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Regular Articles

The dependence of persistent phosphorescence on annealing temperatures in $CaTiO_3$: Pr^{3+} nanoparticles prepared by a coprecipitation technique

Xianmin Zhang, Jiahua Zhang, Xinguang Ren and Xiao-Jun Wang page 393



The dependence of fluorescence and phosphorescence on annealing temperature in $CaTiO_3:Pr^{3+}$ nanoparticles prepared by a coprecipitation technique.

Hydrothermal synthesis of two copper helical coordination polymers with acentric three-dimensional framework constructing from mixed pyridine carboxylates

Shuai Zhang, Yanning Cao, Hanhui Zhang, Xiaochuan Chai and Yiping Chen

page 399



Two copper helical coordination polymers, $[Cu(2-pc)(3-pc)]_n$ **1** and $[Cu(2-pc)(4-pc)]_n$ **2** have been obtained by hydrothermal synthesis. Both two compounds crystallized in non-centrosymmetric space groups, $P_{21}_{21}_{21}$ and Pna_{21} , respectively. The 3D framework of **1** is constructed from right-handed helical Cu(2-pc) chains and lefthanded Cu(3-pc) helices. As for **2**, Cu(2-pc) helical chains, in which left- and right-handed helices are coexisting, and Cu(4-pc) zigzag chains combined together to form 3D architecture of 2 as well.

Regular Articles—Continued

Pyrochlore formation, phase relations, and properties in the CaO–TiO₂–(Nb,Ta)₂O₅ systems

R.S. Roth, T.A. Vanderah, P. Bordet, I.E. Grey, W.G. Mumme, L. Cai and J.C. Nino *page 406*



Crystal structures of the pyrochlores Ca_{1.46}Ti_{1.38}Nb_{1.11}O₇ and Ca_{1.51}Ti_{1.32}V_{0.04}Ta_{1.10}O₇ were refined using single-crystal X-ray diffraction data. Both Ca²⁺ and Ti⁴⁺ occupy the A-type sites; Ca occupies the ideal 16*d* site and Ti is displaced 0.7 Å to partially occupy a ring of six 96*g* sites. The O' oxygens are disordered among a tetrahedral cluster of 32*e* sites displaced 0.48 Å from the ideal 8*b* site. Both pyrochlores display dielectric relaxation similar to that observed for analogous Bi-based systems.

Designing and tuning properties of a three-dimensional porous quaternary chalcogenide built on a bimetallic tetrahedral cluster $[M_4\text{Sn}_3\text{S}_{13}]^{5-}$ (M = Zn/Sn) Min Wu, Thomas J. Emge, Xiaoying Huang, Jing Li and Yong Zhang page 415



A three-dimensional quaternary chalcogenide $[Na_5Zn_{3.5}Sn_{3.5}S_{13}]\cdot 6H_2O$ represents an interesting example of chalcogenide based semiconductor that combines semiconductivity, porosity, and light emission in a single structure. The electronic and optical properties of this compound can be systematically tuned by substitution of metal and chalcogen elements.

Temperature-dependent structural study of microporous $CsAlSi_5O_{12}$

Martin Fisch, Thomas Armbruster and Boris Kolesov page 423



Temperature-dependent structural evolution of microporous CsAl-Si₅O₁₂ has been investigated by single-crystal and powder X-ray diffraction, as well as Raman spectroscopy. Results yielded a phase transition of order–disorder type.

Synthesis and characterization of sulfonated single-walled carbon nanotubes and their performance as solid acid catalyst

Hao Yu, Yuguang Jin, Zhili Li, Feng Peng and Hongjuan Wang

page 432



Sulfonated SWCNTs with 20 wt% $-SO_2OH$ groups were prepared by a high-temperature H_2SO_4 process, which transformed the hydrophobic surface of pristine SWCNTs to a hydrophilic surface and provided an excellent performance as solid acid catalyst.

