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Properties of Synthetic Opal Infiltrated with Phenolic Resin and Periodic Porous Nanostructured Carbon

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Properties of materials are dependent not only on their molecular structure but also on morphology and super-structure especially at the surface and the interface between other materials. It is important how to functionalize and design the structure, surface and interface of materials. In some cases, porous nature of materials is important determining factor of their properties.

Silica opal is a type of naturally occurring photonic crystal [1,2] that consists of well-ordered three dimensional arrays of SiO_2 spheres, which have diameters in the wavelength range of visible light. We have proposed to realize new functionality by infiltration of various materials such as polymer and carbon in the interconnected nano-scale voids of a synthetic opal which was prepared by sedimentation of mono-dispersed SiO_2 spheres of several hundreds nanometer in diameter, and demonstrated various interesting phenomena in this infiltrated opal [3-8].

Synthetic opals with a three dimensional periodic structure in thin films were fabricated by sedimentation of mono-dispersed SiO_2 spheres of 300 nm in diameter [6]. This opal film of 50μm in thickness exhibits beautiful opalescence color. Phenolic resin was used for infiltration into nano-scale voids of the thin opal films in the liquid phase. The optical transmission spectra were measured utilizing a HP8452A spectrophotometer. Figure 1 indicates the transmission spectra of an opal thin film fabricated from SiO_2 spheres of 300nm in diameter and the opal film infiltrated with a phenolic resin. In the both cases, clear stop bands were observed. The stop band shifts remarkably by infiltrating a phenolic resin.

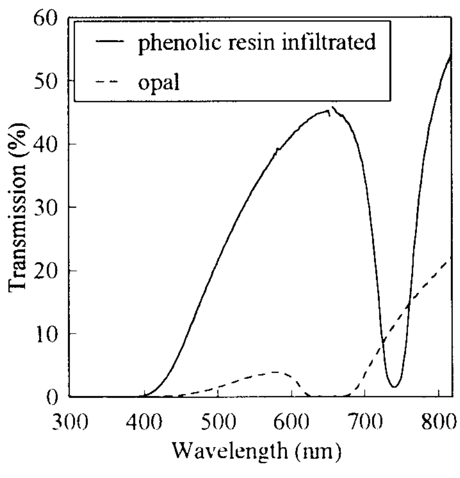


Fig. 1. Transmission spectra of a synthetic opal of 50μm in thickness in air and the opal film infiltrated with phenolic resin.

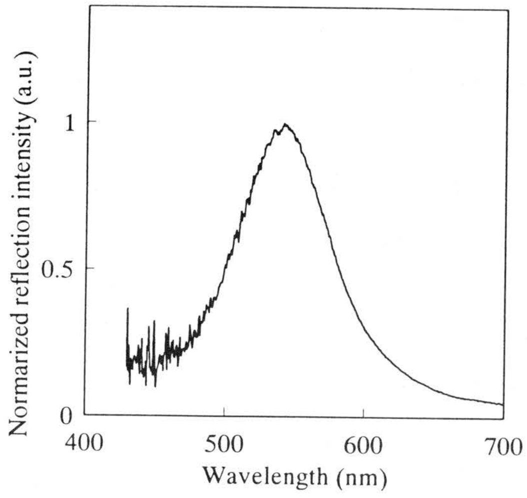
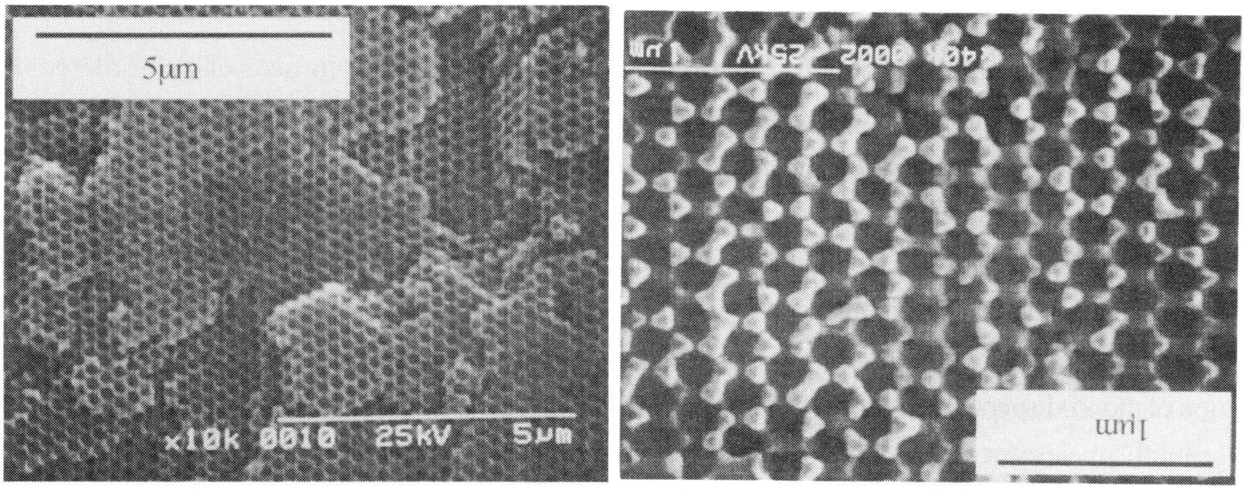


Fig. 2. Reflection spectrum of porous phenolic resin with nano-scale periodic structure.



