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#### **Regular** Articles

One-pot synthesis of superacid catalytic material  $SO_4^{2-}/ZrO_2\mbox{--}SiO_2$  with thermostable well-ordered mesoporous structure

Ruifeng Li, Feng Yu, Fuxiang Li, Meimei Zhou, Bingshe Xu and Kechang Xie *Page 991* 



TEM images of the calcined mesoporous catalyst  $MSC_x$  (x=1.1), in which no aggregated  $ZrO_2$  particles can be observed. The corresponding electron diffraction (insets in figures) indicated the final product was a mesoporous material with a crystalline framework.

Ti<sub>2</sub>GaC, Ti<sub>4</sub>GaC<sub>3</sub> and Cr<sub>2</sub>GaC—Synthesis, crystal growth and structure analysis of Ga-containing MAX-phases  $M_{n+1}$ GaC<sub>n</sub> with M = Ti, Cr and n = 1, 3Johannes Etzkorn, Martin Ade, Dominik Kotzott, Monique Kleczek and Harald Hillebrecht *Page 995* 



Single crystals of Ga-containing MAX-Phases  $(TiC)_n(TiGa)$  (n = 1, 3) were grown from a metallic melt including and characterised by X-ray diffraction.  $Ti_4GaC_3$  is one of the few 413-phase and the first containing Ga.

#### Regular Articles—Continued

Facile fabrication of rutile monolayer films consisting of well crystalline nanorods by following an IL-assisted hydrothermal route

Peng Peng, Xiaodi Liu, Chuansheng Sun, Jianmin Ma and Wenjun Zheng *Page 1003* 



The rutile film consisting of rectangular nanorods is successfully deposited on glass substrate in presence of ionic liquid (IL) of [Bmim]Br. The nanorods were rectangular with width of 100–200 nm and length of more than 1  $\mu$ m, which grew up typically along *c*-axis to form the arrays against the substrate.

### Comprehensive survey of Nd<sup>3+</sup> substitution In La<sub>2</sub>Mo<sub>2</sub>O<sub>9</sub> oxide-ion conductor

Gwenaël Corbel, Pierrick Durand and Philippe Lacorre *Page 1009* 



Metastable–stable phase diagram of the solid solution  $La_{2-x}Nd_x$ Mo<sub>2</sub>O<sub>9</sub> determined from DTA. The topological metastability of the high-temperature cubic  $\beta$ -form which goes back to monoclinic stable  $\alpha$ -form above 450–500 °C is shown in the intermediate range  $0.4 \le x \le 1.2$ . Above x = 1.2, this transition progressively changes into a thermal trace of the postulated Arrhenius/VTF transformation of transport mechanism. Synthesis, structure, and magnetic and electronic properties of Cs<sub>2</sub>Hg<sub>2</sub>USe<sub>5</sub>

Daniel E. Bugaris, Daniel M. Wells and James A. Ibers *Page 1017* 



View of the crystal structure of Cs<sub>2</sub>Hg<sub>2</sub>USe<sub>5</sub>.

### Synthesis, crystal structure, and electrical and magnetic properties of $Ce_3MoO_7$

Philippe Gall and Patrick Gougeon Page 1035



We report the synthesis, crystal structure, and electrical and magnetic properties of the novel compound  $Ce_3MoO_7$  containing infinite chains of trans-corner-sharing  $MoO_6$  octahedra.

### A fast route to obtain manganese spinel nanoparticles by reduction of K-birnessite

F. Giovannelli, T. Chartier, C. Autret-Lambert, F. Delorme, M. Zaghrioui and A. Seron *Page 1021* 



TEM image showing  $Mn_3O_4$  particle after treatment of birnessite with an addition of hydrazine during 24 hours.

Syntheses, structures and properties of a series of photochromic hybrids based on Keggin tungstophosphates Li-Zhi Zhang, Wen Gu, Zhili Dong, Xin Liu, Bing Li and Mei-Ling Liu Page 1040



A series of photochromic hybrids based on Keggin tungstophosphates and lanthanides have been synthesized and characterized by elemental analysis, IR, UV–vis, and single-crystal X-ray diffraction. Their photochromic, magnetic, and luminescent properties have been studied.

