PROPERTIES OF OPTICAL FIBER PREFORMS PREPARED BY INNER COATING OF SUBSTRATE TUBES

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Substrate/deposit high-temperature interactions taking place at the standard MCVD method, MCVD method combined with the solution doping and sol-gel method are experimentally investigated on preform cores composed of $(P_2O_3)/\text{GeO}_2/\text{SiO}_2$ glasses and $Al_2O_3/P_2O_3/\text{SiO}_2$ glass matrices doped with Yb and Er ions. Effects of these interaction processes on the increase of the core radius, the content of dopants in the core and attenuation of fibers are shown. Conditions under which the interaction substantially affects the refractive-index profiles are found by comparing the theoretically estimated and experimentally determined characteristics. Attenuation of optical fibers used in fiber-optic sensors and soliton fiber lasers which have been fabricated on the basis of these results are shown.

INTRODUCTION

Rapid progress achieved in telecommunications is closely related to fabrication of high-quality silica optical fibers. This was possible only after the development of chemical vapour deposition (CVD) methods for the preparation of fiber preforms. However, demands for fibers used in fiber-optic lasers and amplifiers initiated the development of modifications of the CVD methods by novel techniques such as the solution-doping [1 - 3] and the sol-gel method [4 - 6]. The main goal of these approaches was the fabrication of glass matrices containing the highest possible concentrations of rare-earth ions (Er, Nd, Yb) without their clustering. By adapting alumino-phospho-silicate [1, 2, 6] or recently phospho-silicate [3] glass matrices, efficient erbium-doped fiber-optic amplifiers and lasers have been fabricated mainly by the solution-doping technique. The sol-gel method and other special techniques have been used only rarely [4, 5].

All the above techniques can be described by the same general mechanism consisting of four steps. The first step is the preparation of input mixtures of colloidal particles, sols, by chemical reactions (aerosol of oxides in CVD methods, a sol of oxidic chains in the sol-gel method). In the second step, the input sols are deposited on the inner wall of a glass tube (a silica tube) forming thus a gel layer. The composition of this layer can be further modified by soaking the layer with solutions of dopants such as in the solution-doping. The third step is the sintering of the gel layer to a glass layer at temperatures below $T_{\rm m}$ in the atmosphere of oxygen with the addition of drying means (Cl₂, CCl₄) or raw materials (POCl₃) [2 - 4]. The last step is the collapse of the tube with the coated glass layers to a rod, the preform, which increases the thicknesses of the coated layers by transforming them into a preform core via highly viscous flow of glass driven by surface tension.

Every technique for the fabrication of an optical fiber should make possible to produce a structure composed of an optical core surrounded by an optical cladding which exhibits controlled wavelengthdependent attenuation of the fiber materials. However, there are some processes inherently included in the fabrication of optical fibers that may induce departures from these basic characteristics. One of them is the diffusion in glass, or more generally, the interaction of the deposited layers with the substrate. The effect of the diffusion processes on the refractive-index profiles of optical fibers prepared by the MCVD method has been investigated in several papers [7, 8]. The effect of hightemperature treatment of the deposited rare-earth-doped glass layers prepared by the sol-gel method on properties of the layers has also been observed [9].

This work is aimed at experimental comparison of substrate/deposit high-temperature interactions taking place at the standard MCVD method, MCVD method combined with the solution doping and sol-gel

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method. These interactions are studied on preform cores composed of $(P_2O_5)/GeO_2/SiO_2$ glasses and $Al_2O_3/P_2O_5/SiO_2$ glass matrices doped with Yb and Er ions. Effects of the interaction processes on the core radius, refractive index, composition and attenuation of preforms and fibers are shown. Conditions under which the interaction substantially affects the refractive-index profile are found by comparing the theoretically estimated and experimentally determined characteristics.

EXPERIMENTAL PART

MCVD process

In the experiments glass layers with a $(P_2O_5)/GeO_2/SiO_2$ composition were prepared. Aerosols of oxide particles with diameters below 1 µm were prepared by high-temperature oxidation of raw materials, SiCl₄, GeCl₄, POCl₃, in the hot zone of a hydrogen-oxygen burner. The particles were thermophoretically deposited on the inner wall of a rotating silica substrate tube and sintered by the burner traversing around the tube (an inner diameter of 15 mm). The ranges of the flow-rates of raw materials, deposition (sintering) temperatures and number of the deposited glass layers used in the experiments are summarized in table 1.