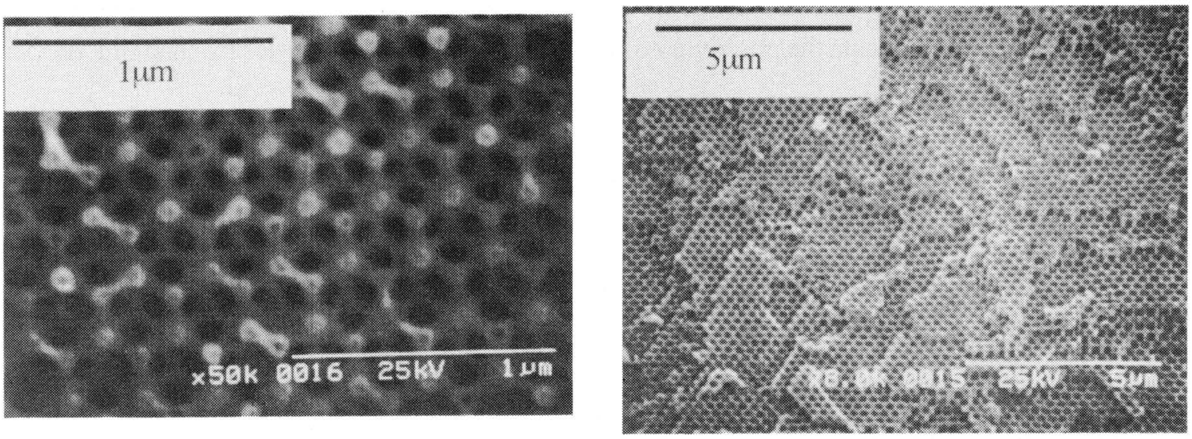
(a) (b)

Fig. 3. SEM images of (a) porous phenolic resin with nano-scale periodic structure and (b) pyrolized one at 700°C.

The reflectance spectrum at normal incidence was evaluated by observing the light reflected from the sample surface irradiated with the light of a wide spectrum in the visible range using a PMA-11 (HAMAMATSU). Porous phenolic resin with nano-scale periodic structure was made by immersing the product infiltrating voids in opal by phenolic resins into the aqueous solution of hydrofluoric acid to remove SiO_2 spheres. The reflection peak of porous phenolic resin with nano-scale periodic structure also was clearly observed as shown in Fig.2.

Next, we have examined the change of pyrolized periodic porous structure and optical properties in a periodic porous nanostructured carbon. There are two methods to obtain a periodic porous nanostuructured carbon which consists of the carbon containing air-filled spheres instead of the SiO_2 spheres. That is, the first were made by pyrolizing the porous phenolic resin with nano-scale periodic structure at various temperatures in a high-purity Ar or N_2 atmosphere for carbonization. The periodicity and therefore the size of voids in the pyrolized porous phenolic resin with nano-scale periodic structure at 700°C varies from

300 nm to 230 ± 10nm with increasing the heat treatment temperature (HTT) as can be seen from SEM images in Fig. 3.



(a) (b)

Fig. 4. SEM images of periodic porous nanostructured carbons with the HTTs of (a) 700 and (b) 2800°C.

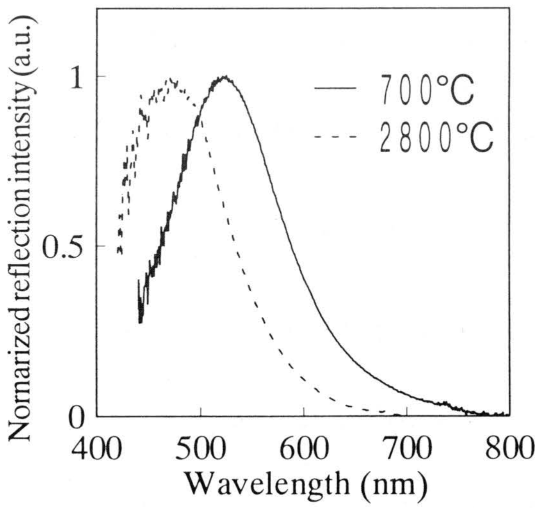


Fig. 5. Reflection spectra of pyrolized periodic porous nanostructured carbons with the HTTs of 700 and 2800°C.

Next, the pristine products infiltrating voids in opal by phenolic resin were pyrolized in a high-purity Ar or N_2 atmosphere for carbonization of samples and subsequently the periodic porous nanostructured carbons were made by immersing the pyrolized product infiltrating voids in opal by carbons into the aqueous solution of hydrofluoric acid to remove SiO_2 spheres. These samples were pyrolized at various temperatures for 1 h in a high-purity Ar or N_2 atmosphere. Figures 4 and 5 show SEM images of pyrolized samples at 700 and 2800°C and the dependence of the reflection spectra for sample on the HTT. The periodicity and therefore the size of voids in pyrolized sample at 700°C are consistent with the diameter of spheres of synthetic opal as can be seen from SEM images in Fig. 4a. As evident in Fig. 5, clear reflection peak is observed also in the pyrolized sample at 700°C. In the case of sample with a HTT of 2800 °C, sharp reflection peak is blue-shifted and periodicity varies from 300 nm to 265 ± 10 nm. It should be noted that even in the case of the HTT of 2800 °C, sample contains a periodic porous nano - scale structure. These results indicate the possibility of control of the optical properties by changing the periodicity with pyrolysis technique.

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