### High temperature phase transition in SOFC anodes based on $Sr_2MgMoO_{6-\delta}$

D. Marrero-López, J. Peña-Martínez, J.C. Ruiz-Morales, M.C. Martín-Sedeño and P. Núñez *Page 1027* 



The double perovskite  $Sr_2MgMoO_6$ , recently proposed as an efficient SOFC anode for direct hydrocarbon oxidation, exhibits a reversible structural phase transition from triclinic to cubic at 275 °C.

Solvothermal synthesis and luminescent properties of monodisperse LaPO<sub>4</sub>:Ln ( $Ln = Eu^{3+}$ ,  $Ce^{3+}$ ,  $Tb^{3+}$ ) particles Piaoping Yang, Zewei Quan, Chunxia Li, Zhiyao Hou, Wenxin Wang and Jun Lin *Page 1045* 



Monodisperse rare-earth ion  $(Eu^{3+}, Ce^{3+}, Tb^{3+})$  doped LaPO<sub>4</sub> phosphors with monazite-type structure and uniform oval morphology and strong emission intensity have been prepared through a facile solvothermal process.

#### Cobalt oxide hollow microspheres with micro- and nano-scale composite structure: Fabrication and electrochemical performance

Feifei Tao, Cuiling Gao, Zhenhai Wen, Qiang Wang, Jinghong Li and Zheng Xu Page 1055



 $Co_3O_4$  hollow microspheres self-assembled by nanosheets are successfully fabricated by a template-free wet-chemical approach. The hollow microspheres are in good morphology purity and homogeneous size.  $Co_3O_4$  hollow microspheres constructed by porous nanosheets show the high discharge capacity of  $1048 \text{ mAh g}^{-1}$ , indicating it to be the potential electrode material of Li-ion battery.

### Sonochemical fabrication of fluorinated mesoporous titanium dioxide microspheres

Changlin Yu, Jimmy C. Yu and Mui Chan *Page 1061* 



A novel method for preparing fluorinated mesoporous  $TiO_2$  microspheres was developed by a combined ultrasonic and hydrothermal treatment. The fluorinated  $TiO_2$  microspheres show high crystallinity, stability and enhanced photocatalytic activity.

Precipitation synthesis of lanthanide hydroxynitrate anion exchange materials,  $Ln_2(OH)_5NO_3 \cdot H_2O$  (Ln = Y, Eu–Er) Sheena A. Hindocha, Laura J. McIntyre and Andrew M. Fogg *Page 1070* 



New anion exchangeable layered hydroxynitrates,  $Ln_2(OH)_5NO_3$ .  $H_2O$  (Ln=Y, Eu - Er) have been synthesized via a precipitation route. These materials have been shown to be very flexible intercalation hosts undergoing facile exchange reactions with organic carboxylate and sulfonate anions.

### Syntheses, structures, magnetism, and optical properties of gadolinium scandium chalcogenides

Geng Bang Jin, Eun Sang Choi and Thomas E. Albrecht-Schmitt

Page 1075



A view of the three-dimensional structure of  $Gd_{3.04}Sc_{0.96}S_6$  along the *c* axis.

#### Formation of calcium carbonate films on chitosan substrates in the presence of polyacrylic acid

Linghao He, Rui Xue and Rui Song Page 1082



Chitosan membranes with different degrees of deacetylation (DA) are employed as support to culture calcium carbonate (CaCO<sub>3</sub>). In high concentration of polyacrylic acid (PAA), the CaCO<sub>3</sub> films obtained consisted of vaterite. However, the CaCO<sub>3</sub> film grown on chitosan with 8% DA mainly consisted of vaterite as opposed to aragonite for chitosan with 8% DA.

### CTAB-assisted synthesis and photocatalytic property of CuO hollow microspheres

Shunli Wang, Hui Xu, Liuqin Qian, Xi Jia, Junwei Wang, Yangyi Liu and Weihua Tang *Page 1088* 



Dandelion-like CuO hollow microspheres were fabricated through a hydrothermal method. The prepared products exhibited a high photocatalytic activity for the photocatalytic decolorization of Rhodamine B aqueous solution under UV-light illumination.

