



**RESEAU FRANÇAIS DE
MECANOSYNTHESE**

Lettre N°96

Mars 2003

**189 Groupes de Recherche
(dont 115 à l'étranger / 34 Pays)**

**Bureau du RFM : E. Gaffet (Président)
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Bulletin d'adhésion 2003 / Subscription Print

(à retourner à l'adresse suivante - to be sent at the following address) :

Eric GAFFET

CNRS UMR5060 « Métallurgies et Cultures »

Nanomaterials Research Group

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Nom/Name :Prénom / First Name :

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désire adhérer au Réseau Français de Mécanosynthèse / want to become a member of the French Mechanical Alloying Network

Chèque ci joint / Check enclosed in the amount of **20 Euros (20€)**

The check has to be to the order to : Réseau Français de Mécanosynthèse

Le site web du RFM est :

<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'2001 et quelques éléments mis à jour régulièrement concernant les derniers résultats dans ce domaine.

Lettre RFM N°96 - Mars 2003

Corresp. : <mailto:Eric.Gaffet@utbm.fr>

Ph'D Thesis

Synthesis of Nanoparticulate Zirconia by Mechanochemical Processing

Aaron Dodd,

Department of Mechanical and Materials Engineering,

University of Western Australia, Nedlands, WA 6009 Australia

Abstract

This thesis presents the results of an investigation into the synthesis of ultrafine zirconia powders by mechanochemical processing. The purpose of this investigation was to identify different reaction mechanisms and to establish the effects of process variables on the properties of the final zirconia powders, specifically the average particle size and phase content.

All of the reaction systems that were investigated in this study involved reaction of anhydrous chloride precursors with an exchange reagent. However, despite this fundamental similarity, the systems were found to exhibit significantly different reaction kinetics. Depending on the exchange reagent used and the presence of inert diluent, chemical reaction of the precursors either occurred gradually during milling, by mechanically activated combustion, or only during post-milling heat-treatment.

Reaction of the $ZrCl_4 + 2MgO$ system occurred gradually during milling and resulted in the formation of ultrafine ZrO_2 particles embedded within a matrix of $MgCl_2$. This allowed ultrafine ZrO_2 powders to be synthesised by either milling the precursors until complete reaction was achieved or by milling for a short duration and then driving the reaction to completion by low temperature heat treatment.

The average particle size of the ZrO_2 product formed by reaction of $ZrCl_4 + 2MgO$ was found to decrease with milling time down to a lower limit. In contrast, the addition of $MgCl_2$ diluent to the reactant mixture was found not to have any significant effect on the average particle size. The effect of milling duration and dilution is consistent with previous studies that suggest the average particle size is determined by the effective reaction volume.

Reactant mixtures that used Li_2O as the oxide exchange did not undergo chemical reaction during milling. Milling merely resulted in amorphisation of the $ZrCl_4$ and overall microstructural refinement. Chemical reaction, with the consequent formation of ZrO_2 and $LiCl$, only occurred during post-milling heat-treatment. The mechanism of chemical reaction during heat-treatment was found to depend on the applied heating rate and also on the presence of $LiCl$ diluent in the reactant mixture.

The addition of $LiCl$ diluent to the Li_2O reaction systems was found to decrease the average crystallite size and increase the proportion of single crystal particles in the final ZrO_2 product. For undiluted reactant mixtures, the average particle and crystallite size was found to be independent of the milling duration. In contrast, for diluted reactant mixtures, the average particle and crystallite size decreased with milling time down to a lower limit. The effect of milling duration and dilution is attributable to the high enthalpy change associated with formation of the reaction products, which allowed particle growth to occur during reaction.

The decrease in average particle size with dilution was accompanied by an increase in the tetragonal volume fraction, which is consistent with the view that the stability of the tetragonal phase in nanoparticulate ZrO_2 is due to the lower specific surface energy of the tetragonal phase compared to the monoclinic.

Reaction of the $ZrCl_4 + 4LiOH$ system occurred in a combustive manner following approximately 1 minute of milling and resulted in the formation of highly agglomerated ZrO_2 particles. However, dilution of the reactant mixture with $4LiCl$ suppressed combustive reaction and reaction during milling resulted in the formation of a hydrated gels with an amorphous structure. This allowed ZrO_2 particles to be synthesised either by milling until complete reaction or by combining a short milling time with subsequent low temperature heat treatment.

The average particle size and degree of agglomeration of the ZrO_2 formed by the diluted $LiOH$ reaction system was found to show a strong dependence on the milling duration. The average particle and crystallite size initially decreased with milling as a consequence of increased microstructural refinement of



the reactants. Further milling beyond this initial stage resulted in aggregation of the particles before the attainment of a steady-state particle size.



Post Doc Proposal

Titre : Postdoctoral research fellowship on the elaboration of nanomaterials by supercritical fluid processing – Bordeaux (France)

Contact : <mailto:cansell@icmcb.u-bordeaux.fr>

The Institut of Condensed Matter Chemistry of Bordeaux (ICMCB) proposes a post-doctoral research fellowship. ICMCB (200 people) is a French laboratory of CNRS (French National Center for Scientific Research) with research activities in Solid state chemistry, Material science and Molecular sciences.

The research project concerns the elaboration of nanomaterials by supercritical fluid processing in using a new synthesis reactor equipped with an in situ analysis system by fluorescence spectroscopy. The aim consists to synthesize ferroelectric nanomaterials with controlled size and surface properties. In particular, we plan to study the nanoparticle size evolution as a function of the synthesis process working conditions (Pressure, Temperature, residence time,...) by means of fluorescence spectroscopy. The ferroelectric properties of the obtained nanomaterials will be studied and a correlation between nanoparticle size, nanoparticle surface properties and material ferroelectric properties will be established.

The post-doctoral student will work with two well known teams of ICMCB and so, will dispose of a very important human and technical potential.

The post-doctoral student must have a good expertise in material science, more precisely, in material or nanomaterial synthesis and characterization and in material surface characterization.

The postdoctoral fellowship is supported by CNRS (2050 euros per month) for 12 or 18 months.



Congress and School Announcements

J2IM 2003

Vogüé, ARDECHE (France)
19-21 mai 2003

<http://www.emse.fr/fr/actualites/j2im2003.html>

The 10th Int. Symposium on Metastable, Mechanically Alloyed and Nanocrystalline Materials,

ISMANAM 2003,

**will be held in Foz do Iguacu,
Brazil, on 24-28 August 2003**

<mailto:ismanam2003@dema.ufscar.br>
<http://www.dema.ufscar.br/ismanam2003>

**International Conference
NANOMATERIALS AND NANOTECHNOLOGIES (NN 2003),
Crete, Greece; August 30 - September 6, 2003**

<http://www.ipme.ru/ipme/conf/NN2003/>

**INTERNATIONAL CONFERENCE
"Novel Technologies in Powder Metallurgy and Ceramics"**

September 8-12, 2003
Kiev, Ukraine

**Fourth INTERNATIONAL CONFERENCE
ON MECHANOCHEMISTRY AND MECHANICAL ALLOYING
4th INCOME 2003**

Technical University of Braunschweig, Braunschweig, Germany
September 7-11, 2003

Website : <http://www.tu-bs.de/INCOME2003>

11th FORESIGHT CONFERENCE ON MOLECULAR NANOTECHNOLOGY

October 9-12, 2003
San Francisco Airport Marriott
Burlingame, CA, USA

<http://www.foresight.org/Conferences/MNT11>

XV. International Symposium on Reactivity of Solids:

Nov. 9. - 13. 2003

Website : <http://www.ISRSKYOTO.org/>
<mailto:info@ISRSKYOTO.org>



J2IM 2003
Vogüé, ARDECHE
19-21 mai 2003

<http://www.emse.fr/fr/a>

PROGRAMME

... sous réserve de modifications de dernière minute :

Lundi 19 mai 2003 :

à partir de 10h30 : accueil au Village des Vacances Vogüé
vers 11h15 : départ du car de Montélimar (heure approximative ; sera ajustée en fonction des horaires SNCF)
vers 12h30 : arrivée du car au VVF

13h00 : déjeuner

Session 1 : lundi 19 mai, après-midi

14h45 : **Exposé invité : Yannick CHAMPION (CECM, CNRS, Vitry)**
Les joints de grains dans les nanomatériaux et propriétés nouvelles
15h45 : **Claude MONTY (CNRS, IMP, Font Romeu)**
**Rôle des joints de grains dans la conductivité ionique de
nanomatériaux de zircone yttrée**
16h15 : pause - café
16h45 : **Présentation des posters**
17h30 : **Session posters**
18h30 : Table ronde : **Quel avenir pour le groupe J2IM ?**
20h00 : dîner

Session 2 : mardi 20 mai, matin

9h00 : **Exposé invité : Jean-Luc MAURICE (CNRS/Thales, Orsay)**
Interfaces en microélectronique et spintronique
10h00 : **Abdekebir LAMZATOUAR (CP2M, Marseille) :**
**First correlation between structure, segregation and electrical activity in
Ge(S) system: case of $\Sigma=51$ tilt grain boundary**
10h30 : pause - café
11h00 : **Pierre BASTIE (Laboratoire de Spectrométrie Physique, Grenoble)**
**La transition α - β du quartz : Nature de l'interface entre les phases α et
incommensurable à la transition du premier ordre**

12h00 – 16h30 :
déjeuner servi sous forme de paniers-repas
départ du car pour une visite (les détails seront définis ultérieurement)



Session 3 : mardi 20 mai, après-midi

- 17h00 : *Olivier HARDOUIN DUPARC (LSI, Ecole Polytechnique, Palaiseau)*
Développement contradictoire des concepts d'interfaces dans les matériaux dans les années 20
- 17h30 : **Exposé invité** : *Michel BISCONDI (ENSMSE, St-Etienne)*
Joints de grains et changements d'échelle : un défi !
- 18h30 : **Table ronde**
animation : Didier BLAVETTE (Institut des Matériaux, Rouen)
- 20h00 : dîner "de gala"

Session 4 : mercredi 21 mai matin

- 9h00 : **Exposé invité** : *Pierre COMBRADE (FRAMATOME, Le Creusot)*
Rôle de l'interface métal/film d'oxyde en corrosion sous contrainte
- 10h00 : *Jean-Philippe MONCHOUX (CRMC2, CNRS, Marseille)*
Thermodynamique et cinétique des interfaces dans le système Fe-Pb
- 10h30 : pause – café
- 11h00 : *Jean-Philippe COUZINIE (CECM, CNRS, Vitry)*
Interactions entre dislocations et joints de grains. Comparaison entre métaux à forte et à faible énergie de faute d'empilement
- 11h30 : *Jérôme CREUZE (LURE, Orsay)*
Ségrégation interfaciale : vers une modélisation thermodynamique de la rupture
- 12h00 : **Bilan de la réunion**
Alain THOREL (ENSMP, Centre des Matériaux, Evry)
- 12h30 : déjeuner
- 14h30 : départ du car pour Montélimar



ISMANAM 2003

SECOND ANNOUNCEMENT (February 2003)

Please visit our home-page for all updated information:

<http://www.dema.ufscar.br/ismanam2003/>

Scope

The International Symposium on Metastable, Mechanically Alloyed and Nanocrystalline Materials 2003 (ISMANAM-2003) is the eleventh Symposium in the annual ISMANAM series. Materials classes to be covered in the Symposium include metastable crystalline, quasicrystalline and amorphous phases and nanostructures, including thin films, nanocrystalline materials and nanosized particles. All the main aspects of these materials will be covered, including synthesis, microstructure, properties and performance. As a forum for novel ideas, the symposium is intended to promote contact between basic research and technological needs for industrial applications.

Topics

- 1) Synthesis and Processing
- 2) Nanocrystalline materials
- 3) Bulk metallic glasses
- 4) Mechanochemistry
- 5) Mechanisms and Kinetics
- 6) Characterization
- 7) Thermodynamics and Modelling
- 8) Properties (chemical, catalytic magnetic, electrochemical, mechanical, optical and others)
- 9) Technological Applications

The conference language is English.

Deadlines

April, 15th, Abstract submission

May, 15th, Abstract acceptance

June, 15th, Early Registration

August, 24th, Manuscript deadline

Abstracts

Abstract submission form can be downloaded from our home-page.

<http://www.dema.ufscar.br/ismanam2003/>

Please note, the abstract submission deadline is April, 15th 2003

Manuscripts

Three copies of each manuscript are to be submitted upon arrival at the conference. Instructions for preparation of manuscripts will be sent upon confirmation of acceptance of your abstract. All the papers will be refereed for publication in *Mat.Sc.Forum*.

Proceedings

The proceedings of the conference will appear as a special issue of the international journal *Materials Science Forum*. All the papers will be refereed according to the standards of the journal and will be published by Trans Tech approximately 03 months after the conference. The acceptance of the presentation does not constitute acceptance of the paper for publication. Contributions that have already been or are to be published elsewhere will not be accepted for publication in the proceedings.



Registration

Please complete and submit a separate registration form for each participating person.
Registration form can be also downloaded from <http://www.dema.ufscar.br/ismanam2003/>

Payment method

Payment in advance can be either by direct bank transfer or by cheque.
Cash or travellers cheque can be also accepted for payment at the Conference site.
Accepted currencies: Reais, Euros or USA dollars.
Payment by credit card (VISA) will also be possible and details will be given soon.

Payment by Direct Bank Transfer

Must in name of: **FAI**
Banco do Brasil
Agency number: 1888-0
Account number: 5.996-X
Identifying code: 9.111-1

Payment by Cheques

Cheques payable to: **FAI**, to be sent to the following address:
ISMANAM 2003
Universidade Federal de São Carlos
Departamento de Engenharia de Materiais
Caixa Postal 676
13.565-905 São Carlos
Brazil

Organiser's Contact Details

Symposium Web Site: <http://www.dema.ufscar.br/ismanam2003/>
e-mail: ismanam2003@dema.ufscar.br

Mailing address:

ISMANAM 2003
Universidade Federal de São Carlos - UFSCar
Departamento de Engenharia de Materiais - DEMa
Caixa Postal 676
13565-905 São Carlos – SP, Brazil



General Schedule of ISMANAM Program

	Sunday 24 august	Monday 25 august	Tuesday 26 august	Wednesday 27 august	Thursday 28 august	
MORNING	Registration	Plenary and oral sessions	Plenary and oral sessions	Plenary and oral sessions	Plenary and oral sessions	
		Lunch	Lunch	Lunch	Closing Remarks Lunch	
AFTERNOON		Plenary and oral sessions	Conference Excursion Iguaçu Falls	Plenary and oral sessions		
		Poster Session		Poster Session		
EVENING		Welcome Party and Supper	Dinner	Dinner	Banquet	

Confirmed invited speakers

J.H. Ahn, Andong National University Korea
M.D. Baro, Universitat Autònoma de Barcelona, Spain
M. Baricco, Università di Torino, Italy
R. Bormann, GKSS Research Centre, Germany
A. Calka, University of Wollongong, Australia
B. Cantor, University of York, UK
J. Eckert, IFW Dresden, Germany
S.Enzo, [Università' degli Studi di Sassari](#) Italy
H. Fecht, University of Ulm, Germany
E. Gaffet, Nanomaterials group, CNRS UPR, France
X.B. Gelibert, Universitat Autònoma de Barcelona, Spain
A. Inoue, Tohoku University, Japan
C.C. Koch, North Carolina State University, USA
U. Köster, Universität Dortmund, Germany
A. Kvik, European Synchrotron Radiation Facility, France
E. Ma, The Johns Hopkins University, Baltimore, USA
P.G. MacCormick, Advanced NanoTechnologies, Welshpool, Australia
S. Mathur, Saarland University, Saarbruecken, Germany
D.G. Morris, CENIM-CSIC, Madrid, Spain
M. Pardavi-Horvath, The George Washington University, USA
J. Perepezko, University of Wisconsin, Madison, USA
R.B. Schwarz, Los Alamos National Laboratory, USA
L. Spinu, AMRI & Department of Physics, University of New Orleans, USA
L. Takacs, University of Maryland, Department of Physics, USA
A.R. Yavari, Institut National Polytechnique de Grenoble, France
X.X. Zhang, The Hong Kong University of Science and Technology, Hong Kong



Proposed Schedule for accompanying person's program

	Sunday 24 august	Monday 25 august	Tuesday 26 august	Wednesday 27 august	Thursday 28 august
MORNING	Registration	Iguaçu Falls / Argentina side Nacional Park	Free program	Itaipú Hydroelectric	Free program
		Lunch	Lunch	Lunch	Lunch
AFTERNOON		Shopping and Duty Free Argentina side	Conference Excursion Iguaçu Falls	Shopping Paraguai side	
EVENING	Welcome Party and Supper	Dinner	Dinner	Banquet	

Conference Site

Foz do Iguacu is located in the southwestern corner of Paraná State, near the borders of Paraguay and Argentina. Foz do Iguacu lies on the Paraná River near its confluence with the Iguacu River. Most of the local economy is based on tourism; an international airport is also located in the city. The main attraction is the spectacular Iguacu Falls, which is divided into 275 cascades, spanning over four kilometres, with a drop of more than 80 metres.