Table 1. Processing conditions used in the MCVD method.

Flow rates of raw materials (mmol min ⁻¹)	SiCl₄ GeCl₄	0.0 - 5.9 0.3 - 2.7
	POCl ₃	0.07 - 0.27
Number of deposited layers		1 - 25
Deposition/sintering temperatures		1400-1700
(°C)		

MCVD and solution-doping technique

Glass layers with a Al₂O₃/P₂O₅/SiO₂ composition doped with Er and Yb ions were prepared in the experiments. At first, a porous layer composed of SiO₂ and P_2O_5 was deposited on the inner wall of the substrate silica tube by the reverse deposition MCVD technique at 1400 °C [2, 3, 10]. The layer was presintered in order to fix the deposit. The deposited layer was soaked with an aqueous solution of AlCl₃, ErCl₃ and YbCl₃ by filling in the solution into the tube at a determined velocity followed by the deposit/solution interaction taking place for one hour. The solution was drained from the tube at the same velocity as during the filling in. The wet porous layer was dried under a flow of nitrogen at the laboratory temperature. The heattreatment of the porous layer was carried out in a glassworking lathe under the rotation of the tube and heating it by a burner traversing around it. Typical conditions used at the frit deposition are shown in table 2 and

Table 2. Processing conditions used in the solution doping.

Frit			
Flow rates of raw materials (mmol min ⁻¹)	SiCl ₄ POCl ₃	0.45 0.09 - 0.53	
Soaking solutions			
Solvent	Water (F	Water (HCl)	
Solvent	mater (1	/	
Concentration of chlorides	AlCl ₃	0.4 - 1.7	
Concentration of chlorides (mol l ⁻¹)	AlCl ₃ YbCl ₃	0.4 - 1.7 0.05 - 0.10	
Concentration of chlorides (mol 1 ⁻¹)	AlCl ₃ YbCl ₃ ErCl ₃	0.4 - 1.7 0.05 - 0.10 0.005 -0.01	

Table 3. High-temperature treatment used in the solution doping and the sol-gel methods.

	Temperature (°C)	Atmosphere
Oxidation Sintering	800 - 1000 1000 - 1600	$O_2 O_2 + POCl_3$

The sol-gel technique

Glass layers composed of $Al_2O_3/P_2O_5/SiO_2$ glass matrices doped with Er and Yb ions were prepared in the experiments. The input sols were mixed from tetraethoxysilane (TEOS), POCl₃, AlCl₃, ErCl₃.6H₂O and YbCl₃ .6H₂O. Isopropanol was used as a solvent and hydrochloric acid as a catalyst. A procedure published elsewhere was used at the preparation of the input sols [9]. The viscosity of these sols was characterized with a capillary viscosimeter and ranged from 3 to 6 mPa s. The surface tension was determined by the hanging-drop technique. Basic characteristics of the input sols are shown in table 4.

Table 4. Basic characteristics of the sols used in the sol-gel method.

C - locant	T	1	
Solvent	Isopropa	Isopropanol	
Catalyst	HCl $(pH < 1)$		
Concentration	TEOS	0 - 1.5	
of components	AlCl ₃	0.3 - 0.8	
(mol 1 ⁻¹)	POCl ₃	0.04 - 0.30	
	YbCl ₃	0.01-0.03	
	ErCl ₃	0.001-0.003	
water/(Si+Al+P+Yb) (mol mol ⁻¹)		1 - 1.5	
Draining velocity (cm min ⁻¹)		10	

The input sols were applied on the inner wall of a silica tube by rising and lowering the sol level at a velocity of 10 cm min⁻¹. As a typical sol was prepared with the water/alkoxide ratio (R_w) approximately equal to unity, the applied gel layers were brought into contact with a stream of oxygen saturated with water for 10 min.

A low-temperature treatment of the gel layers included their drying at 100 °C for 20 min and then heating in a furnace up to 700 °C at a rate of 5 °C min⁻¹ for burning-out organic species. Then the deposited layers were exposed to the high-temperature treatment shown in table 3.

