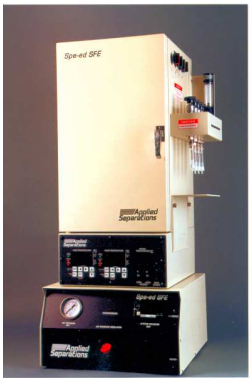
SCF Application #530

Impregnation of Organometallic Silver into Polyimide Film using Supercritical Carbon Dioxide

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 CO_2 is an alternative infusion technology that eliminates the use of solvents and need for high temperatures. Supercritical CO_2 is harmless ecologically, readily available, non-toxic and non-explosive. When placed above the critical point (31.1°C and 73 atm) CO_2 becomes a remarkable solvent for many additives. The high diffusivity of supercritical CO_2 allows the additives dissolved in the CO_2 to be absorbed by the polyimide.

As the system is rapidly depressurized, the CO_2 is released and the precipitated additive is left in the polyimide.

This application describes the use of supercritical CO_2 to produce a reflecting film by the infusion of silver.

Equipment

- ✓ Applied Separations' *Spe-ed*™ SFE II

Materials

- ✓ BTDA-ODA polyimide films
- ✓ 1,5-cycloocta-dienesilver(I) 1,1,1,5,5,5-hexafluoroacetylacetonate, (COD) AgF_6acac
- ✓ Carbon dioxide –supercritical grade

Method

Curl the fully cured polyimide film and place in the vessel followed by the additive. Pump SC-CO_2 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 ºC
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 miscroscopy (SEM) and transmission electron microscopy (TEM). Optical reflectivity can be measured by thermogravimetric analyses (TGA).

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Conclusion

The supercritical CO_2-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, CO_2 can be recycled for re-use. In addition, since supercritical CO_2 has higher diffusivity than liquid solvents, the SC-CO_2 infusion technique requires only a one step process.

References

Boggess, R. and L. Taylor. "Infusion of Silver into Polyimides by the use of Supercritical Carbon Dioxide." *J. Appl. Polym. Sci.*, **64**,1309 (1997).

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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