

RESEAU FRANÇAIS DE MECANOSYNTHESE

Lettre N°46

Janvier 1999

132 Groupes de Recherche
(dont 69 à l'étranger)

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

2 Nouvelles Adhésions

I. Dekany - Dept of Colloid Chemistry - Szeged - Hongrie
Yijun Sun - Shangai Institute of Metallurgy - Chine

Happy New Year !!!

Prière de Penser à votre Cotisation au titre de l'année 1999 !!

**Pour ceux qui n'auraient pas réglé leur cotisation 1998 (voire 1997...), Prière de
le faire rapidement.**

La liste de diffusion sera remise à jour pour 1999

!!!

Le site web du RFM est le suivant

<http://www.bls.fr/amatech>

Rubrique Pages Sciences et Techniques pour l'Ingénieur (Rubrique Sciences)

⇒ vous y trouverez les anciennes lettres du RFM (accessible par Adobe Acrobat)

les statuts du RFM ainsi que les annonces concernant les JRFM'99 et quelques éléments mis à jour régulièrement concernant les derniers résultats dans ce domaine.

Une Rubrique "Forum"

**est ouvert sur le site web du RFM afin de permettre
de discuter sur tout sujet**

"Mécanosynthèse et/ou Nanomatériaux"

**L'inscription en ligne aux JRFM'99
est possible sur ce site Web !!!!**

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JRFM'99

4 èmes Journées du Réseau Français de Mécanosynthèse
Dijon, les 2-3 Juin 1999
1ère circulaire

Programme des JRFM'99

- 3 conférenciers invités sont d'ores et déjà programmés :
- D.LOUER (Directeur de Recherches CNRS- Rennes).
 - JF.BERAR (Ingénieur de Recherches CNRS- Grenoble / ESRF).
 - J.C.MUTIN (Directeur de Recherches CNRS - Dijon).

Appel à Communications
à renvoyer avant le **30 Décembre 1998**

Nom:Prénom:

Adresse:.....

.....

Téléphone : Fax :E-mail :

Souhaite présenter une communication lors des JRFM99 sous forme de :
communication orale Communication par affiche

Titre :

Fiche d'inscription à retourner à

Frédéric BERNARD (Journées RFM99), Laboratoire de Recherches sur la Réactivité des Solides (UMR5613 CNRS - Université de Bourgogne), 9 avenue Alain Savary, BP 400, 21011 Dijon Cedex. (Tel : 03.80.39.61.25 - fax : 03.80.39.61.67, E-mail : fbernard@u-bourgogne.fr)

Comité d'organisation :

Frédéric Bernard et toute l'équipe " Matériaux à Grains Fins " :
Laboratoire de Recherches sur la Réactivité des Solides (UMR5613 CNRS
Université de Bourgogne), 9 avenue Alain Savary, BP 400, 21011 Dijon Cedex.
Tel : 03.80.39.61.25 - Fax : 03.80.39.61.67, E-mail : fbernard@u-bourgogne.fr)

Eric Gaffet :

Président du Réseau Français de Mécanosynthèse
Groupe "Nanomatériaux" - CNRS UPRA423/806 - Institut Polytechnique de Sévenans
90100 Belfort Cedex.
Tél : 03 84 58 31 02 - Fax : 03 84 58 30 27 - E-mail : Eric.Gaffet@utbm.fr

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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
Nanostructured Hybrid Materials
Symposium TMS Annual Meeting - San Diego CA - USA - 28 Février 4 Mars 1999
Contact : gmchow@anvil.nrl.navy.mil`

XXV JEEP
Journées d'Etudes des Equilibres entre Phases 1999
11 - 12 Mars 1999 - Annecy France
E-Mail : Conference.Jeep@univ-savoie.fr

VII International Seminary
Defects, structure and properties of Nanocrystalline Materials obtained by Nanocrystallization of Amorphous Solids
and of Metals with Extreme Distortion of the Lattice
Mars 1999 - Ekaterinburg - Russie
E-Mail : Noskovaimp.uran.ru

Nanocomposite Materials : Design and Applications
28 Mars - 2 Avril 1999 - Alyeska Resort - Alaska
E-Mail : Engfnd@aol.com

4th International Workshop on Metastable Phases (IV IWOMP)
7 - 9 Avril 1999 - Bologne - Italie
Contact : Bonetti@df.unibo.it

12th International Conference on Wear of Materials
Atlanta - Georgie / USA - 25 - 29 Avril 1999
contact : Amy Richardson E-Mail A.Richardson@elsevier.co.uk
or web site : <http://www.elsevier.nl/locate/wom99>

E MRS - Spring Meeting
1 - 4 Juin 1999 - Strasbourg - France
Web Site <http://www-emrs.C-strasbourg.fr>
Symposium A : Phot - Excited Process and Applications
Symposium B : Protective Coating adn Thn Films 99
Symposium C : Progress in Computational Materials Science
Symposiym D : Plasma and Ion Surface Engineering
Symposium E : Advanced Silicon Substrates
Symposium F : Process induced defects in Semiconductors
Symposium G : Material Physics Issue and Applications on Magnetic Oxides
Symposium H : Strain in Materials : Analysis, Relaxation and Properties
Symposium I : Microcrystalline and Nanocrystalline Semiconductors
Symposium J : Materials for Coherent Optics
Symposium K : Materials, Process and Technology for Optical Interconnect
Symposium L : Ab - Initio Approaches to Microelectronics Materials...
Symposium M Basic Models to enhance Reliability in Si based devices and ..
Symposium N : Molecular Optoelectronics : Materials, Physics and Devices
Symposium O : Chalcogenide Semiconductors for Photovoltaics
Symposium P : Optical Characterization of Semiconductor layers and Surfaces

JRFM'99
4èmes Journées du RFM
2 & 3 Juin 1999 - Dijon - France
Web Site : <http://www.bls.fr/amatech> - Web SubSite : Sciences

**Nanostructured Materials Symposium at the 5th IUMRS International Conference
on Advanced Materials
(IUMRS - ICAM'99)**
Beijing - Chine - 13 - 18 Juin 1999
Contact : Kelu@imr.ac.cn
WebSite - <http://www.chimeb.edu.cn>

PM2 Tec 98
1999 International Conference
on Powder Metallurgy and Particulate Materials
Vancouver - 20 / 24 Juin 1999
E-Mail : Info@mpif.org - Website: www.mpi.org

4th Int. Conf. on Materials Chemistry
13 - 16 Juillet 1999 - Trinity College _ Univ. Dublin - Irlande
Web Site : <http://www.rsc.org/conferences>

Themes :
Inorganic Nano and Micro Particles
Functional Polymers
Magnetic Materials
Organic Nanostructures
Molecular Crystals and Crystal Engineering
Computational Chemistry and Materials for Electronic

Advanced Materials - Nanostructured Systems

15 - 17 Juillet 1999 - Hong Kong
1st workshop of the new IUPAC series :
"New Directions in Chemistry
Theory, Nanoparticles, Quantum Dots,
Bio - Inspired Structures, Applications to Nanotechnology
Organizing Committee A. El - Sayed - Georgia Tech - Atlanta - USA
J. Portner - President of IUPAC - Tel Aviv - Israel
N. Teng Yu - HKUST - Hong Kong
S. Williams - Hewlett - Packard Co., California USA
Web Site : <http://www.iupac.org/symposia/conferences/wam1>

NATO Advanced Research Workshop

Investigations and Applications of Severe Plastic Deformation
2 - 6 Aout 1999 - Moscou - Russie
E-Mail : TLow@lanl.gov and Valiev@ippm.rb.ru

RQ10

10th International Conference on Rapidly Quenched and Metastable Materials

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

ISMANAM 99

International Symposium on Metastable Mechanically Alloyed and
Nanocrystalline Materials
NOUVEAU and Euro Conference on Gas Phase Synthesis of Nanocrystalline Materials.
Org. : L. Schultz, J. Eckert, H. Hahn
Dresden - 30 Aout - 3 Septembre 1999
E-Mail : ISMANAM99@ifw-Dresden.de
WebSite: <http://www.ifw-dresden.de/imw/ismanam/>

SMM14

14th International Conf. on Soft Magnetic Materials

8 - 10 Septembre 1999 Balatonfüred - Hongrie

web site : <http://www.kfki.hu> - Subsite : [smm14](http://www.kfki.hu/smm14)

Int. Symp. Cluster and Nanostructure Interfaces (ISCANI)

25 - 29 Octobre 1999 - Richmond USA
website : <http://www.vcu.edu/ISCANI/>

NANO 2000

NOUVEAU

5th International Conference on Nanostructured Materials
Sendai - 20 - 25 Aout 2000
E-Mail : nano2000@imr.tohoku.ac.jp

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Annonces de Soutenance de Thèses
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Caractérisation et réactivité de la surface de poudres nanométriques d'oxydes métalliques: Analyse par spectrométrie IR-TF et application à l'étude des mécanismes de détection de gaz par capteurs résistifs.