In the Guarani Indian language Foz do Iguacu translates: "great water."

Visit Foz do Iguacu Web Site: <http://www.fozdoiguacu.pr.gov.br/turismo>

Travel information

The most convenient way to reach **Foz do Iguacu** (Iguassu Falls) is to arrive at São Paulo airport (Guarulhos International Airport, GRU).

From Europe: Flight time from Europe to São Paulo is just over eleven hours depending on your starting point. Most flights leave from Europe in the evening and arrive early morning in São Paulo. On the return leg they leave late afternoon or early evening to get to Europe the following day around lunchtime or earlier. Air France, Alitalia, British Airways, Iberia, KLM, Lufthansa, Swissair, TAM, TAP and Brazilian flag carriers VARIG and TAM all run regular services between Europe and São Paulo. This means that travelers have a choice of several different airlines linking the two continents daily.

From the US: Flights by American Airlines, Continental, Delta, United and the Brazilian flag carriers VARIG and TAM link the US with Brazil Flight time is eight and a half hours from Miami to São Paulo, just over nine from New York, eleven from Atlanta, thirteen from Los Angeles and sixteen from San Francisco. The vast majority of flights between North America and Brazil are overnight flights that arrive in Brazil in the early morning.

From Latin America: São Paulo receives daily flights from most of the major Latin American cities and is served by Aerolineas Argentinas, Aero Peru, Avianca, Cubana de Aviacion, Ladeco, Lan Chile, LAB, Pluna, Viasa, and the Brazilian flag carriers VARIG, TAM and VASP.

From Asia: São Paulo receives also daily flights from many cities from Asia; usually the flights are via Europe (e.g., Frankfurt, London, Amsterdam) or via US (e.g. Los Angeles or New York) and flight time depends on your starting point. The following companies make regular flights to / from São Paulo: JAL, Nippon Airways, Korean Air, United Airlines, Lufthansa, Air France and VARIG.

You can then fly from the same arriving airport in São Paulo (Guarulhos International Airport, GRU) to Foz do Iguacu according to the following timetable



Flights from São Paulo (GRU) to Foz do Iguaçu (Cataratas, IGU).

Company	Flight	Frequency	Departing	Arriving
VASP	4261	Daily	11:45	13:25
VARIG	2251	Daily	12:50	14:25
TAM	3557	Daily	13:05	14:30
VARIG	2148	Daily	21:45	23:20

Flights from Foz do Iguaçu (Cataratas, IGU) to São Paulo (GRU).

Company	Flight	Frequency	Departing	Arriving
VARIG	2149	Daily	06:30	08:20
VARIG	2250	Daily	15:30	17:00
TAM	3556	Daily	16:00	17:35
VASP	4260	Daily	17:00	18:25

Pos-conference tours

The ISMANAM's official travel agency (Pinhal Turismo - mario@pinhalturismo.com.br) can make the necessary arrangements for Pos-Conference Tours.

We present below some suggested tours and packages. For those who are interested, it is recommended to contact Pinhal Turismo before buying the international flight ticket; usually it is more convenient and economic to buy the Brazilian domestic flights associated with the international ticket. Another possibility is to buy Brazilian Air Pass (see below).

Brazilian Air Pass: Visitors to Brazil which are interested in visiting different regions of Brazil, should consider the possibility of buying in advance the **Brazilian Air Pass**. The Air Pass can only be sold outside Brazil, and to non-residents with a return air ticket, and can be purchased for the flights of Varig, TAM and VASP, each pass only being valid for the flights of the issuing carrier. The price of the Air Pass varies, but considering the size of Brazil (larger than Europe), and the cost of internal Brazilian air travel (a return flight from Rio to Manaus can be over US\$1,000 and to Foz de Iguaçu, US\$550), it offers excellent value for money.

Currently the cost of the Airpass is US\$530 for up to 5 internal flights and is valid for 21 days after the first flight. There is also a South American Airpass which allows you to travel economically throughout Brazil, Argentina, Chile, Uruguay and Paraguay. The cost of the South American Airpass is based on the number of miles flown. Prices start at just US\$225.

For more details about the Air Pass, please see:

TAM : <http://www.tamgetaways.com/airpass.html>

VARIG: <http://www.varigbrasil.com/english/>

Information about VISA

Citizens of the following countries **do not need** a visa to come to Brazil: (**)

ANDORRA, ARGENTINA, AUSTRIA;
BAHAMAS, BARBADOS, BELGIUM, BOLIVIA;
CHILE, COLOMBIA, COSTA RICA;
DENMARK;
ECUADOR;
FINLAND, FRANCE;
GERMANY, GREECE;
HUNGARY;
ICELAND, IRELAND, ISRAEL, ITALY;
LIECHTENSTEIN, LUXEMBOURG;
MALAYSIA, MALTA, MOROCCO, MONACO;
NAMIBIA, NETHERLANDS, NORWAY;
PANAMA, PARAGUAY, PERU, PHILIPPINES, POLAND, PORTUGAL;
REPUBLIC OF KOREA;
SAN MARINO, SOUTH AFRICA, SPAIN, SURINAME, SWEDEN; SWITZERLAND;
THAILAND, TRINIDAD AND TOBAGO;
UNITED KINGDOM, URUGUAY;
VATICAN, VENEZUELA.

(**) Please confirm this information with the Brazilian Embassy or Consulate in your country.



Brazil follows the reciprocity principle concerning VISA requirements for foreign citizens from all the other countries, which ask VISA for Brazilian citizens.

The type of required visa is the one for **ACADEMIC, CULTURAL and SCIENTIFIC Activities** (*Temporary One*). Such visas are meant for non-Brazilian experts and authorities in particular fields of knowledge traveling to Brazil to make presentations at meetings or to engage in teaching, training, research and other specialized activities. Details concerning the REQUIREMENTS to get a TEMPORARY VISA can be obtained with the Brazilian Embassy or Consulate in your country.

Vaccinations

An international certificate of vaccination against polio is compulsory for children aged between three months and six years.

An international certificate of vaccination against yellow fever **is compulsory** for travelers who, within the three months prior to their arrival in Brazil, have visited or been in transit through any of the following countries: *Angola, Benin, Bolivia, Burkina Faso, Cameroon, Colombia, the Democratic Republic of Congo, Ecuador, French Guyana, Gabon, Gambia, Ghana, Guinea Bissau, Liberia, Nigeria, Peru, Sierra Leone, Sudan, Venezuela and Zaire.*

Additionally, vaccination against yellow fever **is recommended** for all travelers when visiting the following states of Brazil: *Acre, Amazonas, Amapá, Distrito Federal, Goiás, Maranhão, Mato Grosso do Sul, Pará, Rondônia, Roraima and Tocantins.* Please note that yellow fever vaccinations take approximately 10 days to become effective. If there is any doubt about the need for vaccinations, please contact the Brazilian Embassies or Consulates.



INTERNATIONAL CONFERENCE
"Novel Technologies in Powder Metallurgy and Ceramics"
September 8-12, 2003
Kiev, Ukraine

Under auspices of EPMA
Under auspices of ECerS

Devoted to the 100th Birthday of Mikhail Bal'shin, known scientist in the Powder Metallurgy

Ukrainian Materials Science Association "Composites"
Polish Ceramic Society
National Academy of Sciences of Ukraine (NASU)
Frantsevich Institute for Problems of Materials Science of NASU
INTEM LTD (Ukraine)

International programme committee

Skorokhod V. (Ukraine) - co-chairman	Koval'chenko M. (Ukraine)
Haberko . (Poland) - co-chairman	Krstic B. (Canada)
Wroe J. (EPMA) - co-chairman	Krut'ko N. (Belarus)
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Belous A. (Ukraine)	Mironovs V. (Latvia)
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Brewin P. (United Kingdom)	Nakamura T. (Japan)
Dariel M. (Israel)	Novikov N. (Ukraine)
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Firstov S. (Ukraine)	Pampuch R. (Poland)
Frage N. (Israel)	Parilak L. (Slovakia)
Froes S. (USA)	Petrovskij V. (Ukraine)
Glinchuk M. (Ukraine)	Ragulya A. (Ukraine)
Gnesin G. (Ukraine)	Ristich ?. (Yugoslavia)
Gogotsi Yu. (USA)	Rustichelli F. (Italy)
Grigoriev O. (Ukraine)	Shafran N. (Poland)
Ilyuschenko A. (Belarus)	Sheleg V. (Belarus)
Ivasishin O. (Ukraine)	Shevchenko V. (Russia)
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Konyashin I. (Germany)	Stepanchuk A. (Ukraine)
Kostornov A. (Ukraine)	Ternovoi Yu. (Ukraine)
	Uskokovich D. (Serbia)

Conference topics

- A. Modelling of powder metallurgy processes
- B. Novel technologies for production of powders and fibers
- C. Processes of forming, presintering, sintering and thermal-chemical treatment of materials and products from powders, including: composite, structural, tribotechnical, highly porous, electrotechnical, tool, high-temperature
- D. Nanotechnologies and nanomaterials
- E. Engineering of surface and coatings
- F. Novel methods for testing and evaluation powder metallurgy and ceramics technologies and materials

Special information



Lettre RFM N°96 - Mars 2003
Corresp. : <mailto:Eric.Gaffet@utbm.fr>

The outstanding scientist M. Bal'shin (1903-1980) was one of the founders of modern powder metallurgy. As far back as 1936 he performed systematic researches in the main fields of powder metallurgy - compaction and sintering, as a result of which the principles for control of these technological operations have been formulated. M. Bal'shin had great concern with development of powder metallurgy industry and new scientific directions in designing new materials and products. The industrial production of electrolytic copper powder, bearings based on iron and porous iron based on technologies developed with participation of M. Bal'shin started as early as in the middle of last century. M. Bal'shin proposed also new constructions of large-dimension press moulds. Wide fame and recognition M. Bal'shin won with his monographs: from the first fundamental in the world in domain PM "Metaloceramics" (1938) to his last book "Principles of Powder Metallurgy" (1978, co-author Prof. S. Kiparisov). He was the author of about 100 scientific publications many of that were translated into various languages.

GENERAL INFORMATION

It has become a tradition to hold in autumn large international conferences devoted to the most important problems of materials science, under the aegis of the Frantsevich Institute for Problems of Materials Science of the National Academy of Sciences of Ukraine in Kiev. This time the conference is devoted to powder metallurgy and ceramics. Our city was chosen for this event not accidentally. There are numerous research institutes in Kiev, which deal with conference topics. Among them there are academic Institute for Problems of Materials Science, Institute of Metal Physics, Institute of Superhard Materials, Institute of General and Inorganic Chemistry etc. National Technical University of Ukraine "Kiev Polytechnical Institute", Shevchenko National Kiev University train young specialists for those research institutes. Not far from Kiev there is one of the largest powder metallurgy plants (Brovary town). There are also production areas at a number of Kiev machine-building enterprises, which produce ceramic and powder metallurgy products. The time for holding conference is early autumn. This season is still rather warm (23-25° C), and numerous parks attract by their freshness and comfort. Kiev is known by its memorials, places of worships, historical buildings, among which are worldwide known Kievo-Pecherskaya Lavra and Sent Sofia Cathedral. We hope that you will not only spend your time usefully by meeting colleagues at the conference, but also have opportunity to see one of the most beautiful cities in the world.

Conference regulations

For the first day of conference it is planned to present submissions, which will be selected by Program Committee and will contain the most important results in the fields of powder metallurgy and ceramics (duration of these is 30-40 min). During the next days the sections will be held, at which 2-3 key (20-25 min) and several oral (up to 15 min) contributions will be presented. Most contributions will be presented as posters. At each section enough time will be preserved for discussion of posters. It is planned to hold "Day of Hgans firm" where the contributions of firm employees concerning the firm's latest and most important achievements will be presented. It is also planned to hold special seminar devoted to one of the most prospective direction in materials science, namely "nanotechnology and nanomaterials".

The monitoring seminar for current INTAS projects will be planned. Full papers presented at conference will be published in a special issue of a journal "Powder Metallurgy" (Kiev), which is published in English by one of the American publishers.

An abstract book containing papers presented at the conference will be given to all participants of the event. Extended abstracts have to be prepared according to guidance for abstract preparation in English (up to 2 pages). Please send also information about authors according Supplement Form. Only electronic version of abstracts (E-mail file or diskette) should be submitted. Abstracts submitted after deadline will be rejected.

English and Russian are official languages of the Conference.

Important Dates

Submission of presentations - before April 1, 2003.

Dissemination of the second information issue - before June 1, 2003.

Fee payment - before September 1, 2003.

Additional information

Full information about financial conditions of conference participation will be given in the second information issue. Tentative conference fee will be \$180.



The conference will be held in halls of the Ukraine National Academy of Sciences in the should be very center of Kiev. The participants will be accommodated in Kiev hotels.

Guidance for abstract preparation

Author A.B., Author C.D.(1), Author E.F.(2) etc.

Full title of organization, postal and e-mail addresses

(1)Full title of organization, postal and e-mail addresses

(2)Full title of organization, postal and e-mail addresses

Abstract book will contain extended abstracts prepared in English for the Microsoft Word 6.0-7.0, "Times New Roman". Abstracts should be up to two pages of A4 format (29.7x21cm).

Abstract title printed with capital letters with #14 bold type should be placed in the first line expanded for the whole width of the two columns.

The next line is blank.

Further, centered are printed names (Surname and Christian name) of the authors with the #12 bold type. The name of presenting author should be underlined.

Then also centered are placed the full name of organization, postal address, E-mail (for each organization in individual line), printed with #12 type.

The next line is blank.

The text of abstract should be arranged in two columns and one-space printed with #11 type. Margins: top - 3 cm; down, left and right - 2 cm each.

Spacing between the columns - 1 cm.

The text of abstract can contain high-quality graphs, figures, photos.

References should be numbered in the sequence of their appearance in the text. Reference numbers should be placed in square brackets. Full information about references is placed at the end of text.

Supplement Form

information about authors

Title: _____

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11th FORESIGHT CONFERENCE ON MOLECULAR NANOTECHNOLOGY

October 9-12, 2003
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TOPICS INCLUDE:

supramolecular chemistry * mechanosynthesis * molecular machines
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*** IMPORTANT DATES ***

CALL FOR ABSTRACTS - DEADLINE - June 2, 2003

Authors of Abstract and Poster submissions received on or before June 2, 2003 will receive their acceptance notices by July

1. Abstracts for oral presentations and poster session may be submitted via the web at:

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SCIENTIFIC TUTORIAL:

October 9, 2003 - If you have substantial science background relevant to nanotechnology, but want to get up to speed on areas you're unfamiliar with, attend this tutorial. The focus is on getting you oriented on recent research on key topics.

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Tutorial space is limited, therefore early registration is recommended. For additional information, see Registration Information, or contact the

Conference Office at +1(650) 917-1122, foresight@foresight.org.

*** GENERAL CONFERENCE HIGHLIGHTS ***

ORAL PRESENTATION OPPORTUNITIES:

Speaker sessions scheduled for Friday through Sunday.

POSTER PRESENTATION OPPORTUNITIES:

The poster session reception will be held on Friday afternoon. On Saturday there will be additional time for attendees to view and discuss the posters.

PRIZE OPPORTUNITIES:

The 2003 Foresight Feynman Prizes in Nanotechnology will be presented at a gala banquet on Saturday night.

SPONSORSHIP OPPORTUNITIES:

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Périodiques / Congrès

[90] MECHANOCHEMICAL SOLID REACTION OF YTTRIUM OXIDE ALUMINA LEADING TO THE SYNTHESIS OF YTTRIUM ALUMINUM GARNET

Zhang QW. Saito F. - Powder Technology. 129(1-3):86-91, 2003

Dry grinding of Y₂O₃ and transition aluminas was conducted, using a planetary ball mill under atmospheric conditions, to synthesize yttrium aluminum garnet (Y₃Al₅O₁₂, YAG). The Y₂O₃ reacts mechanochemically with the transition aluminas to form YAlO₃ and YAG after 120 min of grinding. No reaction occurs when the Y₂O₃ was ground with hard corundum or soft gibbsite. Grinding the Y₂O₃ for 360 min with an alumina prepared by heating at 400 degreesC leads to a nearly amorphous phase of YAG. A well-crystallized phase of YAG was obtained easily by calcining the ground sample at 700 degreesC. The calcined YAG fine powders contain agglomerates that consist of nanosized primary grains

[89] DIRECT OBSERVATION OF AGGREGATES AND AGGLOMERATES IN ALUMINA GRANULES

Tanaka S. Kato Z. Uchida N. Uematsu K. - Powder Technology. 129(1-3):153-155, 2003

Aggregates and agglomerates in granules prepared from milled alumina powder. were studied with new confocal laser scanning fluorescence microscopy (CLSM) using the liquid immersion method. Two kinds of ground powders were prepared by agitation bead milling and conventional ball milling and spray drying. The observation by CLSM was very effective for detecting aggregates and agglomerates of very low amounts in alumina granules. Aggregates and agglomerates were absent in the granule made from powder milled by agitation bead milling, whereas they remained in the granule made from powder milled by ball milling. The volume fractions of these particles were about 2.0 vol.% and under 0.1 vol.% for the granules made from ball-milled and bead-milled powders, respectively.