Collapse of the tube and drawing of fibers

In all the techniques described above the tube with deposited layers was collapsed at 2000 °C to a rod, the preform. Fibers with a diameter of 0.125 mm were drawn from the preforms at temperatures of about 1950 °C. During the drawing they were coated by UV-curable acrylate (DeSotto, USA).

Characterisation of preforms and fibers

The refractive-index profiles of the preforms were measured by the commercial refractive-index profiler P101 (PK Technology, GB). With this device the refractive index was measured with an accuracy better than 0.0005.

The spectral attenuation of the fibers was determined by the cut-back method by a laboratory analyser SPM (Safibra Ltd., Prague). By using the lock-in amplifier technique an accuracy better than 0.005 dB and a dynamic range of 100 dB can be achieved with this device.

The concentration profiles of Al_2O_3 , P_2O_5 , GeO_2 , SiO_2 , Yb_2O_3 in the preforms were measured on polished samples of preforms by X-ray analysis (EDAX) with an accuracy better than 5 rel.%. The thickness of the deposited layers was estimated either from the refractive-index profiles of preforms or from microscopic measurements on samples of the tubes or preforms.

THEORETICAL INTERPRETATION OF EXPERIMENTAL DATA

In order to show some features in the experimental results, certain characteristics of the deposited glass layers such as the composition, thickness *etc.* were calculated by using theoretical or semiempiric formulas. The composition of glass layers containing GeO₂ and/or P_2O_5 and prepared by the MCVD method was determined either from chemical analysis or calculated from the chemical equilibrium. Approaches used in the calculations have been published elsewhere [11].

The thickness t of a glass layer deposited by the MCVD process on the inner wall of a tube with a diameter D in a single application cycle can be

estimated from

$$t = N'(gl) V(gl) \frac{E_{\rm T}}{\pi D U_{\rm T}}$$
(1)

where N'(gl) is the molar flow of oxides in the tube, V(gl) is the molar volume of the deposited glass, U_{T} is the burner velocity (15 - 18 cm min⁻¹) and $E_{\text{T}} \approx 0.5$ - 0.6 is the thermophoretic efficiency [11]. The parameters N'(gl) and V(gl) were calculated from flow rates of raw materials into the tube and the composition of the glass.

A linear relationship between the refractive index of the glass n(gl) and its composition expressed by equation (2) was used [11].

$$n(gl) = n(SiO_2) + F(GeO_2) x(GeO_2) + F(P_2O_5) x(P_2O_5)$$
(2)

In equation (2), $n(SiO_2)$ is the refractive index of glassy silica, $x(P_2O_5)$ and $x(GeO_2)$ are the molar fractions of P_2O_5 and GeO_2 , respectively, and F(Comp) is a factor that expresses the contribution of the particular component (Comp) to the refractive index of the glass.

On the basis of the mass-conservation law, the radius R of the core formed by viscous transformation from a layer with the thickness t deposited on a wall with the diameter D can be expressed as

$$R = \sqrt{Dt} \tag{3}$$

In the case of the solution-doping method, the thickness t of a fully sintered layer can be expressed as

$$t = \frac{N'(\text{gl}) V(\text{gl})E_{\text{T}}}{\pi D U_{\text{T}}} \left(1 + \frac{P}{1 - P} \sum_{\text{comp}} V(\text{comp}) C(\text{comp}) \right) + t_{1} \sum_{\text{comp}} V(\text{comp}) C(\text{comp})$$
(4)

In equation (4), P is the porosity of the porous layer (a value of 0.9 was used), t_1 is the thickness of the liquid layer remaining on the frit boundary after draining the soaking solution, C(comp) is the molar concentration of a component in the soaking solution (expressed as the concentration of oxides) and V(comp) is the molar volume of a corresponding oxide component.

The thickness t_i of a liquid layer applied on a vertical substrate by flowing of the liquid around the substrate surface with the draining velocity U can be estimated as [12]

$$t_{\rm l} = 0.944 \ \sigma^{1/6} \ \eta^{2/3} \ U^{2/3} \frac{1}{\sqrt{\rho g}} \tag{5}$$

In equation (5), g is the gravitational constant and σ , η and ρ are the surface tension, dynamic viscosity and density of the liquid, respectively.