Jérôme Tribout - Université de Limoges, Limoges, France, 14 décembre 1998.
Directrice: Marie-Isabelle Baraton

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Distinct Element Modelling of a Planetary Ball Mill
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M.P. Dallimore - Dpt of Mechanical and Materials Engineering - University Western Australia - Australie

The distinct element method of modelling has been used to develop a model of a laboratory scale planetary ball mill. The model has been used to evaluate variations in processing parameters such as the impact frequency and energy associated with different milling configurations. These results have been compared to the kinetics of the Ni/CuO displacement reaction propagated under these conditions.

The model is two dimensional in nature and incorporates a modified Kelvin viscoelastic spring / damper system to describe the impact process using experimentally derived impact parameters. The simulation enables quantitative analysis of the collision energies and their spatial distribution. Validation of the model was achieved through the use of high speed video analysis of actual ball trajectories.

The results of the modelling study have demonstrated that kinematic considerations alone are insufficient to predict the variation in ball motion and energy dissipation that occur for different mill configurations. The amount of slip between the balls and the vial wall plays a significant role in determining both the tumbling trajectories and the level and mode of energy dissipation.

When comparing the model predictions to the experimental milling results, it was found that reaction rate was particularly sensitive to the spatial distribution of the impacts. Only when the tumbling trajectories remained relatively unchanged, could the reaction rate be tied to the total level of energy dissipated. Both the energy dissipated through impacts and attrition play an important role in promoting the reaction. The results suggest that the spatial distribution of the impact energy must be considered with respect to the distribution of powder within the vial to fully explain the variations in reaction rates.

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Synthèse et Propriétés de Ferrites Nanométriques : Influence de l'énergie de surface sur les propriétés structurales et magnétiques de ferrites de titane synthétisés par chimie douce et mécanosynthèse

N. Guigue - Millot - 26 Novembre 1998 - LRRS UMR 5613 CNRS - Univ. Bourgogne - Dijon - France
Jury : J. Etourneau, A. Rousset, G. Bertrand, D. Stuerger, G. Le Caër, M. Guyot, O. Isnard, P. Perriat

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Transformations antiferromag - ferromag - paramagnétiques - verre de spin dans les alliages de Fe Rh nanocristallisés par Broyage

E. Navarro - Université de Complutense - Madrid - Espagne - 18 Mai 1998
Co directeurs : A. Hernando - A.R. Yavari

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Modifications morphologiques et microstructurales du matériau actif des cathodes de batteries à l'ion lithium induites par broyage et traitement thermique

Ph. Perrot - Université de Poitiers - 6 Mai 1998
Co - Directeurs : E.L. Mathe, M. Grosbras

Jury : J. Mimault, H. Van Damme, A. Dager, M. Broussely, P. Goudeau, E.L. Mathe, M. Grosbras

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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., Rouzaud JN, Tarascon JM.

=====
"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 (0<x<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

=====
"The Preparation of Nitrides and Carbides by Mechanical Treatment - Phases and Structures"

G.M. Wang - School of Physics, University College, The University of New South Wales - Australian Defence Force Academy - Canberra, ACT 2600 - Australia - 10/12/97

Supervisor - S.J. Campbell - **Co - Supervisors** : W.A. Kaczmarek and A. Calka

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"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

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Sites internet à découvrir

Site sur la cristallographie / Soft + Littérature

<http://www.lmcp.jussieu/sincris-top/logiciel>

N.B. : si vous connaissez d'autres sites en relation avec les thèmes développés par le RFM, faites nous les connaître

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Post Doc Position Proposals

Brésil

Post doc to work in electron microscopy characterization of nano - structured materials.
Contact : Walter J. Botta. F. - Federal Univeristy o Sao Carlos - Sao Paulo State - Brésil
Adresse : DEMa - UFSCar - CP 676, 13565 - 905 Sao Carlos SP Brésil.
tél : 016 - 2608251 - Fax : 016 2615404.

Belgique

The Department Metallurgy and Materials Engineering (MTM) of the K.U.Leuven (Belgium) has a research position available. Candidates are asked to contact the responsible staff member.

Area of research :

Metals and Alloys, Polymer Matrix Composites, Intelligent Processing of Materials, Surface Engineering and Tribology, Metal Forming and Mechanical Behaviour of Materials, Quality Control and Non-Destructive Testing of Materials, Ceramics, Thermodynamics, Corrosion, Nuclear Engineering

Description of research task

Tailor made powders by mechanical alloying of Fe and Cu based materials. Application field: specific composite materials, to be prepared by conventional PM consolidation techniques. Research activities: parametric study of MA, alloy design, microscopic

Staff member to be contacted

Prof. Dr. Ir. L. Froyen

Katholieke Universiteit Leuven - Dept. MTM

de Croylaan 2 - B-3001 Leuven (Belgium)

Tel. +32/16/22.09.31

Japon

Our group: Nanocomposite Group, Department of Composite Materials, National Institute of Materials and Chemical Research, Tsukuba, Ibaraki, Japan is now looking for post-doc researchers

The candidates would be integrated in the Nanocomposite Group of the Department of Composite Materials. The research interests of the group are mainly focused on nanocomposite preparation and its optical/chemical functionalities. Research projects currently under way aim to develop nanostructured and optically/chemically active thin films by sputtering, laser ablation and so on. For additional information about the Institute and group :

<http://www.nimc.go.jp/>

<http://www.aist.go.jp/NIMC/fcg/index.html>

Experience in the fields of materials science (ceramic or metal) is required.

There are two types of post-doc positions.

1. Long-term: from 6 months to 2 years

2. Short-term: from 1 to 3 months

If you or someone in your laboratory is intersted in this fellowship, please contact as soon as possible to:

Dr. Naoto Koshizaki - Department of Composite Materials

National Institute of Materials and Chemical Research(NIMC) 1-1 Higashi, Tsukuba, Ibaraki 305-8565 JAPAN

Tel: +81-298-54-6335 - Fax: +81-298-54-6252 - E-mail: koshizaki@nimc.go.jp - <http://www.aist.go.jp/NIMC/fcg/index.html>

Bibliographie Récente

Livres ou "Special Issues"

Surface-Controlled Nanoscale Materials for High-Added-Value Applications.

Editors: Kenneth E. Gonsalves, Marie-Isabelle Baraton, Rajiv Singh, Heinrich Hofmann, Jerry X. Chen, and Joseph A. Akkara.

Materials Research Society, Symposium Proceedings Volume 501, 1998
MRS, Warrendale, Pennsylvania, USA (website: <http://www.mrs.org/>)

"Nanomatériaux"

Auteurs : E. Gaffet, S. Begin - Colin, O. Tillement

Editeur : Innovation 128 - 24 Rue du Quatre Septembre - 75002 Paris - France

Les dernières années ont vu apparaître dans le monde des matériaux avancés le préfixe "nano" (nanostructuré, nanocristallins, nanophase ou nanométrique) ; les conférences et les forums sur Internet se multiplient où s'échangent des informations sur les avancées scientifiques et technologiques dans ce domaine des matériaux nanostructurés qui se distinguent des matériaux polycristallins conventionnels par la dimension des cristallites les composant ou par la dimension des hétérostructures présentes : ces dimensions sont de quelques dizaines d'angströms, voire de quelques nanomètres. A ces dimensions, les propriétés des matériaux changent radicalement.

Au début des années 90, les japonais ont été les premiers à lancé d'ambitieux programmes de R & D puisque le MITI a consacré aux nanomatériaux près de 200 millions de dollars pour la période 1990 - 2000 et que la Science & Technology Foundation a investi presque la même somme pour co - financer des projets de laboratoires publics et privés. Les Etats Unis puis les pays européens ont investi plus tardivement mais déjà ont obtenu des résultats prometteurs (.....) Certaines applications existent déjà au niveau international, quelque 400 sociétés se partagent aujourd'hui un marché voisin de 1 milliard de dollars mais qui devrait tripler, voire quintupler à l'horizon 2001.(.....)

(...) Pour aider les industriels concernés à imaginer les applications qu'ils pourraient s'approprier et identifier les acteurs internationaux, la présente étude dresse un état de l'art complet des nanomatériaux en décrivant leurs procédés d'élaboration actuels ou envisagés et en détaillant leurs différentes propriétés physico - chimiques et les géométries que l'on peut obtenir.

Enfin l'étude permet de cerner les applications actuelles et potentielles...

CHEMISTRY FOR SUSTAINABLE DEVELOPMENT

Vol. 6, No. 2-3, MARCH-JUNE 1998

Proceedings of 2d International Conference on Mechanochemistry
(INCOME-2), which was held in Novosibirsk in 1997.

Contact : Prof. • N.Z. Lyakhov, Inst. Sol. State Chem.- Russian Acad Sci. - Kutaleladze, 18 - Novosibirsk - 630128 Russia - The Proceedings will be available by the price 80 USD.