Exfoliated nanoplatelets of an Aurivillius phase, Bi_{3.25}La_{0.75}Ti₃O₁₂: Characterisation by X-ray diffraction and by high-resolution electron microscopy Virginie Chevallier, Geneviève Nihoul and Véronique Madigou page 439



 $Bi_{3.25}La_{0.75}Ti_3O_{12}$ nanoplatelets, of which the thickness downs to one cell parameter along the *c*-axis, are obtained through a soft chemical lithiation process followed by exfoliation. Li⁺ cations intercalate in the intermediate $(Bi_2O_2)^{2+}$ layers. However, not all of them actually accommodate lithium. Moreover, exfoliation is destructive for the host layers.

Novel three-dimensional dandelion-like TiO₂ structure with high photocatalytic activity

Xuelian Bai, Bin Xie, Nan Pan, Xiaoping Wang and Haiqian Wang

page 450



Rutile-phase TiO_2 powders with novel 3D dandelion-like structures were synthesized. This kind of 3D artificial hierarchical titania structure has the advantage of reserving the novel nanometer-scale properties while providing us the easiness of storing and handling as we routinely enjoyed for the micrometer-scale materials. A different oxidation process of Ti(III) to Ti(IV) during hydrothermal process was suggested.

2D parallel interpenetration of $[M_2(bpp)_4X_4]$ [M, Fe(II)/Co(II); bpp, 4,4'-trimethylenedipyridine; X, SCN⁻, SeCN⁻ and N₃⁻] complexes: Pseudohalide-dependent conformation of bpp

Subal Chandra Manna, Atish Dipankar Jana,

Georgina M. Rosair, Michael G.B. Drew, Golam Mostafa and Nirmalendu Ray Chaudhuri

page 457



Complexes of $[M_2(\text{bpp})_4X_4]$ [M, Fe(II)/Co(II); bpp, 4,4'-trimethylenedipyridine; X, SCN⁻, SeCN⁻ and N3⁻] have been synthesized.The structural analysis reveals undulated 2D network with (4,4) nettopology adopting two different conformations of bpp alternately.Two such networks undergo parallel interpenetration. Pseudohalides are observed to help in stacking the interpenetrated netsthrough hydrogen bonding. The structure of manganese dioxide and position of proton studied by neutron diffraction with isotopic substitution Cédric Pitteloud, Miki Nagao, Keiji Itoh and Ryoji Kanno page 467



Proposed model for the position of 'Ruetschi' and 'Coleman' proton inside manganese dioxide.

Synthesis and characterization of 2-phosphonoethanesulfonic acid and a barium-hydrogenphosphonatoethanesulfonate — $BaH(O_3P-C_2H_4-SO_3)$

Andreas Sonnauer and Norbert Stock page 473



The synthesis of the ligand $H_2O_3P-C_2H_4-SO_3H$ and its use in the systematic investigation in the system $BaCl_2/H_2O_3P-C_2H_4-SO_3H/NaOH/H_2O$ led to the new barium phosphonatosulfonate $BaH(O_3P-C_2H_4-SO_3H)$.

A facile two-step modifying process for preparation of poly(SStNa)-grafted Fe₃O₄/SiO₂ particles Zhongli Lei, Yanli Li and Xiangyu Wei page 480



Fig. 2b shows TEM images of silica-coated Fe₃O₄ particles. The magnetic silica particles with well-defined core/shell structures were rather monodisperse, even though silica shells have trapped more than one magnetic core. The Fe₃O₄/SiO₂ particles used in this case for the production of composite particles had an average diameter of 70 ± 10 nm obtained by TEM images.

Synthesis and characterization of environmentally benign calcium-doped Pr₂Mo₂O₉ pigments: Applications in coloring of plastics

Giable George, L. Sandhya Kumari, V.S. Vishnu, S. Ananthakumar and M.L.P. Reddy *page 487*



A new class of environmentally benign rare earth pigments of general formula $Pr_{2-x}Ca_xMo_2O_{9-\delta}$ (x ranges from 0 to 1.0) displaying colors ranging from green to yellow were synthesized by traditional solid-state route, as alternatives to lead, cadmium and chromium colorants. The yellow–green pigments were found to be interesting alternatives to existing toxic pigments for coloration of plastics.

