

Supercritical Fluid Application Notes

**SCF
530**

Drying of Aerogels with Supercritical Carbon Dioxide

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Introduction

Numerous industries use polyimides in applications as varied as aerospace devices and integrated electronic circuits. Unlike polymers that can be easily infused with multiple substances, once formed, polyimides are impervious to many materials. Infusing additives into commercial polyimides, however, greatly increases the range of their available applications. For example, a polyimide that has a metallic or metallic oxide layer on its surface can be used in aerospace applications that require high reflectivity.



Current methods of infusion rely on using liquid solvents at high temperatures. Among the drawbacks of liquid solvent infusion is that it is time consuming, produces a large volume of waste material, and can leave behind potentially toxic residual solvent in the products.

Supercritical CO₂ is an alternative infusion technology that eliminates the use of solvents and need for high temperatures. Supercritical CO₂ is harmless ecologically, readily available, non-toxic and non-explosive. When placed above the critical point (31.1°C and 73 atm) CO₂ becomes a remarkable solvent for many additives. The high diffusivity of supercritical

CO₂ allows the additives dissolved in the CO₂ to be absorbed by the polyimide.

As the system is rapidly depressurized, the CO₂ is released and the precipitated additive is left in the polyimide.

This application describes the use of supercritical CO₂ to produce a reflecting film by the infusion of silver.

Equipment

- ✓ Applied Separations' *Spe-ed*TM SFE-2 or Helix



Materials

- ✓ BTDA-ODA polyimide films
- ✓ 1,5-cycloocta-dienesilver(I) 1,1,1,5,5,5-hexafluoroacetylacetonate, (COD)
- AgF₆acac
- ✓ Carbon dioxide –supercritical grade

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Method

Curl the fully cured polyimide film and place in the vessel followed by the additive. Pump SC-CO₂ into the vessel at the desired pressure and heat the vessel to the desired temperature until the end of the infusion sequence. Upon completion of film infusion, cool the cell to room temperature. Once the vessel is cool, decompress the vessel to atmospheric pressure. Remove the film and cure a second time at 300 °C.

Infusion Conditions

Vessel: 10 mL
Pressure: 5000 psi
Temperature: 110 °C
Additive: 10% of polyimide film weight
Time: 120 minutes

Analysis

Analyze the distribution of silver on the surface of the film and in the polyimide via scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Optical reflectivity can be measured by thermogravimetric analyses (TGA).

Conclusion

The supercritical CO₂-assisted impregnation process is a fast, ecologically safe, and economic method for infusing a silver additive into a polyimide film. The resulting films were reflective and retained their flexible nature. When used in large scale production, CO₂ can be recycled for re-use. In addition, since supercritical CO₂ has higher diffusivity than liquid solvents, the SC-CO₂ infusion technique requires only a one step process.

References

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Nazem, N.; Taylor, L.; Rubira, and Rubira, A. "Supercritical Fluid Infusion of A Silver-Containing Compound into Poly(Ether Ether Ketone) for the Purpose of Making Reflective Films."

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