

unique structure, size and shape-dependent phenomena. Therefore, systematic directions for synthetic methods and an understanding of the mechanisms by which the structure, size and shape of the nanostructural material can be controlled are of particular interest. In this work, we report the controllable synthesis of ferric oxide nanostructural materials utilizing a simple hydrothermal method. We also examine the roles of parameters critical to the size and shape guiding processes and properties of the synthesized nanostructural materials. In a typical synthesis, added the ferric solution and additive to NaOH solution with various concentrations under vigorous magnetic stirring, and then transfer the mixture to a stainless steel autoclave. The products were characterized by XRD, SEM, TEM, ED, IR, TG-DTA, and SQUID. The XRD patterns of the as-prepared products were shown that, all of the peaks can be easily indexed as the rhombohedral α -Fe₂O₃. The SEM images indicate all products are composed of a large number of individual nanoparticles. The structure, size and shape of nanostructures could be control by adjusting hydrothermal conditions.

Keywords: hydrothermal synthesis, ferric oxide, nanostructural materials

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Growth and characterization of Nd_{1.85}Ce_{0.15}CuO₄ samples

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Recently several attempts have been made of looking at the physics of electron-doped and hole-doped cuprates under a unifying point of view, possibly elucidating the general mechanism for high-T_c superconductivity [1]. The fabrication method of Nd_{1.85}Ce_{0.15}CuO₄ bulk samples, based on a systematic control of the microstructural and phase homogeneity, is of fundamental importance in order to obtain sintered targets and precursors for high quality thin films and single crystals fabrication, respectively. Structural and electrical characterization of samples fabricated in different ways (bulk, films and single crystals) as a function of growth conditions and thermal treatments will be presented. We investigated morphology and composition by Scanning Electron Microscopy, Energy Dispersive Spectroscopy and Wavelength Dispersive Spectroscopy to verify the right stoichiometry, the presence of spurious elements, clusters and undesired phases. High resolution x-ray diffraction allowed to measure the structural parameters and to detect possible different phases and grain orientations. Standard four-probe resistance versus temperature measurements have been performed to study the transport properties of all the samples. Moreover, x-ray excited photoemission experiments performed at the European Synchrotron Radiation Facility have been used for obtaining information on the electronic configuration of the superconducting state as well as in the normal state [2,3].

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Crystal structure and superconducting properties of monoclinic perovskite BaPb_{0.7-x}Sb_xBi_{0.3}O₃

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Starting materials BaCO₃, PbO₂, Bi₂O₃ and Sb₂O₅ were pressed into pellets and sintered in slowly flowing oxygen at the temperature of 1123 K for 15 hours. Polycrystalline BaPb_{0.7-x}Sb_xBi_{0.3}O₃ pellets were powdered. X-ray diffraction analysis was performed at room temperature using Siemens D5000 X-ray Powder Diffractometer with K α radiation. The space group was determined to be *I*2/*m* (No. 12), which was the same as for BaPbO₃ [1]. The lattice parameters were calculated using Powder Cell for Windows program [2]. The upper limit of lead replacement by antimony in monoclinic BaPb_{0.7-x}Sb_xBi_{0.3}O₃ was found to be $x = 0.4$. The lattice parameters *a*, *c* and the volume of the unit cell decrease with increasing *x* parameter up to $x = 0.2$. Then increasing the *a* and *c* lattice parameters and the volume of the unit cell in the *x* range $0.2 < x < 0.4$ is observed. The *b* lattice parameter decreases with increasing *x* parameter in the whole examined range. The monoclinic angle beta corresponds to the angle between the cubic directions [110] and [1-11]. Electrical resistivity measurements were performed to examine superconducting properties of the samples. Maximum transition temperature T_c equal to 13.2 was reached for the $x = 0.2$. The minimum of the unit cell volume in the BaPb_{0.7-x}Sb_xBi_{0.3}O₃ system is also observed for the $x = 0.2$. Oxygen deficiency leads to a loss of superconducting properties and change in the space group. Semiconductor BaPb_{0.7-x}Sb_xBi_{0.3}O_{3-d} compounds have space group *I*4/*mcm* at room temperature.

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Keywords: superconducting oxides, perovskites, resistivity

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Role of O atom modulation in the self-doped spin-ladder compound Sr₁₄Cu₂₄O₄₁

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The Sr_{14-x}(Ca, La, Y)_xCu₂₄O₄₁ series are so-called spin-ladder compounds with two-legged Cu₂O₃ ladder and one-dimensional CuO₂ chain. Especially, Sr_{14-x}Ca_xCu₂₄O₄₁ series form a self-doped system, because the formal valence of Cu is +2.25. The ternary Sr₁₄Cu₂₄O₄₁ as the parent material of Sr_{14-x}(Ca, La, Y)_xCu₂₄O₄₁ forms incommensurate composite crystal structure with the CuO₂ subsystem and the Cu₂O₃ subsystem. After modulated structure analysis with the BVS calculations of Sr₁₄Cu₂₄O₄₁, it has become apparent that most of the holes are located in the CuO₂ chain. The valence of Cu atoms in the ladder and the chain are well

