

THE EFFECTS OF SiC CONCENTRATION ON MEMBRANE THICKNESS PREPARED BY CHEMICAL SUSPENSION TECHNIQUE

Z. A. WAHID, R. RAMLI

Ceramics Technology Group, SIRIM Berhad, Persiaran Dato' Menteri, P.O. Box 7035, 40911 Shah Alam, Malaysia

A. MUCHTAR, A. W. MOHAMMAD

Faculty of Engineering, Universiti Kebangsaan Malaysia, 43600 UKM Bangi, Selangor, Malaysia

Abstract Ceramic membrane filtrations are increasingly important as they exhibit tremendous improvement in their capabilities as filtration systems compared to polymeric based systems. Having excellent mechanical properties, chemical and thermal stability, ceramics can resist hostile temperatures as well as harsh solvent environment. The objective of this research is to investigate the effects of the SiC concentration on the thickness of the membrane prepared by the chemical suspension technique. Six SiC concentrations were prepared for the membrane development i.e. 1.5 g, 2.5 g, 3 g, 4 g, 5 g, and 6 g. The SiC in the form of whiskers were suspended in chemical solution and then circulated inside the alumina tube using a special coating assembly for membrane formation and deposition. The porous structure of the alumina support and the SiC membranes were studied using Scanning Electron Microscope (SEM) to determine the microstructure and membrane thickness.

KEYWORDS: Chemical suspension, membrane, thickness, ceramic, microfiltration

Introduction

Ceramic filters, first introduced in the 1980s, have shown a lot of improvement in their performance from microfiltration to ultrafiltration and just recently nanofiltration (Bolduan and Latz, 2000; Sondhi *et al.*, 2003). This is due to the exploitation of new innovative membrane materials and depositing techniques. Even though polymer membrane filters are flexible, cost effective, highly permeable and easy to produce, polymer filters have many limitations that confine their ability in wider applications particularly concerning high temperatures and harsh chemical conditions. Ceramic membrane filters have distinct advantages compared to those of polymeric membrane filters in that they can withstand high temperatures, high operating pressures and extreme chemical conditions (Bolduan and Latz, 2000; Jones *et al.*, 2001; McCool *et al.*, 2003; Sondhi *et al.*, 2003).

Membrane thickness and coating uniformity are crucial in achieving consistency in membrane performance. Various techniques have been developed for ceramic membrane development including sol-gel dip coating, sol-gel spin coating, and chemical vapor deposition (CVD), slip casting and chemical suspension technique (Chu and Anderson, 1996; So *et al.*, 1998; Rakib *et al.*, 2001; McCool *et al.*, 2003). Among these, the chemical suspension technique is the least studied but it was reported that this technique could easily achieve consistency in membrane thickness compared to other methods (Chu and Anderson, 1996).

The ceramic microfiltration performance relies on the thickness and uniformity of the membrane. The membrane layer is made as thin as possible to achieve high flux during the filtration process (Rakib *et al.*, 2001). In this study, silicon carbide (SiC) whiskers (1 μ m diameter) are coated on

ceramic tubes using the chemical suspension technique for microfiltration applications. The thickness of the membrane depends not only on the particle size of SiC whiskers but also on the amount of SiC used during coating process. Thus, the current work attempts to study the effects of the SiC concentrations on the thickness of the membrane layer.

Material and Methods

In this study, alumina tubes with dimensions of 10 mm outer diameter, 6 mm inner diameter and 800 mm length with average pore size of 5 μm were used as supports. 16 g of SiC whiskers (Ceramicfilter Co. Ltd) was added to 3 L of distilled water with 20 ml of 25% ammonia solution (BDH Analar) in a mixing vessel. The pH of the mixture was kept between 10 to 11 for good suspension of the SiC. The mixture was stirred vigorously for one hour at 3000 rpm (Glas-Col GKH High Speed). Next, the mixture was ultrasonicated (Honda Electronics W-220R) with constant stirring for another 30 minutes. The mixture was then transferred to a closed container and was allowed to settle overnight.