[88] EFFECT OF GRINDING ON THE STRUCTURE AND CHEMICAL EXTRACTION OF METALS FROM SERPENTINE

Kim DJ. Chung HS. - Particulate Science & Technology. 20(2):159-168, 2002

A serpentine mineral was treated in a planetary ball mill, and the effect of grinding was studied on the crystalline structure, average size, and metal extraction rate by sulfuric acid leaching. The crystalline peaks of the serpentine were gradually reduced with increase of grinding time, and after 120 min the serpentine turned into the amorphous phase. It was found that the extraction rate of the metals contained in the mineral by sulfuric acid was rapidly increased by the mechanochemical treatment. For the mineral treated for 240 min, Mg and Fe metal elements were extracted at 100% within 5 min, while 83% and 58% of Ca and Al, respectively, were obtained within 10 min.

[87] THE OXIDATION BEHAVIOR OF UNACTIVATED AND MECHANICALLY ACTIVATED SPHALERITE

Chen QY. Hu HP. Yin ZL. Zhang PM. Ye LS. - Metallurgical & Materials Transactions B-Process Metallurgy & Materials Processing Science. 33(6):897-900, 2002

The oxidation behaviors of unactivated and mechanically activated sphalerites were investigated using the thermogravimetry method (TG) in flowing highly pure oxygen at the heating rate of 10 K min⁻¹. It is found that the remaining mass between 400 and 873 K in the TG curves of mechanically activated sphalerites rises with increasing grinding time. The difference in oxidation reactivity of unactivated and mechanically activated sphalerites was also discussed. The specific granulometric surface area (S-G), the structural disorder, and the content of elemental sulfur of unactivated and mechanically activated sphalerites were determined by X-ray diffraction (XRD) laser particle size analysis, X-ray diffraction, and the gravimetric method, respectively. The results show that the specific granulometric surface areas of mechanically activated sphalerites remain almost constant after a certain grinding period. The elemental sulfur contents of unactivated and mechanically activated sphalerites were determined to be 0.5 mg/g, and the lattice distortions (epsilon) increase but the crystallite sizes (D) decrease with increasing grinding time. All the results imply that the mass increase between 400 and 873 K in the TG curves of mechanically activated sphalerites depends mainly on the increase of lattice distortions (epsilon) and the decrease of the crystallite sizes (D) with increasing grinding time. It was concluded that TG is a useful method for characterizing mechanically activated sphalerites

[86] ANNEALING BEHAVIOR OF SUBMICROCRYSTALLINE OXIDE-BEARING IRON PRODUCED BY MECHANICAL ALLOYING

Belyakov A. Sakai Y. Hara T. Kimura Y. Tsuzaki K. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 34(1):131-138, 2003

The structural changes upon annealing of an ultrafine-grained iron containing dispersed oxides were studied. The starting material was subjected to mechanical milling followed by consolidating bar rolling. The fine-grained microstructures were essentially stable against discontinuous grain growth and/or primary recrystallization during annealing at temperatures up to 800 degreesC, where the specimens maintain a very fine ferrite grain size. The annealing behavior is mainly characterized by normal grain growth accompanied by recovery. The grain-growth kinetics correlates with the oxide coarsening. Both the grain growth and the oxide coarsening can be enhanced by an increase in the temperature. The static restoration/recrystallization processes operating in the ultrafine-grained matrices, as well as the effect of dispersed oxides particles, are discussed in some detail

[85] EFFECT OF MECHANICAL MIXTURE OF ALUMINA AND SILVER SUPPORTED CATALYSTS ON THE ACTIVITY FOR THE SELECTIVE REDUCTION OF NO [JAPANESE]

Shinriki M. Haneda M. Kintaichi Y. Nagao Y. Sato K. Yoshinari T. Hamada H. - Journal of the Japan Petroleum Institute. 45(6):368-374, 2002



The catalytic activity of alumina for NO reduction by propene was considerably enhanced by mixing mechanically with Ag/SiO₂. Based on catalyst characterization, it was concluded that the activity enhancement was due to the formation of Ag/Al₂O₃ by migration of silver from Ag/SiO₂ to alumina under the reaction conditions. The migration of silver was found to occur in oxidizing atmospheres, and accelerated by the presence of H₂O. The same performance of mechanically mixed catalysts was also observed for NO reduction by decane

[84] INVESTIGATION OF THE MACHINABILITY BEHAVIOUR OF AL₄C₃ REINFORCED AL-BASED COMPOSITE PRODUCED BY MECHANICAL ALLOYING TECHNIQUE

Ozcatalbas Y.- Composites Science & Technology. 63(1):53-61, 2003.

In this study, by the technique of mechanical alloying (MA), aluminium-based composite reinforced contained various amounts of Al₄C₃ particles, depending on MA time, were produced. The machinability of metal-matrix composites (MMCs) was characterised by the investigation of chip formation, cutting forces and surface roughness. XRD, SEM and mechanical properties of the block samples produced by powder metallurgy technique were investigated. It was found that increasing the duration of mechanical alloying resulted in the formation higher amounts of Al₃C₄ particles which therefore raised the hardness, but on the contrary decreased the transverse rupture strength of the samples. During the machining of MMCs, elemental and arc chips formation were observed. High volume fraction of Al₄C₃ in the matrix decreased formation of built-up edge (BUE) and surface roughness at high cutting speeds. Furthermore, it was concluded that the effect of Al₄C₃ on the crack formation in shear plane, reduced the cutting force, shortened the chip contact length and the chip segment thickness

[83] NEW LITHIUM ION CONDUCTING GLASS-CERAMICS PREPARED FROM MECHANOCHEMICAL Li₂S-P₂S₅ GLASSES

Tatsumisago M. Hama S. Hayashi A. Morimoto H. Minami T. - Solid State Ionics. 154(Part B Special Issue SI):635-640, 2002

Amorphous solid electrolytes in the system Li₂S-P₂S₅ were prepared from a mixture of crystalline Li₂S and P₂S₅ using a mechanical milling technique at room temperature. In the composition range x less than or equal to 87.5 of xLi₂S.(100-X)P₂S₅, conductivities of the glassy powders mechanically milled for 20 h were as high as 10⁽⁻⁴⁾ S cm⁽⁻¹⁾ at room temperature. The heat treatment of the 80Li₂S(.20P₂S₅) glassy powders at around 220 degreesC produced dense glass-ceramics with high conductivity around 10⁽⁻³⁾ S cm⁽⁻¹⁾ at room temperature. The crystallization of conductive phases of Li₇PS₆, Li₃PS₄ and unknown crystals from the glass matrix and the decrease of grain boundaries by the softening of the glassy powders are simultaneously achieved at relatively low temperatures, around 220 degreesC.

[82] TRIVALENT ION CONDUCTION IN NASICON TYPE SOLID ELECTROLYTE PREPARED BY BALL MILLING

Tamura S. Imanaka N. Adachi G. - Solid State Ionics. 154(Part B Special Issue SI):767-771, 2002

Trivalent cation conducting solid electrolytes with NASICON type structure, R₁/3Zr₂(PO₄)₃ (R=Rare Earths), were synthesized by a ball milling method and trivalent R³⁺ cation conducting properties were investigated. The trivalent ion conductivity of R₁/3Zr₂(PO₄)₃ was enhanced ca. three times compared to the samples prepared by a conventional sol-gel method due to the improvement of the crystallinity of the solids. The macroscopic Sc³⁺ cation conducting behavior in Sc-1/3 Zr-2(PO₄)₃, which shows the highest trivalent ion conductivity in R₁/3Zr₂(PO₄)₃ series, was directly demonstrated by a dc electrolysis method.

[81] VISIBLE CATHODOLUMINESCENCE FROM MECHANICALLY MILLED GERMANIUM

Nogales E. Montone A. Cardellini F. Mendez B. Piqueras J. - Semiconductor Science & Technology. 17(12):1267-1271, 2002

There has been interest in the past years in the visible luminescence properties of germanium nanocrystals. Most of the previous works refer to Ge nanocrystals embedded in an oxide matrix. In this work nanocrystalline germanium has been prepared by high-energy ball milling of Ge single crystals and Ge powder. The structure of the milled samples has been assessed by x-ray diffraction (XRD) and transmission electron microscopy (TEM) while the luminescence has been investigated by cathodoluminescence (CL) in the scanning electron microscope (SEM). The samples consist of a powder of particles with sizes of hundreds of nanometres, normally aggregated to form larger particles. TEM reveals that the particles consist of nanocrystals with a wide range of sizes including crystallites of some nanometres. Milled Ge shows two CL bands at about 2.4-2.5 eV and 3.1 eV, respectively. Both emissions appear to be related to the presence of Ge nanocrystals. The CL of untreated and milled GeO₂ powder has been also investigated for comparison

[80] STRUCTURAL STUDIES OF COBALT SELENIDES PREPARED BY MECHANICAL ALLOYING

Campos CEM. de Lima JC. Grandi TA. Machado KD. Pizani PS. - Physica B: Condensed Matter. 324(1-4):409-418, 2002

The structural evolution of a binary mixture with nominal composition Co₂₅Se₇₅ prepared by ball milling was investigated as a function of milling time using X-ray diffraction (XRD), differential scanning calorimetry and Raman spectroscopy. The XRD patterns show that for milling times up to 30 h, orthorhombic and cubic CoSe₂ phases are nucleated but there is also some amorphous selenium. For milling times between 30 and 38 h, the volume fraction of the orthorhombic CoSe₂ phase decreases and the volume fraction of the cubic CoSe₂ phase increases. For milling times greater than 38 h the cubic CoSe₂ phase starts to decompose and a hexagonal CoSe phase begins to form. For long milling times, the XRD patterns show only the hexagonal CoSe phase

[79] ANNEALING BEHAVIOR OF SUBMICROCRYSTALLINE OXIDE-BEARING IRON PRODUCED BY MECHANICAL ALLOYING



Belyakov A. Sakai Y. Hara T. Kimura Y. Tsuzaki K. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 34(1):131-138, 2003

The structural changes upon annealing of an ultrafine-grained iron containing dispersed oxides were studied. The starting material was subjected to mechanical milling followed by consolidating bar rolling. The fine-grained microstructures were essentially stable against discontinuous grain growth and/or primary recrystallization during annealing at temperatures up to 800 degreesC, where the specimens maintain a very fine ferrite grain size. The annealing behavior is mainly characterized by normal grain growth accompanied by recovery. The grain-growth kinetics correlates with the oxide coarsening. Both the grain growth and the oxide coarsening can be enhanced by an increase in the temperature. The static restoration/recrystallization processes operating in the ultrafine-grained matrices, as well as the effect of dispersed oxides particles, are discussed in some detail.

[78] EFFECTS OF PROCESS-CONTROL AGENTS ON MECHANICAL ALLOYING OF NANOSTRUCTURED ALUMINUM ALLOYS

Shaw L. Zawrah M. Villegas J. Luo H. Miracle D. - Metallurgical & Materials Transactions A-Physical Metallurgy & Materials Science. 34(1):159-170, 2003

The effects of the process-control agents (PCAs) stearic acid and methanol on the mechanical alloying (MA) of a nanostructured aluminum alloy (Al93Fe3Ti2Cr2) have been investigated. The dependency of the powder-particle sizes, grain sizes, atomic-level strains, lattice parameters, formation of solid solutions, and microstructural evolution of the aluminum alloy on the types of PCAs and their concentrations have been studied using a variety of analytical instruments including X-ray diffraction scanning electron microscopy, and transmission electron microscopy. The results clearly indicate that prevention of excessive cold welding of Al particles can be achieved by the addition of a PCA at the expense of reductions in the grain size, formation rate of solid solutions, and rate of microstructural refinement, all of which are desired in MA of the Al alloy. Furthermore, a PCA that is more effective in preventing excessive cold welding will also impose more hindrance to the MA process. These phenomena have been discussed in the light of the adsorption of the PCA on the metal surface and the lubricating function of the PCA

[77] LEAD ZINC NIOBATE (PZN)-BARIUM TITANATE (BT) CERAMICS FROM MECHANOCHEMICALLY SYNTHESIZED POWDERS

Kong LB. Ma J. Huang H. Zhang RF. - Materials Research Bulletin. 37(15):2491-2498, 2002

Nanosized PZN-BT powders were synthesized directly from their constituent component oxide mixture via a high-energy ball milling process. XRD patterns showed that perovskite phase could be formed after, milling for 5 h, while single phase perovskite was achieved when the milling was prolonged for 12 h. Further increase in milling time (20 h) led to the formation of pyrochlore phase. PZN-BT ceramics were obtained by sintering the milled powders at temperatures from 1000 to 1100degreesC for 1 h. The 1100degreesC-sintered PZN-BT samples derived from the 12 h milled powders have a density of similar to 99% of the theoretical density with an average grain size of about 4 μm , a dielectric constant of 2300 and a dielectric loss of 0.03, being in good agreement with the reported results for PZN-BT prepared by the conventional solid-state reaction process.

[76] EFFECT OF COMPOSITION ON PROPERTIES OF ALUMINA/TITANIUM SILICON CARBIDE COMPOSITES

Luo YM. Li SQ. Chen J. Wang RG. Li JQ. Pan W. - Journal of the American Ceramic Society. 85(12):3099-3101, 2002

In the present study, the room-temperature properties of Al₂O₃-Ti₃SiC₂ composites with different Ti₃SiC₂ contents are determined. The composites are prepared by attrition milling Al₂O₃ and Ti₃SiC₂ mixture powders followed by spark plasma sintering (SPS) under vacuum. From a closer examination of the dependencies of the electrical conductivity on compositions in this system, we determined the percolation threshold at which an interconnected network of electrically conductive phase arises. Since the hardness of Ti₃SiC₂ is lower than that of Al₂O₃, the Vickers hardness decreased with the increasing of Ti₃SiC₂ content while the fracture toughness and the strength increased. The maximum strength (673 MPa) and the maximum toughness (9.3 MPa.m^{1/2}) were reached in the pure Ti₃SiC₂ material

[75] STRUCTURES AND MAGNETIC PROPERTIES OF IRON-NITROGEN ALLOY POWDERS PREPARED BY MECHANICAL ALLOYING PROCESS

Yamaguchi T. Saihata H. - Journal of Magnetism & Magnetic Materials. 254(Special Issue SI):20-22, 2003

An investigation of forming iron-nitrogen solid solution was made by applying a mechanical alloying process to the mixture of Fe and Fe₄N powders. Following the collapse of the FCC structure of Fe₄N at an early stage of milling process, a single phased structure which could be regarded as BCC-like Fe-N solid solution was formed. A certain amount of increase in magnetization due to formation of this Fe-N alloy was observed

[74] MAGNETIC PROPERTIES OF NANOSCALE Fe_xCu_xAg_{100-2x} (x=15, 35) GRANULAR ALLOYS

Calderon RG. Ucko DH. Barquin LF. Pankhurst QA. - Journal of Magnetism & Magnetic Materials. 254(Special Issue SI):79-81, 2003

Granular alloys of Fe_xCu_xAg_{100-2x} (x = 15, 35) have been produced by mechanical alloying. The x = 15 alloy is composed of Fe(Cu) and Ag nanocrystals, whereas the Fe₃₅Cu₃₅Ag₃₀ is amorphous. DC magnetisation and AC susceptibility (5-300 K) show a maximum about 150 K for the x = 15 alloy. The magnetisation has been fitted using log-normal distribution of grain sizes. The effect of interparticle interactions is discussed.

[73] MAGNETIC PROPERTIES OF MNZN FERRITE WITH ULTRA-FINE GRAIN STRUCTURE

Moulin J. Champion Y. Greneche JM. Mazaleyrat F. - Journal of Magnetism & Magnetic Materials. 254(Special Issue SI):538-540, 2003



Nanostructured ferrites have been prepared by milling starting from commercial ferrite powders. Subsequent compaction and sintering were applied to obtain cylindrical pieces. The magnetization as a function of the grain size follows the non-magnetic grain boundary model. The boundary thickness is estimated as about 1.5nm, which is consistent with that calculated from the Mossbauer absorption areas. Due to the high lattice distortion, the small exchange length impedes the averaging of the anisotropy.

