2000
DOI: 10.1017/S1431927605500783
Microsc Microanal 11(Suppl 2), 2005

TEM INVESTIGATION OF GeO_2 CONCENTRATION PROFILES IN GeO_2-SiO_2 SOOT PARTICLES FROM PREFORMS OF OUTSIDE VAPOR DEPOSITION

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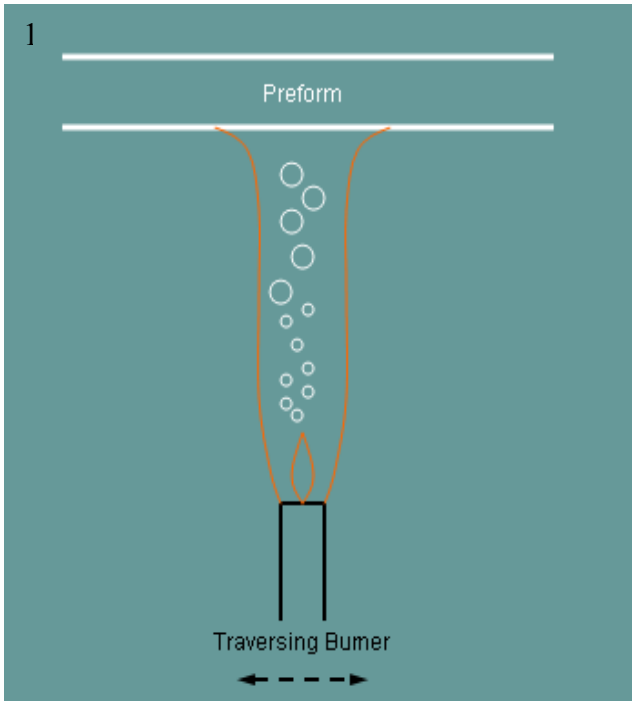
The optical attributes of optical fiber are determined by the refractive index profile (RIP), which depends on the GeO_2 concentration profile along the radial direction. High-data-rate optical fiber products often have complex RIP's, demanding high accuracy in the fabrication of core preforms with GeO_2 profiles that closely match the target. It is important to have fundamental understandings of GeO_2 deposition mechanisms in order to improve the manufacturing process. In the process of outside vapor deposition (OVD), the GeO_2 and SiO_2 are produced from the corresponding halides via oxidation and/or hydrolysis reactions in the flame described by the following chemical reactions:

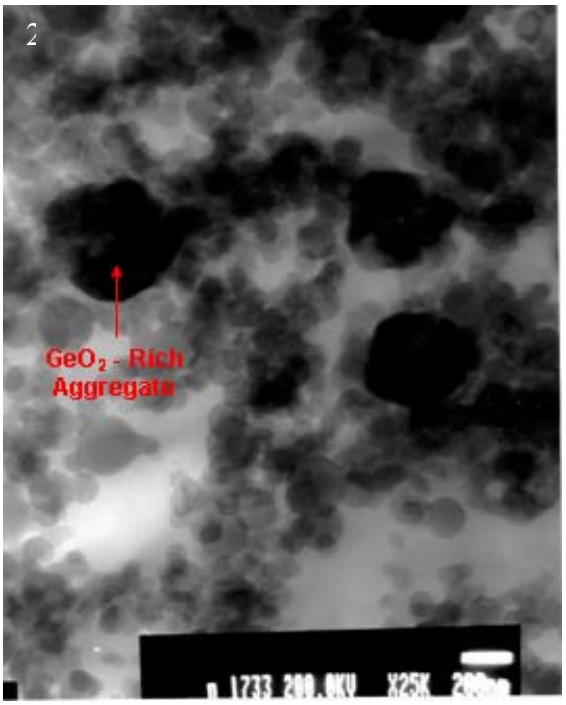
$MCl_4 + O_2 = MO_2 + 2\ Cl_2$ and $MCl_4 + 2\ H_2O = MO_2 + 4\ HCl$

