURING MECHANICAL ALLOYING OF A MULTICOMPONENT Zr-POWDER MIXTURE

Sagel A. Wanderka N. Wunderlich RK. Schubertbischoff P. Fecht HJ. - *Scripta Materialia*. 38(1):163-169, 1997

[47] SYNTHESIS OF WC AND WC-CO CERMETS BY MECHANICAL ALLOYING AND SUBSEQUENT HOT ISOSTATIC PRESSING

Mi S. Courtney TH. - *Scripta Materialia*. 38(1):171-176, 1997

[46] ENHANCED MAGNETIZATION AND CATION DISTRIBUTIONS IN NANOCRYSTALLINE ZnFe₂O₄ - A CONVERSION ELECTRON MOSSBAUER SPECTROSCOPIC INVESTIGATION

Clark TM. Evans BJ. - *IEEE Transactions on Magnetics*. 33(5 Part 2):3745-3747, 1997

The synthesis of nanocrystalline zinc ferrite (ZnFe₂O₄) has been accomplished by a variety of chemical and mechanochemical techniques. An enhanced magnetization, 4-5 times that of bulk zinc ferrite, is observed for the nanoparticles regardless of synthesis method. The hypothesis that the enhanced magnetization is due to partially

inverted nanocrystalline zinc ferrite has been examined by transmission Fe-57 Mossbauer spectroscopy (TMS), conversion x-ray Mossbauer spectroscopy (CXMS) and conversion electron Mossbauer spectroscopy (GEMS). Both TMS and CXMS data indicate differences in local structure between bulk and nanocrystalline samples, however they do not suggest that the samples are partially inverted, GEMS data are similar for both bulk and nanocrystalline samples, indicating similar cation distributions near the surface of nanocrystalline and bulk particles.

[45] CONTACT RESISTANCE INCREASE IN PALLADIUM MATERIAL AS A RESULT OF MECHANOCHEMICAL REACTION

Karasawa K. Chen ZK. Sawa K. - Electronics & Communications in Japan, Part 2: Electronics - 80(2):33-44, 1997
In small-size relays used under low current, mechanical actions of contacts at make-break, such as impact and wipe, can affect contact deterioration more than electric factors such as arcing and Joule heat. In this article, the effects of vertical impact and horizontal wipe were studied in separate experiments in order to clarify the mechanism by which a palladium oxide film is formed on the contact area and the contact resistance is rapidly increased by make-break operations under no load in air. The experiments show that the main factor of contact resistance increase is the formation of an accumulated layer made of insulation powders by wipe action. Based on this fact, models of contact surface condition during operations are suggested to explain the variation of contact resistance. In addition, the effect of passing current through the contacts is also explained by the models.

[44] MECHANOCHEMICAL SYNTHESIS OF NANOCRYSTAL COMPLEX OXIDES

Grigoreva TF. Barinova AP. Ivanov EY. Boldyrev VV. - Doklady Akademii Nauk. 354(4):489-492, 1997

[43] PREPARATION OF NI POWDER BY MECHANOCHEMICAL PROCESS

Baburaj EG. Hubert KT. Froes FHS. - Journal of Alloys & Compounds. 257(1-2):146-149, 1997

A solid state displacement reaction between NiCl₂ and Mg, induced by mechanical milling, has been employed for the production of fine nickel powder. The reaction ignites after a definite incubation period, leading to a sharp temperature rise of the vial. The observed overall temperature rise during milling is due to two effects (i) mechanical working, and (ii) chemical reaction. Ignition has been shown to occur as a result of a critical combination of chemical mixing, lattice strain and milling induced temperature rise. This is related to the incubation time observed after a room temperature hold. The reaction product (Ni+MgCl₂) has been leached with dilute HCl to obtain nickel powder which is in the size range of 10-500 nm.

[42] NONTRADITIONAL PATHWAYS OF EXTRATERRESTRIAL FORMATION OF PREBIOTIC MATTER

Goldanskii VI. - Journal of Physical Chemistry. 101(19):3424-3432, 1997

Mechanisms of solid-phase reactions were investigated experimentally and theoretically during recent decades that can have direct connections to the formation of organic substances in space and their delivery to Earth and to the problems of prebiotic evolution. Among such mechanisms are molecular tunneling (hypothesis of the cold prehistory of life), polycondensation of solid monomers by shock waves (problem of the delivery of organic substances to Earth by meteorites), thermal and thermal-wave explosions and oscillations of temperature and radical concentrations in small cold particles under radiation exposure, arid mechanochemical explosions and autowave propagation of chemical reactions due to the positive feedback between the fragile destruction of solids and reactions at freshly formed surfaces.