Hydrothermal synthesis, structure, and magnetic properties of Pu(SeO₃)₂

Travis H. Bray, S. Skanthakumar, L. Soderholm, Richard E. Sykora, Richard G. Haire and Thomas E. Albrecht-Schmitt *page 493*



A depiction of the three-dimensional structure of $Pu(SeO_3)_2$ formed from the interconnection of one-dimensional chains of edgesharing PuO_8 dodecahedra by selenite anions.

Crystal structure, stoichiometry, and dielectric relaxation in Bi_{3.32}Nb_{7.09}O_{22.7} and structurally related ternary phases I.E. Grey, T.A. Vanderah, W.G. Mumme, R.S. Roth, J. Guzman, J.C. Nino and I. Levin *page 499*



[110] polyhedral projection of the structure of $Bi_{3,32}Nb_{7,09}O_{22.7}$. Heavy arrows show location of chemical twin planes.

Kinetics study on phase transformation from titania polymorph brookite to rutile

Jason Huberty and Huifang Xu page 508



The transformation kinetics from titania brookite to rutile can be described by both the standard first-order model and the JMAK model. The obtained activation energy for micron-sized brookite crystals is much higher than that for brookite nano-crystals.

The effect of replacement of Sr by Ca on the structural and luminescence properties of the red-emitting $Sr_2Si_5N_8$:Eu²⁺ LED conversion phosphor

Y.Q. Li, G. de With and H.T. Hintzen *page 515*



The temperature dependence of the luminescence efficiency of $M_{1.9}\text{Eu}_{0.1}\text{Si}_5\text{N}_8$ (M = Ca, Sr, Ba) and $\text{Sr}_{1.3}\text{Ca}_{0.6}\text{Eu}_{0.1}\text{Si}_5\text{N}_8$ ($\lambda_{\text{exc}} = 465 \text{ nm}$).

Effects of Mg doping on the properties of highly transparent conductive and near infrared reflective $Zn_{1-x}Mg_xO$:Ga films Quan-Bao Ma, Hai-Ping He, Zhi-Zhen Ye, Li-Ping Zhu, Jing-Yun Huang, Yin-Zhu Zhang and Bing-Hui Zhao *page 525*



The figure shows transmittance and reflectance spectra of $Zn_{1-x}Mg_xO:Ga$ films measured in the wavelength range of 300–2500 nm. In the visible region the films are highly transparent, and their spectra are like those of dielectrics regardless of Mg content. In the IR region the films behave like metals and have high reflectance and low transmittance.

Stepwise conversion of a *single source precursor* into crystalline AlN by transamination reaction

Stephan Schulz, Tillmann Bauer, Wilfried Hoffbauer, Jörn Schmedt auf der Günne, Markus Doerr, Christel M. Marian and Wilfried Assenmacher *page 530*



Ammonolysis reactions of $Me_3N-Al(NHDipp)_3$ in liquid NH_3 yielded Al-N oligomers, which can be transformed into nanocrystalline aluminum nitride particles under thermolysis conditions at 1000 °C. Theoretical calculations were performed in order to identify potential reaction intermediates.

Mononuclear, trinuclear, and hetero-trinuclear supramolecular complexes containing a new tri-sulfonate ligand and cobalt(II)/ copper(II)-(1,10-phenanthroline)₂ building blocks

Yunfang Yu, Yongqin Wei, Ria Broer, Rongjian Sa and Kechen Wu

page 539



Novel mononuclear, trinuclear, and hetero-trinuclear supermolecular complexes, $[Co(phen)_2(H_2O)(HTST)] \cdot 2H_2O$ (1), $[Co_3(phen)_6$ $(H_2O)_2(TST)_2] \cdot 7H_2O$ (2), and $[Co_2Cu(phen)_6(H_2O)_2(TST)_2] \cdot$ $10H_2O$ (3), have been synthesized by the reactions of a new trisulfonate ligand (2,4,6-tris(4-sulfophenylamino)-1,3,5-triazine, H₃TST) with the M^{2+} (M=Co, Cu) and the second ligand 1,10phenanthroline (phen). The study shows the flexible multifunctional self-assembly capability of H₃TST ligand presenting in these supramolecular complexes.