[72] SYNTHESIS OF NANO-SIZED LANTHANUM OXYFLUORIDE POWDERS BY MECHANOCHEMICAL PROCESSING

Lee J. Zhang QW. Saito F. - Journal of Alloys & Compounds. 348(1-2):214-219, 2003

Lanthanum oxide (La₂O₃) was subjected to grinding with polytetrafluoroethylene (PTFE, (CF₂CF₂)_n) in air using a planetary mill followed by thermal treatment to synthesize lanthanum oxyfluoride ultra-fine particles. The grinding operation causes mechanochemical (MC) reaction between the two materials. The MC reaction is almost finished by 240 min, and the products ground for 240 min or more are composed of LaOF, amorphous La(CO₃)F and amorphous carbon (C). Heating this MC reaction product at 600 degreesC enables us to eliminate amorphous C and decompose La(CO₃)F into LaOF, so that pure LaOF material can be obtained as the final product. The average particle size of the final product (purified LaOF) is around 10 nm

[71] ON THE PRODUCTION OF ULTRA-FINE TITANIUM HYDRIDE POWDER AT ROOM TEMPERATURE

Bobet JL. Even C. Quenisset JM. - Journal of Alloys & Compounds. 348(1-2):247-251, 2003

A reactive mechanical grinding (RMG) method has been proposed for producing fine titanium hydride powders. The kinetics of the hydrogen/metal interaction are significantly increased by the high metal deformation under balls shocks and by the reduction of the particles size thanks to metal embrittlement by work hardening and hydriding. Also important is the significant local temperature increase related to the high rate of deformation. On the other hand, RMG tends to increase slightly the powder contamination by the metallic elements constituting the milling tools. The efficiency of the method is dependent on the rotation speed of the planetary system of milling and depends also on sufficient hydrogen pressure during RMG. The important density of defects generated in the grains by the technique is expected to facilitate the hydrogen extraction during dehydriding.

[70] MECHANOCHEMICAL TRANSFORMATION OF ALPHA-Fe₂O₃ TO Fe₃-xO₄-MICROSTRUCTURAL INVESTIGATION

Hofmann M. Campbell SJ. Kaczmarek WA. Welzel S. - Journal of Alloys & Compounds. 348(1-2):278-284, 2003

The effects of wet-milling alpha-Fe₂O₃ in vacuum for up to 144 h have been investigated by neutron diffraction measurements at room temperature and in situ at similar to 950 K. Rietveld refinements show that the main product is iron-deficient magnetite of approximate stoichiometry similar to Fe_{2.8}O₄. Comparison of the phases derived from the neutron data with results of the Fe²⁺/Fe³⁺ oxidation states as determined by chemical analysis reveals that a significant fraction of the unreacted alpha-Fe₂O₃ occurs in an amorphous-like or disordered state. The wet-milled products are also found to contain similar to 8% gamma-Fe₂O₃. The transformation from the alpha-phase to the gamma-phase occurs as a result of the shearing during the low-energy milling, with the further collisions and impacts leading to defect magnetite on extended milling. While other contributing effects take place, the transformation process from alpha-Fe₂O₃ to Fe₃-xO₄ occurs mainly as a result of rupturing the oxide surface layers of alpha-Fe₂O₃ and releasing oxygen with consequent reduction to Fe₃-xO₄.

[69] THE ELECTRONIC AND ELECTROCHEMICAL PROPERTIES OF THE TIFE-BASED ALLOYS

Szajek A. Jurczyk M. Jankowska E. - Journal of Alloys & Compounds. 348(1-2):285-292, 2003

Ti(Fe,M)-based alloys (M=Ni, Mo, Cr, Co) were mechanically alloyed (MA) under an argon atmosphere to synthesize nanostructured materials. XRD analysis showed that, after 25 h of milling, the starting mixture of the elements had decomposed into an amorphous phase. Following the annealing in high purity argon at 750 degreesC for 0.5 h, XRD confirmed the formation of CsCl-type structures with crystallite sizes of about 30 nm. These materials were used as negative electrodes for a Ni-MHx battery. The alloying elements of the 3d transition metals, Ni, Mo, Cr and Co, were substituted for iron atoms, and the structural, electronic and electrochemical properties were studied. With increasing nickel content in TiFe_{1-x}Ni_x, the material showed an increase in discharge capacity which passed through a maximum for x=3/4. In the nanocrystalline TiNi_{0.6}Fe_{0.1}Mo_{0.1}Cr_{0.1}Co_{0.1} powder, a discharge capacity of up to 135 mA h g⁻¹ (at 40 mA g⁻¹ discharge current) was measured. For this composition the capacity degraded much more slowly with cycling in comparison to other studied compositions. The electronic structure was studied by the tight-binding version of the linear muffin-tin method in the atomic sphere approximation (TB-LMTO ASA). In the TiFe_{1-x}Ni_x alloys, increasing the content of the Ni impurities extended the valence bands and increased the density of states at the Fermi level. Similar effects were observed for the TiNi_{1/2}Fe_{1/8}Mo_{1/8}Cr_{1/8}Co_{1/8} system. Mechanical alloying proved to be a suitable procedure for obtaining TiFe-based alloy electrodes for Ni-MHx batteries

[68] A STUDY ON THE IMPROVEMENT OF THE CYCLIC DURABILITY BY CR SUBSTITUTION IN V-TI ALLOY AND SURFACE MODIFICATION BY THE BALL-MILLING PROCESS

Kim JH. Lee H. Lee PS. Seo CY. Lee JY. - Journal of Alloys & Compounds. 348(1-2):293-300, 2003

The Ni-MH battery has been developed as a potential power source with a high energy density and excellent performance for mobile electrical appliances and hydride electric vehicles (HEVs). Research and development are, however, still essential for further improvement of the energy density and cycling life. Recently, vanadium-based solid solution hydrogen storage alloys have been considered as promising candidates for negative electrode materials because of their higher reversible hydrogen storage capacities. However, for the practical use of V-based alloy as a negative electrode, it is necessary to improve both the electrocatalytic activity and cyclic durability in alkaline solution. As a novel method for changing the surface properties of an



alloy electrode to obtain a higher electro-catalytic activity, we have employed the ball-milling process with Ni powder without deterioration of the alloy bulk properties. In order to overcome the poor cyclic durability of V-Ti alloy, elemental Cr, which forms a protective oxide layer against corrosion in alkaline solution, was partially substituted for V. Through systematic experimentation, a V_{0.68}Ti_{0.20}Cr_{0.12} alloy electrode ball-milled with Ni powder was found to have an advanced discharge capacity of 420 mAh/g and a good cycle life (80% after 200 cycles). For further promotion of the surface catalytic activity, instead of normal Ni powder (spherical Ni powder), filamentary Ni with a larger specific surface was used as a new surface modifier. Under optimum ball-milling conditions (7 wt% filamentary Ni for 25 min), V_{0.68}Ti_{0.20}Cr_{0.12} alloy was effectively coated with Ni particles and showed an increased discharge capacity of 460 mAh/g.

[67] AMORPHIZATION, OF INTERSTITIAL ELEMENTS CONTAINING IRON MATERIALS BY MECHANICAL ALLOYING

Ogawa H. Miura H. - Japanese Journal of Applied Physics Part 1-Regular Papers Short Notes & Review Papers. 41(8):5311-5316, 2002

In mechanical alloying (MA) of Fe-C and Fe-N materials with additive elements A such as Nb, Ti, Cr, and Co in an Ar atmosphere using a ball mill, the amorphization of MA powders is most crucially dependent on the interaction parameter WAX (X = C or N) among the several properties of additives A, including their melting points and atomic sizes, and the milling energy supplied to MA materials. The parameter WAX represents the difference between the bonding energies of the atomic A-X pair (U-AX) and the Fe-X pair (U-FeX) in the Fe-A-X solution, i.e., $W-AX = U-AX - U-FeX$. Additives such as Cr and Nb with negative WAX values markedly promote the amorphization of MA materials, in contrast to Co or Ni which have positive W-AX values. When V-VI group elements with negative and moderate W-AX values, such as Nb, Ta, and Cr, are employed as the ternary additives, the amorphization of Fe-C and Fe-N materials is readily attained.

[66] AMORPHIZATION AND NANOSTRUCTURE SYNTHESIS IN AL ALLOYS

Perepezko JH. Hebert RJ. Tong WS. - Intermetallics. 10(11-12):1079-1088, 2002

The recent innovations in metallic glasses have led to new alloy classes that may be vitrified and a re-examination of the commonly used criteria for glass formation and stability. In one case large, bulk volumes may be slowly cooled to the glassy state that signifies a nucleation-controlled synthesis. The other important case is represented by Al and Fe based marginal glass formers that have been synthesized under growth controlled kinetic conditions mainly by rapid solidification processes. With marginal glass forming alloys the termination of the amorphous state upon heating is often characterized by a primary crystallization reaction that yields a high number density in the range of 10^{21} - 10^{22} m⁻³ of Al nanocrystals (15-20 nm in diameter) dispersed within a residual amorphous matrix. At the same time the results from alternate synthesis strategies involving intense cold rolling reveal that the primary crystallization reaction can be bypassed during deformation alloying of elemental multilayers or enhanced during deformation bonding of amorphous ribbons. The kinetics analysis of the crystallization behavior provides insight into the origin of the dispersed nanocrystal and amorphous matrix micro structures, the important influence of heterogeneous catalysts and an effective assessment of the overall stability that are essential for microstructure control

[65] INVESTIGATIONS OF MECHANICALLY ALLOYED NI-ZR-TI-SI AMORPHOUS ALLOYS WITH SIGNIFICANT SUPERCOOLED REGIONS

Lin CK. Feng YB. Lee PY. Wang LY. Lin HM. Chen GS. - Intermetallics. 10(11-12):1149-1155, 2002

This study examined the amorphization behavior of Ni₅₇Zr₂₀Ti_{23-x}Si_x (x=0, 1, 3) alloy powders synthesized by mechanical alloying technique. According to the results, after 5 h of milling, the mechanically alloyed powders were amorphous at compositions of Ni₅₇Zr₂₀Ti_{23-x}Si_x (x=0, 1, 3). The amorphization behavior of Ni₅₇Zr₂₀Ti₂₀Si₃ was examined in details. The conventional X-ray diffraction and synchrotron EXAFS results confirm that the fully amorphous powders formed after 5 h of milling. The thermal stability of the Ni₅₇Zr₂₀Ti_{23-x}Si_x amorphous powders was investigated by differential scanning calorimeter (DSC). As the results demonstrated, the amorphous powders were found to exhibit a large supercooled liquid region before crystallization. The supercooled liquid regions, defined by the difference between T_g and T_x, (i.e. $\Delta T = T_g - T_x$), are 95 K, 66 K, and 88 K, for Ni₅₇Zr₂₀Ti₂₃, Ni₅₇Zr₂₀Ti₂₂Si₁, and Ni₅₇Zr₂₀Ti₂₀Si₃, respectively.

[64] FORMATION OF QUASICRYSTALS IN ZR-PD-(CU) MELT SPUN RIBBONS AND MECHANICALLY MILLED POWDERS

Sordelet DJ. Rozhkova E. Besser MF. Kramer MJ. - Intermetallics. 10(11-12):1233-1240, 2002

Amorphous Zr₇₀Pd₃₀ and Zr₇₀Pd₂₀Cu₁₀ alloys were prepared by mechanical milling and melt spinning to compare their devitrification behaviors. The devitrification of mechanically milled Zr₇₀Pd₃₀ and Zr₇₀Pd₂₀Cu₁₀ powders occurs via a single-step, first-order transformation to a stable Zr₂Pd tetragonal structure. This is in sharp contrast to the devitrification of the same amorphous alloys prepared by melt spinning, in which a primary meta-stable quasicrystalline phase forms. Since the mechanical milling process does not involve direct liquid phase formation of an amorphous structure, it is inferred that the short-range order in the solid state derived amorphous powder is different from that in the melt spun ribbon. During mechanical milling of an amorphous melt spun ribbon, crystallization of the quasicrystalline phase appears to precede disordering into an amorphous structure having an different short range order. Deformation of an amorphous melt spun ribbon by repetitive rolling at ambient temperature crystallizes the meta-stable quasicrystalline phase.

[63] MECHANICAL ALLOYED TI-CU-NI-SI-B AMORPHOUS ALLOYS WITH SIGNIFICANT SUPERCOOLED LIQUID REGION

Jeng IK. Lee PY. Chen JS. Jeng RR. Yeh CH. Lin CK. - Intermetallics. 10(11-12):1271-1276, 2002



This study examined the glass formation range of $Ti_{94-x-y}Cu_xNi_ySi_4B_2$ alloy powders synthesized by mechanical alloying technique. According to the results, after 5-7 h of milling, the mechanically alloyed powders were amorphous at compositions with $(x + y)$ equal to 20-40%. For the compositions with $(x + y)$ larger than 45% or smaller than 10%, the structure of ball-milled powders is a partial amorphous single phase or coexistent partial amorphous and crystalline phases, respectively. The thermal stability of the amorphous powders was also investigated by differential thermal analysis. As the results demonstrated, several amorphous powders were found to exhibit a wide supercooled liquid region before crystallization. The temperature interval of the supercooled liquid region defined by the difference between T_g and T_x i.e. $\Delta T (T_x - T_g)$, are 52 K for $Ti_{74}Ni_{20}Si_4B_2$, K for $Ti_{64}Ni_{30}Si_4B_2$, 58 K for $Ti_{64}Cu_{20}Ni_{10}Si_4B_2$, and 61 K for $Ti_{74}Cu_{10}Ni_{10}Si_4B_2$

[62] CONSOLIDATION OF AMORPHOUS NI-ZR-TI-SI POWDERS BY VACUUM HOT-PRESSING METHOD

Lee PY, Hung SS, Hsieh JT, Lin YL, Lin CK. - Intermetallics. 10(11-12):1277-1282, 2002

In the current study, we investigated the feasibility of fabricating amorphous $Ni_{57}Zr_{20}Ti_{20}Si_3$ powders by mechanical alloying, and consolidating them into bulk metallic glasses by a vacuum hot pressing technique. The as-milled and hot-pressed samples were examined by X-ray diffraction, scanning electron microscope, and differential thermal analysis. According to the results, amorphous $Ni_{57}Zr_{20}Ti_{20}Si_3$ powders were prepared after milling elemental powder mixtures for 5 h. The amorphous powders were found to exhibit a wide supercooled liquid region of 88 K before crystallization. Bulk metallic glasses were prepared successfully by consolidating the as-milled $Ni_{57}Zr_{20}Ti_{20}Si_3$ amorphous powders using vacuum hot pressing in the supercooled liquid region. Vickers microhardnesses of the as-pressed samples are in the range of 788-830 kg/mm²

[61] AMORPHOUS PRESSURE-INDUCED ALLOYS - FORMATION, STABILITY, AND CRYSTALLISATION

VF Degtyareva - NEW KINDS OF PHASE TRANSITIONS: TRANSFORMATIONS IN DISORDERED SUBSTANCES (Series: NATO SCIENCE SERIES, SERIES II: MATHEMATICS, PHYSICS AND CHEMISTRY), 2002, Vol 81, pp 505-514 - NATO ADVANCES RESEARCH WORKSHOP ON NEW KINDS OF PHASE TRANSITIONS - TRANSFORMATIONS IN DISORDERED SUBSTANCES; RUSSIA. MAY 24-28, 2001

An amorphous state of materials is a metastable state which can be produced by different ways, as for instance, by quenching from the liquid, sputtering thin films, ion implantation, ball milling or action of high pressure. Intensively studied amorphized materials, both experimentally and theoretically, are tetrahedral semiconductors-group IV elements and III-V compounds. This paper concerns a special case of pressure-induced amorphization in binary alloy systems. Discussed are some general features of amorphous pressure-induced state in binary alloy systems, the conditions providing alloy amorphization and crystallization processes of amorphous phases. An ability of binary alloy systems to form an amorphous phase is regarded to valence electron concentration in spirit of known Hume-Rothery phases. The subject of discussion is amorphous state primarily in alloys with a two-phase mixture at ambient conditions as in Al-Ge, Cd-Sb and Zn-Sb alloys. Alloy amorphization is considered by pressure quenching and in situ under pressure.

[60] REACTIVE MILLING - AN IMPROVED PROCESS FOR SYNTHESIS OF SILVER-TIN OXIDE COMPOSITE POWDERS FOR AUTOMOTIVE CONTACTS

PB Joshi, GR Marathe, VL Gadgeel, GH Upadhyay, VK Kaushik, P Ramakrishnan - POWDER METALLURGY IN AUTOMOTIVE APPLICATIONS - II, 2002, pp 113-121 - INTERNATIONAL CONFERENCE ON POWDER METALLURGY FOR AUTOMOBILE COMPONENTS (PM02); INDIA. , 2002

In last few years silver/tin oxide contact materials have started replacing silver/cadmium oxide materials in both AC as well as DC applications in view of their greater resistance to arc erosion and superior anti-welding characteristics, besides being an environment-friendly substitute to conventional silver/cadmium oxide contacts, The present work deals with the synthesis of silver-tin oxide composite powders for automotive contact application by a novel technique of reactive milling (RM) followed by their consolidation using conventional powder metallurgy (PM) process. The synthesized powders are characterized by techniques such as x-ray diffraction (XRD), scanning electron microscopy (SEM) and electron spectroscopy for chemical analysis (ESCA). The consolidated bulk solid contacts are evaluated for their density, microhardness and electrical conductivity and also subjected to evaluation of resistance to arc erosion on a commercial contactor under DC load.