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

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

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

[40] MECHANOCHEMICAL PREPARATION OF DRUG-CARRIER SOLID DISPERSIONS

Shakhtshneider TP. Vasilchenko MA. Politov AA. Boldyrev VV. - J. Thermal Analysis. 48(3):491-501, 1997

The method of mechanical activation was used to obtain solid-state dispersions of some drugs in polyvinylpyrrolidone, polyethylene glycol and talc as carriers. Solid dispersions obtained by mechanical activation were found to have higher apparent solubilities and dissolution rates than mechanically activated drugs or their physical or eutectic mixtures with carriers used. It was shown by IR-spectroscopy and fluorescence measurements that mechanical treatment gave rise to an interaction between components which was apparently responsible for the solubilization effects observed.

[39] REGULARITIES OF CATALYTIC OXIDATION OF CARBON BY SULPHUR DIOXIDE

Babenko VS. Buyanov RA. - Kinetics & Catalysis. 38(1):44-47, 1997

Basic features of catalytic oxidation of dissimilar carbonaceous materials of various origin by sulphur dioxide were investigated. The compounds of alkali, alkaline-earth, and transition metals were found to be effective catalysts for carbon oxidation in the SO₂ medium; they also lower the temperature of the reaction onset by similar to 200 K. The rate of carbon oxidation depends on the carbon material type. The mechanochemical activation of carbonaceous materials was shown to increase the rate of their oxidation with sulphur dioxide. At 823-973 K, the rate of carbon catalytic oxidation nonadditively increases when small amounts of oxygen or water vapor were added to SO₂.

[38] SYNTHESIS OF CALCIUM SILICATE HYDRATE WITH CA/SI=2 BY MECHANOCHEMICAL TREATMENT

Sasaki K. Masuda T. Ishida H. Mitsuda T. - Journal of the American Ceramic Society. 80(2):472-476, 1997

An amorphous, C-S-H-like phase with Ca/Si = 2 was synthesized from amorphous precipitated silica, calcium oxide,

and water by mechanochemical treatment using a vibration mill at room temperature. The product was studied by XRD, Si-29 MASNMR, TEM, analytical TEM, and TGA-DTA. After 14 h of treatment, the starting materials react to form an amorphous phase (C-S-H-like phase). The XRD pattern of C-S-H-like phase resembles the C-S-EI formed hydrothermally or by cement hydration except it has no reflection at 0.182 nm. The Si-29 NMR results revealed a silicate anion structure of C-S-H-like phase consisting mainly of a mixture of a monomer and a dimer. On heating, the C-S-H-like phase decomposed into beta-dicalcium silicate below 1000 degrees C.

[37] CHEMICAL ANALYSES OF MECHANOCHEMICAL REACTION PRODUCTS OF ALPHA-Si3N4 IN ETHANOL AND OTHER LOWER ALCOHOLS

Hibi Y. Enomoto Y.- Journal of Materials Science Letters. 16(4):316-319, 1997

[36] SOLID-STATE TRANSFORMATION MECHANISMS OF ASSOCIATED MINERALS TO ALUMINOSILICATES

Mendelovici E.- Journal of Thermal Analysis. 48(1):141-144, 1997

Thermochemical and mechanochemical transformation mechanisms of associated minerals in clay (quartz, calcite, Al and Fe oxyhydroxides) are comparatively examined. The phase transformation sequence resulting from heating and, on the other hand from dry grinding of each of the associated minerals, depends on the structure and physicochemical properties of the starting materials. Grinding treatments may cause local heating and local pressure effects in some selective metal oxides and in calcite, respectively, accounting for their structural conversions.

[35] MECHANOCHEMICAL TRANSFORMATIONS OF AG, HG AND PB CYANOMETALLATES IN KBR PRESSED DISKS

Reguera E. Fernandezbertran J. - Spectroscopy Letters. 30(1):89-98, 1997

The mechanochemical transformations of Ag, Hg and Pb cyano complexes (ferro- and ferricyanides, cobaltcyanides, nitroprussides, hexacyanoplatinates and tetracyanonickellates) during milling and pressing with KBr have been studied using IR, XRD and Mossbauer techniques as sensors. Exchange of K⁺ and the heavy metal cations is observed in all cases.