Mechanochemistry of Materials

Cambridge International Science Publishing

Emmanuel Gutman - Materials Eng. Dpt - Ben Gurion University - Beer Sheva - Israel

Considerable advances have been made in mechanochemistry in the last couple of decades. Training of experts in this field with a background in materials science, chemical and mechanical engineering, etc. requires study of the fundamentals of mechanochemistry. There is a need for a textbook in the general and compressed form which would cover many aspects and would be used as a basis for understanding the fundamental principles to control mechanochemical phenomena. This textbook is based on lectures given by Prof. Gutman in a graduate course in the mechanochemistry of materials at the Ben - Gurion University of the Negev. The book contains examples of experimental results to illustrate the mechanochemical phenomena and technologies.

BIBLIOGRAPHY ON MECHANICAL ALLOYING AND MILLING

Suryanarayana (Inst for Materials and Advanced Processes, University of Idaho, USA)

The present bibliography covers information on mechanical alloying and milling of materials starting from 1970 (when it was recognized that MA has become a commercial/viable material processing technique instead of just a grinding method) to 1996. All the available references will be presented in a chronological fashion. Under each year, (.....)

Please send your order to: Book Department - Cambridge International Science Publishing 7 Meadow Walk, Great Abington, Cambridge CB1 6AZ, England Fax: +44 1223 894 539; tel +44 1223 893295, email: orders@cisp.demon.co.uk / <http://www.demon.co.uk/cambsci/homepage.htm>

Proceeding du Congrès "Mechanically Alloyed, Metastable and Nanocrystalline Materials"- Barcelone (1997)

Editor : M.D. Baro, S. Surinach - Materials Science Forum 269 - 272 (1998)

PERIODIQUES

(Rubrique assurée grâce au concours de M^{me} TAUZIN - FIN BiPSé)

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

[46] PHASE TRANSFORMATION OF HEPTANE ADDED TI-AL MECHANICALLY ALLOYED POWDERS DURING HEATING

Fujii T. Sodeoka S. Ameyama K. - Journal of the Japan Institute of Metals. 62(10):945-951, 1998

Phase transformation behaviors of Ti-Al mechanically alloyed (MA) powders during heating were investigated with analytical TEM, DSC and XRD. Elemental Ti and Al powders with overall compositions of 33, 41 and 46 mol% Al were blended for mechanical alloying. N-heptane was used as a process control agent for the mechanical alloying. Irregular shaped and dislocation free Al supersaturated alpha-Ti nanocrystals were formed in the MA powders after 720 ks of milling. The mean grain size is about 25 nm. An hcp->fcc->hcp->DO19 transformation in the Ti rich MA powders and an hcp->fcc->L1(0) transformation in the Al rich MA powders were observed during heating. The morphological features are as follows: (1) an fcc/hcp thin layered structure, (2) a composition invariant, (3) a large number of defects, and (4) rapid grain growth. They strongly suggest that the grains are formed by shear mechanism as well as short range diffusion, i.e., a massive transformation during heating. The fee phase formation can be explained by an hcp->fcc structure change by the motion of a $\frac{a}{3}[1\ 1\ \overline{1}]\text{type}$ Shockley partial dislocation on every second plane of the basal plane of the hcp crystal. Since an hcp structure is stable in Ti rich MA powders, the fee phase, which in turn transforms to the hcp phase by the motion of a $\frac{a}{6}[\overline{1}1\ 2]\text{type}$ partial dislocation. Finally, the DO19 (alpha(2)) or L1(0) (gamma) phases form from the hcp or fee phases by the short range diffusion.

[45] RAMAN SPECTROSCOPY OF MONOLAYER-TYPE CATALYSTS - SUPPORTED MOLYBDENUM OXIDES [Review]

Mestl G. Srinivasan TTK. - Catalysis Reviews-Science & Engineering. 40(4):451-570, 1998

[44] THE FUNDAMENTALS OF MECHANOCHEMICAL PROCESSING

Mccormick PG. Froes FH. - JOM-Journal of the Minerals Metals & Materials Society. 50(11 Suppl S):61-65, 1998

It has been demonstrated that the activation of chemical reactions by mechanical energy can lead to many interesting applications, ranging from waste processing to the production of advanced materials with novel microstructures and enhanced mechanical properties. In this article, the status of three types of mechanochemical processing is reviewed-mechanical milling, mechanical alloying, and reaction milling.

[43] X-RAY MICROSCOPY OF NOVEL THERMOPLASTIC/LIQUID CRYSTALLINE POLYMER BLENDS BY MECHANICAL ALLOYING

Smith AP. Bai C. Ade H. Spontak RJ. Balik CM. Koch CC. - Macromolecular Rapid Communications. 19(11):557-561, 1998

Incorporation of liquid crystalline polymers (LCPs) into commodity polymers remains a challenge in the design of high-performance, low-cost polymeric blends. Blends of a thermoplastic polymer and a nematic LCP are produced here by mechanical alloying. Functionality sensitive X-ray microscopy reveals LCP dispersions as small as 100 nm in diameter. Intimate mixing remains upon subsequent melt processing, indicating that mechanical alloying is suited for applications such as recycling.

[43] ABOUT VACANCY AND FE²⁺ CONTENTS IN LiZnTi FERRITES

Arias AG. Delcueto A. Munoz JM. Defrancisco C. - Materials Letters. 37(4-5):187-191, 1998

A thermogravimetric method is applied to the evaluation of the cationic vacancy delta and Fe²⁺ contents in LiZnTi ferrites with nominal composition $\text{Li}_{0.5(1-x+y)}\text{Zn}_{x2}\text{Ti}_{y4}\text{Fe}_{2.5-0.5(x+3y-z)}\text{Mn}_{z3}\text{O}_4$, where some iron excess is provided by the contamination taking place in the milling stages of the ceramic procedure used to synthesize the ferrite. Experimental results for samples with $x = 0.25$, $y = 0.83$ and $z = 0.05$ are shown. The single phased samples, with a cell parameter of 8.36 Angstrom, were analysed in an automatic thermobalance in the form of 5 x 20 mm rods and sintered in O-2, air and CO₂, suspended in a platinum wire. Changes in delta due to atmosphere changes are much more significant than those in [Fe²⁺]. When sintering in O-2, an increase of the iron defect in the nominal composition practically does not affect [Fe²⁺], but reduces delta drastically, and this reduction can be correlated to changes in the ferrite magnetic properties. To our knowledge, this is an unexpected and significant result, since most discussions in the literature on iron excess ferrites usually attribute the changes in magnetic properties to the influence of [Fe²⁺], ignoring delta.

[42] STRAIN-INDUCED STRUCTURAL CHANGES AND CHEMICAL REACTIONS - I - THERMOMECHANICAL AND KINETIC MODELS

Levitas VI. Nesterenko VF. Meyers MA. - Acta Materialia. 46(16):5929-5945, 1998

Strain-induced chemical reactions were observed recently (Nesterenko et al., Appl. Phys. Lett., 1994, 65(24), 3069; Metall. Met. Trans. A, 1995, 26, 2511) in experiments in the shear band in both Ti-Si and Nb-Si mixtures. Reactions can start in the solid state or after melting of at least one component. One of the aims is to find theoretically, whether there are possible macroscopic mechanisms of mechanical intensification of the above and other chemical reactions due to plastic shear in the solid state. Continuum thermodynamical theory of structural changes with an athermal kinetics, which includes martensitic phase transformations, plastic strain-induced chemical reactions and polymorphic transformations, is developed at finite strains. The theory includes kinematics, criterion of structural change and extremum principle for determination of all unknown variable parameters for the case with neglected elastic strains. Thermodynamically consistent kinetic theory of thermally activated structural changes is suggested. The concept of the effective temperature is introduced which takes into account that temperature can vary significantly (on 1000 K) during the chemical reactions under consideration. The theory will be applied in Part II of the paper for the description of chemical reactions in the shear band.

[41] IMPLICATIONS OF FINE GRINDING IN MINERAL PROCESSING - MECHANOCHEMICAL APPROACH

Lin IJ. - Journal of Thermal Analysis. 52(2):453-461, 1998

Fine grinding of minerals may change physical and chemical properties of the material to the extent that has to be considered in laboratory verification work or processing work in the industrial plant. The ground material is mechanically activated by increase of both: specific surface energy and elastic strain energy. The activation energy can then be dissipated through different mechanisms, such as: polymorphic transformation, mechanochemical decomposition or synthesis. The thermodynamical principles and kinetics mechanisms responsible for the relaxation modifications are thoroughly discussed. Important factors such as: Reaction triggering dimension, action of shear stresses, surface groups activity, product reactivity, etc. are described. A short survey on comminution by fine grinding is also presented.

[40] ENERGETIC APPROACH TO KINETICS OF MECHANOCHEMICAL PROCESS

Kheifets AS. - Journal of Thermal Analysis. 52(2):463-473, 1998

Kinetics of a mechanochemical process is proposed to describe quantitatively with a problem set-up scaled to energy instead of time. This makes possible the comparability of experimental data obtained in machines with different intensities of treatment.