The thickness of a fully densified layer t_d can be derived on the basis of the mass-conservation law as

$$t_d = t_l \sum_{\text{comp}} C(\text{comp}) \ V(\text{comp})$$
(6)

RESULTS AND DISCUSSION

Preforms prepared by all the three techniques used in the experiments exhibit properties affected by the substrate/deposit interactions.

MCVD method

The effect of the interaction on the refractive index of preforms can be seen from the data in figure 1, which relate the factor $F(\text{GeO}_2)$ (see equation (2)) to the theoretical thickness of the deposited layer that was estimated from equation (1) for *D* equal to 15 mm. Despite experimental errors, two distinct areas can be distinguished in the data set. In the first area for the thickness of the layer below the limit of 8 µm, the factor increases with the layer thickness, while above this limit, the factor $F(\text{GeO}_2)$ is nearly constant.

Below the limit of 8 µm, the actual thickness of the deposited layer, which was calculated from equation (3), experimental radius of the core R and for D equal to 15 mm, exceeds the thickness calculated from the mass conservation law, i.e. from equation (1) for $E_{\rm T} = 1$ (see the dashed line in figure 2). The effect of the interaction can also be seen on the refractive-index profile of a preform prepared by depositing pure GeO₂ into a silica tube (figure 3). The actual profile exhibits by about twofold increase of the core diameter and by about 13-fold decrease of the refractive index in comparison with the same quantities of glassy GeO₂ (see figure 3, solid line). A content of about 11 mol.% was determined by chemical analysis in the prepared cores. All these experimental data show that the substrate/deposit interaction in the MCVD process takes place in part of the deposited layer and the core with thickness of about 10 µm.



Figure 1. The dependence of the factor $F(GeO_2)$ on the theoretical thickness of the layer deposited by the MCVD method into a tube with a diameter of 15 mm.

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Figure 2. The relationship between the thickness of the deposited layer calculated from the measured core radius and the theoretical thickness for a tube with a diameter of 15 mm.



Figure 3. Comparison of the theoretical refractive-index profile of glassy GeO_2 .

 $(n(gl)-n(SiO_2) = 0.0193)$ and the actual refractive-index profile of the core prepared by the deposition of pure GeO₂.

Solution doping

A more complex picture can be observed in the case of the MCVD technique combined with the solution doping. The diameter of the prepared $Al_2O_3/P_2O_5/SiO_2$ preform cores ranged from 0.9 to 1.5 mm, which on the basis of equation (3) corresponds to the thickness of the deposited layer in a range from 13 to 37 µm (*D* equal to 15 mm). This thickness is much larger than that calculated from equation (4), which ranged from 0.5 to 1.5 µm for the parameters summarized in table 2. The difference between the theoretical and experimental layer thicknesses can hardly be explained by the remaining porosity of the sintered layer because in this case a porosity value of about 0.9 would have to be assumed. This is a rather high value for a densified transparent glass layer.

The content of dopants entrapped in a porous frit from a soaking solution depends on the frit porosity. By changing the processing parameters during the frit deposition and soaking, glass cores with the Al₂O₃ content ranging from 1.5 to 14 mol.% were prepared. These concentration values correlate well with the values of the refractive index shown in figure 4. For a constant flow rate of SiCl₄ in the tube, the refractive index of the glass depends on the pre-sintering temperature, velocity of draining the soaking solution from the tube, concentration of the soaking solution and flow rate of POCl₃ in the tube. The higher the flow rate of POCl₃ (a higher value of n'(P)/n'(Si)), the lower presintering temperatures are necessary for attaining the same value of the refractive index. However, there is a limit of the pre-sintering temperature below which the



Figure 4. The effects of the processing parameters on the refractive index of cores with $Al_2O_3/P_2O_5/SiO_2$ glass matrices prepared by the solution doping.

■ - U = 5 cm min⁻¹, \blacktriangle - U = 20 cm min⁻¹, \blacktriangledown - c(Al) = 1.5 mol l⁻¹, \blacklozenge - c(Al) = 1.7 mol l⁻¹, \blacklozenge - c(Al) = 0.9 mol l⁻¹



Figure 5. The refractive-index profiles of the preform cores prepared by the solution doping showing the effect of changes of the velocity at which the soaking solution was drained from the tube.

frit peels off the substrate by the effect of the soaking solution. On the other hand, above a certain temperature limit, the concentration of dopants strongly decreases, which can be related to a strong decrease of the porosity (see figure 4, n'(P)/n'(Si) = 0.2).