[34] MECHANOCHEMISTRY - THE ACTIVATION METHOD OF VPO CATALYSTS FOR N-BUTANE PARTIAL OXIDATION

Haber J. Zazhigalov VA. Stoch J. Bogutskaya LV. Batcherikova IV. - Catalysis Today. 33(1-3):39-47, 1997

The paper describes the influence of the introduction of alkali and alkaline-earth metals into the VPO precursor and the effect of bismuth additive introduced into the VPO precursor by the mechanochemical treatment. XRD, XPS, DTA, TPD of NH, and determination of catalytic activity and selectivity in n-butane oxidation have been carried out. The preparations studied after mechanochemical treatment in a planetary mill were: (a) VPO, composed of VOHPO4 0.5H(2)O, (b) VPBiO, containing the bismuth additive introduced during synthesis, (c) VPO/Bi2O3 and VPO/BiPO4, mechanical mixtures of the VPO precursor and Bi2O3 or BiPO4, respectively. Introduction of alkali and alkaline-earth metals additives increases the basicity of surface oxygen in vanadium pyrophosphate, which entails increase in the rate of n-butane oxidation. The mechanochemical treatment of the preparations changed their morphology, favouring the formation of the vanadyl (001) plane in the phosphate precursor. The XRD peaks of bismuth phosphate appeared instead of those of bismuth oxide and the +0.8 eV chemical shift in the XPS Bi4f band was observed. The change in the chemical state of VPO/Bi2O3 are an evidence of the occurrence of chemical reaction. Mechanochemical treatment of VPO/BiPO4 and VPO/Bi2O3 catalysts increases their activity in n-butane oxidation and the selectivity to maleic anhydride. Thus, mechanochemistry may be a promising method for introduction of promoting additives into the basic VPO composition.

[33] STUDY OF STRUCTURAL TRANSFORMATIONS IN POTASSIUM-EXCHANGED ZEOLITE A INDUCED BY THERMAL AND MECHANOCHEMICAL TREATMENTS

Kosanovic C. Subotic B. Smit I. Cizmek A. Stubicar M. Tonejc A. - J. Materials Science. 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.

[32] MECHANOCHEMICAL EFFECTS OF GRINDING ON THE YBa2Cu3Ox SUPERCONDUCTOR

Wu XS. Chen WM. Jin X. Jiang SS. - Physica C. 273(1-2):99-106, 1996

The mechanical grinding effects on the powders of YBa2Cu3Ox superconductor are examined. The grain size, the oxygen content of the powders decrease, and the content of the amorphous phase, the crystal strain increase, with the increasing of grinding time. Except that for amorphous phase, the crystallized phase can be refined by the orthorhombic and tetragonal phases. The refined results show that the diffraction peaks broaden after continuous grinding. This broadening can be explained from the crystal strain and the small grain size due to prolonged grinding. The lattice constants of the refined phases vary with the grinding time.

[31] SYNTHESIS OF PYROCHLORE-FREE 0.9PB(MG1/3NB2/3)O-3-0.1PBtIO(3) CERAMICS VIA A SOFT MECHANOCHEMICAL ROUTE

Baek JG. Isobe T. Senna M. - Journal of the American Ceramic Society. 80(4):973-981, 1997

Single-phase perovskite 0.9Pb(Mg1/3Nb2/3)O-3-0.1PbTiO(3) (0.9PMN-0.1 PT) from a stoichiometric mixture of starting materials was synthesized by applying a mechanochemical technique to the stage of a precursor. A stoichiometric mixture of PbO, TiO2, Mg(OH)(2), and Nb2O5 was milled for 60 min and heated at temperatures as low as 850 degrees C for 4 h to obtain a single phase. The maximum dielectric constant of the samples from the milled mixture increased as the sintering temperature increased, with the remarkable grain growth, and attained 24600 at 1200 degrees C. In contrast, poor densification and coexistence of the pyrochlore phase were observed on the

samples from the nonmilled mixture. Further observation suggested that the pyrochlore phase concentrated near the surface during sintering and then migrated into the PbZrO₃ packing powder, leading to a pyrochlore-free phase at 1250 degrees C. The dielectric constant of the latter ceramics was explained by the series mixing rule for the dielectric constant of a biphasic solid.

[30] MECHANOCHEMICAL DEGRADATION OF EVA-EPDM BLENDS

Mishra S. Naik JB. - Polymer-Plastics Technology & Engineering. 36(2):231-240, 1997

An internal mixing mastication of EVA-EPDM blends was studied in the temperature range 80 degrees-230 degrees C. The extent of degradation was determined by torque measurement. The breakdown of EVA and EPDM in the mixer is minimum in the temperature range 110 degrees-120 degrees C and 150 degrees-160 degrees C, respectively.