[39] MECHANOCHEMICAL ADSORPTION OF PHENOL BY TOT SWELLING CLAY MINERALS I - THERMO-IR-SPECTROSCOPY AND X-RAY STUDY

Ovadyahu D. Yariv S. Lapidés I. - Journal of Thermal Analysis. 51(2):415-430, 1998

The mechanochemical adsorption of phenol by laponite, saponite, montmorillonite, beidellite and vermiculite was studied by IR and X-ray spectroscopy. Mixtures containing phenol and clay in the ratio of 6:10 were manually ground by a mortar and pestle for 1, 3, 5 and 10 min and the ground mixtures were investigated. Depending on the basicity of the clay mineral and the time of grinding, two different associations between interlayer cations, water and phenol were identified. In these associations phenol can act either as a proton acceptor or donor (Configurations I and II, respectively). The phenol is more acidic than water and in most cases phenol acts as a proton donor. With montmorillonite and beidellite phenol acts as a proton acceptor. In this association the aromatic ring forms pi bonds with atoms of the oxygen planes of the tetrahedral sheers which donate electrons to the anti-bonding pi orbitals of the phenol.

[38] MECHANOCHEMICAL ADSORPTION OF PHENOL BY TOT SWELLING CLAY MINERALS II - SIMULTANEOUS DTA AND TG STUDY

Ovadyahu D. Yariv S. Lapidés I. Deutsch Y. - Journal of Thermal Analysis. 51(2):431-447, 1998

The mechanochemical adsorption of phenol by laponite, saponite, montmorillonite, beidellite and vermiculite was studied by simultaneous DTA and TG analysis. Fitting of the DTA curves showed four endothermic peaks in the r.t. - 250 degrees C temperature range. The first peak represents the melting of the excess crystalline phenol. The second peak represents the evolution of water. The third peak represents the boiling of the free phenol. The fourth peak represents the evolution of adsorbed phenol. In the DTA curves of montmorillonite the fourth peak is not observed as a separate peak but as part of the third peak. Laponite, saponite and montmorillonite show a very small exothermic effect which follows the endothermic events. Iaponite adsorbs the greatest amounts of phenol and shows a strong exothermic peak at 390 degrees C. None of the other clays show this exothermic effect at this temperature.

[37] NEW APPROACH TO FILLED POLYMERS - MECHANOPOLYMERIZATION ON FILLERS

Gutman EM. Bobovitch AL. - Journal of Thermal Analysis. 51(1):245-250, 1998.

Mechanopolymerization of pentabromobenzyl (mono)acrylate (PBB-MA) on the surface of inorganic fillers Mg(OH)(2) and CaCO₃ was studied. The role of activated surface of fillers was investigated using DSC and FTIR. The influence of milling time and of the filler chemical content on polymerization starting temperature and polymerization enthalpy was studied using DSC. It was shown that an increase of the filler concentration leads to a higher maximum conversion degree. The correlation between conversion kinetics and polymerization enthalpy of the material notpolymerized during milling was shown.

[36] FORMATION OF CARNALLITE TYPE DOUBLE SALTS BY GRINDING MIXTURES OF MAGNESIUM AND ALKALI HALIDES WITH THE SAME ANIONS

Shoval S. Yariv S. - Journal of Thermal Analysis. 51(1):251-263, 1998

The formation of carnallite type double salts by grinding mixtures of hydrated magnesium halide and alkali halides with the same anions was investigated by X-ray diffraction, infrared spectroscopy and thermal analysis. Carnallite (KMgCl₃ · 6H₂O), cesium-carnallite (CsMgCl₃ · 6H₂O), bromo-carnallite (KMgBr₃ · 6H₂O) and cesium-bromo-camallite (CsMgBr₃ · 6H₂O) were formed by grinding mixtures of MgCl₂ · 6H₂O with KCl or CsCl and MgBr₂ · 6H₂O with KBr or CsBr, respectively. Hydrated solid solutions of magnesium in potassium or cesium halides were obtained from that portion of potassium and cesium halides which did not take part in the formation of the double salt.

[35] EFFECT OF MECHANICAL ACTIVATION ON THE PHYSICO-CHEMICAL PROPERTIES OF MOO₃

Poluboyarov VA. Kiselevich SN. Kirichenko OA. Pauli IA. Korotaeva ZA. Dektyarev SP. Ancharov AI. - Inorganic Materials. 34(11):1152-1158, 1998

The effect of mechanical activation on the sintering behavior and strength of MoO₃ was studied. Mechanical activation of MoO₃ powder was found to produce particles similar or equal to 10 nm in size, whose number amounts to 50% of the total number of particles, whereas the weight does not exceed 5%. EPR, IR, synchrotron XRD, and EXAFS data demonstrate that the 70-nm fraction of the MoO₃ powder, including similar or equal to 10-nm particles, consists of defect-free orthorhombic MoO₃ and crystallographic-shear structures.

[34] SOOT DEEP OXIDATION CATALYZED BY MOLYBDENA AND MOLYBDATES - A THERMOGRAVIMETRIC INVESTIGATION

Hasan MA. Zaki MI. Kumari K. Pasupulety L. - Thermochimica Acta. 320(1-2):23-32, 1998

Molybdena (MoO₃) and molybdates of bismuth (Bi₂Mo₃O₁₂), chromium (Cr₂Mo₃O₁₂), barium (BaMoO₄),

manganese (MnMoO₄) and copper (Cu₃Mo₂O₉) were synthesized and characterized by X-ray powder diffractometry and infrared spectroscopy. They were then assessed as 'loose contact' catalysts for soot deep oxidation (combustion) in air by thermogravimetry. A similar assessment was carried out on commercial chromia (Cr₂O₃) and tungsta (WO₃). Observed high oxidation activity of MoO₃, as compared to both Cr₂O₃ and WO₃, is attributed to the higher volatility (mobility) of MoOx species. On similar grounds, observed high activity of MoO₃ and Cu₃Mo₂O₉, as compared to the other test molybdates, is explained. Relatively speaking, however, a higher activity was observed for Cu₃Mo₂O₉ than MoO₃, whereby soot ignition temperature decreased from 571 degrees C (uncatalyzed oxidation) to 430 degrees C, to occur within the temperature range of diesel exhaust (200-450 degrees C). This observation is ascribed to copper-promoted redox conduct of Mo(VI) in the oxidation reaction of soot. Kinetics of the reaction was studied non-isothermally, and the kinetic parameters (A, k, Delta E and the reaction order) were calculated.

[33] ABNORMAL GRAIN GROWTH OF NIOBIUM-DOPED STRONTIUM TITANATE CERAMICS

Bae C. Park JG. Kim YH. Jeon H. - Journal of the American Ceramic Society. 81(11):3005-3009, 1998

We have investigated the influence of the Ti/Sr ratio and particle size on abnormal grain growth of Nb-doped SrTiO₃ ceramics. As the Ti/Sr ratio increases, the onset time and temperature of abnormal growth increase, for specimens sintered at temperatures above 1440 degrees C. When the particle size is decreased by milling, the onset and completion temperatures of the abnormal growth and the final grain size are decreased. The abnormal grains growing during isothermal heating are nearly tetrakaidecahedral in shape, whereas those growing during rising temperature have the morphology of an edge-cut cube.

[32] SUPERACIDS BY METAL OXIDES, X - REACTION OF BUTANE CATALYZED BY SULFATED METAL OXIDES, ZEOLITES, OR SILICA ALUMINAS MIXED WITH PT-ZRO2

Hino M. Arata K. - Applied Catalysis A-General. 173(1):121-124, 1998

Active catalysts for the butane conversion were obtained by mechanically mixing Pt-ZrO₂ and sulfated metal oxides (SO₄-MeOx), zeolites, or silica-aluminas. Pt-ZrO₂ was prepared by the impregnation of zirconia gel with aqueous H₂PtCl₆. 6H₂O, followed by calcination at 750 degrees C (0.5 wt.% Pt). The activity order of SO₄-MeOx was SO₄-TiO₂>SO₄-Al₂O₃>SO₄-Fe₂O₃>SO₄-SiO₂; the alumina catalyst gave the highest selectivity for the skeletal isomerization, i-C-4, 78% with 25% conversion at 220 degrees C under the pulse reaction conditions. No effect of the Pt addition was found when prepared by co-impregnation of metal oxides with sulfate and Pt materials. The effect of mixing with zeolites and silica-aluminas showed satisfactory conversions of butane. Mordenites were highly active, but the selectivity for i-C-4 was relatively low, similar to 50% at 200 degrees C. Silica-aluminas and other zeolites, ZSM-5 and Y-type, were also active at 200 degrees C-240 degrees C, but most of the products were cracked materials: methane, ethane, and propane. The selectivity for i-C-4 highly increased by sulfation of the silica-aluminas to >53%.