controlled by the O atom modulation in the CuO_2 . We have further investigated the temperature dependence of the atomic modulations in $\text{Sr}_{14}\text{Cu}_{24}\text{O}_{41}$, particularly in the CuO_2 chain in which the spin-gap behavior accompanied by the formation of the spin-dimerized state is realized at low temperature. By single-crystal x-ray-diffraction method, we have confirmed that superspace group of the modulated structure remains unchanged from room temperature to 150K. The hole distribution has been considered on the basis of the changes of lattice constants, the atomic modulation and the interatomic distances between Cu in the Cu_2O_3 and O atom in the CuO_2 . It is indicated that the small amount of holes doped in the Cu_2O_3 have been back-transferred to the CuO_2 and that almost all of the holes are localized in the CuO_2 at low temperature. Moreover, the possible hole-ordered structure with the Zhang-Rice singlet in the CuO_2 are mainly due to the O atom modulation in the CuO_2 and the ZR-singlet site with rectangular CuO_4 unit is possible in the CuO_2 , which is analogous to the local CuO_4 coordination in the CuO_2 plane of high- T_c cuprates.

Keywords: superconductor oxides, composite crystals, incommensurate modulated structures

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Structural study on the rattling phenomena in the β -pyrochlore oxides and filled skutterudites

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β -pyrochlore oxides and filled skutterudites exhibit a wide variety of physical properties; superconductivity, heavy fermion, valence fluctuation, non-Fermi liquid behavior. The alkali ions in the β -pyrochlore oxides and the rare earth ions in filled skutterudites are located inside the oversized cages, and these ions are rattling heavily with large thermal displacements. In order to elucidate the relation between the rattling and the physical properties, we carried out X-ray diffraction measurements on single crystals of $\beta\text{-KOs}_2\text{O}_6$ and $\text{NdOs}_4\text{Sb}_{12}$ using a CCD area detector and a curved imaging plate. It is known that the two compounds exhibit the largest rattling among each series. The values of the atomic displacement parameter U_{eq} are estimated at $U_{\text{eq}}=0.0735(8)$ for K in $\beta\text{-KOs}_2\text{O}_6$ (Fd-3m) and $U_{\text{eq}}=0.0558(1)$ for Nd in $\text{NdOs}_4\text{Sb}_{12}$ (Im-3) at 300 K, which are significantly large in comparison with the other atoms. It is found that the electron density of the K atom in $\beta\text{-KOs}_2\text{O}_6$ is not spherical but extended considerably along the $\langle 111 \rangle$ direction in spite of the high point symmetry of the site ($-43m$), giving evidence for a large anharmonic vibration of the K atom. In contrast, the Nd atom in $\text{NdOs}_4\text{Sb}_{12}$ shows isotropic electron density, consistent with the m-3 site symmetry. Thus, it is concluded that the anharmonicity, which is the key issue for the rattling, is more pronounced in $\beta\text{-KOs}_2\text{O}_6$. In addition, on the first-order transition for $\beta\text{-KOs}_2\text{O}_6$ at $T_p = 7.5$ K below the superconducting transition at $T_c = 9.6$ K, we found a clear stepwise change in the X-ray intensity of some selected reflections, which must be relevant to some sort of changes in the rattling of the K atom.

Keywords: structural studies, superconducting materials, heavy fermions

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Microstructure and superconductivity in polycrystalline boron-doped diamonds

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The discovery of superconductivity in polycrystalline boron-doped diamond (BDD) synthesized under high pressure and high temperatures (Ekimov et al. (2004) Superconductivity in diamond. Nature 428: 542) has raised a number of questions on the origin of the superconducting state. It was suggested that the heavy boron doping of diamond eventually leads to superconductivity. To justify such statements a more detailed information on the microstructure of the composite materials and on the exact boron content in the diamond grains is needed. For that we utilized high-resolution transmission electron microscopy as well as electron energy loss spectroscopy. For the studied superconducting BDD samples synthesized at high pressures and high temperatures the diamond grain sizes are about 1-2 microns with a boron content between 0.2(2) and 0.5(1) at.%. The grains are separated by 10-20 nm thick layers and triangular-shaped pockets of predominantly (at least 95 at.%) amorphous boron. Our results render superconductivity caused by the heavy boron doping in diamond highly unlikely.

Keywords: superconducting materials, microstructure, TEM characterization

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Superconductivity and charge-density wave in ring- or Moebius-shaped NbSe_3 and TaS_3 single crystals

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NbSe_3 and TaS_3 single crystals of ring- or Moebius-shape have been fabricated by Tanda et al. and an intriguing possibility to investigate superconductivity or charge-density wave (CDW) in these topological spaces has been opened. In this paper, we predict several new phenomena in these systems based on both phenomenological Ginzburg-Landau theory and microscopic Bardeen-Cooper-Schrieffer theory. First we study the physical properties of superconductivity in a Moebius ring, which is obtained by applying pressure or doping atoms to NbSe_3 . Most interesting phenomenon appears when a magnetic field is applied to this system: an ordinary Little-Parks oscillation, which is an oscillation of transition temperature as a function of magnetic flux (F) threading the ring, is modified especially when F is close to a half-odd-integer times a superconducting magnetic flux quantum, and novel superconducting states appear which have a gap node along the center line of the Moebius ring. This kind of state has never been achieved in other