# Montmorillonite-based porous clay heterostructures (PCHs) intercalated with silica-titania pillars—synthesis and characterization

Lucjan Chmielarz, Barbara Gil, Piotr Kuśtrowski, Zofia Piwowarska, Barbara Dudek and Marek Michalik

Page 1094



Synthesis of the montmorillonite based porous clay heterostructures (PCHs) intercalated with silica-titania pillars has been performed. The mechanism of the thermal degradation of organic templates in the pore system of PCHs was studied. PCHs were characterized with respect to structure, texture, composition, surface acidity, thermal stability and form of introduced titanium.

### XPS spectra of the $U^{5+}$ compounds $KUO_3,\,NaUO_3$ and $Ba_2U_2O_7$

J.-H. Liu, S. Van den Berghe and M.J. Konstantinović *Page 1105* 



U4f X-ray photoelectron spectroscopy (XPS) spectra of NaUO<sub>3</sub>, KUO<sub>3</sub> and Ba<sub>2</sub>U<sub>2</sub>O<sub>7</sub>, indicating single valence state of uranium in these compounds. U4f XPS spectra of UO<sub>2</sub> (U<sup>4+</sup>) and Cs<sub>2</sub>U<sub>4</sub>O<sub>13</sub> (U<sup>6+</sup>) are shown for a comparison.

Coupled  $Li^{1+}/Nb^{5+}$  and  $O^{2-}/F^{-}$  ordering on the Na and Cl sites of the average NaCl structure of  $Li_4NbO_4F$ Lasse Norén, Ray L. Withers, Darren J. Goossens, Margaret Elcombe and Gordon J. Kearley *Page 1109* 



An  $\langle 001 \, \rangle$  zone axis EDP typical of Li\_4NbO\_4F.

# Crystal structure and thermal expansion of the low- and high-temperature forms of $BaM^{IV}(PO_4)_2$ compounds (M = Ti, Zr, Hf and Sn)

D. Bregiroux, K. Popa, R. Jardin, P.E. Raison, G. Wallez, M. Quarton, M. Brunelli, C. Ferrero and R. Caciuffo *Page 1115* 



The layered high-temperature form of  $BaM(PO_4)_2$ , only expands along the *c*-axis.

### Thermodynamic quantities and defect equilibrium in $La_{2-x}Sr_xNiO_{4+\delta}$

Takashi Nakamura, Keiji Yashiro, Kazuhisa Sato and Junichiro Mizusaki

Page 1121



In order to elucidate the relation between thermodynamic quantities, the defect structure, and the defect equilibrium in  $La_{2-x}Sr_xNiO_{4+\delta}$ , statistics thermodynamic calculation is carried out and calculated results are compared to those obtained from experimental data.

Barium aluminides  $Ba_xAl_5$  (x = 3, 3.5, 4) Michael Jehle, Harald Scherer, Marco Wendorff and Caroline Röhr *Page 1129* 



Al<sub>5</sub> layers of Kagomé nets in the new binary electron precise *Zintl* compound Ba<sub>3.5</sub>Al<sub>5</sub>, also found in Ba<sub>3</sub>Al<sub>5</sub> and Ba<sub>4</sub>Al<sub>5</sub>.

# The polar mixed-valent lanthanum iron(II, III) sulfide $La_3Fe_{2-\delta}S_7$ : Synthesis, crystal and electronic structure, <sup>57</sup>Fe Mößbauer spectra, magnetic susceptibility and electrical resistivity

Allison M. Mills, Daniel Bräunling, Helge Rosner, Walter Schnelle, C. Peter Sebastian, Rainer Pöttgen and Michael Ruck

Page 1136



Rods of face-sharing  $[Fe^{II}S_6]$ -octahedra and  $[Fe^{III}S_4]$ -tetrahedra, all pointing in the same direction, dominate the polar structure. Vacancies in the octahedral Fe positions downgrade one-dimensional metallic conductivity to an activated semi-conducting behavior.

Phase equilibria and crystal chemistry of the R-Cu-Ti-O systems (R = lanthanides and Y)

Z. Yang, W. Wong-Ng, J.A. Kaduk, M. Jang and G. Liu *Page 1142* 



Phase diagram of the  $Y_2O_3\mathchar`-CuO\mathchar`-TiO_2$  system prepared in air at 960  $^\circ\mbox{C}.$ 

### Template-free polyoxometalate-assisted synthesis for ZnO hollow spheres

Qiuyu Li, Enbo Wang, Siheng Li, Chunlei Wang, Chungui Tian, Guoying Sun, Jianmin Gu and Rui Xu *Page 1149* 



ZnO hollow spheres with porous shell were synthesized by a onestep polyoxometalate-assisted solvothermal route at low temperature. Room temperature photoluminescence spectrum of the ZnO hollow spheres exhibits exciting emission features with a broad band covering nearly all the visible region.