[31] SYNTHESIS AND CHARACTERIZATION OF NANOCRYSTALLINE 316-STAINLESS STEEL COATINGS BY HIGH VELOCITY OXY-FUEL SPRAYING

ML Lau, HG Jiang, EJ Lavernia - THERMAL SPRAY, VOLS 1 AND 2, 1998, pp 379-384 - 15TH INTERNATIONAL THERMAL SPRAY CONFERENCE; NICE, FRANCE. MAY 25-29, 1998

The present paper describes the synthesis of nanocrystalline 316-stainless steel coatings by high velocity oxy-fuel (HVOF) thermal spraying. The feedstock powders were synthesized by mechanical milling to produce flake-shaped agglomerates with an average grain size of less than 100 nm. The powders were introduced into the HVOF spray to successfully produce nanocrystalline coatings. X-ray diffraction analysis and transmission electron microscopy were used to determine the average grain size of the milled powders. Scanning electron microscopy and transmission electron microscopy were used to study the morphology of the nanometric particles and the microstructure of the as-sprayed coatings. The properties of various coating materials were characterized by microhardness measurements performed on the polished surface of the cross section.

[30] ELECTROCHEMICAL CHARACTERISTICS OF AN AMORPHOUS MG0.9V0.1NI ALLOY PREPARED BY MECHANICAL ALLOYING

Nohara S. Hamasaki K. Zhang SG. Inoue H. Iwakura C. - J. Alloys & Compounds. 280(1-2):104-106, 1998

Electrochemical characteristics of an amorphous MgNi alloy with Mg partially substituted by V were investigated. A Mg_{0.9}V_{0.1}Ni alloy prepared by mechanical alloying (MA) exhibited much better cycle life than MgNi alloy. It was found that the partial substitution of Mg in MgNi with V could suppress the formation of Mg(OH), on the alloy surface during the charge-discharge cycling in alkaline solution. This may have unveiled an important factor to improve cycle life of the Mg-based alloy for use in nickel-hydrogen batteries.

[29] A STUDY ON THE PREPARATION OF IRON ALUMINIUM BASED INTERMETALLIC ALLOY BY ALUMINOTHERMIC SMELTING TECHNIQUE

Chakraborty SP. Sharma IG. Bose DK. - Journal of Alloys & Compounds. 280(1-2):255-261, 1998

Iron aluminium based intermetallic alloys or iron aluminide alloys are usually made by conventional melting and powder processing routes. Melting is carried out either in vacuum induction melting or are melting furnaces. However, the use of high vacuum and high temperature expensive furnaces, high cost associated with the procurement of pure individual metal components, operational problems during melting stages are some of the most deterrent factors in making these alloys by melting route. Powder processing route utilises atomised, carbonyl or rapidly solidified pre alloyed powders as starting materials and alloying is carried out either by mechanical alloying or reaction synthesis or by consolidation through hot isostatic pressing. This process also involves many processing steps and considerable expense. In the present investigation, it has been found possible to eliminate the above problems by adopting a simple aluminothermic reduction (thermit) smelting technique for the preparation of iron aluminide alloy of targetted composition Fe-16Al-5.46Cr-0.5V-0.05C (wt %) by the direct co-reduction of oxides of iron, chromium and vanadium in presence of carbon and excess aluminium. Incorporation of slag fluidizer CaO amounting to 10 wt % of total charge, aluminium, 10 wt % excess over stoichiometric requirement and carbon, double the theoretical amount have been found to be adequate to form the above alloy with maximum overall yield

of 99.4 wt %. The compositional variation of alloying components Like iron, chromium, vanadium, aluminium and carbon in the aluminothermally produced iron aluminide alloy from the target composition have been minimized under optimum experimental conditions. The reactions have been found to be well controlled, rapid and self-sustaining and are conducted in an off-furnace manner using a simple experimental set-up. Studies on composition and microstructure characterization, hot rolling behaviour and oxidation resistance of the said alloy points towards its suitability for use.

[28] COBALT- AND COPPER-SUBSTITUTION EFFECTS ON THERMAL STABILITIES AND HYDRIDING PROPERTIES OF AMORPHOUS MGNI

Ikedo K. Orimo S. Zuttel A. Schlapbach L. Fujii H. - Journal of Alloys & Compounds. 280(1-2):279-283, 1998
Amorphous (a-)Mg(Ni_{1-x}T_x) with T=Co and Cu were formed by mechanical alloying in the composition range x=0-0.5. The crystallization temperatures increase/decrease by Co/Cu substitution, while the enthalpies of amorphous formation are little changed by substitution. On the other hand, the p-e isotherms indicate that the hydrogen-site energies become partially unstable with substitution. The relation between the enthalpy of amorphous alloy formation and subsequent hydride formation is discussed quantitatively on the basis of thermal analysis and the rule of reversed stability.

[27] DIRECT SYNTHESIS OF MG₂FEH₆ BY MECHANICAL ALLOYING

Huot J. Boily S. Akiba E. Schulz R. - Journal of Alloys & Compounds. 280(1-2):306-309, 1998
The hydride Mg₂FeH₆ was synthesized by high-energy ball milling of MgH₂ and Fe under argon atmosphere without subsequent sintering. After 60 h of milling, 56% wt. of Mg₂FeH₆ was synthesized. This yield was deduced from Rietveld analysis of the X-ray powder measurements and confirmed by pressured differential scanning calorimeter (PDSC). Hydrogen capacity measurements indicated that the loss of capacity with cycling is minimal.

[26] PREPARATION OF MG₂NI BASE ALLOY BY THE COMBINATION OF MECHANICAL ALLOYING AND HEAT TREATMENT AT LOW TEMPERATURE

Aymard L. Ichitubo M. Uchida K. Sekreta E. Ikazaki F. - Journal of Alloys & Compounds. 280(1-2):321, 1998

[25] MAGNETIC PROPERTIES OF NANOSCALE SM_{0.25}ZR_{0.75}FE₃ PRODUCED BY MECHANICAL ALLOYING

Seifu D. Oliver FW. Hoffman E. Aning A. Babu VS. Sehra MS. - J. Magnetism & Magnetic Materials. 189(3):305-309, 1998

Mechanical alloying of Sm, Zr, and Fe with the composition of Sm_{0.25}Zr_{0.75}Fe₃ was studied as a function of milling time up to 20 h using X-ray diffraction (XRD), Mossbauer spectroscopy, and magnetometry. In XRD, initial crystalline Bragg reflections due to Sm, Zr, and Fe changed to a single broad maximum after 20 h milling, and in Mossbauer spectroscopy, the initial sextet changed to a broad singlet. These results and the temperature and magnetic-field dependent studies of the 20 h milled sample showed the material to be a superparamagnet with considerable mechanical strains and a wide particle-size distribution with average particle size of about 33 Angstrom and blocking temperature of about 220 K. Comparison of these results with those in mechanically alloyed SmFe₃ shows that Zr accelerates the rate of mechanical synthesis.

[24] STRUCTURE AND MAGNETIC PROPERTIES OF PRFE_{11.5}-XVXTi_{0.5} COMPOUNDS AND THEIR NITRIDES

Tang SL. Yang CP. Wang BW. Jin XM. Zhang SY. Du YW. - Journal of Magnetism & Magnetic Materials. 189(3):341-345, 1998

Phase formation and magnetic properties of PrFe_{11.5-x}V_xTi_{0.5} compounds (x = 0.5-1.5) and their nitrides have been investigated by X-ray diffraction, differential thermometric analysis (DTA) and magnetic measurements. It is found that PrFe_{11.5-x}V_xTi_{0.5} compounds with ThMn₁₂-type structure can be formed by annealing the as-cast samples in a fairly wide temperature range. The substitution of V for Fe in the compounds results in a decrease of the saturation magnetization and Curie temperature. Upon nitrogenation, the Curie temperature is increased to around 739-763 K, the saturation magnetization increases by about 10%, and the anisotropy fields are all larger than 80 kOe. The PrFe_{11.5-x}V_xTi_{0.5} compounds have excellent intrinsic magnetic properties favorable for permanent magnet materials. The hard magnetic properties of PrFe_{10.5}V₁Ti_{0.5} magnets prepared by mechanical alloying technique have been assessed.

[23] MECHANOCHEMICAL SYNTHESIS OF PBTiO₃ FROM PBO AND TiO₂

Durovic D. Kostic E. Kiss SJ. Zec S. - Journal of Alloys & Compounds. 279(2):L 1-L 3, 1998

Data on mechanochemical synthesis of PbTiO₃ from PbO and TiO₂ without subsequent heating of activated mixtures are presented. Physical and chemical processes occurring during mechanical activation are followed by the X-ray diffraction (XRD) method, scanning electron microscope (SEM) analysis and measurement of BET specific surface area. It was shown that during vibro milling of 10 g oxide mixture, formation of PbTiO₃ was ended after 60 min. The progression of the chemical reaction was detected by the XRD method and chemical analysis.

