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Material Structure of Germanium-Doped Optical Fibers and Preforms

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The structural characteristics of preforms and optical fibers fabricated by modified chemical vapor deposition were studied by optical, interference, and scanning electron microscopy. It was observed that the structural features resulting from the deposition process are preserved through subsequent processing and appear in the fiber with the exception of a region at the center of the fiber. Here, selective evaporation of dopant material from the inner surface of the deposit results in a refractive index depression on the axis of the optical waveguide.

I. INTRODUCTION

The chemical vapor deposition process, in which oxides are deposited and simultaneously fused on the inner surface of a fused silica tube, has become a valuable technique for fabricating low-loss[1,2] and graded-index optical fibers.[3] In modifications and refinements of this technique, higher depositional rates and very low-loss single-mode fibers[4] have also been achieved.

An important question that arises in utilizing this process concerns the correlation of the deposited material structure in the preform to that in the resulting optical fiber. Can one be confident, for example, that the same distribution of refractive index that is introduced into the preform by changing the material composition of the deposited layers exists in the fiber pulled from this preform? This determination is necessary if one is to reliably fabricate those graded-index profiles required to achieve a minimum of pulse dispersion.[5] This is due to the fact that the shaping of the index profile is quite critical because the reduction-in-pulse-dispersion-vs-profile curves exhibit a singularity-like behavior in the region of the optimum index distribution.[6]

Evidence for the preservation of the deposited profile has recently been reported, based on the observation of a linear increase in refractive index in a fiber which was pulled from a preform in which the

dopant concentration was increased in the same manner.[7] In this paper, we present results of optical, interference, and scanning electron microscope studies[8] of a graded, near-parabolic, index fiber and preform as further aid in understanding the transition of material from the preform to the fiber state. A main conclusion of this study is that the structural features resulting from the preform deposition process are preserved and, after suitable scale transformation, appear in the fiber. Due consideration should therefore be given to depositional characteristics that may ultimately affect transmission behavior.

II. OBSERVATIONS

The preform originates from an approximately $\frac{1}{2}$-m-long 12 × 14-mm fused-quartz tube which is collapsed into a rod after the deposition process. In the structure studied here, the deposition started with an initial layer of borosilicate to prevent impurity diffusion into the core. This layer was deposited with 41 traversals of a oxyhydrogen burner which provides the heat to react the BCl_3, $SiCl_4$, and O_2 starting materials. The core deposition process consists of systematically increasing the flow of $GeCl_4$ while holding the flows of $SiCl_4$ and BCl_3 constant, thus producing an increasing GeO_2 content and associated increased refractive index with increasing deposit thickness. The $GeCl_4$ flow was increased 11 times, in such a manner as to produce a near-parabolic index variation from the cladding interface to the center of the core. The number of torch traversals during each of the 11 steps was controlled to make the thickness of each step approximately equal. After collapse, a length of preform was pulled into a fiber with an overall diameter of $\sim$100 μm by the use of an electric furnace.

A slice transverse to the axis of the remaining length of preform was made and then polished to a thickness of approximately 10 mills for interference and optical microscopic observations. Transverse samples of the fiber were also prepared for interference-microscope and scanning-electron-microscope studies.[8] In the latter case, after a short length of fiber is scored and broken to ensure a flat end, it is etched in a 25-percent solution of hydrofluoric acid for several minutes and then flash-coated. The last step is performed to prevent charge build-up on the sample during scanning-electron microscope observations.