Synthesis, structure, and magnetic properties of Ba₂Cu₂US₅ Hui-yi Zeng, Jiyong Yao and James A. Ibers *page 552*



 \mathbf{O} Ba \mathbf{O} Cu \mathbf{O} U \mathbf{O} S Unit cell of Ba₂Cu₂US₅ viewed down [010].

An unprecedented extended architecture constructed from a 2-D interpenetrating cationic coordination framework templated by $SiW_{12}O_{40}^{4-}$ anion

Xiuli Wang, Hongyan Lin, Yanfeng Bi, Baokuan Chen and Guocheng Liu

page 556



Compound [Cu₂(*bpp*)₄(H₂O)₂](SiW₁₂O₄₀)~6H₂O (1) represents the first 2-D interpenetrating cationic metal–organic frameworks (MOFs) templated by Keggin-type anions. These MOF layers are stacked together along the crystallographic *c* axis exactly to construct large cubic-like channels (with dimensions of 12.3×13.6 Å) occupied by SiW₁₂O⁴₄₀ clusters.

Synthesis and luminescence properties of hybrid organic-inorganic transparent titania thin film activated by *in-situ* formed lanthanide complexes

Yige Wang, Li Wang, Huanrong Li, Peng Liu, Dashan Qin, Binyuan Liu, Wenjun Zhang, Ruiping Deng and Hongjie Zhang

page 562



Novel stable luminescent organic–inorganic hybrid titania thin film with high transparency activated by *in-situ* formed lanthanide complexes have been obtained at room temperature via a simple one-pot synthesis approach by using TTFA-modified titanium precursor with amphiphilic triblock copolymer P123. The obtained hybrid thin film displays bright red (or green), near-monochromatic luminescence due to the *in-situ* formed lanthanide complex.

Syntheses, structures, luminescence, and magnetism of four 3D lanthanide 5-sulfosalicylates

Rui-Sha Zhou, Ling Ye, Hong Ding, Jiang-Feng Song, Xiao-Yu Xu and Ji-Qing Xu *page 567*

Syntheses and crystal structures of four 3D lanthanide-5-sulfosalicylates, $Ln(SSA)(H_2O)_2$ [Ln = Ce(III) (1), Pr(III) (2), Nd(III) (3), and Dy(III) (4)], have been reported. In complexes 1–4, Ln(III) acting as 5-connected inorganic node and SSA³⁻ ligand acing as 5-connected organic node interlink into rarely reported 4⁶6⁴ topology network. Luminescence and magnetism properties have also been studied.

Phase stability of BSCF in low oxygen partial pressures

James Ovenstone, Jae-II Jung, Jeffery S. White, Doreen D. Edwards and Scott T. Misture *page 576*



The phase stability of the fuel cell cathode $Ba_{0.5}Sr_{0.5}Fe_{1-x}Co_xO_{3-\delta}$ in low pO_2 and high temperature has been investigated using *in situ* X-ray diffraction. Both stability and thermal expansion coefficient were found to increase with increasing iron content. Decomposition products under reducing conditions have been identified.

Shape evolution and thermal stability of Ag nanoparticles on spherical SiO₂ substrates

Shaochun Tang, Shaopeng Zhu, Haiming Lu and Xiangkang Meng

page 587



The shape evolution and thermal stability of Ag nanoparticles (NPs) on spherical SiO₂ substrates were investigated by means of *in situ* TEM imaging and DSC analysis. A possible mechanism for the desquamation of Ag NPs from the SiO₂ sphere surface is proposed. Here, a simple sketch is shown to describe the shape evolution and desquamation process of the Ag NPs.