[22] PREPARATION OF NITRIDES DISPERSED AL-TI ALLOY BY REACTIVE BALL MILLING IN N₂ GAS

Oh MS. Ilmoon K. Lee YS. - Journal of Alloys & Compounds. 279(2):201-208, 1998

The nitride dispersed Al-Ti alloys have been prepared by reactive ball milling (RBM) of elemental powders of Al and Ti in nitrogen gas. XRD and TEM analyses showed that when Al-10, 20 wt. % Ti elemental powder mixtures were ball milled in nitrogen gas, no nitrides could be observed in Al-Ti alloy powders. In order to accelerate the formation of Ti nitrides, when only Ti powder was milled in nitrogen gas at the first stage and then a second stage milling was carried out with as-milled Ti powder and elemental Al powder in the two-step RBM, TiN readily formed and dispersed in the Al matrix. The particle size and grain size were effectively reduced by the hard and brittle TiN and dissolved nitrogen atoms. High resolution TEM analysis showed that the TiN, having the grain size of 5 nm, existed between the Al matrix. The hardness of Al-Ti alloys milled in nitrogen gas was higher than that of Al-Ti alloys milled in Ar gas. It seems that the TiN is an effective dispersion strengthener in Al-Ti alloys. After annealing treatment at 500 degrees C, the hardness of Al-Ti alloys hardly decreased with annealing time and they exhibited a good thermal stability.

[21] PHASE TRANSFORMATION AND MAGNETIC PROPERTIES OF MECHANICALLY ALLOYED NDFE12-XMOXNY

Tang SL. Jin XM. Wang BW. Jin ZQ. Zhang SY. Du YW. - *J. Alloys & Compounds*. 279(2):229-232, 1998
The structure and permanent magnetic properties of isotropic powders of NdFe_{12-x}Mo_x (1 less than or equal to x less than or equal to 2) and their nitrides were investigated by using mechanical alloying. It was found that heat treatment of as-milled powders at 850 similar to 900 degrees C resulted in the formation of the Nd(Fe,Mo)(12) phase. The metastable Nd(Fe,Mo)(7) phase and a large amount of alpha-Fe were present at 800 degrees C. After nitrogenation, the volume expansion of NdFe_{12-x}Mo_x phases was about 3.5% and the Curie temperature of the samples was enhanced by 157 similar to 207 degrees C. In NdFe_{12-x}Mo_xN_y compounds the values of T_c fall in the range of 272 similar to 75 degrees C. It has been found that the intrinsic coercivities strongly increased with increasing Mo content. The maximum coercivity, H_{i(c)}=7.8 kOe, was achieved at 850 degrees C for the sample with x=2. The difference between the coercivities of the mechanically alloyed Nd(Fe,Mo)(12)N_y powders with different Mo content was attributed to their different crystallization behavior.

[20] MECHANICAL SOLID STATE MIXING FOR SYNTHESIZING OF SICP/AL NANOCOMPOSITES

Eleskandarany MS. - *Journal of Alloys & Compounds*. 279(2):263-271, 1998
High-energy ball milling was successfully employed to fabricate the metal matrix composite (MMC) of Al reinforced with SiC particulate (SiCp) with distinct nanocrystalline characteristics. The powders of the end-products (86 ks of milling time) of the mechanically-mixed nanocomposites SiC_xAl100-x (x=2, 5, 7, 10 vol. %) that have spherical-like morphology (less than 0.5 mu m in diameter) were consolidated into bulk compacts, using a plasma activated sintering method. Density measurements show that this consolidation step leads to the formation of fully dense bulk samples. Moreover, the consolidated SiC_xAl100-x samples reveal a nanocrystalline structure with a homogeneous reinforcement distribution of SiCp, in the Al matrix. Reactive products, i.e. Al₄C₃ and Si, could not be detected on the interfaces between the reinforcement and the matrix in both mechanically mixed powders and consolidated samples. The mechanically mixed powders and consolidated compacts have been characterized by means of X-ray diffraction, scanning and transmission electron microscopes, optical metallography and chemical analyses. The hardness and some mechanical properties of SiCp/Al have been determined as a function of SiCp content.

[19] EFFECTS OF PHASE COMPOSITION AND OF POTASSIUM PROMOTION ON COBALT MOLYBDATE CATALYSTS FOR THE SYNTHESIS OF ALCOHOLS FROM CO₂ AND H₂

Calafat A. Vivas F. Brito JL. - *Applied Catalysis A-General*. 172(2):217-224, 1998
Cobalt molybdate and cobalt molybdate-potassium catalysts were found to exhibit activity for alcohol formation from CO₂ and H₂ at 3.0 MPa and 250 degrees C and 300 degrees C. Tetrahedrally coordinated Mo⁶⁺ ions favor the selectivity to alcohols. Addition of K₂CO₃ greatly enhances the selectivity of the reaction to methanol. The extent of this effect was dependent on the way the alkali metal was mixed with the catalyst. Catalysts obtained by mechanical mixing of alpha-CoMoO₄ and K₂CO₃ deactivate faster than the beta-CoMoO₄/K mixture calcined at 550 degrees C, where formation of a K₂MoO₄ phase was detected by XRD. Both catalysts show a similar maximum in selectivity to alcohols at around 90 min of reaction. Addition of potassium by coprecipitation along with Co and Mo, which leads to the formation of K₂CO₂(MoO₄)₃, also enhances alcohol production, but the effect is more attenuated than for the other promoted samples.

[18] PHYSICAL MIXTURE OF CUO AND CR₂O₃ AS AN ACTIVE CATALYST COMPONENT FOR LOW-TEMPERATURE METHANOL SYNTHESIS VIA METHYL FORMATE

Ohyama S. Kishida H. - *Applied Catalysis A-General*. 172(2):241-247, 1998
Slurry-phase methanol synthesis via methyl formate was carried out at 373 K and an initial pressure of 5 MPa using alkoxide and a physical mixture of CuO and Cr₂O₃ as a catalyst. Methanol productivity was enhanced with prolonged milling of the mixture. Characterization of the mixtures by physical adsorption, X-ray diffraction and transmission electron spectroscopy suggested that long-term milling brought about a lattice disorder in the crystals of CuO and/or Cr₂O₃ as well as an enlarged surface area, which contribute to an increase in catalytic activity.

[17] IRON ALUMINIDE AL₂O₃ COMPOSITES BY IN SITU DISPLACEMENT REACTIONS - PROCESSING AND MECHANICAL PROPERTIES

Subramanian R. Mckamey CG. Schneibel JH. Buck LR. Menchhofer PA. - *Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing*. 254(1-2):119-128, 1998
Composites of an intermetallic alloy matrix (such as iron aluminide) and ceramic particle reinforcement (such as alumina) have potential for high-temperature structural applications requiring excellent oxidation resistance. In contrast to conventional processing methods (e.g. mechanical alloying), in situ processing techniques can be inexpensive and can also lead to unique microstructures, such as very fine dispersions of the reinforcements or interconnecting phases. The feasibility of processing Fe-Al alloy/Al₂O₃ composites by an in situ displacement reaction between Fe-40at.%Al and the iron oxide Fe₂O₃ was investigated and the results are presented in this paper. Specimens were prepared by hot pressing of the powder mixtures at 1300 degrees C. The final volume fractions of Al₂O₃ were determined by the initial ratio of Fe-40%Al to Fe₂O₃ and composites with 7 and 20 vol.% Al₂O₃ were investigated in this study. Compression creep tests performed at 700 degrees C showed that composites with 7 and 20 vol.% Al₂O₃ had higher compressive strengths than monolithic iron aluminide (Fe-28at.%Al). In particular, the compressive strength of the composite with 20 vol.% Al₂O₃ was three times that of the monolithic iron aluminide. Further increases in compressive strength, as well as significant improvements in bend and fracture strength, were obtained when the particle size of the starting Fe-40%Al powders was less than 10 mu m. Improved fracture toughness with the smaller Fe-40%Al powders could be attributed to the ductile failure of fine iron aluminide ligaments. The effect of Cr and Ti additions of up to 8 at.% on the strength of the interface between the iron aluminide and alumina phases was also investigated. The compressive strength of a composite processed with < 10-mu m diameter iron aluminide powders and 8 at.% Cr was the highest of all the specimens. However, within the range of compositions studied in this investigation, only slight improvements in the mechanical properties could be

obtained with these alloying additions.

[16] CHANGE OF STRUCTURE AND CATALYTIC PROPERTIES OF V₂O₅ UNDER ITS MECHANOCHEMICAL TREATMENT

Zazhygalov VO. Kharlamov OI. Bacherykova IV. Komashko GA. Khalameida SV. Bogutska LV. Byl OG. Stoch J. Koval A. - Teoreticheskaya i Eksperimental'Naya Khimiya. 34(3):180-183, 1998.

It has been established by means of X-ray diffraction. and tunnelling microscopy that the mechanochemical treatment of vanadium(V) oxide in ethanol medium using a planetary mill leads to not only crushing of its particles with specific surface area increase but also to the structure changing. Increase of the treatment time causes a growth (more than by 5 times) of the relative content of (010) vanadyl plane in the sample. XPS study shows partial reduction of vanadium ions on the surface. Modification of structure and composition of the surface of vanadium oxide leads to the change of its catalytic properties in the reactions of n-butane and benzene oxidation. An enhancement (about twice) of hydrocarbon conversion can be connected with the increase of the sample specific surface area that confirmed by the constant value of the specific rate of n-butane oxidation, An abrupt improvement in the selectivity towards maleic anhydride (by 6 and 3 times, respectively, at n-butane and benzene oxidation) is connected with the increase of the content of vanadyl plane and with an appearance of the groups containing vanadium in different oxidation state on the surface.