An overall view of the preform sample observed with conventional optical microscopy is shown in Fig. 1a. The sample is $\sim$7.2 mm in diameter with a core diameter of 4 mm. The irregular shape is due to pieces of the cladding which broke off during the cutting and polishing procedure. It should be noted that the preform is under considerable stress because of the difference in the expansion coefficients of the

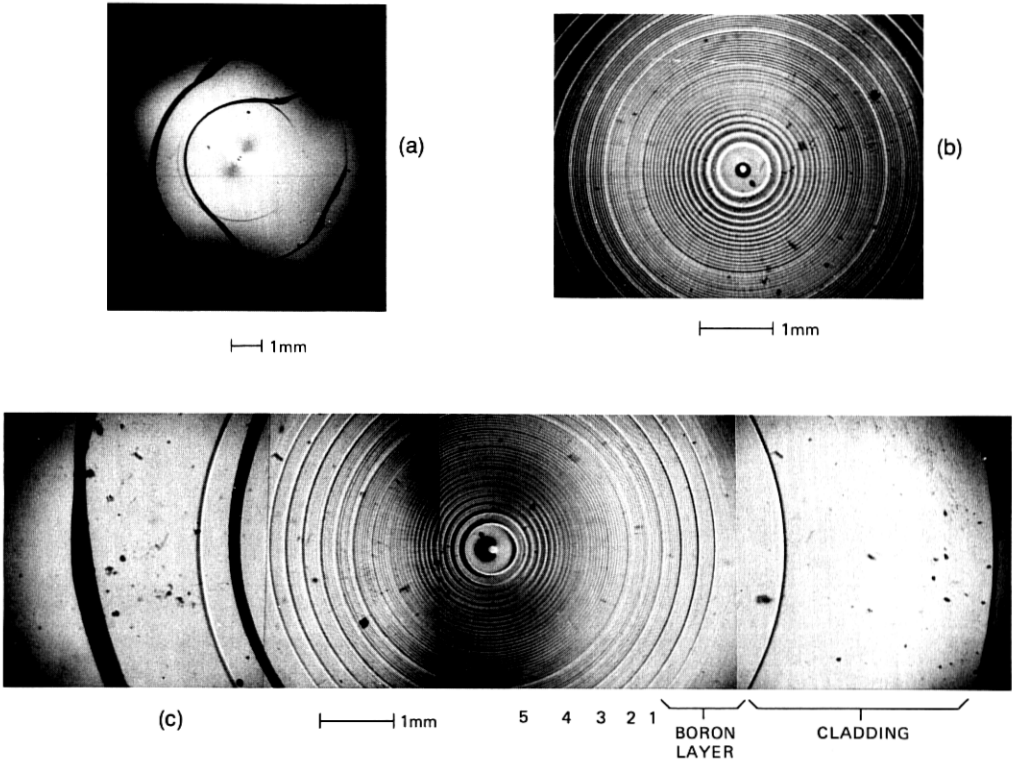


Fig. 1—Transmitted light photomicrographs of transverse section of preform. (a) Entire section. (b) Central core region. (c) Entire section at higher magnification.

core and the cladding. If this stress is not to be relieved by shattering upon cutting, extreme care must be used. In this case, the preform was cut with a diamond wire saw with controlled lubrication. Even so, the preform did crack, as seen by the dark curved line in the left-hand section of the sample.

Figures 1b and 1c obtained by optical microscopy show expanded views of the central region and of a portion of the entire cross section. The cladding, the borosilicate layer, and the first five germania-borosilicate steps are labeled. The regions between the steps are quite distinct, as are the individual layers within each step. Each of these layers, as noted, corresponds to a traversal of the oxyhydrogen burner along the tube. During step 3, for example, the $GeCl_4$ flow was maintained constant for nine traversals of the burner, producing the nine layers observed in Fig. 1c.

An expanded view of one-half the preform sample as observed by interference microscopy is shown in Fig. 2. The refractive-index difference between the cladding and a point in the core of the preform is given by the fringe displacement at that point times the wavelength of observation and divided by the thickness of the sample. One observes the straight parallel fringes in the fused silica cladding on the right indicating the uniform composition of this region, as expected. The drop in the level of the fringes indicates the termination of the cladding and the start of the borosilicate step that has a lower index of refraction than pure fused silica. Again, in this region, which extends for about 375 μm, the composition is relatively uniform and no evidence is seen of the 41 layers which comprise this step. This tends to indicate that some boron diffusion occurs, smoothing out the individual layers. It does not appear, however, that boron diffuses into the cladding, as evidenced by the relatively sharp transition occurring $\sim$10 μm between the cladding and this step.