[59] AGING BEHAVIOUR OF SIC - PARTICULATE REINFORCED 7075 AL ALLOY COMPOSITES PREPARED BY MECHANICAL ALLOYING

R Sankar, S Paramanand - POWDER METALLURGY IN AUTOMOTIVE APPLICATIONS - II, 2002, pp 219-225 - INTERNATIONAL CONFERENCE ON POWDER METALLURGY FOR AUTOMOBILE COMPONENTS (PM02); INDIA. , 2002

Al based metal matrix composites because of their specific strength are the focus of attention of the automotive industry. Alumina or alumina and silica in Al-Si matrix as replacement for nickel cast iron in the diesel piston crowns, carbon fibre and alumina fibers in an aluminum matrix as cylinder liners are important applications of MMCs in automotive sector. Automotive drive shafts, valve spool and oil pump rotors are few potential commercial applications of particulate reinforced aluminum composites. The addition of reinforcement influences the heat treatment behaviour and hence, its properties. In the present study aging behaviour of 7075 Al/SiCp composites have been investigated. 7075 Al alloy (Al-5.6 Zn 2.5 Mg 1.5 Cu) - base alloy and the metal matrix composites (MMC) of 7075 aluminium alloy reinforced with 15 v/o SiC particulate (SiCp) were prepared by hot pressing of canned and degassed mechanically alloyed elemental powders. The base alloy and the composite were solutionized at 475degreesC and age-hardened at 80degreesC, 120degreesC and 170degreesC. This paper deals with the overall age hardening behaviour of the base alloy and the composites using microhardness tester and also



analyses the age hardening characteristics of SiCp/matrix interfaces in the composites by ultra-low-load dynamic microhardness testing techniques. It has been observed that maximum hardness occurs at the particle/matrix interface and hardness decreases with increasing distance from the interface. Variation in hardness of the interface and the matrix with increasing distance from particle has been correlated with the dislocation density, which has been found to decrease with increasing distance from the interface. Compared to the base alloy, the composite showed accelerated aging and this has been correlated to activation energy of the system. The retardation in aging kinetics with increasing distance from interface has been further confirmed by TEM observations, which reveal the precipitates and network of dislocations.

[58] STRUCTURAL CHANGES AND THERMAL STABILITY OF MECHANICALLY ALLOYED TI-AL POWDERS WITH ADDITIONS OF MN AND NB

L Lu, F Zhang, MO Lai - ADVANCED MATERIALS FORUM I (Series: KEY ENGINEERING MATERIALS), 2002, Vol 230-2, pp 130-135 - 1ST INTERNATIONAL MATERIALS SYMPOSIUM (MATERIALS 2001); COIMBRA, PORTUGAL. APRIL 9-11, 2001

Quaternary Ti-48Al-2Mn-2Nb (at.%) alloys have been prepared by mechanical alloying. The effects of addition of alloying elements (Mn and Nb) on both the structural evolutions during mechanical alloying and thermal stability in the subsequent heat treatment were investigated. Grain size at different milling stages was calculated. The results showed that although the equilibrium solubility of both alloying elements is limited in Al and Ti, Nb was totally dissolved into the matrix of the parent elements before 10 hours of milling, while dissolution of Mn occurred only at later stage of milling. This behavior was mainly due to the difference in mechanical properties between the two alloying elements.

[57] MG-RICH LIGHT ALLOYS SYNTHESISED BY MECHANICAL ALLOYING

L Dias, C Coelho, B Trindade, FH Froes - ADVANCED MATERIALS FORUM I (Series: KEY ENGINEERING MATERIALS), 2002, Vol 230-2, pp 283-286 - 1ST INTERNATIONAL MATERIALS SYMPOSIUM (MATERIALS 2001); COIMBRA, PORTUGAL. APRIL 9-11, 2001

In this work, Mg_xTi_{1-x} (x = 18, 50 and 95 at.%) and Mg₈₈Ti₄Si₇ alloys were synthesised by mechanical alloying. Phase transformations occurring in the samples as a function of milling time and during subsequent heating were studied by means of x-ray diffraction, scanning electron microscopy, differential scanning calorimetry and electron probe microanalysis. Concerning the Ti-Mg binary system, super-saturated Ti(Mg) solid solutions with low degree of structural order were obtained after milling. The Mg₈₈Ti₄Si₇ mechanical alloyed sample was formed by a Mg₂Si intermetallic phase in a Mg matrix. During heating up to 600degreesC, there was the formation of further Mg₂Si at 380degreesC, followed by the formation of Ti₅Si₃ at 540degreesC. No decomposition of either Mg₂Si or Ti₅Si₃ phases was detected during cooling. The final structure of mechanically alloyed and heat treated Mg₈₈Ti₄Si₇ sample consist of a fine precipitation of these two intermetallics in a Mg matrix.

[56] STRUCTURE AND PROPERTIES OF MG METAL-METAL COMPOSITE

Authors L Lu, MO Lai, L Froyen - ADVANCED MATERIALS FORUM I (Series: KEY ENGINEERING MATERIALS), 2002, Vol 230-2, pp 287-290 - 1ST INTERNATIONAL MATERIALS SYMPOSIUM (MATERIALS 2001); COIMBRA, PORTUGAL. APRIL 9-11, 2001

Mg_{10.3%}Ti and Mg_{5%}Al_{10.3%}Ti metal-metal composites have been fabricated using mechanical milling. Strain to failure was found to have dramatically increased from 2.6% to 41.7% after the milling process. The increase in strain to failure is associated to the smaller grain size. Fractograph study shows ductile failure.

[55] MECHANICAL ALLOYING OF FE-CU ALLOYS FROM AS-RECEIVED AND PREMILLED ELEMENTAL POWDER MIXTURES

FM Lucas, B Trindade, BFO Costa, G LeCaer - ADVANCED MATERIALS FORUM I (Series: KEY ENGINEERING MATERIALS), 2002, Vol 230-2, pp 631-634 - 1ST INTERNATIONAL MATERIALS SYMPOSIUM (MATERIALS 2001); COIMBRA, PORTUGAL. APRIL 9-11, 2001

We have investigated the effect of premilling elemental powders of Fe and Cu on the mixture mechanism of a Fe₅₀Cu₅₀ alloy. The alloying process was compared and studied using electron probe microanalysis, X-ray diffraction, scanning electron microscopy and Mossbauer spectroscopy. In both processing routes (with and without premilling), a broad distribution of different local environments of the iron atoms was observed in the Mossbauer spectra of the fcc-FeCu phase. Mossbauer spectra show further that a fcc non magnetic Fe-rich phase is formed transiently besides the bcc-Fe phase. The reactions are slowed down when premilled powders are used as starting powders but similar fcc solid solutions are formed after 16h and 32h in our milling conditions when as-received and premilled powders are used respectively as starting powders.

[54] HIGH-TEMPERATURE STABILITY OF A NANOSTRUCTURED CU-AL₂O₃ ALLOY

MT Marques, JB Correia, JM Criado, MJ Dianez, P Matteazzi - ADVANCED MATERIALS FORUM I (Series: KEY ENGINEERING MATERIALS), 2002, Vol 230-2, pp 652-655 - 1ST INTERNATIONAL MATERIALS SYMPOSIUM (MATERIALS 2001); COIMBRA, PORTUGAL. APRIL 9-11, 2001

A nanostructured oxide dispersion strengthened (ODS) copper alloy powder, containing an alumina dispersion (about 1% vol.) was prepared by mechanical alloying and subsequently consolidated by hot extrusion. The high temperature stability of the nanostructured and consolidated powders was studied. The stability of the nanostructure was checked with 1 hour heat treatments in the range 400-800degreesC. Powders were vacuum encapsulated in copper cans and extruded at temperatures in the range 500-600degreesC. The crystallite size of both the as-milled and the heat treated powders was determined with X-ray diffraction (XRD). The line broadening was used to calculate grain (crystallite) size. Two methods were used: the



Scherrer equation (SE) and the Williamson-Hall plot (WH). The microstructures of both loose powders and extruded powders were characterised with Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM). The mechanical properties were assessed with microhardness measurements. The results obtained via XRD with both methods are within the same order of magnitude. A reasonable agreement is also obtained with SEM and TEM observations. The X-ray determination of grain size and the microhardness measurements indicate negligible grain coarsening up to 800degreesC. On the other hand, the consolidated powders showed lower hardness values than those of the as-milled and annealed powders. The nanostructured copper alloy studied shows good thermal stability at temperatures up to 800degreesC.

[53] KINETICS OF MECHANOSYNTHESIS OF THE FE70NI30 ALLOY USING PARE COMPONENTS

Cherdyntsev VV. Kaloshkin SD. Tomilin IA. Shelekhov EV. Baldokhin YV. - Physics of Metals & Metallography (English Translation of Fizika Metallov i Metallovedenie). 94(5):459-465, 2002

Scanning electron microscopy, X-ray diffraction, and Mossbauer spectroscopy were used to study transformations that occur at the macroscopic and microscopic levels upon the production of the Fe70Ni30 alloy by mechanical alloying using powders of pure metals in a planetary ball mill. The intense mechanical treatment causes a coarsening of the powder particles with time. The results obtained by X-ray diffraction demonstrate a gradual increase in the content of an fcc: phase in this alloy. Mossbauer spectroscopy also indicates a gradual increase in the iron content in the fcc phase to values that correspond to the nominal composition of the alloy; the considerable concentration differences observed between the phases coexisting at the intermediate stages of mechanosynthesis were explained by the development of the reverse bcc --> fcc martensitic: transformation in the course of mechanical alloying

[52] EFFECTS OF CARBON CRYSTALLINITY ON HYDRIDING-DEHYDRIDING AND CHARGE-DISCHARGE CHARACTERISTICS OF MGNI ALLOY-CARBON MATERIAL COMPOSITES

Iwakura C. Inoue H. Furukawa N. Nohara S. - Materials Transactions. 43(11):2706-2710, 2002

Hydriding-dehydriding and charge-discharge characteristics of amorphous MgNi alloys modified by ball-milling with different carbon materials were investigated with the intention of using them in nickel-metal hydride batteries. It was found that the ball-milling with carbon materials markedly improved hydriding-dehydriding and charge-discharge characteristics of the MgNi alloy, and an optimum ball-milling time was shortened by utilization of carbon material with lower crystallinity. However, ball-milling for longer than the optimum time led to a decrease in the amount of absorbed hydrogen and the discharge capacity, which was ascribed to the formation of MgNi3C. These results indicate that the characteristics of MgNi alloy-carbon material composites greatly depend on both crystallinity of carbon material used and ball-milling time

[51] SURFACE MODIFICATION OF MGNI BY PERYLENE

Ma T. Hatano Y. Abe T. Watanabe K. - Materials Transactions. 43(11):2711-2716, 2002

Amorphous MgNi was modified by ball milling with perylene and its effects on the charge/discharge capacity were examined by using a conventional two-electrode cell. It was found that both the ball milling time and perylene/MgNi ratio had great influence on the discharge capacity and cycle life of MgNi. Three types of effects were identified, depending on ball-milling conditions. One of them was the increase in the discharge capacity at the first cycle, the second type was the deceleration of the degradation of the discharge capacity with charge/discharge cycle, and the last type was the reduction in the charge/discharge capacity. Chemical states of modified surfaces were analyzed by Auger electron spectroscopy (AES) as well as ab-initio calculation. Both AES and ab-initio calculations indicated that carbon atoms can form bonding with both magnesium and nickel atoms, but bonding with magnesium atoms is most preferable. The change in the charge/discharge capacity is attributed to such kind of reactions, and the three distinct effects are ascribed to the presence of different MgNi-perylene composites, formed during the ball milling on the surface, resulting in the retardation or acceleration of Mg(OH)(2) formation on the electrode

[50] PROTIUM ABSORPTION/DESORPTION CHARACTERISTICS OF MG2NI/LANI5 COMPOSITE

Okumura H. Matsui A. Yamagiwa S. Kamado S. Kojima Y. - Materials Transactions. 43(11):2728-2733, 2002

In this study, an attempt is made to form composites by mechanical alloying (MA) or pulse-electric-current-sintering (PECS) for utilization of synergy effect to acquire both high capacity and ability to absorb/desorb protium at low temperatures. The composites have well-bonded structure consisting of Mg2Ni alloy that has high hydrogenation capacity and LaNi5 alloy that is capable of protium absorption/desorption at relatively low temperatures. During the absorption process, the protium content of MA + PECS and MA specimens at 80degreesC is over the theoretical value corresponding to the ratio of LaNi5 phase. After absorption of the maximum protium content at 40degreesC by MA + PECS specimen, the desorption process was evaluated by TG/DTA analysis and the specimen starts desorbing protium at about 100degreesC. Consequently, the result suggests the occurrence of synergy effect during the absorption/desorption process of the obtained composites.

[49] EMERGENCE OF HYDROGEN ABSORPTION ABILITY IN METASTABLE HCP, FCC AND AMORPHOUS TI-AL ALLOYS PREPARED BY MECHANICAL GRINDING

Hashi K. Ishikawa K. Suzuki K. Aoki K. - Materials Transactions. 43(11):2734-2740, 2002

Titanium aluminides [L1(0)-TiAl (Ti0.50Al0.50 and Ti0.40Al0.60). TiAl2 and D0(22)-TiAl3] have been mechanically ground (MG) in an argon atmosphere. In addition, hydrogen absorption and desorption properties of the MG-alloys have been investigated. Hcp- and amorphous (a-) TiAl, fcc- and a-TiAl2 and fcc-TiAl3 are formed by mechanical grinding (MG) of TiAl, TiAl2 and TiAl3, respectively. Although original Ti-aluminides do not absorb hydrogen. hcp- and a-TiAl. and fee- and a-TiAl2, prepared by MG, having a fresh surface absorb hydrogen of the amount of 0.4 - 0.1 (H/M). The present work clearly demonstrates that MG is a useful technique for an emergence of the hydrogen absorption ability of intermetallic compounds containing much hydride-forming elements



[48] SOLID-STATE FORMATION OF LITHIUM FERRITES FROM MECHANICALLY ACTIVATED $\text{Li}_2\text{CO}_3\text{-Fe}_2\text{O}_3$ MIXTURES

Berbenni V. Marini A. Matteazzi P. Rieceri R. Welham NJ. - Journal of the European Ceramic Society. 23(3):527-536, 2003

The formation of lithium ferrites (LiFe_5O_8 and LiFeO_2) from mechanically activated mixtures of $\text{Li}_2\text{CO}_3\text{-Fe}_2\text{O}_3$ has been studied using thermal analysis (TGA, DSC), evolved gas analysis (TG/FT-IR), X-ray powder diffraction (XRD), scanning electron microscopy (SEM) and particle size analysis. It is shown that mechanical activation of the precursors considerably enhances the reactivity of the solid system analysed and makes it possible to obtain reaction products with a much lower expense of thermal energy. In particular, lithium ferrites can be obtained at temperatures at least 160 degreesC lower than those necessary in the absence of mechanical activation. Moreover, both the microstructure and the allotropic ratio of the products, as well as the reaction path, are affected by mechanical activation

[47] NANOCRYSTALLIZATION IN FE-C ALLOYS BY BALL MILLING AND BALL DROP TEST

Todaka Y. Umemoto M. Tsuchiya K. - ISIJ International. 42(12):1430-1437, 2002.

Microstructural evolution and nanocrystallization in various carbon steels by ball milling and ball drop test has been studied. In ball milling, nanocrystallization was observed in all the carbon steels irrespective of the carbon content (up to 0.9 mass% C) or starting microstructure (ferrite, martensite, pearlite or spheroidite). In ball drop test, nanocrystallization was observed in high carbon steels or ultrafine grained low carbon steels. It is realized that high strength before ball drop test is required for the nanocrystallization. The nanocrystalline structure obtained by ball milling and ball drop test has similar microstructure with dark smooth contrast. The morphologies such as pearlite lamellar, spheroidite cementite, ferrite grain boundary disappeared by nanocrystallization. The boundary between the nanocrystalline and work-hardened regions is quite sharp. The hardness of the nanocrystalline region is about two times higher than that of work-hardened region. The annealing of nanocrystalline region shows substantially slow grain growth and re-precipitation of fine cementite. This annealing behavior is quite different from the work-hardened region which is characterized by recrystallization and fast grain growth. From the present study, it was confirmed that the nanocrystalline structure produced by ball milling and ball drop test has quite similar in structure, hardness and annealing behavior although the number of deformation applied is substantially different.

[46] FABRICATION OF FINE-GRAINED HIGH NITROGEN AUSTENITIC STEELS THROUGH MECHANICAL ALLOYING TREATMENT

Tsuchiyama T. Uchida H. Kataoka K. Takaki S. - ISIJ International. 42(12):1438-1443, 2002.

Mechanical alloying (MA) treatment was applied for the fabrication of fine-grained high nitrogen stainless steels. Chromium nitride (Cr_2N) powder was mixed with Fe-Cr binary alloy powder to control its mean chemical composition to be Fe-23mass%Cr-1mass%N which is enough to stabilize austenitic structure at room temperature. The powder mixture was mechanically alloyed up to 360 ks in an argon gas atmosphere (MA powder). The MA powder was packed in a stainless steel tube in a vacuum and consolidated by warm rolling at 1 073 K. The consolidated materials were finally heated to various temperatures (1 173-1 473 K) for austenitizing and then quenched without holding at the temperatures. Although the materials heat-treated below 1 323 K had bcc (martensitic) matrix, those heat-treated above 1 373 K had stable austenitic structure with a small amount of Cr_2N . The grain size of matrix was maintained to be fine due to dispersed oxide particles within matrix in all steels. For example, the materials heat-treated at 1 473 K had fine austenitic structure in which the grain size was 2.2 μm and the solute nitrogen concentration was 0.86 mass%. The steel had very high yield strength of 1.1 GPa and moderate elongation of 30 %. Such a high strength of the steel was explained by the combined strengthening mechanism of nitrogen solid solution strengthening and grain refining strengthening.

