GLASSY CARBON COATED GRAPHITE FOR NUCLEAR APPLICATIONS

Delpeux S. Cacciaguerra T, Duclaux L
CRMD,CNRS-University of ***Orléans,*** *IB rue de la Férollerie 45071 Orléans cedex 2*

Taking into account the problems caused by the treatment of nuclear wastes, the molten salts breeder reactors are expected to a great development. They use a molten fluorinated salt (mixture of LiF, BeF_2, ThF_4, and UF_4) as fuel and coolant.

The reactor core, made of graphite, is used as a neutrons moderator. Despite of its compatibility with nuclear environment, it appears crucial to improve the stability and inertness of graphite against the diffusion of chemicals species leading to its corrosion. One way is to cover the graphite surface by a protective impermeable deposit made of glassy carbon obtained by the pyrolisis of phenolic resin [1,2] or polyvinyl chloride [3] precursors.

The main difficulty in the synthesis of glassy carbon is to create exclusively, in the primary pyrolysis product, a micropomsity of about twenty Angstrôms which closes later at higher temperature. Therefore, the evacuation of the volatile products occurring mainly between 330 and 660°C, must progress slowly to avoid the material to crack.

In this study, the optimal parameters for the synthesis of glassy carbon as well as glassy carbon deposits on nuclear-type graphite pieces are discussed. Both thermal treatment of phenolic and PVC resin have been performed. The structure and microtexture of glassy carbon have been investigated by X-ray diffraction, scanning and transmission electron microscopies and helium pycnometry.

Glassy carbon samples (obtained at 1200°C) show densities ranging from 1.3 to 1.55 g/cm^3 and closed pores with nanometric size (- 5 to 10 nm) appear clearly on the TEM micrographs. Then, a thermal treatment to 2700°C leads to the shrinkage of the entangled graphene ribbons (Fig. 1), in good agreement with the nanosed texture model for glassy carbon (Fig. 2) [4].

Glassy carbon deposits on nuclear graphite have been developed by an impregnation method. The uniformity of the deposit depends strongly on the surface texture and the chemistry of the graphite substrate. The deposit regions where glassy carbon presents a good adhesion appear smooth and non porous, therefore suggesting that glassy carbon deposition is an interesting route to shut off the open porosity on the surface of graphite pieces (Fig. 3). Nevertheless, numbers of parameters have to be better understood: i.e. the optimal protocol to impregnate graphite by the resin, the role of the interface and the possibilities to control the resin shrinkage by adding additives (ie. carbon black, graphite particles, carbon char, etc ...) to the glassy carbon precursor.

Fig. 1. TEM micrographs of glassy carbon prepared from phenolic resin at 1227°C (left side) and 2700°C (right side).

Acknowledgement: the authors thank Bernard Tahon, from the SGL Carbon Group (Passy, France) for providing nuclear-type graphite samples.

[1] H. Maleki, L. R. Holland, G. M. Jenkins, R L. Zimmerman., Carbon, *vol 35 (2),* 227-234, 1997.
[2] L.L. Kreb, P. A. Watkins, US Patent 799479, *1985.*
[3] Terasaki et al, US Patent 048754,*1986.*
[4] G. M. Jenkins and K.Kawamura, Polymeric Carbons-Carbons Fiber, Cambridge, Cambridge University Press, *1976.*