At the termination of the pure borosilicate layer, germania deposition commences. The first three of these steps are labeled. Note, in particular, that step 3 exhibits nine sinusoid-like variations which, as discussed previously, correspond to nine torch traversals used in depositing this step. The reason for this index variation within each layer may be due either to a difference in composition of the particles reacting homogeneously (i.e., in the gas phase) and heterogeneously (i.e., on the surface of the tube),[7] or to temperature variation effects, depositing different concentrations as the torch passes. These compositional variations become quite sizable as the number of layers in each step decreases towards the center of the core. Note that in all steps the integrity of these layers is maintained and that relatively sharp boundaries exist between the steps. The transitions are most

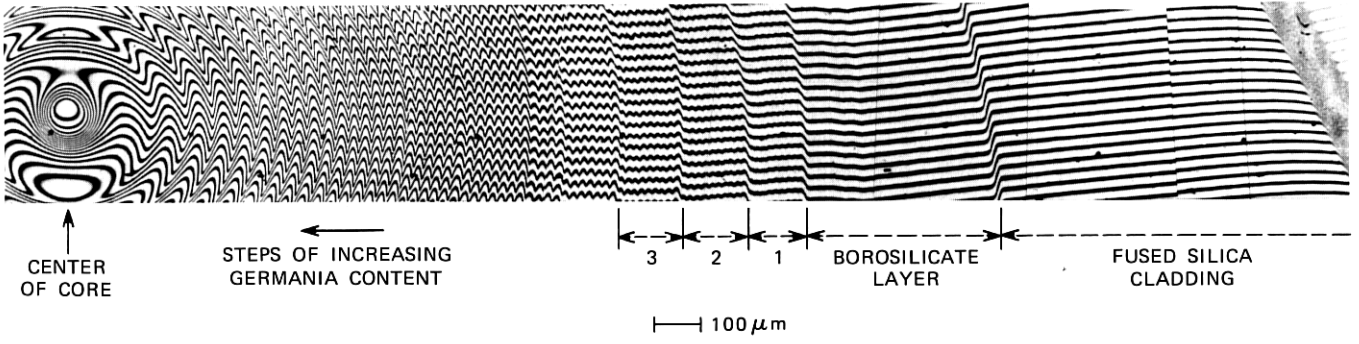


Fig. 2—Composite microinterferrogram of segment of one-half the preform sample.

clearly seen between the first few steps in which the change in germanium content is largest. These observations indicate that little, if any, germanium diffusion occurs between adjacent steps or within a given step itself.

The situation is quite different at the center of the core. Despite the fact that the germanium concentration was varied in a smoothly increasing manner in the last several steps, a large disturbance in the center is observed, with a corresponding dip in the refractive index. We believe this is due to the evaporation of germania at the inner surface of the deposit during the elevated temperatures experienced in the collapse process. The fact that the last two or three layers appear to broaden towards the center also indicates the existence of some germanium flow extending beyond the immediate central segment. It may be possible to compensate for this effect by a germanium overdoping in this region.

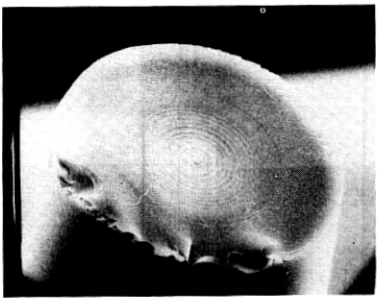
Quantitative index measurements indicate a maximum index difference between core and cladding of approximately $\Delta n_m = 0.016$ and an index difference between the cladding and the borosilicate layer of $\Delta n = 0.004$. Both values are in very good agreement with the corresponding measurements made from the microinterferrogram of the fiber, which is shown in Fig. 3a, and indicate a preservation, for the most part of the material composition through the pulling process.