The schematic drawing of the coating assembly is indicated in Fig. 1. The alumina tubes were put together into a specially made housing and were placed inside a coating chamber. Then, 15 L of distilled water and 40 ml of ammonia solution were mixed together inside the mixing vessel. Using a pump, the solution was circulated into the tubes for one minute. Next, the SiC slurry with the known SiC concentration (1.5 g, 2.5 g, 3 g, 4 g, 5 g, 6 g) together with 25 ml of 10% ZrCl_2 solution (Fluka) was added into the vessel. The addition of the ZrCl_2 solution is to facilitate adhesion between the SiC particles and the alumina tubes. The mixture was circulated into the tubes for 10 minutes until the water inside the vessel was as clear as the water coming out of the tubes indicating that all the SiC particles have been deposited onto the tubes. The tubes were dried before the firing step. Sintering was done at 500°C with a soaking period of one hour (Shin Sung High Temperature Furnace 1800 s). The sintered ceramic membrane filter tubes were characterized in terms of microstructure and membrane thickness using a Hitachi S2500 Scanning Electron Microscope (SEM).

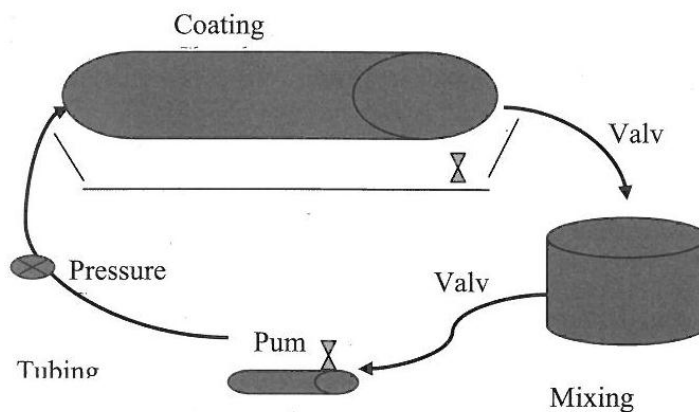


Figure 1. Flow drawing of coating assembly for the chemical suspension technique

Results and Discussion

Figure 2(a) exhibits the cross-sectional view of the alumina membrane filter which consists of two parts: the outer part is the alumina tube and the inner part is the SiC membrane. Figure 2(b) demonstrates the graph of the measured thickness of the membrane layers versus SiC concentrations. As expected, the higher the SiC concentrations, the thicker the SiC membrane.

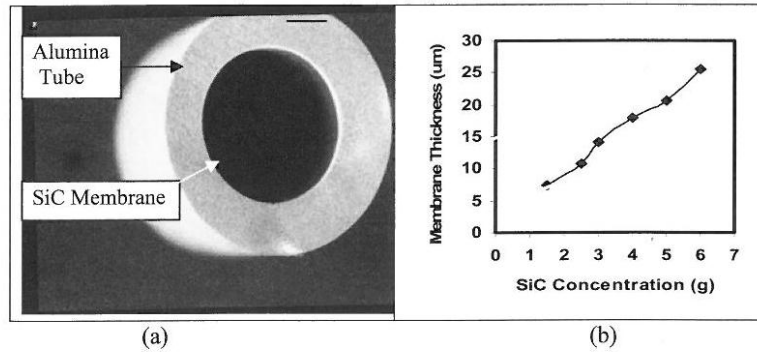


Figure 2. (a) A cross-section of the alumina filter tube; (b) Effect of SiC concentration on the membrane thickness

Figures 3 exhibit cross-sectional views of the tube and the membrane. The pore size of the membrane is incredibly small compared to the pore size of the tube. Both figures demonstrate the microstructure of SiC coating which was of fiber like surface. The SiC coating is thicker for samples prepared with 6 g of SiC compared to those prepared with 1.5 g of SiC. The measured thickness for all the samples is tabulated in Fig. 2(b). The sample with 1.5 g SiC which has a membrane thickness of only 7.5 µm, shows very thin SiC coating layer and its distribution is uneven. This indicates that the SiC concentration used in the coating process is not sufficient to cover the whole tube.