#### Effect of sodium to barium substitution on the space charge implementation in thermally poled glasses for nonlinear optical applications

Artem Malakho, Marc Dussauze, Evelyne Fargin, Olivier Bidault, Vincent Rodriguez, Frederic Adamietz and Bertrand Poumellec *Page 1156* 



The sodium to barium substitution strongly influences the space charge implementation ( $E_{int}$ ) at the anode side of the poled glass and thus its nonlinear optical properties.

### Synthesis and magnetic properties of rare earth ruthenates, $Ln_5Ru_2O_{12}$ (Ln = Pr, Nd, Sm-Tb)

M. Bharathy, W.R. Gemmill, A.H. Fox, J. Darriet, M.D. Smith, J. Hadermann, M.S. Remy and H.-C. zur Loye *Page 1164* 



Single crystals of  $Ln_5Ru_2O_{12}$  (Ln = Pr, Nd, Sm–Tb) were grown out of NaOH/KOH fluxes in sealed silver tubes. The crystal structure consists of one-dimensional chains of edge-sharing RuO<sub>6</sub> octahedral pairs along the *b* axis, separated by a two dimensional  $LnO_x$ polyhedral framework. Each RuO<sub>6</sub> octahedral pair is separated alternately by Ln atoms and its disordered component, observed as stacking faults in the HRTEM images.

Transition alumina phases induced by heat treatment of boehmite: An X-ray diffraction and infrared spectroscopy study

A. Boumaza, L. Favaro, J. Lédion, G. Sattonnay, J.B. Brubach, P. Berthet, A.M. Huntz, P. Roy and R. Tétot *Page 1171* 



Infrared spectra of alumina sequence  $\gamma \rightarrow \delta \rightarrow \theta \rightarrow \alpha - Al_2O_3$  obtained from 24h calcinations of boehmite at 873 K (a), 1123 K (b), 1223 K (c), 1273 K (d), 1293 K (e), 1383 K (f), 1573 K (g).

## Ab initio structure determination and Rietveld refinement of $Bi_{10}Mo_3O_{24}$ the member n=3 of the $Bi_{2n+4}Mo_nO_{6(n+1)}$ series

J. Galy, J. Hernández-Velasco, A.R. Landa-Cánovas, E. Vila and A. Castro *Page 1177* 



This work reports on the *ab initio* determination of Bi<sub>10</sub>Mo<sub>3</sub>O<sub>24</sub> structure, as well as its refinement by using the Rietveld method, from the combination of X-ray and neutron diffraction powder data. It belongs to the monoclinic system, space group *C*2, with cell parameters: a=23.7282(2)Å, b=5.64906(6)Å, c=8.68173(9)Å,  $\beta=95.8668(7)^{\circ}$  with Z=2.

#### Structural properties of Pb<sub>3</sub>Mn<sub>7</sub>O<sub>15</sub> determined from highresolution synchrotron powder diffraction

Julia C.E. Rasch, D.V. Sheptyakov, J. Schefer, L. Keller, M. Boehm, F. Gozzo, N.V. Volkov, K.A. Sablina, G.A. Petrakovskii, H. Grimmer, K. Conder and J.F. Löffler *Page 1188* 



The crystal structure of  $Pb_3Mn_7O_{15}$  has been reinvestigated by synchrotron powder diffraction. The compound crystallizes in the orthorhombic space group *Pnma* and shows no structural transition between 15 and 295 K.

#### Thermal, solution and reductive decomposition of Cu–Al layered double hydroxides into oxide products Sylvia Britto and P. Vishnu Kamath Page 1193



SEM image of (a) the  $Cu_2O-Al(OH)_3$  composite obtained on reductive decomposition of  $CuAl_4$ -LDH and (b)  $Cu_2O$  obtained on leaching of  $Al(OH)_3$  from (a).