[29] MECHANOCHEMICAL SYNTHESIS OF ULTRAFINE ZrO₂ POWDER

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

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

[28] REGULARITIES OF CATALYTIC OXIDATION OF CARBON BY SULPHUR DIOXIDE

Babenko VS. Buyanov RA. - Kinetics & Catalysis. 38(1):44-47, 1997

asic features of catalytic oxidation of dissimilar carbonaceous materials of various origin by sulphur dioxide were investigated. The compounds of alkali, alkaline-earth, and transition metals were found to be effective catalysts for carbon oxidation in the SO₂ medium; they also lower the temperature of the reaction onset by similar to 200 K. The rate of carbon oxidation depends on the carbon material type. The mechanochemical activation of carbonaceous materials was shown to increase the rate of their oxidation with sulphur dioxide. At 823-973 K, the rate of carbon catalytic oxidation nonadditively increases when small amounts of oxygen or water vapor were added to SO₂.

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[26] CHEMICAL ANALYSES OF MECHANOCHEMICAL REACTION PRODUCTS OF ALPHA-SI₃N₄ IN ETHANOL AND OTHER LOWER ALCOHOLS

Hibi Y. Enomoto Y. - Journal of Materials Science Letters. 16(4):316-319, 1997

[25] 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 on 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.

[24] STUDY OF STRUCTURAL TRANSFORMATIONS IN POTASSIUM-EXCHANGED ZEOLITE A INDUCED BY THERMAL AND MECHANOCHEMICAL TREATMENTS

Kosanovic C. Subotic B. Smit I. Cizmek A. Stubicar M. Tonejc A. - J. Materials Science. 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.

[23] EFFECT OF MECHANOCHEMICAL DEGRADATION ON GELATION AND MECHANICAL PROPERTIES OF PVC

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

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

undegraded PVC. The mechanical properties of PVC are improved quite a lot after blending it with a small amount of mechanochemically degraded PVC.

[22] EFFECT OF MODIFICATION OF ACID SITES LOCATED ON THE EXTERNAL SURFACE OF A GALLIUM-SILICATE CRYSTALLINE CATALYST ON REDUCING COKE DEPOSIT IN PARAFFIN AROMATIZATION

Inui T. Yamada T. Matsuoka A. Pu SB. Industrial & Engineering Chemistry Research. 36(11):4827-4831, 1997
Acid sites located on the external surface of a MFI-type gallium-silicate crystals were selectively neutralized by a mechanochemical method using cerium oxide supported on spherical silica particles in micrometer size. The CeO₂-modified catalyst exhibited longer catalyst life and higher yield of aromatics than a non-modified catalyst in an octane conversion reaction, and the coke deposited on a modified catalyst was much less than that on a non-modified one. This is attributed to that decrease in acid sites on the external surface of Ga-silicate crystals reduce the deep condensation of aromatic rings. The effect was further enhanced by the combination with Pt modification. The multifunctions of this catalyst are due to the aromatization ability of Ga species incorporated into the silicate framework, neutralization of external acid sites by CeO₂ species, and dehydrogenation-cyclization of Pt particles on the external surface of crystals, respectively

[21] CO AND CO₂ HYDROGENATION UNDER MECHANOCHEMICAL TREATMENT

Morozova OS. Streletskii AN. Berestetskaya IV. Borunova AB. - Catalysis Today. 38(1):107-113, 1997
The effect of mechanochemical treatment on the hydrogenation of CO and CO₂ was studied using a flow milling vial as a catalytic reactor. Traditional catalysts for the methane formation (Ni, Zr, Zr-Ni-containing samples, Zr hydride and Zr-Ni hydrides of various genesis), as well as inactive NiO and ZrO₂, were examined. The following features of the mechanically driven CO hydrogenation were observed: (i) Zr and Zr-Ni hydrides demonstrated the highest activity in the formation of CH₄; (ii) the hydrogen present in the reaction mixture suppressed the formation of CH₄; (iii) Ni was found to be ineffective in the formation of CH₄ but active in the reaction of CO disproportionation; (iv) on the oxide samples, a small amount of CH₄ was formed owing to the mechanical activation. Deep structural transformations of the metal and hydride samples under milling in the reaction mixture were found to be responsible for the changes in their catalytic activity. Only zirconium hydride exhibited catalytic activity for CO₂ hydrogenation. In this case, no phase transformation was observed.

[20] HEMATITE POWDERS SYNTHESIZED BY MECHANOCHEMICAL PROCESSING

Ding J. Tsuzuki T. McCormick PG. - Nanostructured Materials. 8(6):739-747, 1997
The mechanochemical synthesis of ultrafine hematite powders via solid-state reactions involving FeCl₃ and CaO or Ca(OH)₂, respectively, has been investigated. Mechanical milling of 2FeCl₃+3CaO led to a nanocrystalline mixture of these two compounds. Fe₂O₃ formed only after heat treatment at 150 degrees C or above. A dilution of CaCl₂ into the starting mixture caused a significant decrease in particle size, resulting in 10-30 nm mono-disperse particles of Fe₂O₃. Nanocrystalline akaganeite, FeOOH, together with calcium chloride was formed by milling 2FeCl₃+3Ca(OH)₂. Conversion of FeOOH to Fe₂O₃ by dehydration occurred during subsequent heat treatment. Ultrafine Fe₂O₃ particles of 20-50 nm were obtained after removal of the CaCl₂ by-product phase.