On the contrary, the sample prepared with 6 g SiC exhibits well distributed SiC whiskers and the coating layer is consistent in thickness. This is due to the fact that the applied pressure was uniform throughout the tube. However, a thicker membrane layer will decrease the flux and the flux velocity. For normal filtration applications, the optimum thickness is 15 – 20 µm (Sondhi *et al.*, 2003). Therefore, as indicated in Fig. 2(b), at least 4 g of SiC is required to achieve the optimum membrane thickness.

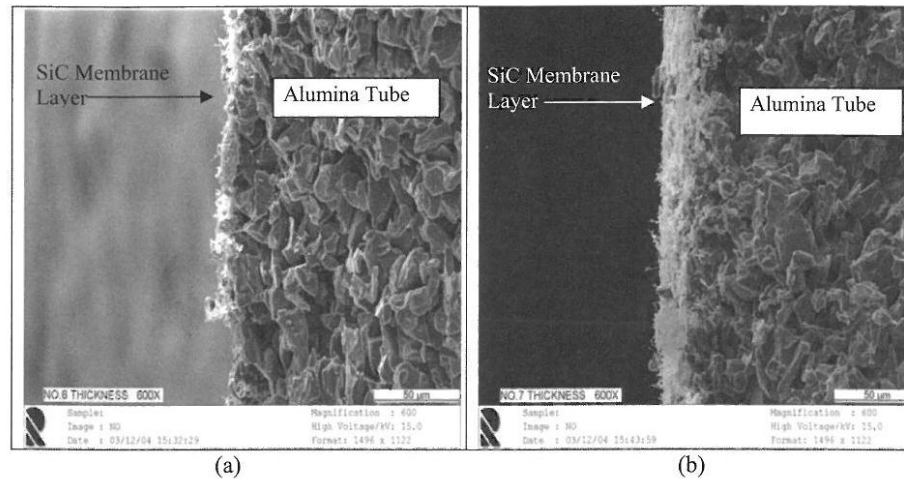


Figure 3. (a) Microstructure of sample prepared with 1.5 g SiC and (b) Microstructure of sample prepared with 6 g SiC

Conclusion

In developing the SiC membrane for microfiltration, the chemical suspension technique can be used to achieve well distributed and uniform coating thickness. However, if the concentration of the SiC used for the coating is too low, there will not sufficient SiC to coat the whole tube and as a consequence the coating layer is uneven. The SiC concentration has to be at least 4 g to achieve the optimum thickness for normal filtration applications.

References

- Bolduan, P. and M. Latz. 2000. *Filtration and Separation* 4:36-38.
- Burggraaf, A. J. and L. Cot. 1996. *Fundamentals of Inorganic Membrane Science and Technology*, Elsevier Science, B.V., Amsterdam. 29-31 pp.
- Chu, L. and M. A. Anderson. 1996. *J. Membr Sci.* 110:141-149.
- Jones, C. D., M. Fidalgo, M. R. Wiesner and A. R. Barron. 2001. *J. Membr Sci.* 193:175-184.
- McCool, B. A., N. Hill, J. Dicarlo and W. J. DeSisto. 2003. *J. Membr Sci.* 218:55-67.
- Rakib, S., M. Sghyar, M. Rafiq, A. Larbot and L. Cot, L. 2001. *Sep Purif Technol.* 25:385-390.
- So, J-H., S-M. Yang and S. B. Park. 1998. *J. Membr Sci.* 147:147-158.
- Sondhi, R., R. Bhawe and G. Jung. 2003. *Membrane Technology* 11:5-8.