# A convenient sol-gel route for the synthesis of salicylate-titania nanocomposites having visible absorption and blue luminescence

Atanu Mitra, Asim Bhaumik, Mahasweta Nandi, John Mondal and B.K. Roy *Page 1200* 



A new titania–salicylate nanostructure material has been synthesized, which exhibit a considerable red shift towards the visible region vis-à-vis nanocrystalline (organic-free)  $TiO_2$  and blue luminescence at room temperature.

Mesoscale assembly of NiO nanosheets into spheres Meng Zhang, Guojin Yan, Yonggai Hou and Chunhua Wang *Page 1206* 



The mesoscale assembly of NiO nanosheets into spheres have been achieved by a solvothermal method. N<sub>2</sub> adsorption/desorption isotherms show the  $S_{BET}$  of NiO is tunable. NiO spheres show large discharge capacity and slow capacity-fading rate.

### Some clues about the interphase reaction between ZnO and $MnO_2$ oxides

F. Rubio-Marcos, A. Quesada, M.A. García, M.A. Bañares, J.L.G. Fierro, M.S. Martín-Gonzalez, J.L. Costa-Krämer and J.F. Fernández *Page 1211* 



Recently new room temperature interphase magnetism has been reported to appear in  $ZnO-MnO_2$  system. Raman spectroscopy is used to evidence both the nature of the interphase reaction and the kinetic. The interphase evolved towards complete formation of the spinel phase. The reactivity of the ZnO plays an important role in the formation of this interphase. Finally, a clear correlation between the amount of the intermediate valence state and the interphase magnetic properties has been established.

Comparative XANES study on the two electron-doped high- $T_c$  superconductor systems, (Sr,La)CuO<sub>2</sub> and (Nd,Ce)<sub>2</sub>CuO<sub>4</sub> Y. Tanaka, M. Karppinen, J.M. Chen, R.S. Liu and H. Yamauchi *Page 1217* 



High-quality samples with systematically varied substitution levels of the two electron-doped copper-oxide systems,  $(Sr_{1-x}La_x)CuO_2$ and  $(Nd_{2-x}Ce_x)CuO_4$ , have been synthesized and thoroughly characterized by means of XANES spectroscopy at O-*K*, Cu- $L_{2,3}$ and Ce- $M_{4,5}$  edges to gain comprehensive understanding of the electronic structure and doping in *n*-type high- $T_c$  superconductors. Not only common but also slightly different features are revealed for the two systems.

#### Structure of $\delta$ -Bi<sub>2</sub>O<sub>3</sub> from density functional theory: A systematic crystallographic analysis

Dilpuneet S. Aidhy, Susan B. Sinnott, Eric D. Wachsman, Simon R. Phillpot and Juan C. Nino *Page 1222* 

(½)\*11.19761 Å

1/8 of a 2 × 2 × 2 δ-Bi<sub>2</sub>O<sub>3</sub> superstructure having Fm3̄ space group. Every oxygen (black) has three possible positions, only one of which is filled either by O1 (red) or O<sub>2</sub> (blue).

Sol-gel synthesis of nanocomposite materials based on lithium niobate nanocrystals dispersed in a silica glass matrix Elisa Marenna, Carmela Aruta, Esther Fanelli, Mario Barra, Pasquale Pernice and Antonio Aronne Page 1229



Sol-gel synthesis of nanocomposite materials in the  $Li_2O-Nb_2O_5-SiO_2$  system is reported. The goal was to synthesize thin films containing lithium niobate nanocrystals embedded in a silica matrix. Starting from LiNO<sub>3</sub>, NbCl<sub>5</sub> and Si(OC<sub>2</sub>H<sub>5</sub>)<sub>4</sub>, it was possible to obtain LiNbO<sub>3</sub> as only crystallizing phase, nanocrystals size was 27 nm for a film 10Li<sub>2</sub>O-10Nb<sub>2</sub>O<sub>5</sub>-80SiO<sub>2</sub> heated 2 h at 800 °C.

#### A general protocol to coat titania shell on carbon-based composite cores using carbon as coupling agent

Rongbo Zheng, Xianwei Meng and Fangqiong Tang Page 1235



A general protocol was developed to coat titania shell on carbonbased composite cores based on the carbonaceous surface. Typically,  $Ag/C/TiO_2$  core/shell spheres were formed via this method.