Visible-light responsive dye-modified TiO₂ photocatalyst Dong Jiang, Yao Xu, Dong Wu and Yuhan Sun *page 593*



Dye-modified TiO₂ photocatalysts were synthesized via the reaction between Chrysoidine G (CG), Degussa P25 (TiO₂), and tolylene-2,4-diisocyanate (TDI) as a bridging molecule. As a result, π -conjugated surface organic complexes were formed on TiO₂ surface. Due to the existence of organic complexes, dye-modified TiO₂ catalysts showed great visible absorption and high activity under the visible light irradiation.

Lattice effects in cubic $La_2Mo_2O_9$: Effect of vacuum and correlation with transport properties

Cristina Tealdi, Lorenzo Malavasi, Clemens Ritter, Giorgio Flor and Giorgio Costa page 603



Anti-tetrahedral unit centred in O1 and linked through La ions to form a cage where the partially occupied O2 and O3 ions are placed.

Neutron powder diffraction study of the crystal and magnetic structures of BiNiO₃ at low temperature

Sandra J.E. Carlsson, Masaki Azuma, Yuichi Shimakawa, Mikio Takano, Alan Hewat and J. Paul Attfield *page 611*



A neutron diffraction study shows that the perovskite $BiNiO_3$ retains the unusual charge distribution $Bi_{0.5}^{3+}Bi_{0.5}^{5+}Ni^{2+}O_3$ down to 5 K. The Ni^{2+} moments are ordered in the *G*-type antiferromagnetic arrangement shown; however, $BiNiO_3$ is ferrimagnetic due to the inexact cancellation of the four inequivalent moments in the triclinic unit cell.

Flash microwave synthesis of trevorite nanoparticles C. Bousquet-Berthelin, D. Chaumont and D. Stuerga page 616



At the end of the 20th century, a new concept of battery was introduced, named "Li ion", where electrodes are both lithium-storage materials. Compounds with a spinel structure are so investigated and microwave heating appears as an efficient source of energy to produce nanoparticles in a very short time and at low temperature, with controlled size (4–5 nm) and high specific area ($240 \text{ m}^2/\text{g}$).

Legend: Pictogram represents our original microwave reactor, the RAMO (French acronym of Réacteur Autoclave Micro-Onde), containing the reactants and submitted to the microwave irradiation. Multicolor candy represents obtained material.

Synthesis, structure, magnetic properties and structural distortion under high pressure of a new osmate, Sr₂CuOsO₆ Michael W. Lufaso, William R. Gemmill,

Samuel J. Mugavero III, Seung-Joo Kim, Yongjae Lee, Thomas Vogt and Hans-Conrad zur Loye *page 623*



Relative compressibility of the lattice parameters of a new osmate, $\mathrm{Sr}_2\mathrm{CuOsO}_6.$

Facile synthesis of multifunctional multiwalled carbon nanotubes/ Fe_3O_4 nanoparticles/polyaniline composite nanotubes

Lirong Kong, Xiaofeng Lu and Wanjin Zhang page 628



The work on preparing nanocomposites has been too much, but few reports were about synthesizing one-dimensional nanocomposite of three different nanoscale materials. In our work, we prepared one-dimensional multiwalled carbon nanotubes/ Fe_3O_4 particles/polyaniline composite nanotubes and studied their conductive and magnetic properties.

Hydrothermal synthesis and structural characterization of two 1-D and 2-D Dawson-based phosphotungstates Jun-Wei Zhao, Shou-Tian Zheng, Wei Liu and Guo-Yu Yang

page 637



Two Dawson-based phosphotungstates $(H_2en)_{0.5}H[Cu(en)_2(H_2O)]_2 \{[Cu(en)_2](\alpha_1-P_2W_{17}CuO_{61})\} \cdot 8H_2O$ (1) and $[4,4'-H_2bpy]_2\{[Cu(4,4'-bpy)_3][Cu(4,4'-bpy)_3][Cu(4,4'-bpy)_4(H_2O)_2]_2[Cu(4,4'-bpy)][\alpha-P_2W_{18}O_{62}]_2\} \cdot 6H_2O$ (2) have been hydrothermally synthesized and structurally characterized. 1 consists of a 1-D linear chain structure constructed from monocopper-substituted Dawson polyoxoanions, while 2 represents the first 2-D sheet-like structure with a (4,4)-connected topological net built up from saturated Dawson-type polyoxanions and Cu^{II}-4,4'-byy complex cations in polyoxometalate chemistry.