[15] MECHANOCHEMICAL REACTION BETWEEN SULFUR AND ILMENITE

Welham NJ. - Australian Journal of Chemistry. 51(10):947-953, 1998.

An ilmenite (FeTiO₃) concentrate has been milled with sulfur in a laboratory-scale ball mill for 100 h under a variety of conditions. X-Ray diffraction and thermal processing have shown that reaction occurs within the mill forming pyrite (FeS₂) and rutile (TiO₂). The reaction was of greatest extent in a mill run under 400 kPa of inert gas; some reaction occurred in a mill under 10(-2) Pa, whereas little reaction was observed when either water or air was present. Annealing of the product powders showed that reaction to TiO₂ and FeS₂ could be achieved in a closed system after 1 h at 400 degrees C but with the evolution of SO₂, whereas annealing at 800 degrees C also gave TiO₂ and FeS₂, but without formation of SO₂ which was thought to have disproportionated to form iron sulfate(VI). Closed-tube annealing of unmilled sulfur and milled ilmenite showed the same reactions as milled powders but of much greater extent.

[14] EFFECT OF BALL MILLING MATERIALS AND METHODS ON POWDER PROCESSING OF Bi2223 SUPERCONDUCTORS

Yavuz M. Maeda H. Vance L. Liu HK. Dou SX. - Superconductor Science & Technology. 11(10):1153-1159, 1998

Various milling systems consisting of agate and polypropylene grinding containers, agate and YSZ balls, and dry and wet milling were used in planetary ball-milling and YSZ balls and YSZ container were used in wet and dry attrition milling. The differently milled powders were then evaluated by measurements of particle size, surface area, porosity, size distribution and chemical analysis of the Si, Zr and C contents. The results show that dry milling is much more efficient for particle size reduction in planetary milling than wet milling, whereas wet milling and dry milling gave quite similar results in attrition milling. Meanwhile SiO₂ contamination was found in powder milled with an agate container with agate balls. Some C contamination from the polypropylene container was detected after milling, but negligible Zr from YSZ balls and C from the grinding carrier(hexane). It was found that after 1 h milling in the planetary mill fracture mechanisms transform from the elastic to the plastic region. Therefore, further milling is not very effective. it was also shown that the Bi2212 phase decomposes into several non-superconducting oxides such as Ca₂PbO₄, (Sr, Ca)(₂)CuO₃, CuO and a main amorphous phase after extensive dry milling.

[13] STRUCTURE AND ELECTROCHEMISTRY OF LiMO₂, (M = TI, MN, FE, CO, NI) PREPARED BY MECHANOCHEMICAL SYNTHESIS

Obrovac MN. Mao O. Dahn JR. - Solid State Ionics. 112(1-2):9-19, 1998

LiMO₂ (M = Ti, Mn and Fe) were prepared by ball milling mixtures of lithium transition metals and transition metal oxides. The resulting oxides were found to have the rocksalt structure with the transition metal and lithium ions randomly ordered in the cation sites, except for LiMnO₂ which had some short range cation order. LiCoO₂ and LiNiO₂ samples prepared at high temperature were also observed to transform to the disordered rocksalt structure after subsequent milling. However, during milling these samples lost lithia and oxygen to form Li_xM_{1-x}O (0.25 < x < 0.5). The electrochemical performance of the milled samples as cathodes in lithium batteries was poor, presumably due to the disorder of the cation lattice. However, mechanochemical synthesis may prove to be a useful synthesis step in the preparation of novel active cathode materials.

[12] MAGNETIC HARDENING OF MECHANICALLY ALLOYED NdFe₁₂-xVx (1-LESS-THAN-OR-EQUAL-TO-X-LESS-THAN-OR-EQUAL-TO-2) AND THEIR NITRIDES

Tang SL. Wu CH. Jin XM. Wang BW. Li GS. Ding BZ. Chuang YC. - Journal of Magnetism & Magnetic Materials. 189(2):202-206, 1998

The structure and permanent magnetic properties of isotropic powders of NdFe₁₂-xV_x compounds were investigated by using mechanical alloying. It was found that heat treatment of as-milled powders at 700-900 degrees C resulted in the formation of Nd(Fe,V)(₁₂) phase except for the sample with x = 1, for which the crystallization was not complete and a large amount of alpha-Fe(V) was present at temperatures lower than 750 degrees C. After nitrogenation, the volume expansion was about 5% and the Curie temperature enhancements were 140 to 160 degrees C. In NdFe₁₂-xV_x the values of T_c fall in the range 469-485 degrees C. It has been found that the intrinsic coercivities strongly increased with increasing V content. The maximum coercivity of H_i(c) = 10.96 kOe is achieved at 750 degrees C with x = 2.00. The difference between the coercivities of the mechanical alloyed Nd(Fe,V)(₁₂)N-delta powders with different V content is attributed to their different crystallization behavior and phase stability during nitrogenation.

[11] PHYSICAL MIXTURE OF CUO AND CR₂O₃ AS AN ACTIVE CATALYST COMPONENT FOR LOW-

TEMPERATURE METHANOL SYNTHESIS VIA METHYL FORMATE

Ohyama S. Kishida H. - Applied Catalysis A-General. 172(2):241-247, 1998

Slurry-phase methanol synthesis via methyl formate was carried out at 373 K and an initial pressure of 5 MPa using alkoxide and a physical mixture of CuO and Cr₂O₃ as a catalyst. Methanol productivity was enhanced with prolonged milling of the mixture. Characterization of the mixtures by physical adsorption, X-ray diffraction and transmission electron spectroscopy suggested that long-term milling brought about a lattice disorder in the crystals of CuO and/or Cr₂O₃ as well as an enlarged surface area, which contribute to an increase in catalytic activity.

[10] INVESTIGATION OF THE EFFECT OF PARTICLE SIZE ON THE 10 GHZ MICROWAVE PROPERTIES OF BA1-XSRXTIO3/MGO COMPOSITE CERAMICS

Synowczynski J. Sengupta LC. Chiu LH. - Integrated Ferroelectrics. 22(1-4):861-872, 1998

The objective of this study is to determine the effect of milling on the X-Band microwave properties of Ba_{0.6}Sr_{0.4}TiO₃/MgO {wt% MgO = 0 to 60 wt%} composites. At low frequency, the effect of the reduction of the overall particle size of the materials from attritor milling is dependent on the additive oxide content; for samples with oxide content > 5.0 wt%, the dielectric constant, and tunability (%) are decreased. Whereas, milled Ba_{0.6}Sr_{0.4}TiO₃/MgO [1] {wt% MgO = 0 to 1 wt%} composites exhibit low loss tangents, higher tunabilities, and higher dielectric constants than unmilled samples. The present study is focused on the effect of milling on the dielectric properties of Ba_{0.6}Sr_{0.4}TiO₃/MgO composites at microwave frequency. The samples were evaluated in a 10 GHz resonator cavity. Additionally, the effect of barium content and MgO content on the 10 GHz dielectric properties of Ba_xSr_{1-x}TiO₃ (x = 0.45, 0.50, 0.55, 0.60)/MgO (MgO = 1 - 60 Wt%) are investigated. These results are correlated with relevant material characterization techniques including FT-Raman studies.

[9] INVESTIGATION OF THE EFFECTS OF MILLING CERAMIC TARGETS ON THE OPTICAL AND ELECTRICAL PROPERTIES OF BST THIN FILMS DEPOSITED BY RF SPUTTERING

Stowell SC. Zhu YF. Zhang XB. Sengupta S. Sengupta LC. Hsieh AJ. - Integrated Ferroelectrics. 21(1-4):441-449, 1998

The RF sputtering method was utilized to deposit thin films of Ba_{1-x}Sr_xTiO₃ (BST). The targets utilized in these experiments were prepared from ceramic powders with different particle size. The goal of this work is to examine whether the particle size distribution of the target can affect the properties of the thin films fabricated by the sputtering method. The Atomic Force Microscope (AFM) was used to examine the grain size in the thin films. The composition of the thin films and the bulk materials were examined by Fourier Transform Infrared (FTIR) spectroscopy. The dielectric properties of the thin films were measured and compared to its bulk counterparts. It was found that on lattice matched electrodes of SrRuO₃ on LaAlO₃ substrates, the thin films deposited from ceramic targets manufactured from ball-milled powders had finer grain size than those deposited from targets made from unmilled powders. However, this phenomenon was not observed in the case of polycrystalline films deposited on platinized silicon wafers.