### Growth of group III nitride films by pulsed electron beam deposition

J. Ohta, K. Sakurada, F.-Y. Shih, A. Kobayashi and H. Fujioka

Page 1241



We have grown group III nitride films by pulsed electron beam deposition (PED) and found that the films of group III nitrides grow epitaxially on 6H–SiC and  $Al_2O_3$  substrates. We also found that the use of PED allows us to reduce the epitaxial growth temperature for GaN down to 200 °C.

# Syntheses, crystal and electronic structures of three new potassium cadmium(II)/zinc(II) tellurides: $K_2Cd_2Te_3$ , $K_6CdTe_4$ and $K_2ZnTe_2$

Min-Jie Li, Chun-Li Hu, Xiao-Wu Lei, Yong Zhou and Jiang-Gao Mao

Page 1245



Three new semiconducting K–Zn(Cd)–Te phases were synthesized. They feature 2D, 1D or 0D anionic structure made of corner- and edge-sharing or isolated MTe<sub>4</sub> tetrahedra.

#### Determination of the Mössbauer parameters of rare-earth nitroprussides: Evidence for new *light-induced magnetic excited state (LIMES)* in nitroprussides

V. Rusanov, S. Stankov, A. Ahmedova and A.X. Trautwein

Page 1252



Rare-earth nitroprussides are studied by Mössbauer spectroscopy. Population of metastable states in a thin surface layer, and another state which remains stable at room temperature, are detected. The latter is a photoproduct which is called *light-induced magnetic excited state (LIMES)* and explained with a photochemical redox reaction, which changes the valence, spin, and magnetic state of 4f-3d bimetallic complexes.

### Synthesis, crystal structure and thermal behavior of a novel oxoborate $SrBi_2B_4O_{10}$

M.G. Krzhizhanovskaya, R.S. Bubnova, A.V. Egorysheva, M.S. Kozin, V.D. Volodin and S.K. Filatov *Page 1260* 



A fragment of  $\mathrm{SrBi}_2\mathrm{B}_4\mathrm{O}_{10}$  structure showing isolated borate anion  $[\mathrm{B}_4\mathrm{O}_9]^{6-}$  composed of a triborate group and a triangle, Sr atoms and a part of Bi–O chains involving oxocentred OBi<sub>3</sub> triangles.

#### Single-crystalline $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> with hierarchical structures: Controllable synthesis, formation mechanism and photocatalytic properties

Jianmin Gu, Siheng Li, Enbo Wang, Qiuyu Li, Guoying Sun, Rui Xu and Hong Zhang *Page 1265* 



A dual iron precursors system in a hydrothermal process was developed for controllable fabrication of various  $\alpha\text{-}Fe_2O_3$  hierarchical structures. Micro-pines, snowflakes and bundles were successfully fabricated by simply tuning the total concentration of the two iron precursors and their molar ratio. A possible formation mechanism of these structures was proposed.

#### Improvement by heating of the electronic conductivity of cobalt spinel phases, electrochemically synthesized in various electrolytes

Myriam Douin, Liliane Guerlou-Demourgues, Michel Ménétrier, Emilie Bekaert, Lionel Goubault, Patrick Bernard and Claude Delmas *Page 1273* 



In "Co<sub>3</sub>O<sub>4</sub>" type spinel phases synthesized by electrooxidation, the nature of the alkaline electrolyte allows to monitor the amounts of hydrogen and lithium, inserted in spinel framework and therefore the electronic conductivity. Whatever the initial synthesis electrolyte, a moderate thermal treatment of the materials induces a significant increase of the electronic conductivity, due to a structural reorganization (illustrated by the evolution of the cell parameter) and an increase of the  $Co^{4+}/Co^{3+}$  ratio in the octahedral framework.

#### Corrigendum

Corrigendum to "Solid-state synthesis, characterization and luminescent properties of  $Eu^{3+}$ -doped gadolinium tungstate, molybdate phosphors:  $Gd_{(2-x)}MO_6:Eu_x^{3+}$  (M=W, Mo)" [Journal of Solid State Chemistry 181 (2008) 2845–2851] Fang Lei, Bing Yan and Hao-Hong Chen *Page 1281* 

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