[19] HYDROTHERMAL REACTIONS UNDER MECHANOCHEMICAL ACTIVATION CONDITIONS

Boldyrev VV. Khabibullin AK. Kosova NV. Avvakumov EG. - Inorganic Materials. 33(11):1144-1147, 1997
The optimal moisture content of solid mixtures is estimated for setting up hydrothermal conditions in mechanochemical activators. The data on the mechanochemical reaction between calcium hydroxide and hydrated silica support the feasibility of hydrothermal synthesis. It is shown that the reaction gives poorly crystalline tobermorite-like calcium silicate.

[18] MECHANOCHEMICAL SYNTHESIS OF HYDRATED CALCIUM SILICATES BY ROOM TEMPERATURE GRINDING

Saito F. Mi GM. Hanada M. - Solid State Ionics. 101(Part 1):37-43, 1997
Room temperature grinding of a mixture of calcium hydroxide and silicagel with different water contents was conducted in a planetary ball mill. A willite was synthesized mechanochemically by controlling the water weight rate at about 0.23-0.30. Two-hours grinding of the mixture containing with a water weight rate of more than 0.38 enabled the synthesis of other different types of calcium silicate hydrates. On the other hand, tobermorite was synthesized by three-hours grinding of the mixture having a Ca/Si molar ratio of about one and a water rate at about 0.8, while calcium silicate hydrates-(B) were mechanochemically synthesized by grinding during about 1.5 h. The water amount in the mixture plays a significant role to achieve the mechanochemical synthesis of these compounds.

[17] CHARACTERIZATION AND REACTIVITY OF ZINC SULPHIDE PREPARED BY MECHANOCHEMICAL SYNTHESIS

Balaz P. Balintova M. Bastl Z. Briancin J. Sepelak V. - Solid State Ionics. 101(Part 1):45-51, 1997
This paper describes the mechano-synthesis of ZnS by high intensive milling of elementary sulphur and zinc in a planetary mill. Using complex physical and chemical methods (XRD, surface area measurement, IR and XPS) the probable occurrence of sphalerite was proved. The sphalerite was contaminated by iron (up to 0.14% of Fe) and the product of surface oxidation (hydrated ZnSO₄). The reaction of mechano-synthesised ZnS with H₂SO₄ (170 g l⁻¹) at 40-80 degrees C has a higher temperature sensitivity. A shift of the rate-controlling step to surface chemical control was observed, too.

[16] HYDROTHERMAL REACTIONS UNDER MECHANOCHEMICAL TREATING

Kosova NV. Khabibullin AK. Boldyrev VV. - Solid State Ionics. 101(Part 1):53-58, 1997
The mechanochemical treating of solids containing some amount of free or chemically bound water in high-energetic activators enable the hydrothermal processes (as in autoclaves). Estimations of the optimal value of the water content were carried out. The data on the investigation of the mechanochemical reaction between calcium hydroxide and hydrated silica are presented as the experimental confirmation of the hydrothermal regime bring realized.

[15] ENHANCED DENSIFICATION OF INDIUM-TIN OXIDE CERAMICS FOR SPUTTER TARGET THROUGH

WET MECHANOCHEMICAL PROCESSING

Iwasa K. Isobe T. Senna M. - Solid State Ionics. 101(Part 1):71-78, 1997

Changes in the sinterability of indium-tin oxide (ITO) due to mechanical stressing of In₂O₃ 95/SnO₂ 5 mixture have been studied. Starting composite powders were prepared by in situ sol preparation method using an agitation beads mill. The enhanced sinterability due to mechanical treatment is explained by the dissolution-precipitation to form a layer of hydrogels, resulting in the decrease in the vaporization rate of SnO₂ above 1200 degrees C.

