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Original Research

Synthesis, structure, and optical properties of manganese phthalocyanine thin films and nanostructures[☆]Lu Meng^a, Kai Wang^a, Yuyan Han^b, Yi Yao^a, Pin Gao^a, Chao Huang^a, Wenhua Zhang^a, Faqiang Xu^{a,*}^a National Synchrotron Radiation Laboratory, University of Science and Technology of China, Hefei, Anhui 230029, China^b High Magnetic Field Laboratory, Chinese Academy of Sciences, Hefei 230031, China

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ABSTRACT

Manganese phthalocyanine (MnPc) nanostructures with different morphologies were prepared on porous anodic alumina oxide (AAO) at different substrate temperature (T_s) = 50 °C, 80 °C, 120 °C, 180 °C, 240 °C) in an organic molecular beam deposition (OMBD) system. The nanostructures morphologies were studied using scanning electron microscopy (SEM) and the results showed that the nanostructures morphologies could be modulated by the control of T_s , as a result, the continuous film was obtained at 50 °C, whereas the nanorods (NRs), nanoribbons (NBs), nanowires (NWs), nanosheets (NSs) and nanoparticles (NPs) were facily generated as T_s increased. At the same time, the density and the uniformity of the nanostructures decreased. The results of X-ray diffraction (XRD) indicated that only the β -phase polymorph formed throughout the growth process irrelevant to the T_s . Additionally, the ultraviolet visible (UV–Vis) absorption spectra demonstrated that the main absorption bands of MnPc nanostructures showed a remarkable band broadening as the T_s was increased.

1. Introduction

In recent years, the functional organic molecules, especially their nanostructures, have attracted considerable attention motivated by their unique excellent optical and electrical properties [1,2]. Organic nanostructures possess some more competitive properties such as relative ease of chemical doping [3,4], good processability [4], high reactivity [5], and high flexibility [6,7] contrast with the inorganic materials due to their weak intermolecular forces (π – π conjugation, van der Waals forces, H-bonds and charge-transfer interactions) [8].

Metal phthalocyanine (MPc), one of the most typical functional organic molecules, has become an extremely attractive option for applications in light-emitting diode (OLED) [9–13], organic field-effect transistor (OFET) [14–19], organic solar cells (OSCs) [20–24], and optical waveguides [25], etc. Looking back on the previous works, a good deal of researches had been made on MPcs films to fulfill the requirements for high-performance devices, while few of works focused on the synthesis and modulation of the MPcs nanostructures. Nevertheless, MPcs nanostructures are still the best objects for fulfilling the organic semiconductor devices properties owing to their absence of grain boundaries, perfect molecular order and minimized concentration of charge traps [26].

A recent work demonstrates that NRs, NBs, NWs, NSs and NPs of perylene-3,4,9,10-tetracarboxylic dianhydride have been prepared on porous anodic alumina oxide (AAO) [8]. There are numerous active sites with smaller curvature radius on the AAO surface which can provide nanostructures with high surface energy (E_s). And the proposed “site-selective” mechanism has been proved to be suitable for the synthesis of organic nanostructures.

In this paper, what we concern most is the modulation of morphology and optical properties corresponding to MnPc nanostructures. Immediately following the synthesis of MnPc NRs, NBs, NWs, NSs, and NPs, the internal correlation among the morphologies, optical properties and T_s is investigated so that a method of preparing MPcs nanostructures with controllable structures is developed. Simultaneously, it has been found that the MnPc nanostructures have excellent crystallinity and enhanced optical properties, which will open new possibilities for the application of organic nanostructures.

2. Material and methods

2.1. Synthesis

MnPc powder (Sigma-Aldrich, 97%) was purified three times before

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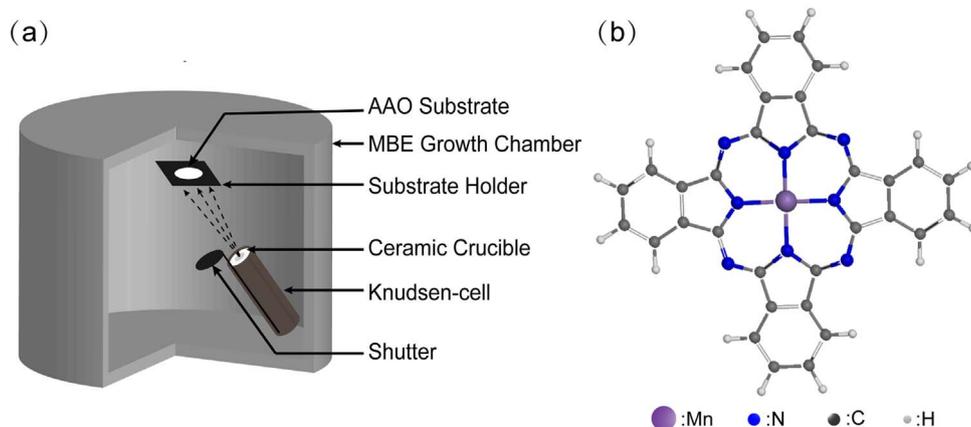


Fig. 1. (a) Cross-sectional schematic diagram of the experiment apparatus. (b) Schematic structure of MnPc molecule.

the use. The purchased AAO template was selected as the substrate with the pore diameter and depth of 50 nm and 60 μm , respectively.