The effect of the draining velocity on the refractive index profiles can be seen from figure 5 as a central peak of the refractive index (figure 5, U = 20 cm min⁻¹). This peak can be explained by coating a thin solution layer on the frit surface during draining. This layer contains only chlorides, which are converted to hardly sinterable oxides during the frit sintering. Slow densification and poor glass-forming properties of these oxides resulted in some cases in the formation of a thin white "thread" in the center of the preforms.

Sol-gel method

Data in figure 6 show that the content of Al_2O_3 in the preform core is lower than that calculated from the composition of the input sols and increases with the number of deposited layers. These results support the assumption of the substrate/deposit interaction. Direct evidence of this interaction was obtained in experiments in which glass cores were prepared from a sol containing only AlCl₃, POCl₃, YbCl₃ and ErCl₃, and no TEOS. The concentrations of Al₂O₃ measured in these cores are shown in figure 6 (data "Al/P/Yb/Er sol"). A content of about 77 mol.% of SiO₂ was determined by chemical analysis in the cores sintered under the conditions shown in table 3. However, this content was further increased to about 83 % by collapsing the tube with a deposited layer partially sintered at 1750 °C (see figure 6, "rapid sintering"). The increase of the refractive index of silica corresponding to these concentration values ranged from 0.016 to 0.02.



Figure 6. Comparison between the content of Al_2O_3 determined by chemical analysis in cores with $Al_2O_3/P_2O_5/SiO_2$ matrices and the content of Al_2O_3 calculated from the composition of the input sol. **•** - 3 layers, • - 6 layers, O - 3 + 3 layers, • - 9 layers, • - Al/P/Yb, Er sol

The effect of the substrate/deposit interaction on the thickness of the deposited layer is shown in figure 7. Similarly to the solution doping, the actual thickness, which was calculated from equation (3), experimental core radius and for the value of D equal to 15 mm, is much larger than the theoretical thickness estimated from equations (5) and (6). For theoretical thicknesses higher than the limit of about 1 mm, the actual thickness increases with the theoretical one. Of course, this conclusion is correct only for the same composition of the input sol, as can be seen from the deviation of the point corresponding to the core prepared with sols containing no TEOS. Similar trends have been observed for the refractive index. These experimental results allow us to explain why the thickness of the sol-gel coated layer estimated from equation (3) and experimental core radius highly exceed the critical thickness of the densified layer prepared by a single application (of about 0.5 μ m) [5, 6].

The effect of sintering conditions on the refractiveindex profiles is shown in figure 8. Here the notation "6 layers" corresponds to the preparation of multiple layers described by a processing sequence

6 times (application of a gel layer + low temperature*treatment*) + *high-temperature treatment*,

and the notation "3+3 layers" represents a processing sequence

3 times (application of a gel layer + low temperaturetreatment) + high-temperature treatment +

+ 3 times (application of a gel layer + low temperature*treatment*) + *high-temperature treatment*.

In the "3+3 layers" process, the second deposited multiple layer interacts with the first multiple layer with the content of dopants higher than that of the substrate.



Figure 7. The dependence of the thickness of the layers of Al₂O₃/P₂O₅/SiO₂ glass matrices calculated from the actual core radius and theoretical thickness for the layer deposited by the sol-gel process into a tube with an inner diameter of 15 mm. ■ - $c(Si) = 1.4 \mod 1^{-1}$, $c(Al) \in (0.3; 0.7 \mod 1^{-1})$, \checkmark - c(Si) = 0, $c(Al) = 0.8 \text{ mol } l^{-1}$

Al₂O₃/P₂O₅/SiO₂core matrices 1.0 0.8



This may explain the central peak of the refractive index in the "3+3 layers" process. However, the refractive

index is also increased by the effect of P₂O₅ contained

in the sintering atmosphere. Comparing the refractive-

index profiles in figure 8 and the concentration profiles

in figure 9 one can say that they exhibit some similarity.

method and their heat treatment allow us to overcome

trouble with cracking and peeling-off the layer from the

substrate which can occur at the application of thick

layers [13]. In any case, the effect of the substrate/deposit interaction on the refractive index

does take place in such preparations.