[14] MECHANOCHEMICALLY ASSISTED PREPARATION PROCESS OF BARIUM HEXAFERRITE POWDERS

Abe O. Narita M. - Solid State Ionics. 101(Part 1):103-109, 1997

A new process of solid state synthesis of BaFe₁₂O₁₉ with the assistance of mechanochemical hydration of Fe₂O₃ under planetary mill treatment is proposed. The increased surface polarity of hydrated Fe₂O₃, induced coagglomeration with Ba(OH)₂ · H₂O in less-hydrophilic organic liquids. Concurrent progress in size reduction enabled elaboration of micro-coagglomerates less than 1 μm in size. The organic liquid media with adequate specific capacity and low viscosity resulted in enhanced reactivity and led BaFe₁₂O₁₉ powders with excellent magnetic properties and sufficient sinterability without irregular grain growth.

[13] NMR AND EPR STUDIES ON THE CHARGE TRANSFER AND FORMATION OF COMPLEXES THROUGH INCOMPLETELY COORDINATED STATES

Senna M. Isobe T. - Solid State Ionics. 101(Part 1):387-392, 1997

Changes in the state of coordination and electron distribution in a fine particulate solid mixture under mechanical stressing are studied by solid state nuclear magnetic resonance (NMR) and electron paramagnetic resonance (EPR), coupled with more conventional analytical tools, e.g., X-ray photoelectron spectroscopy or infrared spectroscopy. Systems examined are mainly Ca(OH)₂ + SiO₂ and metallic Al + hydrogel, TiO₂·H₂O. Si-O-Ca or Al-O-Ti bridging bonds are formed either by charge transfer, i.e. an acid-base reaction in a broad definition, or by radical recombination. The charge transfer is also elucidated on the basis of electronegativity equalization, by taking low-coordination states into account.

[12] SYNTHESIS OF CDS QUANTUM DOTS BY MECHANOCHEMICAL REACTION

Tsuzuki T. McCormick PG. - Applied Physics A-Solids & Surfaces. 65(6):607-609, 1997

The synthesis of ultrafine cadmium sulphide quantum dots by mechanochemical reaction has been studied using X-ray diffraction, transmission electron microscopy and UV-VIS absorption spectroscopy. The solid-state displacement reaction of CdCl₂ + Na₂S → CdS + 2NaCl induced by mechanical milling resulted in the formation of CdS particles with an average diameter of < 8 nm. The average particle size was controlled within the range of 4 to 8 nm by varying the size of the grinding media. The onset energy of optical absorption showed a blue shift with decreasing particle size.

[11] ON THE NATURE OF PHASES FORMED DURING SOFT MECHANOCHEMICAL SYNTHESIS OF CALCIUM TITANATE

Kosova NV. Avvakumov EG. Malakhov VV. Devyatkina ET. Dovlitova LS. Boldyrev VV. - Doklady Akademii Nauk. 356(3):350-353, 1997

[10] FORMATION OF SUPERCONDUCTING PHASES IN A MECHANOCHEMICALLY ACTIVATED BI-SR-CA-CU-O SYSTEM

Shlyakhtina AV. Vishnev AA. Kolbanev IV. Mamsurova LG. Trusevich NG. Shcherbakova LG. - Chemical Physics Reports. 16(7):1225-1240, 1997

It is revealed that the degree and type of the mechanochemical activation effect on the synthesis and formation mechanism of Bi-containing high-temperature superconductors (HTSC) of the 2212 and 2223 composition differ substantially from that observed in synthesis of YBa₂Cu₃O_x. Mechanochemical activation is shown to affect the kinetics of solid-phase interaction and alter the order of entering into the reaction of the mixture components changing their reactivity and, in the final run, the mechanism of formation of Bi-Sr-Ca-Cu-O compounds. As a result, the most favorable conditions arise for formation of the 2201 phase. Formation of compounds 2212 and 2223 can either be substantially promoted or, on the contrary, inhibited, depending on the precursor choice. It is supposed that the reaction should necessarily include the liquid phase (in microscopic amounts) in order HTSC compounds to form in this system and that the mechanism of their formation under mechanical impact is of the intercalation type. Magnetic studies of compound 2212 prepared under different conditions revealed a number of peculiarities of the field dependence of magnetization which suggest the cluster nature of the superconducting Bi-containing phase.

[9] MECHANOCHEMICAL PROCESSING OF REFRACTORY PYRITE

Warris CJ. McCormick PG. - Minerals Engineering. 10(10):1119-1125, 1997

Mechanochemical reactions are those which are initiated by the application of a mechanical force. In recent times it has been shown that solid state mechanochemical reactions can be initiated by milling the reactants in a high energy ball mill - a process known as mechanical milling. Oxidation/reduction reactions have been shown to occur at room temperature during mechanical milling, and this has led to the idea that it may be possible to refine metallic ores via room temperature mechanochemical processes. In this study, it has been found that pyrite (FeS₂) can be mechanochemically processed in a high energy ball mill. Pyrite is the most common sulfide host for gold and in refractory pyrite samples gold is encapsulated in the pyrite matrix, so this matrix must be broken down prior to cyanidation. X-ray diffraction, Mossbauer spectroscopy and thermal analysis techniques have shown that pyrite can be oxidised mechanochemically, with the sulfur retained as a sulfate without the production of sulfur dioxide gas. Potential exists for this process to form the basis for an economically viable, environmentally friendly process for the treatment of refractory pyritic gold ores.