A new promising scintillator Ba₃InB₉O₁₈

Gemei Cai, X.L. Chen, W.Y. Wang, Y.F. Lou, J. Liu, J.T. Zhao and H.H. Chen

page 646



A new compound Ba₃InB₉O₁₈ was synthesized with space group $P6_3/m$, lattice parameters a=7.1359(3)Å, c=16.6151(8)Å, Z=2 and V=723.697Å³. Its crystal structure is made up of planar B₃O₆ groups, regular InO₆ octahedra, irregular BaO₆ hexagons and BaO₉ polyhedra. Its XEL spectra show an intense emission band in the range of 360–500 nm with light yield about 75% of BGO. The projection of the Ba₃InB₉O₁₈ structure along the $\langle 11-20 \rangle$ direction.

Selected-control hydrothermal synthesis and formation mechanism of 1D ammonium vanadate

Nian Wang, Wen Chen, Liqiang Mai and Ying Dai page 652



Selective-controlled structure and shape of ammonium vanadate nanocrystals were successfully synthesized by a simple hydro-thermal method without the presence of catalysts or templates. The final products were $NH_4V_4O_{10}$ nanobelts, $(NH_4)_2V_6O_{16} \cdot 1.5H_2O$ nanowires, and $(NH_4)_6V_{10}O_{28} \cdot 6H_2O$ nanobundles, respectively, when the pH of the growth solution varied from 2.5 to 1.5, then to 0.5.

Luminescence properties of Eu^{2+} and Ce^{3+} -doped $CaAl_2S_4$ and application in white LEDs

Ruijin Yu, Jing Wang, Jianhui Zhang, Haibin Yuan and Qiang Su

page 658



The Eu²⁺- and Ce³⁺-doped CaAl₂S₄ phosphors were comparatively synthesized by two methods. The emission intensity of Eu²⁺ ion in sample synthesized by the evacuated sealed quartz ampoule method is by a factor of 1.7 as strong as that of Eu²⁺ ion in sample prepared by the conventional solid-state reaction method.

Rapid Communications

Formation of Co³⁺ octahedra and tetrahedra in YBaCo₄O_{8.1} O. Chmaissem, H. Zheng, A. Huq, P.W. Stephens and J.F. Mitchell *page 664*



A new mixed-metal oxide of composition YBaCo₄O_{8.1} has been synthesized by controlled oxidation of YBaCo₄O₇ and its structure determined using neutron and synchrotron X-ray powder diffraction. The structure features two alternating layers: a triangular layer of isolated CoO₆ octahedra and CoO₄ tetrahedra that links distorted Kagomé planes of charge-ordered Co ions. In these planes, a rarely observed motif of zigzag rows of edge-sharing octahedra is found, which are connected by ribbons of corner-sharing tetrahedra.

Sixfold self-assembled hierarchical structures synthesized by direct annealing of Zn microtips

C.Y. Kuan, J.M. Chou, I.C. Leu and M.H. Hon page 673



The novel three-dimensional hierarchical structure is composed of single-crystalline Zn microtips with the converted ZnO coating as stem, and ZnO nanowhiskers as branches. The branched ZnO nanowhiskers exhibit preferred growth direction, suggesting that the branched nanostructures might have a preferred orientation, and maintain a well-defined relationship to the Zn stem.

2D and 3D alkaline earth metal carboxyphosphonate hybrids: Anti-corrosion coatings for metal surfaces Konstantinos D. Demadis, Maria Papadaki, Raphael G. Raptis and Hong Zhao

page 679



Syntheses, characterization and crystal structures of metal-hydroxyphosphonoacetate hybrids are reported (Metal = Sr, Ba). 2D and 3D materials were prepared. Their anti-corrosion effects were studied at pH 2.0 and 7.3. It was found that anti-corrosion efficiency was demonstrated only at pH 7.3.

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