The application of several thin layers by the sol-gel

Figure 8. Comparison of the refractive-index profiles of the preform cores prepared by the sol-gel method showing the effect of one-fold (6 layers) and two-fold (3+3 layers) use of sintering during the core fabrication.

 $c(Si) = 1.32 \text{ mol } l^{-1}$, $c(Al) = 0.34 \text{ mol } l^{-1}$, $c(P) = 0.04 \text{ mol } l^{-1}$, -3 + 3 layers, ---- - 6 layers



Figure 9. The concentration profiles of Al₂O₃, P₂O⁵ and Yb₂O₃ in the core of the preform prepared by the "3+3 layers" sol-gel process.

 $c(Si) = 1.32 \text{ mol } l^{-1}, c(Al) = 0.34 \text{ mol } l^{-1}, c(P) = 0.04 \text{ mol } l^{-1},$ $- - Al_2O_3, - - - - P_2O_5, - - - Yb_2O_3$

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Explanation and application of the observed effects

The substrate/deposit interaction may be explained by a combination of several mechanisms including the dissolution of the substrate in a viscous fluid (deposition of pure GeO₂), diffusion effects, viscous sintering and sintering in the presence of melt. Highly viscous oxides, prepared either by oxidation of AlCl₃, YbCl₃ and $ErCl_3$ in the solution doping and by solvolysis, hydrolysis and polycondensation in the solgel method, are in contact with low-viscous parts (particles or chains) of SiO_2 and P_2O_5 . For the content of highly viscous oxides exceeding 3 mol.%, densification of the porous layer and its transparency was achieved at temperatures above 1100 °C. At temperatures of 1300 - 1400 °C, the substrate starts to play a more important role in the densification. The substrate/deposit interaction process is enhanced by the effect of surface tension, which drives the viscous flow of the tube wall towards decreasing the surface area. When isolated and highly viscous islands appeared in the deposit (peeled-off parts of the frit, gel layer), an effect of flowing low-viscous parts of the deposited layer and substrate around these islands was observed and thus the islands moved towards the outer surface of the tube.

Optical fibers doped with rare-earth ions for fiber lasers [2] and optical fibers with special inverted graded-index profiles for fiber-optic chemical sensors [14] were fabricated taking into account the effects of the interaction processes described above. Typical spectral attenuation curves measured on fabricated fibers are shown in figure 10. Higher attenuation measured on fibers prepared by the solution-doping and



Figure 10. Spectral attenuation of optical fibers doped in the core with P_2O_5 and GeO_2 and prepared by the MCVD technique, and fibers with $Al_2O_3/P_2O_5/SiO_2$ cores doped with Yb and Er ions and prepared by the sol-gel and solution-doping techniques.

••••• - sol-gel, —— - solution doping, – – - MCVD (P/Ge/SiO cores)

sol-gel methods can be explained by scattering losses caused by concentration inhomogeneities in the core and variations of the core diameter. These effects can be related to the presence of hardly sinterable dopants.

Ultrashort pulses, optical solitons, of 1.7-ps widths, have been generated in a mode-locked fiber laser based on optical fibers with cores of $Al_2O_3/P_2O_5/SiO_2$ matrices doped with Yb and Er ions [2]. These fibers were pumped at 1060 nm by using the Yb absorption band and emitted light around 1550 nm. Further improvement of the performance of rare-earth doped fibers can be based on decreasing the content of Al_2O_3 . This is the main reason why P_2O_5/SiO_2 matrices are investigated instead of $Al_2O_3/P_2O_5/SiO_2$ matrices for entrapping rare-earth ions in fiber-optic lasers [3].

CONCLUSIONS

The substrate/deposit interaction in the MCVD method, MCVD method combined with the solutiondoping and sol-gel method has been experimentally proved. This interaction increases the diameter of the core of a preform obtained by the collapse of the substrate tube coated on the inner wall by a thin layer. Moreover, the interaction decreases the dopant concentration in the core and consequently the refractive index. In the case of hardly sinterable dopants (rare-earth oxides, Al₂O₃) it may increase the attenuation of the prepared fibers. By taking these effects into account during fabrication, special optical fibers for fiber lasers and amplifiers can be prepared. Future work in this field will be directed to the generalisation of these effects for the fabrication of preforms doped in the core with Bi₂O₃.

