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Mesoporous Ordered Thin Film Formation of Self-Assembled Amphiphilic Copolymer

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An amphiphilic triblock copolymer has been used as a structure-directing agent to produce a mesoporous ordered thin film by a spinning technique. The triblock copolymers consisting of polyethylene oxide (PEO) and polypropylene oxide (PPO) possess amphiphilic properties in a solution and forms self-assembled ordered structures. F108 copolymer, which has the highest value of R (R is the monomer number ratio of ethylene oxide to propylene oxide) among used copolymers, was used to make a highly ordered cubic structure thin film.

Keywords: mesoporous; thin film; triblock copolymer

INTRODUCTION

The structure of mesoporous ordered materials, which have nanometer size pores (5-30 nm), are controlled by a synthesis condition^[1,2]. These materials have attracted considerable interest because they can be applied not only to catalytic materials but also to membranes, chemical sensors, quantum dots, and optical materials^[2,3]. Nowadays, many research groups have tried to make uniform thin films^[4,5] because a thin film has a potential for advanced applications.

In this study, we present the effect of the R value (R is the monomer

number ratio of ethylene oxide to propylene oxide) on the structure ordering of a mesoporous thin film and analyze the internal structure of the highly ordered mesoporous thin film.

EXPERIMENTAL

Amphiphilic triblock copolymers ($\text{EO}_{N/2}\text{-PO}_M\text{-EO}_{N/2}$, Pluronic, BASF Co.: F108 ($R=5.3$), F127 ($R=3.1$), P105 ($R=1.3$), P104 ($R=0.9$), P103 ($R=0.6$), and L101 ($R=0.15$)) were used as a structure-directing agent. TEOS ($\text{Si}(\text{C}_2\text{H}_5\text{O})_4$, Acros, 98%), ethanol (Merck, 99.9%, ACS), double distilled water were used. To obtain a typical reaction solution, 0.008M of triblock copolymer was dissolved by stirring in a solution of 4.7ml of water ($\text{pH}=1.4$, mixed with HCl solution) and 22ml of ethanol, and then 6.4ml of TEOS was added and stirred at room temperature. We used a spin-coating method to prepare a mesoporous thin film on hydrophilic treated Si wafers. The internal structures of the thin films were analyzed by X-ray diffraction and transmission electron microscopy (TEM).

RESULTS AND DISCUSSION

According to the main X-ray diffraction peaks (Fig. 1), the R value affects the ordering of thin films. F108 copolymer system, which has the highest R value (5.3) among the used copolymer systems, had the strongest X-ray peak intensity (180k cps at 1.25 degree). Figure 2 (a) represents the cross-section TEM image of that sample and Fig. 2 (b) its Fast Fourier Transform image. These images reveal that the internal structure of the F108 system is highly ordered cubic. For mesoporous bulk materials, other research groups have reported the effect of R value on the ordering. They indicate that the R value of a

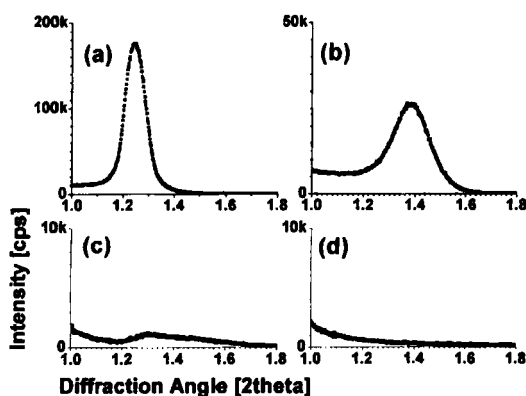


FIGURE 1. X-ray diffraction peaks of maximum intensity for the mesoporous thin films of (a) F108 ($R=5.3$), (b) P105 ($R=1.3$), (c) P104 ($R=0.9$), and (d) P103 ($R=0.6$) copolymer systems (with heat treatment at 400°C , 8hr).

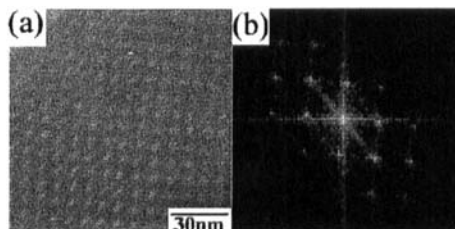


FIGURE 2. (a) TEM image (cross-section with a substrate) of the mesoporous thin film (F108 copolymer system) and (b) its Fast Fourier Transform image.

copolymer controls the mesoporous structure and a higher R value favored a cubic structure^[6].

In the case of mesoporous thin films, the R value also has an effect on the ordering. The higher the R value the more ordered the mesoporous structure. Figure 3. shows the X-ray diffraction peaks and plane indices of the thin film made of F108 copolymer. The results clearly

show that the F108 copolymer system has the mesoporous cubic structure and its d spacing is 11 nm.

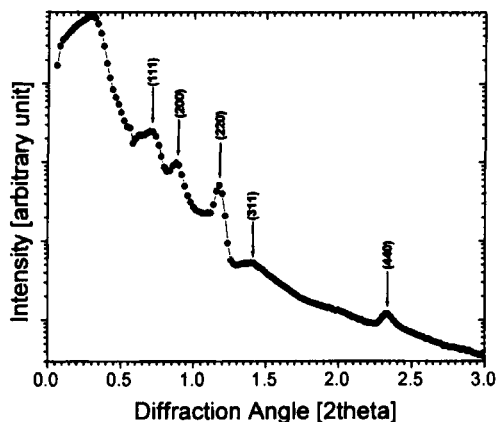


FIGURE 3. The X-ray diffraction peaks correspond to Bragg reflections in the cubic system (F108 copolymer system without heat treatment).

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