Note in Fig. 3a the uniform cladding containing straight parallel fringes, the subsequent drop owing to the borosilicate layer, and the gradual grading of the index profile to a maximum near the axis. The grading was near-parabolic and has been related to a reduction in pulse dispersion for this fiber.[9] These regions are again shown in the transmitted-light photomicrograph of Fig. 3b. The core which appears as the bright central area is surrounded by the relatively dark borosilicate layer. Beyond that is the grayish cladding with an overall diameter of 96 μm.

The resolution of the interference microscope is not sufficient to resolve any layer or step structure in the fiber. To obtain greater resolution, we made use of scanning electron microscopy. Preparation of the fiber samples was described previously, and results of observations are shown in Figs. 4 and 5.

Figure 4 presents three micrographs taken at increasing magnifications centered on the axis, and Fig. 5 is a composite photograph of a section of one-half the fiber at somewhat greater magnification. The main points to be noted are the preservation of the step and layer structure in the fiber and the appearance of the elevated region near the axis. This feature is a region that did not etch as rapidly as the surrounding area, because of a lack of contained dopant and agrees

Fig. 3—(a) Microinterferrogram. (b) Photomicrograph of fiber.



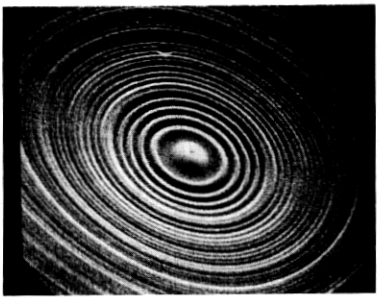


Fig. 4—Scanning-electron-beam-microscope photographs of fiber at increasing magnification.

with the preform observation of germanium departure during collapse. The appearance of the distinct step and layer structure displays the further lack of germanium diffusion even during the pulling process and indicates the maintenance of compositional and structural integrity from the deposition through the pulling process, with the exception of the central region.

We further investigated this latter region in another fiber prepared in the same manner by chemical vapor deposition. In this fiber, the

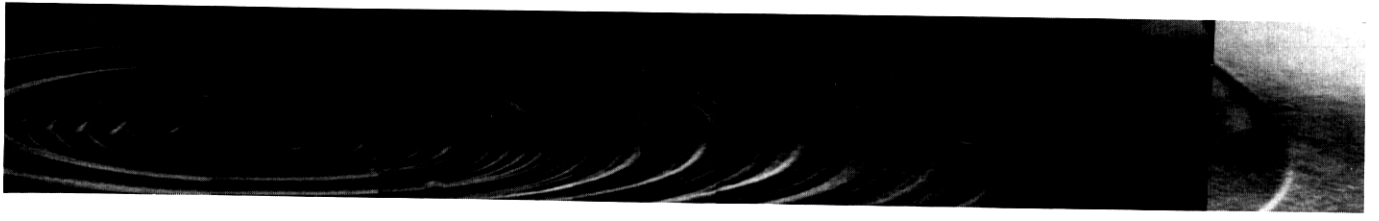


Fig. 5—Composite scanning-electron-beam-microscope photograph of segment of one-half fiber.

$GeCl_4$ flow was increased at each of ten steps to produce a linear variation in concentration. The number of layers in each step varied somewhat from the fiber considered above.[7] Scanning-electron-microscope photographs of etched fiber samples are shown at increasing magnification in Fig. 6.