[8] MECHANOCHEMICAL OXIDATION OF SILICON AND SELECTIVITY OF OXIDE SUPERFICIAL LAYER DISSOLUTION IN AQUEOUS SOLUTIONS OF HF AND KOH

Stevulova N. Suzuki T. Senna M. Balintova M. Sepelak V. Tkacova K. - Sol. St. Ion.. 101(Part 2):681-686, 1997

Mechanochemical oxidation of silicon and selectivity of oxide layer dissolution in diluted solutions of HF and KOH

were investigated. Energy-intensive grinding of Si is accompanied by changes in state and composition of superficial layers and by significant decrease in crystallinity. The effect of the polarity of liquid grinding environment on the amorphisation is not significant. The permittivity of liquid influences oxidation of silicon and surface concentration of damage centers. The porous oxide shell covering the ground particles controls the adsorption behaviour of Si and consequently its dissolution in diluted solutions of HF and KOH. Stimulating or inhibiting effect of mechanically induced pre-oxidation depends upon the mechanism of dissolution. While selective dissolution of oxide in KF is facilitated by the pre-oxidation, the direct dissolution of silicon in alkaline solvents is inhibited by oxide surface layer.

[7] INFLUENCE OF THE MECHANOCHEMICAL TREATMENT ON THE REACTIVITY OF V-CONTAINING OXIDE SYSTEMS

Zazhigalov VA. Haber J. Stoch J. Kharlamov AI. Bogutskaya LV. Bacherikova IV. Kowal A. - Solid State Ionics. 101(Part 2):1257-1262, 1997

Mechanical treatment of V₂O₅ or V-P-O catalysts causes a substantial increase of both catalytic activity in n-butane oxidation and the selectivity to maleic anhydride. Changes in specific surface area and anisotropic deformation take place. Mechanochemical treatment of the initial reagent impairs such properties that they remarkably influence the catalytic properties of the final catalyst.

[6] INFLUENCE OF THE ATMOSPHERE CONTROL DURING THE GRINDING OF TITANIUM POWDER ON ITS REACTIVITY TOWARDS THE CONVERSION INTO TITANIUM NITRIDE

Criado JM. Alcalá MD. Real C. - Solid State Ionics. 101(Part 2):1387-1391, 1997

A grinding vial has been developed to be used with a high grinding energy swinging mill under the pressure of selected gases. This tool has been used for the study of titanium with nitrogen gas. Nanometric titanium nitride with a crystallite size around 10 nm has been obtained. A mechanism for the mechanochemical conversion of titanium into titanium nitride has been proposed.

[5] RESPONSE TO LONG-TIME EXPOSURE AT HIGH TEMPERATURE OF THERMOELECTRIC MODULES OF BI-TE PREPARED BY MECHANICAL ALLOYING

Hasezaki K. Tsukuda H. Yamada A. Nakajima S. - Materials Transactions Jim. 38(11):1022-1026, 1997

Thermoelectric modules assembled with nickel or copper electrodes and bismuth telluride system semiconductors prepared by mechanical alloying were evaluated for long exposure time dependence. Modules exposed at temperatures of 400 and 473 K from 1 to 1000 h in air were measured for thermoelectric motive forces and internal resistances. The properties of nickel and copper electrode modules did not change up to 400 K. However, the internal resistance increased with increasing exposure time at 473 K. This was because the tin element in the soldering material was diffused in the bismuth telluride system semiconductors, making many voids in the junction layer. Furthermore, these results showed that the maximum output could be effectively evaluated using the Larson-Miller parameter ($C=15.2$).

[4] ALUMINUM TITANATE TETRAGONAL ZIRCONIA COMPOSITE WITH LOW THERMAL EXPANSION AND HIGH STRENGTH SIMULTANEOUSLY

Shimada T. Mizuno M. Katou K. Nurishi Y. Hashiba M. Sakurada O. Mizuno D. Ono T. - Solid State Ionics. 101(Part 2):1127-1133, 1997

Aluminium titanate was strengthened by alloying with 3 mol% yttria stabilized zirconia to attain a small expansion coefficient of 2×10^{-6} and a high strength of 100 MPa, simultaneously. Long milling and optimization of the composition of the alloy to an equal weight of the component oxides and also of the firing temperature at 1400 degrees C were important for the purpose. In the optimal conditions, aluminum titanate grains of 3 μ m were surrounded by zirconia matrix of ca. 0.3 μ m grains containing zirconium titanate where would be a balance between the tension in the matrix and the expansion force of aluminum titanate grains. Formation of zirconium titanate was important for the strengthening to decrease the lowering of the strength brought about by the unstabilization of PSZ.

