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Keywords: periodicity, nuclides, Farey-sequence

P11.05.128

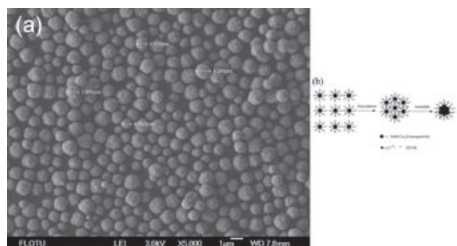
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Preparation of cuprous oxide microspheres via HTAB-assisted glucose reduction approach

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Cuprous oxide microspheres are widely used as catalysis, coatings, disinfection and anodic materials due to their unique assemble structures and their perfect dispersion and fluid properties. Cuprous oxide microspheres are usually fabricated by the thermal oxidation and the chemical vapor deposition which need high temperature or by the solution reduction which use the expensive or toxic materials as formaldehyde, sodium hypophosphite or KBH_4 as the reducing agents. Herein a moderate method was suggested to synthesis cuprous oxide microspheres via the hexadecyltrimethyl ammonium bromide (HTAB)-assisted glucose reduction approach, using CuSO_4 as the raw material. Dispersive uniform cuprous oxide microspheres (800-900nm) composed of crystalline nano-particles (8.5-13 nm) were synthesized by mixing CuSO_4 and glucose solutions in the presence of HTAB at room temperature followed by aging of the mixture at 70-80°C for 0.5 h (Fig. 1a). The presence of HTAB promoted the formation of cuprous oxide nano-particles as well as the subsequently assembly of the nano-particles to the cuprous oxide microspheres (Fig. 1b).



Keywords: microspheres, cuprous oxide, glucose

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Crystal structures and high-temperature phase transition of Sr_2MSbO_6 (M=Sc,Cr,Fe) double perovskites

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The Sr_2MSbO_6 (M=Sc,Cr,Fe) materials have been elaborated by the standard solid state reaction method, and their structures studied with X-ray powder diffraction method, at room temperature (RT) and at high temperatures (HT). At RT, the crystal structures of these materials were found to have a monoclinic symmetry: $\text{Sr}_2\text{ScSbO}_6$, with $P2_1/n$ space group, and cell parameters $a=5.6783(1)$, $b=5.6923(1)$, $c=8.0257(1)$ Å, $\beta=89.98(1)$; $\text{Sr}_2\text{FeSbO}_6$, with $I2/m$

space group, and with $a=5.6148(1)$, $b=5.5954(4)$, $c=7.9002(1)$ Å, $\beta=90.02(4)$; and, finally, $\text{Sr}_2\text{CrSbO}_6$, with $I2/m$ space group, and cell parameters $a=5.5574(1)$, $b=5.5782(1)$, $c=7.8506(1)$ Å, $\beta=90.06(2)$. Depending on the M cation size, the M^{3+} and Sb^{5+} cations can be entirely or partially ordered, in the two distinct B-sites of the double perovskite structure. In the Sc compound, the Sc^{3+} and Sb^{5+} cations are totally ordered; in Cr and Fe, the M^{3+} ($\text{Cr}^{3+}, \text{Fe}^{3+}$) and Sb^{5+} cations are partially ordered: the degree of the ordering is 99.4% and 92%, respectively. At HT, $\text{Sr}_2\text{ScSbO}_6$ undergoes three structural phase transitions: $P2_1/n \rightarrow I2/m \rightarrow I4/m \rightarrow \text{Fm-}3m$, at about 400 K, 560 K and 650 K, which, to the best of our knowledge, is the first time that such a rich phase-transition sequence has been observed in this kind of materials. $\text{Sr}_2\text{CrSbO}_6$ and $\text{Sr}_2\text{FeSbO}_6$ were found to present the $I2/m \rightarrow 4/m \rightarrow \text{Fm-}3m$ PT sequence: the first PT is discontinuous, and the second, continuous. In $\text{Sr}_2\text{CrSbO}_6$, the PT take place at about 500 and 660 K. In $\text{Sr}_2\text{FeSbO}_6$ the first PT starts to take place at 400 K, and shows a temperature interval, 400 to 420 K, in which both phases, monoclinic and tetragonal, co-exist; the second PT, from the tetragonal structure with $I4/m$ to the cubic structure with the $\text{Fm-}3m$, is observed above 500 K.

Keywords: transition-metal perovskites, powder diffraction method, phase transitions

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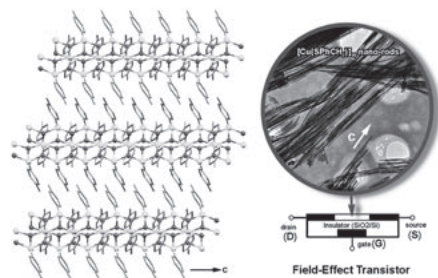
Powder structure determination of a series of homoleptic copper(I)-arythiolate conducting polymers