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References

- 1. Towsend J.E., Poole S.B., Payne D.N.: Electr. Lett. 23, 329 (1987).
- Kasik I., Matejec V., Kanka J., Honzatko P.: Pure Appl. Opt. 7, 457 (1998).
- Vienne G., Caplen J.E., Dong L., Minelly J.D., Nilsson J., Payne D.N.: J. Lightwave Technol. 16, 1990 (1998).
- 4. Wu F., Machewirth D., Snitzer E., Siegel G.H. Jr.: J. Mater. Res. *9*, 2703 (1994).
- Wu F., Puc G., Poy P., Snitzer E., Siegel G.H. Jr.: Mat. Res. Bull. 28, 637 (1993).
- Matejec V., Hayer M., Pospisilova M., Kasik I.: J. Sol-Gel Sci. Technol. 8, 889 (1997).
- Kirchhoff J., Kleinert P., Funke A., Müller H.R.: Cryst. Res. Technol. 22, K105 (1987).
- 8. DiGiovanni D.J., Morse T.F., Cipolla J.W.: J. Lightwave Technol. 71, 914 (1988).

- 9. Matejec V., Kasik I., Berkova D., Hayer M., Kanka J.: J. Sol-Gel Sci. Technol. *13*, 617 (1998).
- 10. Kasik I., Matejec V., J. Aerosol Sci. 26, 399 (1995).
- 11. Spurny K.R.(Editor): Aerosol Chemical Processes in the Environment, p. 271-294, Lewis Publishers, New York 2000.
- 12. Kubeckova M, Sedlar M., Matejec V.: J. Non-Cryst. Sol.147&148, 404 (1992).
- 13. Scherer G.W.: J. Sol-Gel Sci. Technol. 8, 353 (1997).
- Matejec V., Chomat M., Kasik I., Ctyroky J., Berkova D., Hayer M.: Sensors&Actuators *B51*, 347 (1998).

VLASTNOSTI PREFOREM OPTICKÝCH VLÁKEN PŘIPRAVENÝCH NANESENÍM TENKÝCH VRSTEV DO SUBSTRATOVÝCH TRUBIC

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V článku jsou popsány výsledky experimentálního studia interakce mezi tenkou skelnou vrstvou nanesenou na vnitřní stěnu substrátové křemenné trubice při procesech přípravy křemenných optických vláken MCVD metodou, využitím dopování pórézních vrstev z roztoku a sol-gel metodou. Pro všechny tyto metody je charakteristické nanášení pórézních vrstev z koloidních částic získaných chemickými reakcemi a zpracování těchto vrstev při teplotách až 2000 °C, kdy je trubice s nanesenými skelnými vrstvami stažena na tyčinku, preformu. Interakce byla studována na jádrech preforem tvořených (P₂O₃)/GeO₂/SiO₂ skly připravenými MCVD metodou, a jádrech s Al₂O₃/P₂O₅/SiO₂ skelnými matricemi dopovanými Yb³⁺ a Er³⁺ a připravených dopováním z roztoku a sol-gel metodou.

Srovnáním teoreticky vypočtených charakteristik s experimentálními bylo zjištěno, že u všech použitých metod existuje interakce mezi nanesenou vrstvou a substrátovou trubicí. Důsledkem této interakce je zvětšení průměru jádra preforem, snížení koncentrací dopantů a indexu lomu jádra a zvýšení útlumu vláken předevších u vláken dopovaných Al₂O₃ a ionty vzácných zemin. V závěru článku je ukázáno, že zahrnutí výše popsaných efektů do technologie přípravy speciálních optických vláken pro vláknové lasery a vláknověoptické senzory umožnilo připravit vlákna s jejichž pomocí byly v experimentálním vláknovém laseru generovány optické solitony s trváním na úrovni ps a byla připravena vlákna s invertovanými gradientními indexy lomu pro vývoj chemických senzorů.