The depression of the index on the axis because of the loss of dopant is quite pronounced in this fiber and appears as the micron-or-so in

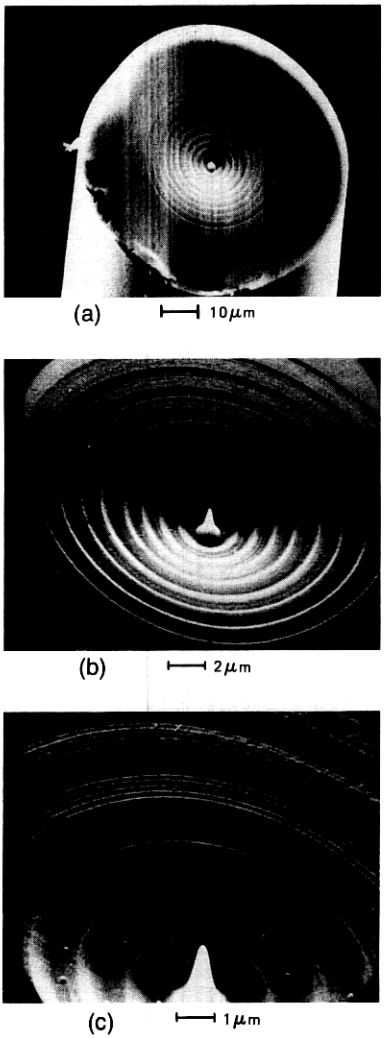


Fig. 6—Scanning-electron-beam-microscope photograph of fiber having linear refractive-index profile.

diameter, raised, tapered pip in the center. Note also the preservation of the distinct step and layer structure. The refractive index depression is also observed in this case in the microinterferrogram of the fiber shown in Fig. 7. It is seen as a dip in the fringe normally passing through the center of the fiber. Slight modulations of the refractive index can also be observed at each of the ten steps in this case owing to the relatively large change in germanium concentration between the steps.

It is important to note that, despite the structural features that exist on such a small scale, the losses of these fibers were less than 5 dB/km in the region of 1.0 μm. This is presumably due to the fact that these features are very uniform in the direction parallel to the axis of the fiber and hence do not contribute in a large way to scattering losses. Small-scale variations of such features if existing, however, could form a lower limit on losses achievable with fibers fabricated by this technique.

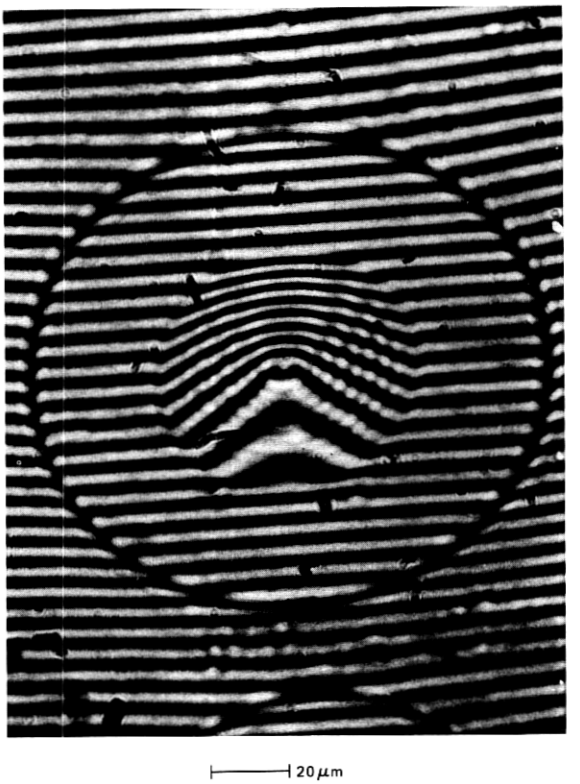


Fig. 7—Microinterferrogram of linear refractive-index profile fiber showing index depression in center.

In summary, structural features resulting from the preparation of both the preforms and graded-index optical fiber by the chemical vapor deposition process have been observed by optical, interference, and scanning electron microscopy. These features can be directly related to steps in the fiber fabrication. It was observed that structures present in the preform were preserved through the drawing and were present in the fiber. For instance, optical and interference observations indicated that germania concentrations varied within each deposited layer, and this variation was not substantially altered by subsequent processing steps except in the centermost layers. Here, flow of the deposit during collapse and vaporization of germania, probably as GeO, led to a depleted region of lower refractive index at the fiber center.

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