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Homoleptic copper(I) arylthiolates $[\text{Cu}(\text{p-SC}_6\text{H}_4\text{X})_n]$ (X = CH_3 , 1, H 2, CH_3O 3, tBu 4, CF_3 5, NO_2 6, COOH 7 and n = infinity) were insoluble crystalline solids.[1] Structure determination using powder diffraction revealed that 1-3 and 6 formed 1-D polymers with Cu atoms bridged by arylthiolate ligands. Weak intra-chain pi-pi stacking interactions are present in 1-3. In 6, the intra-chain pi-pi interactions are insignificant and the chain polymers are associated via non-covalent C-H...O hydrogen bonding interactions. As revealed by TEM, the solid samples of 1-5 and $[\text{Cu}(\text{SCH}_3)_n]$ contained homogeneously dispersed crystalline nano-rods whereas smaller plate nano-crystals were found for 6 and 7. SAED further confirmed that the chain polymers of 1-3 and $[\text{Cu}(\text{SCH}_3)_n]$ similarly grow along the long axes of their nano-rods. The nano-rods of 1-5 and $[\text{Cu}(\text{SCH}_3)_n]$ exhibited p-type field-effect transistor behavior with charge mobility of $10\text{E-}2$ to $10\text{E-}5$ $\text{cm}^2\text{V}^{-1}\text{s}^{-1}$. Their charge mobility is correlated to their unique chain-like copper-sulfur networks, and the para-substituent of arylthiolate ligand alter the ultimate particle morphology and charge transport properties.

[1] Chem. Eur. J. 2008, 14, 2965.



$[\text{Cu}(\text{SPhCH}_3)]_n$ nano-rod FET device

Keywords: *ab initio* powder structure determination,

conducting polymers, structure-properties relationship

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Relationship between crystallinity and surface morphology of blended PHB thin films

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Our concerns about environmental issue have rapidly increased, partly pushed by the recent rise in gasoline prices. Especially, the problem of waste is really serious under the situation in which we should not increase greenhouse gases any more by careless incinerations. Polymers decomposed by bacteria are thus subjected to be extensive researches in the whole world. Poly (3-hydroxyalcanote)s (PHAs) are the only microbial biodegradable polymers with decent thermo-plasticity. Among the PHA family, Poly (3-hydroxybutylate) (PHB) is known to exhibit high crystallinity and excellent biocompatibility. However, PHB may not be considered to completely replace petroleum plastics because of its rigidity and fragility. Polymer blend is a widely-used technique to improve physical properties like flexibility and thermal resistance. Quantitative characterization of surface morphology and physical properties of thin films are crucial in application of polymers. We have exploited surface-sensitive X-ray diffraction techniques to reveal the relationship of surface morphology and crystal structure in the surface region of PHB and its random copolymers [1]. In the present study, we investigate the surface structure and morphology of natural, isotactic PHB thin layers blended with synthesized, atactic PHB by using grazing incidence X-ray diffraction (GIXD), X-ray reflectivity and AFM. Small amount of the synthesized PHB is found to effectively affect the surface morphology of blended PHB, although the out-of-plane GIXD indicates (020) Bragg reflection unchanged.

[1] K. Mori et al., *Macromolecules*

Keywords: biodegradable polymers, surface morphology, X-ray diffraction

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Surface structure of biodegradable polymer blend of poly(hydroxybutyrate) and poly(lactide)

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A treatment of surface is one of the key issues for many practical applications of biodegradable polymer thin films and fibers, since physical properties and biodegradability of films and fibers strongly affected by their surface structure and surface morphology. Polymer blend (polymer alloy) is a widely-accepted technique for improving the physical properties with relatively less development costs. However, it does not seem to be applied for biodegradable polymers effectively due to the small number of biodegradable polymers we have. Instead of the polymer blend, many ingenious plans have been employed: Stereocomplexation of L- and D-configured polymer

chain of biodegradable polylactide (PLA) revealed a substantial improvement of mechanical properties of bulk; for biodegradable polyhydroxybutyrates (PHBs), random copolymers have been synthesized to exploit the possibility of practical use, although the major part of these efforts have been focused on bulk properties. In the present study, we prepared thin films of PHB blended with PLA to investigate how a small amount of PLA affects the surface structure and morphology. The polymers were dissolved in hot chloroform to form spin-coated ultrathin films on Si (100) and thick films with several mm thick. X-ray reflectivity and grazing incident X-ray diffraction were utilized as tools for investigation of surface morphology and crystallinity in the surface region, respectively. Relationship between crystallinity and secondary structure peculiar to the surface will be addressed on a microscopic standpoint.

Keywords: surface morphology, X-ray diffraction, thin films

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Relationship between contact angle and crystallinity in surface region of polyethylene polymer alloy

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Physical properties of polymers, e.g., viscosity, melt temperature, strength strongly depend on its molecular weight. Roles of physical parameters like molecular length on polymer properties can be sharply distinguished from chemical properties by a comprehensive study of a standard polymer blended with the polymer having the same chemical composition (monomer) with different molecular weight. Surface energy, surface morphology, and lamella structure in surface region would also be affected by the molecular weight, which should be crucial for the usage of intelligent polymers for micro fabrication technologies. In the present study, we choose polyethylene (PE) as a standard crystalline polymer, and prepare polymer alloys made from two different molecular weights to serve as quantitative investigations on macroscopic scale as well as those on microscopic standpoints. The former is observation on contact angle between molten PE droplet and a flat Si (100) plane, and that between a droplet of pure water and flat PE surfaces; the latter is surface-sensitive X-ray diffraction (X-ray reflectivity (XR) for characterizing surface morphology, and grazing incidence X-ray diffraction (GIXD) for crystalline lamellae structure). Two PE samples (Mn (number average molecular weight): 33,000 and 480) and a high-precision four-circle diffractometer with rotating-anode X-ray generator (SLX2000+UltraX) were used. Contact angles were fitted from images taken by digital cameras. Although the contact angle between molten PE and Si and crystallinity in the surface region monotonously vary with the mixing ratio, the contact angle between water and PE show and surface roughness show a maximum at an intermediate mixing ratio.

Keywords: surface structure, crystalline polymers, X-ray diffraction