[8] IRON ALUMINIDE AL₂O₃ COMPOSITES BY IN SITU DISPLACEMENT REACTIONS - PROCESSING AND MECHANICAL PROPERTIES

Subramanian R. Mckamey CG. Schneibel JH. Buck LR. Menchhofer PA. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 254(1-2):119-128, 1998

Composites of an intermetallic alloy matrix (such as iron aluminide) and ceramic particle reinforcement (such as alumina) have potential for high-temperature structural applications requiring excellent oxidation resistance. In contrast to conventional processing methods (e.g. mechanical alloying), in situ processing techniques can be inexpensive and can also lead to unique microstructures, such as very fine dispersions of the reinforcements or interconnecting phases. The feasibility of processing Fe-Al alloy/Al₂O₃ composites by an in situ displacement reaction between Fe-40at.%Al and the iron oxide Fe₂O₃ was investigated and the results are presented in this paper. Specimens were prepared by hot pressing of the powder mixtures at 1300 degrees C. The final volume fractions of Al₂O₃ were determined by the initial ratio of Fe-40%Al to Fe₂O₃ and composites with 7 and 20 vol.% Al₂O₃ were investigated in this study. Compression creep tests performed at 700 degrees C showed that composites with 7 and 20 vol.% Al₂O₃ had higher compressive strengths than monolithic iron aluminide (Fe-28at.%Al). In particular, the compressive strength of the composite with 20 vol.% Al₂O₃ was three times that of the monolithic iron aluminide. Further increases in compressive strength, as well as significant improvements in bend and fracture strength, were obtained when the particle size of the starting Fe-40%Al powders was less than 10 μm. Improved fracture toughness with the smaller Fe-40%Al powders could be attributed to the ductile failure of fine iron aluminide ligaments. The effect of Cr and Ti additions of up to 8 at.% on the strength of the interface between the iron aluminide and alumina phases was also investigated. The compressive strength of a composite processed with < 10-μm diameter iron aluminide powders and 8 at.% Cr was the highest of all the specimens. However, within the range of compositions studied in this investigation, only slight improvements in the mechanical properties could be obtained with these alloying additions.

[7] SYNTHESIS AND CHARACTERIZATION OF NANOSIZED NiAl PARTICLES

T Chen, JM Hampikian - NANOSTRUCTURED POWDERS AND THEIR INDUSTRIAL APPLICATIONS (Series: MATERIALS RESEARCH SOCIETY SYMPOSIUM PROCEEDINGS), 1998, Vol 520, pp 205-210

Nanophase NiAl powders were synthesized by solid state reaction via ball milling of elemental Ni and Al powders under an argon atmosphere for 0-8 h, 16 h, 24 h, and 48 h. Structural characterization of the nanophase NiAl was performed by x-ray diffraction and transmission electron microscopy. The oxidation behavior of the powders was studied by thermogravimetric analysis. It was found that the Ni + Al = NiAl solid state reaction took place between 4 and 5 hours of mechanical alloying. After 5 h or more of mechanical alloying, the phase of the ball milled particles was the B2 structure, with average grain sizes which decreased with increasing mechanical alloying times. The relationship between the long-range-order parameters and mechanical alloying time was studied. After 5 h, 6 h, 8 h, 16 h, 24 h, and 48 h mechanical alloying, the long-range-order parameter was determined to be approximately 0.82,

0.75, 0.75, 0.75, 0.71, and 0.63, respectively. Iron contamination was observed, resulting from wear of the steel vial and balls. In the mechanically alloyed NiAl nanoparticles, edge dislocations, shear bands, subgrains, distorted regions, and a large number of grain boundaries were observed.

[6] PRODUCING CLAY-COATED QUARTZ POWDERS FOR SEMIDRY PRESSING OF WALL CERAMICS

GN Kryukova, PA Simonov, GI Storozhenko, GV Boldyrev - NANOSTRUCTURED POWDERS AND THEIR INDUSTRIAL APPLICATIONS (Series: MATERIALS RESEARCH SOCIETY SYMPOSIUM PROCEEDINGS), 1998, Vol 520, pp 233-238

This paper discusses the potential of mechanochemical activation of low grade raw materials with carbonaceous inclusions to achieve high-quality building ceramics. The procedure proposed enabled us to produce clay-coated quartz powders thus facilitating further processing steps of semidry pressing and firing of the bricks, tiles and flagstones.

[5] GRAIN GROWTH OF NANOCRYSTALLINE CRYOMILLED FE-AL POWDERS

Perez RJ. Jiang HG. Dogan CP. Lavernia EJ. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 29(10):2469-2475, 1998

Nanocrystalline Fe-xAl (x = 0, 2.6, or 10 wt pct) powders have been prepared using cryogenic mechanical alloying (cryomilling). The extremely low rate of diffusion of Al in Fe at the process temperature (-196 degrees C) effectively limits dissolution of Al in the nanocrystalline alpha Fe grains. Thermal stability against grain growth in the cryomilled Fe-Al mixtures is found to be much greater than that of identically prepared pure nanocrystalline Fe. Heat treatment of the cryomilled Fe-Al materials results in a highly inhomogeneous distribution of grain size and microchemistry. The observed thermal stability is evaluated in terms of Zener pinning, solute drag, and chemical ordering mechanisms.

[4] CRYSTALLIZATION AND DENSIFICATION OF NANO-SIZE AMORPHOUS CORDIERITE POWDER PREPARED BY A PVA SOLUTION POLYMERIZATION ROUTE

Lee SJ. Kriven WM. - Journal of the American Ceramic Society. 81(10):2605-2612, 1998

A homogeneous and stable amorphous-type cordierite ($2\text{MgO} \cdot 2\text{Al}_2\text{O}_3 \cdot 5\text{SiO}_2$) powder was prepared by a solution-polymerization route employing a Pechini resin or a poly-(vinyl alcohol) (PVA) solution as the polymeric carrier. After calcination at 800 degrees C for 1 h under atmospheric conditions, the bulky precursor changed into a very soft and porous powder. A 30 nm size, amorphous-type cordierite powder was prepared by attrition milling the calcined powder, which was made using a PVA precursor solution. The nano-size powder, which had a high specific surface area of 181 m²/g, was obtained after milling for <1 h. The sintered cordierite grains did not show the presence of any amorphous SiO₂ phase and had a dense microstructure with a relative density of 99% and a thermal expansion coefficient of 2.1×10^{-6} /degrees C.

[3] NMR STUDIES ON MIXING OF INSOLUBLE CONSTITUENTS DURING HIGH ENERGY MILLING

Shaw LL. Xie XQ. Ren RM. Yang ZG. - Scripta Materialia. 39(9):1169-1175, 1998

[3] SURFACE PROPERTIES OF MECHANOSYNTHESIZED AL₂O₃-CR NANOCOMPOSITE POWDERS

Guichard JL. Mocellin A. Simonnot MO. Remy JF. Sardin M. - Powder Technology. 99(3):257-263, 1998

Various Al₂O₃-Cr nanocomposite powders were obtained by high energy milling. Measurements have been made of particle size distribution, zeta potential and acid-base properties on samples which differ by milling conditions or composition. The influence of each factor is assessed. Titration studies show that the composite product has to be handled around neutrality when dispersed in water (slight ionic strength, pH about 5.5-6) because of possible chemical reactions, particularly chromium dissolution which can occur in acidic or basic solutions. Under these conditions, composite powders behave like the pure oxide and could be processed in similar ways.

[2] FORMATION AND CATALYTIC ACTIVITY OF AMORPHOUS Ni₅₀Pd₄₀Si₁₀ ALLOY POWDER BY MECHANICAL ALLOYING

Zhang HF. Li J. Song QH. Hu ZQ. - Journal of Materials Research. 13(10):2779-2782, 1998

Amorphous Ni₅₀Pd₄₀Si₁₀ alloy powder was prepared by mechanical alloying. The surface states and catalytic activities of amorphous and crystalline Ni₅₀Pd₄₀Si₁₀ alloy powder pretreated with hydrofluoric acid (HF) solution were studied. The results show that amorphous Ni-Pd-Si alloy powder was easily prepared by mechanical alloying. After treating with HF solution, the number of Pd atoms on the surface of amorphous Ni-Pd-Si powder was obviously higher than that of the crystalline alloy and catalytic activity of amorphous Ni-Pd-Si powder was higher than that of the crystalline one by three times.

[1] POROUS NITIT ALLOY PREPARED FROM ELEMENTAL POWDER SINTERING

Li BY. Rong LJ. Li YY. - Journal of Materials Research. 13(10):2847-2851, 1998

An elemental powder sintering (EPS) technique has been developed for the synthesis of porous NiTi alloy, in which Ni and Ti powders are used as the reactants and TiH₂ powder is added as a pore-forming agent and active agent. Effects of various experimental parameters (sintering temperature, sintering time, and TiH₂ content) on the porosity, pore size, and pore distribution as well as phase composition in experimental alloys are investigated. It is found that in order to avoid the formation of carcinogenic pure Ni phase, the porous NiTi alloy should be synthesized over a temperature of 1223 K. This gives NiTi as the main phase without any elemental phase. Substitution of Ti by TiH₂ is more economic and more favorable to obtain homogeneous porous NiTi alloy. A proper selection of initial powders, ball-milling, pressing, and sintering process makes it possible to achieve the porous NiTi alloy with desired properties.

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