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SEMIQUANTITATIVE X-RAY MICROANALYSIS ON PREFORMS FOR OPTICAL WAVEGUIDE FIBRES

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Resume - La distribution du dopant GeO2 dans les fibres optiques, qui sont fabriquées par VCVD, est déterminée par analyse par spectrographie X en énergie. On obtient une résolution de 1,5 μm avec la raie Ge(Lα) et une énergie d'excitation de 10 keV. On a observé dans la fabrication des préformes une fluctuation caractéristique de la densité de GeO2 pour chaque couche déposée. Cette distribution initiale du dopant GeO2 est modifiée par la diffusion et l'évaporation lors des phases suivantes de la préparation des préformes et de l'étirage des fibres.

Abstract - The distribution of the GeO2-dopant in optical waveguides prepared by the VCVD-process is determined with an SEM using energy dispersive microanalysis. Operating at 10 keV and analysing the Ge(Lα)-line, a lateral resolution of approximately 1.5 μm is achieved. A strong characteristic fluctuation of the GeO2-density within each freshly deposited layer is observed. This original GeO2-distribution in each layer is changed by interdiffusion and evaporation processes during following steps of preform preparation and fibre drawing.

Introduction

The transmission characteristics of an optical fibre strongly depend on its refractive index profile. Fig. 1 shows two typical profiles of multimode fibres:

i) a step index

ii) a graded index profile.

Fig. 1 - Typical index profiles and doping materials for SiO2 in multimode fibres

The graded index profile is optimized for high transmission bandwidth and therefore of most interest. In actual fibres, however, the index profile is not smooth but has typical defects caused by the manufacturing processes. Most common is an index ripple along the profile and an index dip in the core centre. Such index deteriorations can reduce the fibre bandwidth drastically. Therefore it is of utmost interest to understand the causes for the index deviations to optimize the fabrication process. This paper investigates details of the GeO2-doping distribu-
tion in silica fibres in the various processing steps from the preform preparation to the drawn fibre.

Fibre preparation and measuring method

The fibres were prepared by the VCVD-process /1/ which is similar to the well-known MCVD-process. The process is based on a homogeneous vapour phase reaction of SiCl4 and O2 to produce soot particles of extremely pure silica glass. To change the refractive index of the glass, doping materials GeO2, F, P2O5 or others are used. For graded index fibres the dominant doping material is GeO2.

Fig. 2 - Schematic view of the VCVD-process (a), the drawing process (b) and the dependence of oxide yield on temperature (c)

The starting vapours and gases are fed through a vertically positioned silica glass tube from the top end. In a short high temperature (~1600 °C) zone produced by a graphite resistance furnace surrounding the tube the soot particles are created (Fig. 2a). The particles are driven by thermophoretic forces to the inner tube wall below the reaction zone (left half Fig. 2a) where they are deposited. The slowly downwards moving heat-zone sinters the soot deposit to a glassy layer. The preform preparation includes the deposition of many layers one by the other each layer doped individually to produce the desired index profile. After the deposition process the preform tube is collapsed to a rod by a high temperature process. The optical fibre is drawn from the preform rod. Alternatively the fibre can also be drawn from the preform tube, as shown in Fig. 2b. In this case the collapsing and drawing steps are carried out simultaneously at the lower end of the preform tube.

An important feature of the vapour phase reactions in the deposition process is that SiCl4 reacts completely to SiO2 for temperatures above approximately 1500 °C whereas the GeO2-yield increases with temperature to a maximum value at about 1500 °C and decreases for higher temperatures /2/, Fig. 2c. In a nonuniform temperature field which obviously exists along the tube radius one expects therefore the creation of soot particles with different GeO2-concentration.