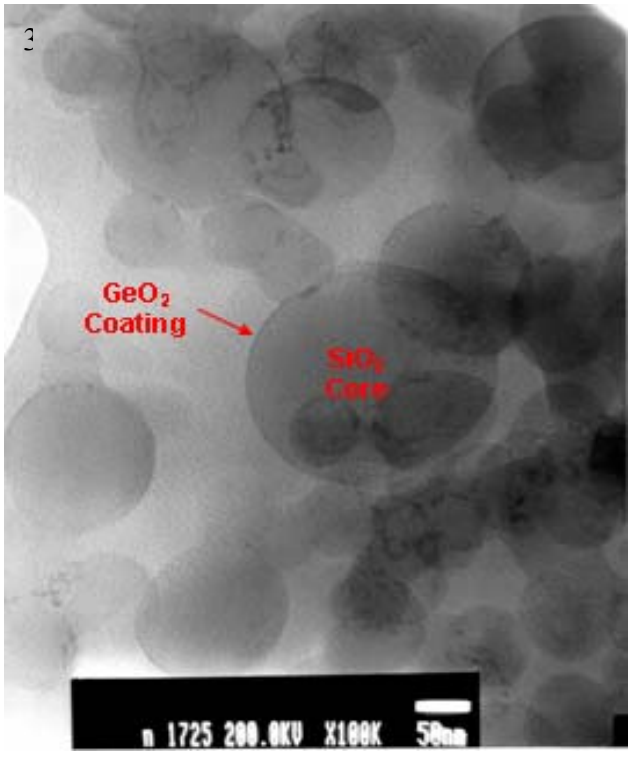
M represents Ge or Si. In the chemical reactions SiO_2 nucleates upstream in the flame jet and forms sub-micron particles and aggregates of particles, which are deposited onto the preform via thermophoresis. The formation of GeO_2 is more complex. Duo to the high vapor pressure of GeO_2 at flame temperatures, reversible reactions are possible. The processes of OVD are illustrated in Fig. 1.

To investigate the GeO_2 concentration profile in GeO_2-SiO_2 soot. TEM experiments were carried out using JEM-2000FX transmission electron microscope equipped with an EVAX ultra-thin window X-ray detector. For the X-ray analysis, the electron probe size was about 10 nm. A small objective lens aperture was used to increase the contrast of the bright field TEM images. In order to obtain the cross sections of the nano-particles, a few layers of specimen of 1 cm^2 in size were taken out from the soot preform sample. These layers were placed on a Si wafer and were glued together with the Si substrate by M-bond. The subsequent procedures of sample preparation are similar to that of cross-sectional TEM sample preparation for thin film specimens.

The EDS results show that the distribution of GeO_2 in the specimen is not uniform and three major types have been observed: (1) Areas of aggregates of nearly pure GeO_2 particles are shown in Fig.2. The dark areas in the image are rich of GeO_2. The aggregates are often as large as 500 nm with irregular shapes. The formation of these aggregates suggests that the vapor phase Ge species nucleate heterogeneously on the preform surface or nucleate homogeneously in the vicinity of the preform surface first and then deposit onto preform via thermophoresis. (2) In contrast to the GeO_2 aggregates, the SiO_2 particles are mostly spherical particles with diameters in the range of 50-200 nm, as shown in Fig. 3. The majority of the particles have a core-shell structure. The shell is very thin with a few nanometers in thickness. The EDS data show that the shell is GeO_2 and the core is SiO_2. The core-shell structure indicates that the vapor phase Ge species condensed on SiO_2 particles in the vicinity of the preform surface. (3) There is a small fraction of particles, in which Ge distributes inside the particles, as shown in Fig. 4. The formation of these particles is different from that shown in Fig. 3. This indicates that the vapor phase Ge species sometime nucleate upstream in the flame jet, together with SiO_2 nucleation and then deposited on the preform.







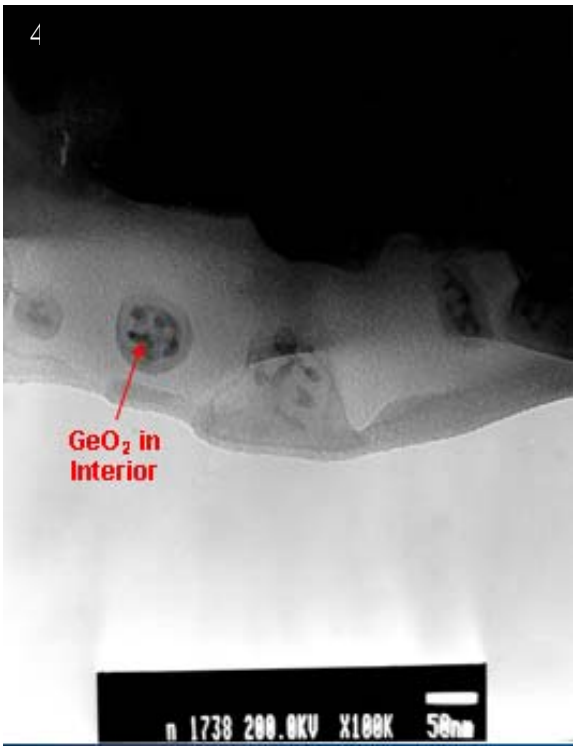


Fig. 1. Illustration of the process of outside vapor deposition.
Fig. 2. Aggregation of nearly pure GeO_2 particles.
Fig. 3. Particles of SiO_2 with GeO_2 coating.
Fig. 4. SiO_2 particles with GeO_2 distributed in the interior of the particles.

https://doi.org/10.1017/S1431927605500783 Published online by Cambridge University Press