[45] ULTRAFINE GRAINED LOW CARBON STEELS FABRICATED BY EQUAL CHANNEL ANGULAR PRESSING: MICROSTRUCTURES AND TENSILE PROPERTIES

Shin DH. Park JJ. Chang SY. Lee YK. Park KT. - ISIJ International. 42(12):1490-1496, 2002.

Equal channel angular pressing (ECAP) was conducted on the two grades of low carbon steel, with or without vanadium, in order to produce an ultrafine grained structure. As a result, the ferrite grains were refined from 30 μm to 0.2 similar to 0.3 μm . The strength of the ECAPed steels increased remarkably, over twice of the strength of the steels before ECAP. A series of static annealing experiments showed that the increment of ECAP strain and the dilute addition of microalloying element such as vanadium (were very effective on enhancing thermal stability of the ultrafine grained low carbon steels produced by ECAP in terms of microstructure and tensile properties. This enhanced thermal stability resulted from, (a) presence of excessive carbon content in the ferrite matrix by carbon dissolution from pearlitic cementite during ECAP; (b) preservation of high dislocation density due to addition of vanadium, providing the effective diffusion path for dissolved carbon atoms; (c) precipitation of excessive carbon as the form of nano-sized cementite particles during subsequent annealing and its effect on suppressing grain growth.

[44] MICROSTRUCTURAL EFFECTS ON THE HIGH TEMPERATURE OXIDATION OF TWO-PHASE CU-CR ALLOYS IN 1 ATM O-2

Fu GY. Niu Y. Gesmundo F. - Corrosion Science. 45(3):559-574, 2003

The oxidation of a Cu-Cr alloy containing about 60 wt% Cr and of two Cu-Cr alloys containing about 40 wt% Cr was studied at 700 and 800 degreesC in 1 atm. O-2. The 60 wt% Cr alloy was prepared by powder metallurgy (PM) and had a phase particle size of 50-150 μm . One of the two alloys containing about 40 wt% Cr was prepared by mechanical alloying (MA) and had a phase grain size ranging from 10-50 nm to 200-300 nm, depending on the location, while the other was prepared



by magnetron sputtering (MS) and had a phase grain size around 5-10 nm. The most important difference between the oxidation behavior of the three alloys is the formation of an exclusive chromia scale on the surface of the Cu-40 wt% Cr alloy prepared by magnetron sputtering and of a continuous chromia layer beneath an outermost layer of copper oxides on the corresponding alloy prepared by mechanical alloying, while the Cu-60 wt% Cr alloy prepared by powder metallurgy formed complex scales composed mostly of CuO, Cu₂O with Some Cu₂Cr₂O₄ and Cr₂O₃. Thus, the microstructure of two-phase binary alloys has a strong effect of their oxidation behavior. In particular, a decrease of the alloy grain size favors the exclusive external oxidation of the most reactive component, reducing the corresponding critical content in the alloy. This effect is attributed to the presence of larger concentrations of rapid diffusion paths for the migration of the components in the alloy as well as to a faster dissolution of the particles of the Cr-rich phase in the copper matrix.

[43] KINETICS OF MECHANOSYNTHESIS OF THE FE70NI30 ALLOY USING PURE COMPONENTS

Cherdyntsev VV. Kaloshkin SD. Tomilin IA. Shelekhov EV. Baldokhin YV. - Physics of Metals & Metallography (English Translation of Fizika Metallov i Metallovedenie). 94(5):459-465, 2002

Scanning electron microscopy, X-ray diffraction, and Mossbauer spectroscopy were used to study transformations that occur at the macroscopic and microscopic levels upon the production of the Fe70Ni30 alloy by mechanical alloying using powders of pure metals in a planetary ball mill. The intense mechanical treatment causes a coarsening of the powder particles with time. The results obtained by X-ray diffraction demonstrate a gradual increase in the content of an fcc: phase in this alloy. Mossbauer spectroscopy also indicates a gradual increase in the iron content in the fcc phase to values that correspond to the nominal composition of the alloy; the considerable concentration differences observed between the phases coexisting at the intermediate stages of mechanosynthesis were explained by the development of the reverse bcc --> fcc martensitic: transformation in the course of mechanical alloying

[42] DISCLINATIONS AND ROTATIONAL DEFORMATION IN FINE-GRAINED MATERIALS

Gutkin MY. Kolesnikova AL. Ovid'ko IA. Skiba NV. - Philosophical Magazine Letters. 82(12):651-657, 2002

A theoretical model is presented which describes a new mechanism of plastic deformation in fine-grained materials. In the framework of the model, rotational deformation occurs via motion of dipoles of grain-boundary disclinations and is associated with the emission of lattice dislocations from grain boundaries into adjacent grain interiors. Ranges of defect system parameters are identified in which the disclination motion is energetically favourable. It is shown that the mechanism can contribute to plastic flow in fine-grained materials prepared by highly non-equilibrium methods such as ball milling, severe deformation and high-pressure compaction

[41] IMPACT-INDUCED DISORDERING OF INTERMETALLIC PHASES DURING MECHANICAL PROCESSING

Delogu F. Cocco G. - Materials Science & Engineering A-Structural Materials Properties Microstructure & Processing. 343(1-2):314-317, 2003

A detailed study on the disordering of the equilibrium compound NiTi, under mechanical processing conditions is reported. The characterization of the milling regime, together with the application of refined X-rays analysis techniques, allowed for the quantitative kinetic description of the amorphization process as a function of the number of impacts. Experimental findings show that the phase transformation follows first-order kinetics. Complete disordering of the crystalline lattice required about ten millions of impacts

[40] SOLID-STATE FORMATION OF LITHIUM FERRITES FROM MECHANICALLY ACTIVATED Li₂CO₃-Fe₂O₃ MIXTURES

Berbenni V. Marini A. Matteazzi P. Rieceri R. Welham NJ. - Journal of the European Ceramic Society. 23(3):527-536, 2003

The formation of lithium ferrites (LiFe₅O₈ and LiFeO₂) from mechanically activated mixtures of Li₂CO₃-Fe₂O₃ has been studied using thermal analysis (TGA, DSC), evolved gas analysis (TG/FT-IR), X-ray powder diffraction (XRD), scanning electron microscopy (SEM) and particle size analysis. It is shown that mechanical activation of the precursors considerably enhances the reactivity of the solid system analysed and makes it possible to obtain reaction products with a much lower expense of thermal energy. In particular, lithium ferrites can be obtained at temperatures at least 160 degreesC lower than those necessary in the absence of mechanical activation. Moreover, both the microstructure and the allotropic ratio of the products, as well as the reaction path, are affected by mechanical activation.

[39] MECHANOCHEMICAL SYNTHESIS OF NANO-SIZED COMPLEX FLUORIDES FROM PAIR OF DIFFERENT CONSTITUENT FLUORIDE COMPOUNDS

Lu JF. Zhang QW. Saito F. - Chemistry Letters. (12):1176-1177, 2002

Complex fluorides ARF₄ (A = Li, Na, K, R = rare earths) are synthesized by the mechanochemical solid reactions of AF and RF₃ powders at room temperature using a planetary ball mill. The reactions proceed with the increase of grinding time and have been completed at 4h. Transmission electron microscope observation shows that the produced ARF₄ powders consist of agglomerates of nano-sized primary particles, which exhibit moderate water solubility. When the ground sample is calcined, the particle size is increased and the water solubility is depressed.

[38] DECOMPOSITION AND THERMODYNAMIC PROPERTY OF METASTABLE FE-ZN SOLID SOLUTIONS PRODUCED BY MECHANICAL ALLOYING

Zhou-F; Chou-YT; Lavernia-EJ - JOURNAL-OF-MATERIALS-RESEARCH. DEC 2002; 17 (12) : 3230-3236.

Thermal decomposition of supersaturated single-phase body-centered cubic (bcc) Fe_{100-x}Zn_x (5 less than or equal to x less than or equal to 65 at.%) solid solutions, processed via mechanical alloying of high-purity metal powders, was investigated using x-ray diffraction and differential scanning calorimetry (DSC). At elevated temperatures the metastable solid solution



decomposed into a stable equilibrium aggregate consisting of the pure bcc Fe phase and an intermetallic compound Fe_4Zn_9 . The decomposition temperature decreased with increasing Zn concentration. The enthalpy of decomposition for various Fe-Zn solid solutions measured by the DSC was in the range of 1.2-3.5 kJ/mol. The enthalpy of mixing of the as-milled solid solutions from elemental Fe and Zn powders was estimated to be 0.5-1.7 kJ/mol. In addition, the activation energies of decomposition for these solid solutions were determined on the basis of the Kissinger analysis, and their values appeared to be independent of the Zn concentration in the alloy, with an average of 147 +/- 17 kJ/mol.

[37] MECHANICAL-DRY COATING OF WAX ONTO COPPER POWDER BY BALL MILLING

Kim-J; Satoh-M; Iwasaki-T - MATERIALS-SCIENCE-AND-ENGINEERING-A-STRUCTURAL-MATERIALS-
Mechanical dry coating process has been applied to formation of the oxidation protecting film of polymer wax onto spherical copper particles (median diameter of 69.1 μm) using a conventional ball milling. The wax used here showed poor adhesiveness to the metal surface and low plasticity at room temperature due to relatively hard and high melting point (446 K). In order to determine the necessary mechanical force for adhering between the metal and the wax without any deformation of metal particles, the coating experiments were carried out with process variables as processing time, wax content and combination of different kinds of wax. The degree of surface coverage of wax film related to the operating conditions was evaluated by means of the dissolution experiment with acid solution in the optical cell. It has been shown that the addition of the soft wax as a sealant material for filling the cavity of the hard wax was effective to stabilize the coating state and to enhance the degree of coverage.

[36] EFFECT OF MECHANICAL ALLOYING ON THE MORPHOLOGY, MICROSTRUCTURE AND PROPERTIES OF ALUMINIUM MATRIX COMPOSITE POWDERS

Fogagnolo-JB; Velasco-F; Robert-MH; Torralba-JM - MATERIALS-SCIENCE-AND-ENGINEERING-A-STRUCTURAL-MATERIALS-PROPERTIES-MICROSTRUCTURE-AND-PROCESSING. 2003; 342 (1-2) : 131-143.

A composite powder with a fine homogeneous distribution of the reinforcement phase in the whole particle can be obtained by mechanical alloying. Aluminium PM6061 unreinforced, and matrix composite reinforced with Si_3N_4 and AlN powder, are milled in a high-energy attritor mill and the powder properties are compared with those of the same composite composition mixed in a horizontal low-energy ball mill. The correlation observed between the apparent densities and the milling time, explained by the morphological and microstructural evolution of the powder particles during the high-energy milling process, is used to determine the steady state of the process. At short milling times, the apparent density decreases as the milling time is extended, due to the deformation dominant at this stage; at longer milling times, it starts to increase with increasing milling time due to the piling up of the flattened particles and fracture of the welded particles. When mechanical alloying reaches the steady state, the apparent density is stabilized. A simple model is proposed to illustrate the mechanical alloying of a ductile-brittle component system. The particle size distribution and the microhardness of the mechanically alloyed particles are determined

[35] STRUCTURAL PROPERTIES OF $\text{CaCu}_3\text{Ti}_4\text{O}_{12}$ OBTAINED BY MECHANICAL ALLOYING

Almeida-AFL; de-Oliveira-RS; Goes-JC; Sasaki-JM; Souza-AG; Mendes-J; Sombra-ASB - MATERIALS-SCIENCE-AND-ENGINEERING-B-SOLID-STATE-MATERIALS-FOR-ADVANCED-TECHNOLOGY. 2002; 96 (3) : 275-283.

Mechanical alloying has been used successfully to produce nanocrystalline powders of $\text{CaCu}_3\text{Ti}_4\text{O}_{12}$ (CCTO), for the first time, using two different experimental procedures. The milled CCTO were studied by X-ray powder diffraction, infrared and Raman scattering spectroscopy. For two different milling procedures, CCTO was obtained after a couple of hours of milling (in average 30 h of milling, depending on the reaction procedure). The X-ray diffraction (XRD) patterns indicate that the crystallite size is within the range of 20-35 nm. After 100 h of milling the formation of CCTO was confirmed by X-ray powder diffraction in both procedures, with good stability. We also prepare the CCTO ceramic using the traditional procedure described in the literature and compared the physical properties of these samples with those ones obtained by milling process and good agreement was observed. The infrared and Raman scattering spectroscopy results suggest that the increase of the milling time leads to the formation of nanocrystalline CCTO, as seen by XRD analysis. These materials are attractive for capacitor applications and certainly for microelectronics, microwave devices (cell mobile phones for example), where the decrease of the size of the devices are crucial. This milling process presents the advantage that melting is not necessary and the powder obtained is nanocrystalline with extraordinary mechanical properties. The material can be compacted and transformed in solid ceramic samples or used in others procedures of film preparation. The high efficiency of the process opens a way to produce commercial amount of nanocrystalline powders. Due to the nanocrystalline character of this powder, their mechanical properties have changed and for this reason a pressure of 1 GPa is enough to shape the sample into any geometry.

[34] PHASE COMPOSITION AND STRUCTURAL FEATURES OF MECHANICALLY ALLOYED IRON-MANGANESE ALLOYS

Cherdyn'tsev-VV; Kaloshkin-SD; Tomilin-IA; Shelekhov-EV; Baldokhin-YV - OF-METALS-AND-METALLOGRAPHY. OCT 2002; 94 (4) : 391-396.

X-ray diffraction and Mossbauer spectroscopy were used to study Fe-Mn alloys prepared by mechanical alloying. It was shown that the mixing of components at the atomic scale during mechanical alloying occurs in a certain correspondence with the equilibrium phase diagram, but the field of solid solutions is broadened and, correspondingly, the two-phase fields are narrowed. The alloys are characterized by a high density of lattice defects. A substantial concentration of stacking faults was observed in fcc alloys.



[33] PHASE TRANSFORMATIONS IN THE ORDERED Ni₃Al (L1(2)) COMPOUND UPON MECHANICAL ALLOYING WITH AL

Portnoi-VK; Tret'-yakov-KV; Fadeeva-VI - PHYSICS-OF-METALS-AND-METALLOGRAPHY. OCT 2002; 94 (4) : 373-377.

Solid-state synthesis of monoaluminide NiAl by mechanical alloying of Ni₃Al with Al is studied. The mechanical alloying-co-milling of Ni₃Al and Al was carried out at room temperature in an argon atmosphere using a planetary ball mill. It was shown that structural changes in both the intermetallic compound and aluminum start immediately after the beginning of co-milling. The structural changes consist in the disordering of nickel aluminide and dissolution of Al in it, which are accompanied by an increase in the lattice parameter of Ni₃Al and decrease in the Al lattice parameter owing to the dissolution of Ni in Al. At the same time, changes in the substructure—a decrease in the block size (D-Ni(Al) = 15 nm and D-Al = 40 nm) and an increase in the micro-distortions [ϵ]/(1/2) to similar to 0.4-0.5 %—are observed. The subsequent milling leads to the precipitation of phases such as Al₃Ni, Al₂Ni₃, and NiAl belonging to the Al-Ni₃Al composition range. These phases are likely to be nucleated at the interfaces between Ni(Al) and Al(Ni) supersaturated solid solutions. Upon subsequent milling, the phases that have the highest negative enthalpy grow preferentially; because of this, two competitive phases—Al₂Ni₃ and NiAl—remain in the solid solution. Subsequently, the increase in the NiAl content is accompanied by an increase in the degree of long-range order from 0.5 (upon the nucleation) to 0.7 after the 120-min milling, when the monoaluminide remains a single phase. The lattice parameter of NiAl also increases. During further milling, the composition of NiAl becomes close to the stoichiometric composition. However, the degree of long-range order remains low because of the high content of point defects typical of the high-energy strain.

[32] INFLUENCE OF GRINDING ON THE CATALYTIC PROPERTIES OF OXIDES

Rougier-A; Soiron-S; Haihal-I; Aymard-L; Taouk-B; Tarascon-JM- POWDER-TECHNOLOGY. DEC 18 2002; 128 (2-3) : 139-147.