[3] INTERACTIONS IN IRON-BASED CERMET SYSTEMS

Aigner K. Lengauer W. Ettmayer P. - Journal of Alloys & Compounds. 262:486-491, 1997

The reactions occurring during sintering cermets were investigated at temperatures up to 1500 degrees C. In the low temperature range (up to 500 degrees C) physical reactions take place, in particular the vaporisation of residual milling agent and desorption of CO from the metal phase which remained from the production of the binder metal. From approximately 500 degrees C up to the melting point of the binder the partial reduction of oxides from the surface of the carbides starts to remove carbon from the carbides and carbonitrides. This improves the wettability of the hard particles and leads to better densification. Below the temperature of formation of the liquid phase there is an intensive solid-state interaction between hard particles and binder metal. Due to the increased mobility of carbon the remaining surface oxides are reduced. This reaction is completed when the binder is molten, because at that time the oxygen from the hard particles' surface and binder is eaten up. In the third temperature range the liquid binder-phase starts to dissolve the hard particles, beginning at the grain boundaries. When the binder is saturated a solution-dissolution reaction takes place, reshaping and increasing the size of the particles. The porosity of Fe-Ti(C_xN_{1-x}) cermets after liquid phase sintering increases with the nitrogen content of the carbonitride. A comparison of the sintering behaviour of mixtures of group IV transition metals with iron shows an increase in porosity from Fe-TiC over Fe-ZrC to Fe-HfC.

[2] SYNTHESIS OF CELSIAN (BaAl₂Si₂O₈) FROM SOLID Ba-Al-Al₂O₃-SiO₂ PRECURSORS - I, XRD AND SEM/EDX ANALYSES OF PHASE EVOLUTION

Allameh SM. Sandhage KH. - Journal of the American Ceramic Society. 80(12):3109-3126, 1997

An intimate Ba-Al-Al₂O₃-SiO₂ powder mixture, produced by high-energy milling, was pressed to 3 mm thick cylinders (10 mm diameter) and hexagonal plates (6 mm edge-to-edge width). Heat treatments conducted from 300 degrees to 1650 degrees C in pure oxygen or air were used to transform these solid-metal/oxide precursors into BaAl₂Si₂O₈. Barium oxidation was completed, and a binary silicate compound, Ba₂SiO₄, had formed within 24 h at

300 degrees C. After 72 h at 650 degrees C, aluminum oxidation was completed, and an appreciable amount of BaAl2O4 had formed. Diffraction peaks consistent with hexagonal BaAl2Si2O8, BaAl2O4, beta-BaSiO3, and possibly beta-BaSi2O5 were detected after 24 h at 900 degrees C, Diffraction peaks for BaAl2O4 and BaAl2Si2O8 were observed after 35 h at 1200 degrees C, although SEM analyses also revealed fine silicate particles, Further reaction of this silicate with BaAl2O4 at 1350 degrees to 1650 degrees C yielded a mixture of hexagonal and monoclinic BaAl2Si2O8. The observed reaction path was compared to prior work with other inorganic precursors to BaAl2Si2O8.

[1] DYNAMICS OF INTERFACIAL AREA CHANGE DURING MECHANICAL ALLOYING OF W WITH NI

Popova TG. Butyagin PY. Streletskii AN. Portnoi VK. - Colloid Journal. 59(6):737-741, 1997

Quantitative X-ray diffraction analysis was employed to study kinetics of the formation of the solid solution of W in Ni during mechanical processing of a mixture of metal powders. The variation in the W content in the mixture, the lattice parameters of the solid solution of W in Ni, the dimensions of the regions of coherent scattering L-rcs, and the root-mean-square microdistortions of crystal lattices for both components were monitored. The kinetics was represented as the N(D) and L-rcs(D) dependences on dose D. It was established that the limiting stage of the formation of the solid solution is the penetration of W atoms through the component contact surface with the area S-c. The dependence of the tungsten consumption N(D) in a mixture of the specified composition can be described by equation $-dN/dD = G(0)S(c)$, where G(0) is the coefficient characterizing the interface permeability. The pattern of the L-rcs(D) dependence is determined by the character of the tungsten particle size distribution. The value of G(0) is equal to 0.04 mol g/MJ m(2).

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