The synthesis of MnPc film and nanostructures were conducted by OMBE method inside an ultrahigh vacuum chamber, in which the base pressure was about 5.0×10^{-10} Torr. The cross-sectional schematic diagram of the experiment apparatus is shown in Fig. 1(a). First of all, the AAO substrate was transported into the ultrahigh vacuum chamber and annealed at 350 $^{\circ}\text{C}$ for 30 min in order to remove the moisture and impurities on the AAO surface, followed by cooled down to the selected T_s . Next, the MnPc powder placed in the evaporator was heated to the evaporation temperature (about 200 $^{\circ}\text{C}$). And the MnPc film and nanostructures were deposited on AAO at different T_s , i.e., 50 $^{\circ}\text{C}$, 80 $^{\circ}\text{C}$, 120 $^{\circ}\text{C}$, 140 $^{\circ}\text{C}$, 180 $^{\circ}\text{C}$ and 200 $^{\circ}\text{C}$. The deposition rate was monitored by a quartz crystal microbalance at about 6 $\text{\AA}/\text{min}$ and the working vacuum was maintained at about 2.0×10^{-8} Torr during the deposition process. The growth time of all samples was 2 h.

2.2. Characterization

The morphology of MnPc nanostructure was examined by scanning electron microscopy (SEM) using a FEI NanoLab 600i SEM/FIB dual-beam system. Prior to the SEM detection the samples were coated with

an ultra-thin layer of Au to avoid charging effect and increase the conductivity of the MnPc nanostructures. The X-ray diffraction (XRD) measurements were carried out on a Rigaku-TTR3, with a Cu K_{α} source ($\lambda=1.541 \text{ \AA}$) to characterize the crystal structure. The ultraviolet visible (UV-Vis) absorption spectra were recorded on a Shimadzu DUV-3700 spectrophotometer. And all the substrate signals have been subtracted during data processing.

3. Results and discussion

3.1. Morphology analysis by SEM

The molecular structure of MnPc is demonstrated in Fig. 1(b). The MnPc molecule possesses planar geometry and a very stable π -conjugated macrocyclic ligand. An unsaturated transition metal-manganese ion with +2 oxidation state is at the center of the molecule.

Fig. 2 shows the SEM images of the MnPc nanostructures prepared at different T_s . The continuous and uniform MnPc film grown at 50 $^{\circ}\text{C}$ was composed of many nanograins with an average dimension of about 70 nm. There were significant similarities between the morphologies of MnPc film and AAO according to Fig. 2(a). Fig. 2(b) shows that when T_s increased to 80 $^{\circ}\text{C}$, the short NRs with an average diameter of 35 nm

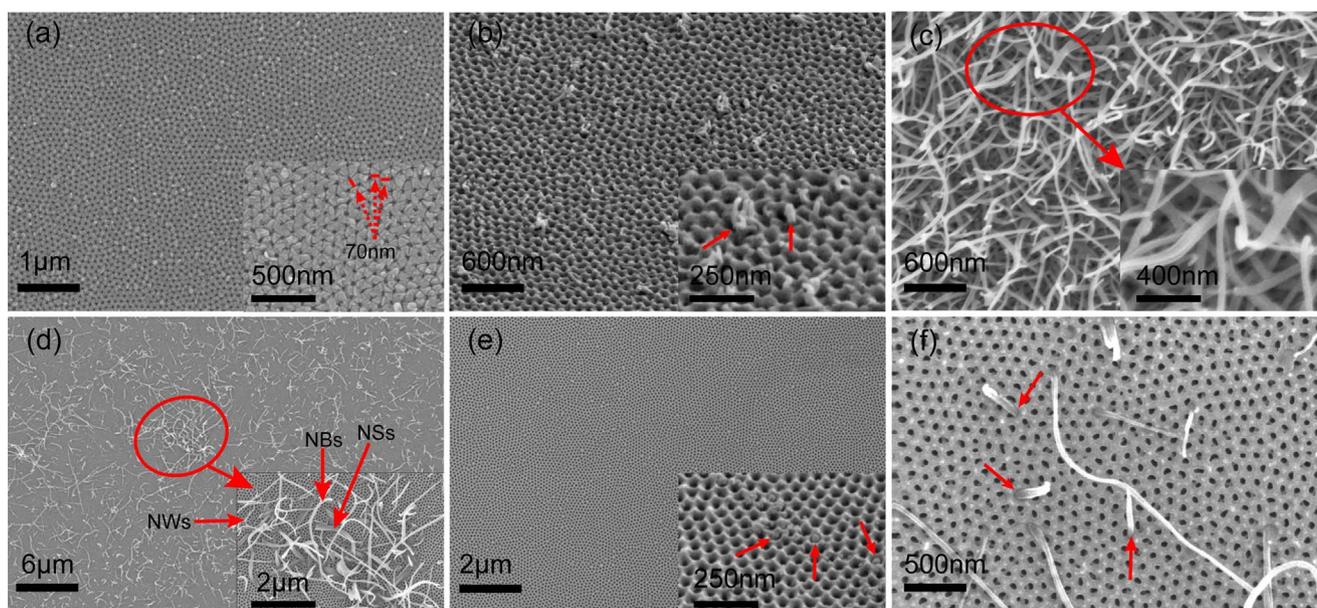


Fig. 2. Schematic structure of MnPc film and nanostructures formed on AAO at (a) $T_s=50^{\circ}\text{C}$, (b) $T_s=80^{\circ}\text{C}$, (c) $T_s=120^{\circ}\text{C}$, (d) $T_s=180^{\circ}\text{C}$, and (e) $T_s=240^{\circ}\text{C}$. A partially enlarged image of nanostructures produced at 180 $^{\circ}\text{C}$ is presented in (f). The insets in (a)-(e) are the enlarged images. And in order to observe the morphology feature more clearly, images of (b) and (e) were taken with a 45 $^{\circ}$ tilted angle.

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