The influence of grinding on the structure and catalytic performances of two families of oxides, namely the perovskite-type La_{0.8}Sr_{0.2}MnO₃ +/- λ and the spinel-type Li-Mn-O, was investigated. Ball-milling of the well-known La_{0.8}Sr_{0.2}MnO₃ (+/- λ) prepared either by a solid state reaction or by a sol-gel method led to (i) a decrease in carbon black combustion temperature of 100 degreesC (T-C = 540 degreesC and of 40 degreesC (TC = 505 degreesC for ceramic and sol-gel ground 5-h samples, respectively, and (ii) to faster kinetics and higher rates of toluene conversion. A greater enhancement of the catalytic performances was obtained by using mechanical milled lithiated manganese oxides that are reported as promising catalyst candidates for the first time. Li-Mn-O catalysts were synthesized by room temperature mechanochemistry of a stoichiometric mixture of Li₂O and MnO₂ using various milling times (0 < t(milling) < 15 h). The nonstoichiometry, large surface area and disorder nature of the ground samples were of great benefit regarding catalytic applications. A remarkable decrease in the carbon black combustion temperature of 200 degreesC (from 650 to 450 degreesC was obtained when using a mixture of Li₂O and MnO₂ ground for 3 or 4 h. This low Tc value favorably compares with the Tc of 500 degreesC of ceramic LiMn₂O₄, which hows, however, better catalytic performances than most of the perovskite-type oxides. The grinding proves to be efficient as well for volatile organic compounds (VOCs) combustion. The inactive ceramic LiMn₂O₄ exhibits a 100% toluene conversion rate for a temperature lower than 200 degreesC when ground 5 h.

[31] LOW TEMPERATURE PREPARATION OF BETA-LiFe₅O₈ FINE PARTICLES BY HYDROTHERMAL BALL MILLING

Ahniyaz-A; Fujiwara-T; Song-SW; Yoshimura-M - SOLID-STATE-IONICS. NOV 2002; 151 (1-4) : 419-423.

Lithium ferrite, beta-LiFe₅O₈, fine particles with a 100-120 nm size were successfully prepared by hydrothermal ball milling at the low temperature of 170 degreesC for 2 h without any post annealing. The lithium ferrite fine particle formation was confirmed by characterization using XRD, SEM, XPS and magnetization measurements. The experimental results strongly support the idea that ball milling can accelerate the reaction not only between the solid and solid phases but also between the solid and liquid phases. We believe hydrothermal ball milling can open a new era for the low temperature synthesis and fabrication of homogeneous fine particles both in composition and size.

[30] MAGNETIC PROPERTIES OF Tb(Fe,Mo)(12) AND Tb(Fe,Mo)(12)C COMPOUNDS

Rixecker-G; Buschow-KHJ; Bakker-H - ZEITSCHRIFT-FUR-METALLKUNDE. OCT 2002; 93 (10) : 997-1001.

Intermetallic compounds in the series TbFe_{12-x}Mo_x, with nominal Mo contents, x, ranging from 1.5 to 3.5, were synthesized and examined for their thermomagnetic properties. Magnetic measurements were performed in the temperature range 5-1130 K by using a SQUID magnetometer and a Faraday balance in combination. Synthesis of interstitially carburized TbFe_{12-x}Mo_xC powders by mechanical alloying with graphite and a subsequent heat treatment was attempted for the single-phase compositions between x = 2 and x = 3. The magnetic behavior of the materials (especially the coercivity) shows a number of peculiarities, which we try to explain in terms of crystal chemical and microstructural features.

[29] PREPARATION AND CHARACTERIZATION OF PLASTICIZED POLYMER ELECTROLYTES BASED ON THE PVDF-HFP COPOLYMER FOR LITHIUM/SULFUR BATTERY

Shin-JH; Jung-SS; Kim-KW; Ahn-HJ; Ahn-JH - JOURNAL-OF-MATERIALS-SCIENCE-MATERIALS-IN-ELECTRONICS. DEC 2002; 13

PVdF-TG-LiX polymer electrolytes comprised of polyvinylidene fluoride (PVdF)-hexafluoropropylene (HFP) copolymer, tetra(ethylene glycol) dimethyl ether as plasticizer, LiCF₃SO₃, LiBF₄ and LiPF₆ as lithium salt and acetone as solvent have been prepared by solvent casting of slurry that mixed PVdF-HFP copolymer with acetone and salt using a ball-milling technique, which was performed for 2 and 12 h with a ball-to-material ratio of 400:1, and their electrochemical and thermal



properties were studied. The ball-milled PVdF-TG-LiX polymer electrolytes have higher ionic conductivity as well as lower glass transition temperature and melting points than the magnetically stirred one. The PVdF-TG-LiPF₆ polymer electrolytes prepared by ball-milling, for, 12 h, in particular, resulted in a maximum value in the ionic conductivity, which was 4.99×10^{-4} S cm⁻¹ at room temperature. The ball-milled PVdF-TG-LiX polymer electrolytes were introduced into Li/S cells with sulfur as cathode and lithium as the anode. The first specific discharge capacities with discharge rate of 0.14 mA cm⁻² at room temperature were about 575 and 765 mA h g-cathode⁻¹ for magnetic stirring and 12 h ball milling.

[28] ENGINEERING PHOSPHORS FOR IMPROVED BRIGHTNESS

Lin-CH; Chiou-BS; Lin-JD - JOURNAL-OF-MATERIALS-SCIENCE-MATERIALS-IN-ELECTRONICS. 2002; 13(12) : 705-711.

Factors affecting the cathodoluminescent (CL) efficiency of phosphors include: morphology, size, stoichiometry, composition, and surface of the phosphors. In the design of phosphors for low-voltage operations, processes such as surface etching are used to enhance phosphor efficiency. In this study, zinc-activated ZnO phosphors were subjected to three processes: acid etching, ball milling, and/or ultrasonic vibration, and the effect of each process on the CL efficiency studied. It is found that the

brightness of phosphors increases 40% after ultrasonic vibration followed by etching twice with 0.005 M HNO₃.

[27] FUNCTIONALIZATION OF MULTIWALL CARBON NANOTUBES: PROPERTIES OF NANOTUBES-EPOXY COMPOSITES

Breton-Y; Delpeux-S; Benoit-R; Salvetat-JP; Sinturel-C; Beguin-F; Bonnamy-S; Desarmot-G; Boufendi-L - MOLECULAR-CRYSTALS-AND-LIQUID-CRYSTALS. 2002; 387 : 359-364

Multiwall nanotubes were functionalized using plasma treatments, chemical oxidation, ball milling and thermal treatments. In optimized conditions, plasmas modify nanotubes surface chemistry with a great selectivity. Vickers microindentation and tension tests performed on epoxy resin loaded with multiwall nanotubes allow comparison of the influence of nanotubes surface chemistry and microtexture on loaded resin mechanical properties

[26] SYNTHESIS OF TITANIUM CARBIDE POWDER FROM TiO₂ AND PETROLEUM COKE BY REACTIVE MILLING

Cui XL. Cui LS. Wang L. Qi M. - Petroleum Science & Technology. 20(9-10):999-1007, 2002.

Synthesis of titanium carbide has been conducted by reactive milling from the blends of titania/titanium/petroleum coke. Two kinds of blend powder, titania/petroleum coke and titania/titanium/petroleum coke, have been milled in the experiment. The results showed that, under the present experiment condition, titanium carbide has been formed in the milled blends of titania/titanium/petroleum coke, but could not be detected in the milled blend of titania/petroleum coke. For the blends of titania/titanium/petroleum coke, the effect of percentage composition of element Ti on the synthesis of TiC has been noticed. The amount of element Ti added played an important role in the transformation, from TiO₂ to TiC

[25] PREPARATION AND CHARACTERIZATION OF PLASTICIZED POLYMER ELECTROLYTES BASED ON THE PVDF-HFP COPOLYMER FOR LITHIUM/SULFUR BATTERY

Shin JH. Jung SS. Kim KW. Ahn HJ. Ahn JH. - Journal of Materials Science-Materials in Electronics. 13(12):727-733, 2002

PVdF-TG-LiX polymer electrolytes comprised of polyvinylidene fluoride (PVdF)-hexafluoropropylene (HFP) copolymer, tetra(ethylene glycol) dimethyl ether as plasticizer, LiCF₃SO₃, LiBF₄ and LiPF₆ as lithium salt and acetone as solvent have been prepared by solvent casting of slurry that mixed PVdF-HFP copolymer with acetone and salt using a ball-milling technique, which was performed for 2 and 12 h with a ball-to-material ratio of 400:1, and their electrochemical and thermal properties were studied. The ball-milled PVdF-TG-LiX polymer electrolytes have higher ionic conductivity as well as lower glass transition temperature and melting points than the magnetically stirred one. The PVdF-TG-LiPF₆ polymer electrolytes prepared by ball-milling, for, 12 h, in particular, resulted in a maximum value in the ionic conductivity, which was 4.99×10^{-4} S cm⁻¹ at room temperature. The ball-milled PVdF-TG-LiX polymer electrolytes were introduced into Li/S cells with sulfur as cathode and lithium as the anode. The first specific discharge capacities with discharge rate of 0.14 mA cm⁻² at room temperature were about 575 and 765 mA h g-cathode⁻¹ for magnetic stirring and 12 h ball milling.

[24] EFFECTS OF HIGH-ENERGY BALL MILLING (HEBM) ON THE STRUCTURE AND ELECTROCHEMICAL PERFORMANCE OF NICKEL HYDROXIDE

Chen H. Wang JM. Pan T. Xiao HM. Zhang JQ. Cao CN. - International Journal of Hydrogen Energy. 28(1):119-124, 2003

A method of high-energy ball milling (HEBM) was first used to modify the structure and electrochemical performance of nickel hydroxide for the active material of Ni/MH battery. The experimental results showed that HEBM is an effective method to improve the electrochemical performance of beta-Ni(OH)₂ such as specific discharge capacity, discharge potential and cycle performance. But the performance of alpha-Ni(OH)₂ was greatly reduced by HEBM processing. The results of electrochemical impedance spectroscopy, powder X-ray diffraction and particle size distribution indicated that the improvement of the performance of beta-Ni(OH)₂ through HEBM was attributed to the reduction of the charge-transfer resistance (R-t), the diffusion impedance (Z(w)), which was resulted from the decrease of the crystallite and particle size and the increase of interlayer spacing. However, for alpha-Ni(OH)₂ samples, those structure changes after HEBM resulted in the increase of the R-1 and Z(w) and the deterioration of its electrochemical performance

[23] MECHANOCHEMICAL SYNTHESIS AND CHARACTERISATION OF NANOPARTICULATE SAMARIUM-DOPED CERIUM OXIDE

Hos JP. McCormick PG. - Scripta Materialia. 48(1):85-90, 2003



The synthesis of 20 mol% samarium-doped cerium oxide nanoparticles was studied from the mechanochemical reaction between samarium trichloride, sodium hydroxide and cerium hydroxide. XRD and high-resolution TEM characterisation showed chemically homogeneous 10-30 nm crystallites having a lattice parameter of 5.435 Angstrom and a surface area of 48.7 m²/g.

[22] NANOSTRUCTURED GRAPHITE-HYDROGEN SYSTEMS PREPARED BY MECHANICAL MILLING METHOD

Orimo SI. Matsushima T. Fujii H. Fukunaga T. Majer G. Zuttel A. Schlappbach L. - *Molecular Crystals & Liquid Crystals*. 386:173-178, 2002.

Nanostructured graphite was prepared by mechanical milling under a hydrogen atmosphere. Several samples obtained after different milling times were systematically examined to get fundamental information about the structures, hydrogen concentrations, and also hydrogen desorption properties. The hydrogen concentration reaches up to 7.4 mass% (CH_{0.95}) after milling for 80 h, and two desorption peaks of hydrogen molecule (mass-number=2), starting at about 600 K and 950 K respectively, are observed in thermal desorption mass-spectroscopy in the sample. Below the temperature of the second desorption peak, at which recrystallization related desorption occurs, the nanostructured graphite is expected to retain its specific defective structures mainly with carbon dangling bonds as suitable trapping sites for hydrogen storage.

[21] HRTEM OBSERVATION OF BALL-MILLED LAMP SHADE CARBON NANOFIBER

Hayashi T. Kim YA. Fukai Y. Endo M. Yanagisawa T. - *Molecular Crystals & Liquid Crystals*. 387:365-368, 2002.

High-resolution transmission electron microscopy study of ball-milled lampshade carbon nanofibers is reported in the present paper. Milled fibers became shorter in length, preserving the lampshade structure with highly ordered graphene layers. We have also shown the simulated model for the shortening process of the fiber.

[20] PZT CERAMICS OBTAINED FROM MECHANOCHEMICALLY SYNTHESIZED POWDERS

Brankovic Z. Brankovic G. Varela JA. - *Journal of Materials Science-Materials in Electronics*. 14(1):37-41, 2003

PZT ceramics were obtained from the mechanochemically synthesized powders. Milling and sintering conditions were optimized based on results of density measurements, as well as on microstructural and electrical characterization. As a result, highly dense and homogeneous ceramics were obtained. Excellent microstructural properties resulted in good electrical properties. Samples showed values of dielectric constants reaching 12800 at the Curie temperature, as well as low dielectric loss under the optimal processing conditions. High values of remanent polarization, reaching 60 μC cm⁻², indicate high internal polarizability.

[19] MICROSTRUCTURAL PROPERTIES OF BULK NANOCRYSTALLINE AG-NI ALLOY PREPARED BY HOT PRESSING OF MECHANICALLY PRE-ALLOYED POWDERS

Wang CL. Lin SZ. Niu Y. Wu WT. Zhao ZL. - *Applied Physics a (Materials Science Processing)*. 76(2):157-163, 2003

A bulk nanocrystalline Ag₅₀Ni alloy has been prepared by hot-pressing the mechanically pre-alloyed powders at 620°C under a normal pressure of 58 MPa in vacuum. The microstructural characteristics of the alloy were studied by X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The results of the precise determination of the lattice parameters of the phases in the powders and in the alloy by XRD show that, after mechanical alloying for 200 h, the solid solubility of Ag in Ni reaches 4.85 ± 0.21 at %, while that of Ni in Ag reaches 0.84 ± 0.30 at %. After hot pressing, the Ag- and Ni-rich phases in the alloy still show a certain degree of supersaturation, with a solid solubility of 0.45 ± 0.11 at % of Ag in Ni. After further annealing of the alloy at 700 °C for 24 h, the solubility decreases to a value of 0.21 ± 0.11 at % for Ag in Ni and to less than 0.1 at % for Ni in Ag. The grain size of the mechanically alloyed powders was of ca 6 nm. After hot pressing, the grain size of the alloy increased to 40-60 nm and then grew further to 100-110 nm after annealing. The influence of the variation of the grain size and the internal stress on the line breadth of the X-ray diffraction peaks has been evaluated in detail. Finally, the role of the nanocrystalline structure in the fast densification process of the powders is also discussed.

[18] MECHANOCHEMICAL EFFECTS ON SYNTHESIS OF RHABDOPHANE-TYPE NEODYMIUM AND CERIUM PHOSPHATES

Onoda H. Nariai H. Maki H. Motooka I. - *Materials Chemistry & Physics*. 78(2):400-404, 2002

Mechanochemical effects were investigated in the mixture of a rare earth compound (Nd(NO₃)(₃).6H₂O, NdCl₃.6H₂O, Nd-2(CO₃)(₃).8H₂O, Ce(NO₃)(₃).6H₂O, CeCl₃.7H₂O, or Ce-2(CO₃)(₃).8H₂O) and (NH₄)(₂)HPO₄. These mixtures were ground and then heated. These ground mixtures and thermal products were characterized by X-ray diffraction (XRD), Fourier transform-infrared spectroscopy (FT-IR), thermogravimetry-differential thermal analyses (TG-DTA), and adsorption of nitrogen. By grinding, Rhabdophane-type rare earth (Nd or Ce) phosphate was formed in the mixture of Nd(NO₃)(₃).6H₂O-(NH₄)(₂)HPO₄ or Ce(NO₃)(₃).6H₂O-(NH₄)(₂)HPO₄. By mechanical treatment, Rhabdophane-type neodymium phosphate was formed at lower temperature in the mixture of NdCl₃.6H₂O-(NH₄)(₂)HPO₄. The mixture of CeCl₃.7H₂O-(NH₄)(₂)HPO₄ indicated XRD peaks of CeO₂ by heating, whereas ground mixtures transformed to Rhabdophane-type cerium phosphate. Specific surface areas of Rhabdophane-type neodymium and cerium phosphates verified from 0.1 to 120 m²/g depending on various synthetic conditions. Rhabdophane-type neodymium and cerium phosphates which had large specific surface area were obtained by grinding with ethanol and then heating

[17] INFLUENCE OF MILLING LIQUIDS AND ADDITIVES ON PARTICLE SIZE REDUCTION AND SINTERING BEHAVIOUR OF AL₂O₃

Sathiyakumar M. Gnanam FD. - *British Ceramic Transactions*. 101(5):200-207, 2002

The grinding process reduces particle size, enhances sinterability, and promotes the development of improved microstructure. A sol-gel derived alumina powder was wet milled at various solids loadings and the optimum loading



(solids/water = 1(.0):1(.0)) determined, with respect to particle size reduction and an optimised milling tune of 6 h. The use of propanol, methanol, and ethanol as milling liquids improved the powder characteristics. Fine particle agglomeration was found to be reduced by the addition of milling additives (magnesium stearate, strontium stearate, and ammonium polyacrylate) at various concentrations. These additives were also found to modify grain growth. Moreover magnesium stearate reduced the grain growth considerably and uniform microstructure was observed. The presence of milling additives increases the fineness of the powder and the sintered density. It is concluded that the Presence of milling additives results in adsorption of the long chain additive molecules over the particle surfaces, thus decreasing frictional and cohesive interactions between them, and thereby altering the flow properties of the mill content during grinding, which in turn enhances the overall grinding process.