For the quantitative analysis of the GeO2-distribution a Leitz AMR 1000 SEM with an energy dispersive X-ray analyser EDAX mod 711 is used. Operating with the Ge(Lα)-line and primary electrons of 10 keV a lateral resolution of approximately 1.5 µm is achieved. The measurements are carried out in the spot mode of the SEM. For the measurements the count rates of five channels are registered for about 200 s at eight energy values between 900 and 2500 eV including the peaks of Ge(Lα), Si(Kα) and P(Kα). With the aid of a special BASIC-program for an HP85 desk computer first the netto intensities and normalized relative intensities are determined with the aid of calculated relative pure element intensities /3/. The ZAF correction program is based on the NBS-program given by Heinrich /4/ with some modifications for the low energy radiation. The background intensities at the eight measured energy values are determined for a pure SiO2 standard and for a sample with high GeO2 dopant (ca. 50 wt.-%). These values are constants in the background subtraction procedure and are linearly interpolated iteratively with respect to the GeO2 content.
Results

Fig. 3 shows SEM-micrographs of an etched cross section of a fibre and of a polished preform disk prepared from the neck-down zone of a preform end, respectively. A characteristic ring structure corresponding to the individual layers from the preform preparation is revealed. The anticipated inhomogeneous distribution of the Ge(O_2) doping within the individual layers results in unequal etching rates. We found an increasing etching rate with increasing Ge-content.

![SEM micrographs of different process steps of fibre fabrication](image)

On the other hand the micrograph of the polished preform disk shows (Fig. 3b) bright zones for Ge-rich and dark zones for Ge-poor regions on the specimen. This contrast behaviour is caused by the different reflection coefficients for high and low Ge-contents.

Fig. 4 and Fig. 5 show the measured distribution of Ge(O_2) and P_2O_5 concentration (a small amount of P_2O_5 was added to the deposition of silica to reduce the sintering temperature) in the layers at the preform tube and in the disk prepared from the collapsed neck-down region when drawing the fibre from the preform tube, respectively.

Fig. 4 - Measured Ge(O_2) concentration profile inside the three innermost layers of the preform tube (a) and SEM micrograph of these layers (b)

Fig. 4a reveals a strong characteristic oscillation of the Ge(O_2) concentration across each layer. In each layer the Ge(O_2) concentration increases towards the tube centre but is zero at both layer borders. In contrast the P_2O_5 concentration level is found to be constant over all layers.

Fig. 4b shows the corresponding SEM micrograph. The dark regions correspond to a low GeO_2 concentration and the brighter regions indicate higher GeO_2 concentrations. The Ge(O_2) concentration profile can be explained by the inhomogeneous reaction of the GeO_2 due to the strong temperature dependence and by the high evaporation rate of GeO_2 at the innermost free layer surface during the sintering of the layer, resulting in a depletion of GeO_2 at the surface region.

Fig. 5 (thick curve) gives the measured Ge(O_2) concentration profile in the neck-down region (sample 2) whereas the thinner curve corresponds to the Ge(O_2) distribution found in the preform tube but transformed into the collapsed form.
Fig. 5 - Ge(O_2)- and P_2(0_5) concentration profiles near the centre of a disk taken from the collapsed preform tube (thick curve) and the transformed Ge(O_2) concentration profile of Fig. 3a (thin curve); P_2(0_5) dashed curve

A comparison of the two profiles clearly demonstrates a change in the Ge(O_2) distribution which obviously occurred during the drawing process when the preform tube was heated up and collapsed to the neck-down region. The change can be explained by dopant-diffusion within the layers, between adjacent layers and by evaporation on the inner free surface. The latter leads to the observed dopant depletion corresponding an index depression at the core centre, the so called index dip.

Conclusion
The GeO_2 distribution in graded index preforms and fibres is neither smooth nor step-like as should be expected theoretically from the deposition of doped layers. Strong oscillations are found within the layers at the preform tube. Details of the distribution in the layers as deposited are essentially a function of the temperature profile in the reaction zone that depends on the heat source and is a function of the temperature dependence of vapour phase reaction for GeO_2.

The oscillating distribution is considerably smoothed out during preform collapsing and fibre drawing. Additional evaporation of dopant material from the inner surface of the preform tube during the collapsing step results in dopant depletion in the core centre and is the reason for the index dip.

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Literature