Synthesis of macroporous diethylphenylene-bridged polysilsesquioxane gels and their mechanical properties

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Introduction

Organically modified siloxane (ORMOSIL) gels are synthesized from alkoxysilane precursors containing *R*-Si or Si-*R'*-Si (*R*, *R'*: non-hydrolyzable organic group) by a sol-gel process. In the presence of appropriate surfactant, ORMOSIL gels bearing macroporous structure with mutually connected gel skeletons. The mechanical strength of monolithic gels depends on the macropore structure as well as the elastic property of gel networks. Compared with silica gels prepared from tetraalkoxysilane, ORMOSIL networks exhibit a lower Young's modulus and higher flexibility. In this study, we have prepared macroporous gels composed of diethylphenylene-bridged polysilsesquioxane(PSQ) by a sol-gel process accompanied by phase separation [1] and their mechanical properties were examined in detail. The effect of molecular structure of gel skeletons will be discussed.

Experimental Procedures

The nonionic surfactant, Pluronic® P123, was dissolved in 0.5 M HCl and methanol mixed solution, and then Bis(trimethoxysilylethyl)benzene was added dropwise with stirring. The mixture was stirred for 10 min at room temperature. The sol was kept 60 °C for 24 h for gelation and aging. The resulting wet gels were washed with distilled water, solvent-exchanged with 2-propanol, and dried to obtain porous monoliths composed of diethylphenylene-bridged PSQ. Some samples were subjected to thermal (hydrothermal) treatment in 1 M urea solution at 60-120 ºC for 24 h to increase the crosslink density. Electron microscopy (SEM), thermogravimetric analysis (TG-DTA), nuclear magnetic resonance spectroscopy, and uniaxial compression were performed on the porous monolithic samples.

Results and Discussion

By optimizing the starting composition, the porous diethylphenylene-bridged PSQ monoliths with 3-D continuous framework were successfully prepared. (Fig. 1). The thermal treatment in urea solution can enhance the crosslinking degree and therefore suppress the shrinkage during drying. As for the mechanical property, the porous silica monolith was fractured with less than 5% compression, whereas the porous diethylphenylene- bridged PSQ monolith without the treatment in urea solution showed more flexible nature recovering its original shape after 40% compressive deformation (Fig. 2). It was however found that the hydrothermal treatment in urea solution spoiled the flexibility rendering the PSQ monolith fragile similar to the porous silica monolith. Hence, it was revealed that the crosslinking degree of 3D networks need to be moderate for acquiring flexibility. In the presentation, we will discuss the relationship between the starting composition and the pore structure of the obtained PSQ gels.

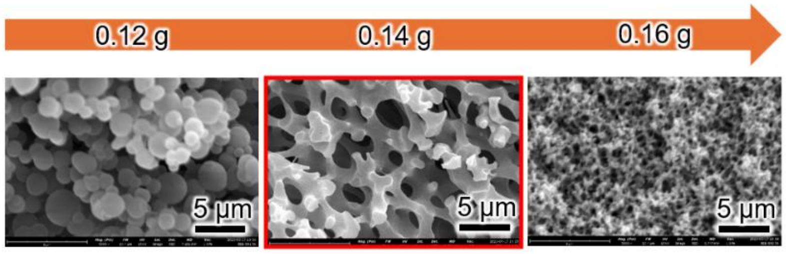

Fig. 1 SEM images of the porous diethylphenylene-bridged PSQ monoliths prepared with varied amounts of P123.

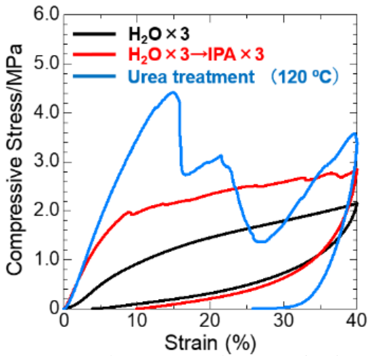

Fig. 2 Compressive stress-strain curves for the porous diethylphenylene-bridged PSQ monoliths.

References

[1] K. Nakanishi, *J. Porous Mater.* **1997**, *4*, 67.