[16] HIGH-ENERGY BALL MILLING OF NiAl(Fe) SYSTEM

Principi G. Spataru T. Maddalena A. Gialanella S. - *Hyperfine Interactions*. 139(1-4):315-324, 2002.

High-energy ball milling was used to promote the solubilization of iron into NiAl powder for an iron concentration range of 10-30 wt.%. The microstructural evolution induced by the intense mechanical deformations, under different milling conditions, was followed by X-ray diffraction and Mossbauer spectroscopy. The Mossbauer spectra are dominated by a magnetic sextet of about 33 T. Increasing the time and the speed of milling gives rise to a non-resolved doublet, having parameters typical of a NiAl compound with Fe atoms in solution. At the same time a reduction of lattice parameter occurs, which can be correlated to composition variations and partial disordering of the NiAl structure. Subsequent annealing modifies the Mossbauer spectra noticeably. In particular, the nonmagnetic component becomes a broad singlet. Both diffraction analysis and Mossbauer spectroscopy indicate that a fcc Ni(Al,Fe) solid solution is forming in samples milled in agate. It is observed that the grain size of the milled products remains in the nanometric range even after thermal treatment, which adds interest to possible applications.

[15] HIGH-TEMPERATURE MOSSBAUER SPECTROSCOPY OF MECHANICALLY MILLED NiFe₂O₄

Helgason O. Jiang JZ. - *Hyperfine Interactions*. 139(1-4):325-333, 2002.

Oxide spinels, in particular those containing iron, often exhibit technically important electrical- and magnetic-properties. We report here on X-ray powder diffraction and Mossbauer studies of nanostructured NiFe₂O₄ particles prepared by high-energy ball milling from bulk NiFe₂O₄, which is an inverse spinel. The Mossbauer spectra were recorded in situ at different temperatures in the range of 300-850 K. The Mossbauer spectra of the milled samples show a broad distribution of magnetic hyperfine fields together with a paramagnetic state at room temperature. Initially, at 700 K the spectrum is mainly paramagnetic, but during the process of annealing, magnetic sextets emerge. The treatment results in a significant change in the B/A area ratio of the ferrite. The Neel temperature of the samples is estimated from the B(T) relation to be in the range of 800-850 K

[14] MOSSBAUER SPECTROSCOPY STUDY OF Fe-Si SOLID SOLUTION PREPARED BY MECHANICAL MILLING

Lehlooh AF. Fayyad SM. Mahmood SH. - *Hyperfine Interactions*. 139(1-4):335-344, 2002.

A specimen of Fe-Si solid solution is prepared by ball milling of proper amounts of the pure elements (3Fe:Si) for different milling times. X-ray diffraction and Mossbauer spectroscopy have been used to characterize the solid solution. The Mossbauer spectra show four different sites corresponding to Fe atoms in a bcc structure having 0, 1, 2 and 3 Si atoms in the 1st nearest-neighbor (nn) shell. The hyperfine magnetic field decreases by 31 kOe for each Si atom in the 1st nn shell. A magnetic component with hyperfine field around (180 kOe) characterized by a broadened sextet was observed which could be due to iron sites having more than 3 Si atoms in the 1st nn. A theoretical model based on the binomial distribution was adopted to analyze the data. Good agreement between the experimental and the theoretical hyperfine field distribution in the high hyperfine field region was found, and the silicon content in the disordered A2 phase is deduced from the parameters which give the best agreement

[13] PARTIAL REDUCTION OF MgFe₂O₄ WITH AL AND MG VIA BALL MILLING

Takacs L. Garg VK. Soika V. Oliveira AC. - *Hyperfine Interactions*. 139(1-4):345-354, 2002.

Nanocomposites consisting of metallic Fe particles and a nonmagnetic oxide were prepared by reducing MgFe₂O₄ with Al or Mg in a ball mill. The reaction takes place as a fast self-propagating process sometime between 0.5 and 1 h of milling. Combining XRD and Mossbauer spectroscopy reveals that the bcc Fe phase contains a few percents of dissolved Al, it has a high defect concentration, and the surface tension of the ultrafine (about 15 nm) grains results in local compressive strains. The magnetization is 25% less than expected for pure ferromagnetic Fe.

[12] MECHANOCHEMICAL TRANSFORMATIONS IN ALPHA-Fe₂O₃ - ICEMS STUDY

Campbell SJ. Klingelhofer G. Kaczmarek WA. Hofmann M. Nagel R. Wang G. - *Hyperfine Interactions*. 139(1-4):407-416, 2002.

Integral conversion electron Mossbauer spectroscopy has been used to investigate the phases and transformations which occur when alpha-Fe₂O₃ is wet-milled in vacuum for up to 144 h. In addition to the transformation to off-stoichiometric Fe_{3-x}O₄ observed previously by transmission Mossbauer spectroscopy, the room temperature ICEMS spectra reveal the presence of similar to 10-15% gamma-Fe₂O₃ on the surfaces of particles milled for more than 24 h. Time-of-flight neutron diffraction of an alpha-Fe₂O₃ sample wet-milled for 200 h also provides evidence of the occurrence of a small fraction (similar to 4%) of gamma-Fe₂O₃ in the milled product. The gamma-Fe₂O₃ is likely to occur as a surface oxide layer and does not appear to play a significant role in the mechanochemical transformation of alpha-Fe₂O₃ to Fe_{3-x}O₄.

[11] HIGH-ENERGY BALL-MILLING OF ALLOYS AND COMPOUNDS

Le Caer G. Delcroix P. Begin-Colin S. Ziller T. - *Hyperfine Interactions*. 141(1-4):63-72, 2002.



After outlining some characteristics of high-energy ball-milling, we discuss selected examples of phase transformation and of alloy synthesis which focus on deviations from archetypal behaviours and throw light on the milling mechanisms. Some contributions of Mossbauer spectrometry to the characterization of ground materials are described

[10] PHASE TRANSFORMATIONS IN FE-MN ALLOYS INDUCED BY BALL MILLING

Cotes SM. Cabrera AF. Damonte LC. Mercader RC. Desimoni J. - *Hyperfine Interactions*. 141(1-4):409-414, 2002.

We have studied the dependence on the milling time of the amounts of metastable fcc and hcp phases produced by ball milling on Fe-13.7 wt% Mn alloys by Mossbauer spectroscopy and X-ray diffraction. The relative proportions of gamma-Fe(Mn)/fcc and epsilon-Fe(Mn) hcp increase with milling times up to 9 h and decline afterwards. A random solid solution of Mn in the alpha-Fe matrix is found for milling times up to 15 h. Mn segregation is observed after 25 h. The evolution of the relative percentages suggests that ball milling brings about processes that on the one hand induce the fcc/hcp martensitic transformation, and on the other favour transformations towards the phases dictated by the thermodynamic phase diagram

[9] NANOTUBE GROWTH DURING ANNEALING OF MECHANICALLY MILLED BORON

Fitzgerald JD. Chen Y. Conway MJ. - *Applied Physics B-Lasers & Optics*. 76(1):107-110, 2003

Boron powder, finely ground in a tungsten carbide ball mill in an ammonia atmosphere, has been annealed at 1200 degreesC in flowing nitrogen to produce small quantities of cylindrical BN nanotubes, both as isolated individuals and grouped into ropes. Thick-walled conical BN tubes are abundant in specimens annealed for longer times, and their growth was catalysed once WC debris was converted into W metal particles. Some catalytic effect of small W nanoparticles could be necessary for nanotube formation, though no tip particles have been imaged here. Given the low temperature of mechanical milling and annealing, BN growth must involve surface diffusion and solid-state reconfiguration. It could be possible to engineer desirable physical and chemical properties by exploiting the variation in cylindrical versus conical BN structures as a function of annealing time

[8] ATOMIC HYDROGEN AS SPIN PROBE IN THERMALLY AND MECHANICALLY ACTIVATED MATERIALS

Scholz G. Stosser R. - *Physical Chemistry Chemical Physics*. 4(22):5448-5457, 2002

Atomic hydrogen H \cdot could be stabilized at T greater than or equal to 293 K in specially prepared fluoride and oxide compounds of aluminium: {AlF(3):OH} and {AlOOH $_x$ }. The formation of atomic hydrogen was accomplished using irradiation including gamma rays, X rays and UV radiation, and even sunlight, that is those wavelengths in the region of lambda less than or equal to 254 nm. The hyperfine coupling constant of H \cdot @ AlF $_3$ could be precisely determined by using H \cdot @ MeT8 as a standard to calibrate the B-0 field. In the samples mentioned above H \cdot acts a spin probe in two ways: (i) that it can be trapped in cages and stabilized with Pauli repulsion indicates sensitively and unambiguously that there are local host nano-cages present in the matrices and, moreover, (ii) that it acts as a spin probe for determining the matrix state of order with its spin dynamics as well as with the kinetic parameters of detrapping.

[7] AN INVESTIGATION ON THE TRANSFORMATION OF THE ICOSAHEDRAL PHASE IN THE AL-FE-CU SYSTEM DURING MECHANICAL MILLING AND SUBSEQUENT ANNEALING

Mukhopadhyay NK. Yadav TP. Srivastava ON. - *Philosophical Magazine A-Physics of Condensed Matter Defects & Mechanical Properties*. 82(16):2979-2993, 2002

Icosahedral quasicrystalline material in the Al-Fe-Cu system was mechanically milled in an attritor ball mill (Szegvari attritor) for 1, 3, 6 and 10 h in dry air, at a speed of 400 rev min $^{-1}$ and with a ball-to-powder ratio of 20 to 1. Structural transformations and the consequent phase evolutions during mechanical milling and subsequent heat treatments were studied by X-ray diffraction, differential thermal analysis (DTA) and transmission electron microscopy techniques. After milling for 1 h, the evolution of disordered B2 phase (a = 0.29 nm) was observed to coexist with the parent icosahedral phase, whereas a microstructure consisting of nanosized B2 particles distributed in an amorphous matrix was observed after further milling (3-10 h). However, no sharp peak corresponding to the phase transformation was identified in DTA, but the microstructure of the powder milled for 10 h after the DTA experiment was found to transform to a mixture of icosahedral and B2 phases, where B2 appears to be the major phase in contrast with that in as-cast material. Isothermal heat treatment of powder mechanically milled at 850degreesC for 10 and 20 h led to complete transformation to a single B2 phase with a high degree of long-range ordering. The implication of these transformations will be discussed with reference to their relative stabilities among the competing phases during milling and subsequent heat treatment.

[6] PHASE TRANSFORMATION AND THERMOELECTRIC PROPERTIES OF N-TYPE Fe $_{0.98}$ Co $_{0.02}$ Si $_2$ PROCESSED BY MECHANICAL ALLOYING

Ur SC. Kim IH. - *Materials Letters*. 57(3):543-551, 2002

N-type Fe $_{0.98}$ Co $_{0.02}$ Si $_2$ powders have been produced by mechanical alloying process and consolidated by vacuum hot pressing. As-milled powders were of metastable state and fully transformed to beta-FeSi $_2$ phase by subsequent isothermal annealing. However, as-consolidated iron silicides consisted of untransformed mixture of alpha-Fe $_2$ Si $_5$ and epsilon-FeSi phases. Isothermal annealing has been carried out to induce the transformation to a thermoelectric semiconducting beta-FeSi $_2$ phase. The transformation behavior of beta-FeSi $_2$ was investigated by utilizing DTA, a modified TGA, SEM and XRD analyses. Isothermal annealing at 830 degreesC in vacuum led to the thermoelectric semiconducting beta-FeSi $_2$ phase transformation, but some residual metallic alpha- and (a) over circle -phases were unavoidable even after 96 h of annealing. Microstructures of iron silicides were investigated using SEM and TEM. Thermoelectric properties of beta-FeSi $_2$ materials before and after isothermal annealing were evaluated in this study. It was shown that thermoelectric properties were remarkably enhanced by isothermal annealing due to the transformation from metallic alpha- and epsilon-phases to semiconducting beta-phases.

[5] COMPARATIVE STUDY OF DENSE BULK MgB $_2$ MATERIALS PREPARED BY DIFFERENT METHODS

Narozhnyi VN. Fuchs G. Handstein A. Gumbel A. Eckert J. Nenkov K. Hinz D. Gutfleisch O. Walte A. Bogacheva LN. Kostyleva IE. Muller KH. Schultz L. - *Journal of Superconductivity*. 15(6):599-601, 2002

We report on the results of a comparative investigation of highly dense bulk MgB $_2$ samples prepared by three methods: (i) hot deformation; (ii) high pressure sintering; and (iii) mechanical alloying of Mg and B powders with subsequent hot compaction. All types of samples were studied by AC susceptibility, DC magnetization, and resistivity measurements in



magnetic fields up to $\mu(0)H = 160$ kOe. A small but distinct anisotropy of the upper critical field $H_{c2}(a,b)/H_{c2}(c)$ similar to 1.2 connected with some texture of MgB₂ grains was found for the hot deformed samples. The samples prepared by high pressure sintering as well as by mechanical alloying show improved superconducting properties, including high upper critical fields $H_{c2}(\mu(0)H_{c2}(0))$ similar to 23 T, irreversibility fields H_{irr} which are strongly shifted towards higher values $H_{irr}(T)$ similar to $0.8H_{c2}(T)$ and high critical current $J(c)(J(c) = 10(5) \text{ A/cm}^2)$ at 20 K and 1 T

[4] MAGNETIC AND MAGNETORESISTANCE PROPERTIES OF LA_{0.825}SR_{0.175}MNO₃ POWDER COMPACT

Li FY. Jin CQ. Zhao HB. Zhu JL. Wang AH. Yu RC. - Journal of Physics-Condensed Matter. 14(44):10709-10712, 2002
The magnetic and magnetoresistance properties of Lao(0.825)Sr(0.175)MnO(3) powder compact prepared by the mechanical alloying method and high temperature high pressure treatment have been investigated. Analysis by means of x-ray diffraction and scanning electron microscopy show that the La_{0.825}Sr_{0.175}MnO₃ powder has an average grain size of 100 nm and has been partly non-crystallized by mechanical alloying. Compared with the crystal and general nanogranular samples of the same material, the powder compact is considerably different in both magnetic and magnetoresistance properties

[3] PREPARATION OF FINE-GRAINED BULK MATERIALS IN THE FE-CO SYSTEM BY SHOCK COMPRESSION

Mashimo T. Fan X. Huang XS. Murata H. Sakakibara M. - Journal of Physics-Condensed Matter. 14(44):10825-10828, 2002

Fine-grained bulk alloys with no crack in the 70:30 mol% Fe-Co system were prepared by means of shock compression of water-atomized powder and mechanical alloying (MA) treated ones. The grain size of the water-atomized bulk body was smaller (<50 μm) than that of the molten bulk body (about 100 μm). The grain size decreased greatly with the MA treatment time, and ones for 21 h were estimated to be about 15 nm from the x-ray diffraction patterns. The coercivity value of the water-atomized bulk body was much larger than that of the molten bulk body. The coercivity value of the MA-treated bulk body increased with the MA treatment time, and then decreased, despite the very small grain size, probably due to the effect of ferromagnetic exchange interaction.

[2] THE EFFECT OF PRESSURE ON THE CURIE TEMPERATURE IN FE-NI INVAR MECHANICAL ALLOYS

Wei S. Duraj R. Zach R. Matsushita M. Takahashi A. Inoue H. Ono F. Maeta H. Iwase A. Endo S. - Journal of Physics-Condensed Matter. 14(44):11081-11084, 2002

Measurements of the temperature dependence of the AC susceptibility were made for Fe-Ni Invar mechanical alloys under hydrostatic pressures up to 1.5 GPa. The Curie temperatures decreased linearly with pressure. The rate of decrease became larger for specimens annealed at higher temperatures. The temperature of annealing after ball milling has been directly related to the extent of the chemical concentration fluctuation, and the extent becomes smaller for specimens annealed at higher temperature. This tendency can be explained by assuming a Gaussian distribution function.

[1] THE DIFFERENCE BETWEEN THE CRYSTALLIZATION PROCESSES INDUCED BY MECHANICAL MILLING AND ANNEALING UNDER NORMAL AND HIGH PRESSURE IN AMORPHOUS FE-N ALLOY

Liu L. Liu SE. Guo XY. Zhao XD. Yao B. Su WH. - Journal of Physics-Condensed Matter. 14(44):11157-11160, 2002
An amorphous Fe-N alloy was prepared by ball milling a mixture of Fe and h-BN. Its crystallization processes induced by mechanical milling (MM) and annealing, under normal and high pressure were studied. The crystallization product of the amorphous Fe-N alloy induced by MM and annealing at temperatures between 690 and 800 K under pressures of 3-4 GPa is ϵ -Fe_xN, while the thermal crystallization product under normal pressure is γ' -Fe₄N. The difference between the crystallization products produced by mechanical and thermal crystallization is attributed to the effects of local pressure and local temperature produced by ball collisions.

