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Title of research project : NEW FLUORIDE GLASSES FOR THE PREPARATION OF INFRARED OPTICAL FIBERS

Principal investigator : Professor J. LUCAS, University of Rennes

Contractor : CENTRE REGIONAL ETUDES BRETAGNE SCIENCES, CREBS
PRESIDENCE DE L'UNIVERSITE
Rue du Thabor - 35000 RENNES (France)

Contract number : DAJA 45-86-C-0050

Fourth periodic report : start sept. 1987 ; end : march 1988

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) New zirconium-free fluoride glasses are investigated for I.R. optical fibers operating up to 5-5.5 μm. A five-component glass having the composition in molar per- cent fluoride Ba ₃₀ In ₃₀ Zn ₂₀ Y ₁₀ Th ₁₀ has been optimized for rods and preforms prepara- tion. F/Cl substitution as well as Na addition allow small variations in the refractive index. Two attempts on rods fibering were positive and demonstrated the possibility of obtaining BIZYT optical fibers without significant crystallization. Purification of indium and Yb based materials has been undertaken. JES		



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FOURTH PERIODIC REPORT

In the three previous periodic reports, we described a new family of fluoride glasses not containing zirconium either aluminium and having consequently a multiphonon edge shifted towards $8 \mu\text{m}$ instead of $6.5\text{-}7 \mu\text{m}$. The expected ultratransparency of such materials was estimated to be around 10^{-3} dB/km at $3 \mu\text{m}$ with losses calculated to be hundred times lower than for the fluorozirconate glasses in the $5 \mu\text{m}$ region.

This report will discuss the following points :

1. Viscosity measurement after T_g
2. Fibering conditions of the BIZYT glass : rod and preform
3. Some preliminary characteristics of the first fibers
4. Purification problems associated with In and Yb fluorides.

1. Viscosity measurements

These results have been obtained in collaboration with Professor MOYNIHAN's group at RPI, N.Y. The method of beam bending has been used on a sample of BIZYT glass prepared in dry and controlled conditions.

The following viscosity η in poises has been measured for different temperatures : $\eta = 10^{13}$ at 332°C , $\eta = 10^{11}$ at 354°C and $\eta = 10^9$ at 377°C . By extrapolation, $\eta = 10^6$ poises is expected at 415°C . Note that the crystallization temperature is 447°C and that the normal viscosity conditions for traditional fluoride glass fibering correspond to $10^5, 10^6$ poises.

2. Fiber preparation from BIZYT glass

In the previous reports, we mentioned that the BIZYT glass was the best crystallization resistant Zr-free fluoride glass. The three main obstacles to face with this glass are :

- a) avoiding small cristallites formation during preform casting and then fiber drawing
- b) combining two glasses with refractive index difference of about 1 % and having the same thermal behaviour
- c) combining two glasses having compatible expansion coefficients in order to get core-clad structure without stress and brake during cooling.

the fibering experiments have been made in collaboration with C.G.E. Marcoussis Laboratories and C.N.E.T. Lannion. The rods and preforms were prepared in Rennes and were respectively one rod of BIZYT, one preform with core BIZYT and clad BIZYI + 5 % NaF, one preform with a core BIZYT in which 4 % BaF₂ was substituted by 4 % BaCl₂ and a clad of BIZYT.

The rod or preform was 10 cm long with a diameter $\Phi = 0.65$ cm. The samples were all surface polished and prepared according to the conditions presented in the third report. The rods and samples were jacketted with PTFE. When rods or preforms are used without protection, crystallization is often observed. The drawing conditions were the following :

- a) the preform is maintained in a He atmosphere with a maximum of 6 ppm H₂O
- b) the drawing furnace giving a sharp gradient temperature zone is preheated at 384° C
- c) the preform is introduced in the heating zone and, after 5-8 min, the extremity of the rod begins to melt and form a drop which falls down in generating the fiber
- d) for this temperature, the drawing rate is about 1 m/minute.
- e) with the rods only , 40 m of fibers at C.G.E. and 160 m at C.N.E.T. have been drawn at the first experiments.

The figure represents the section of fibers with the PTFE coating ; the diameter is 140 μ m.

The Na doped preform has been also successfully fibered while the Cl doped preform showed some crystallization at the interface (note that the preform was already slightly crystallized).

3. Preliminary characterizations of the BIZYT fibers

Some preliminary tests have been undertaken in order to have a rough estimation on those prototype first fibers. The total transmission spectrum shows an almost continuous loss of few dB/m (6-8) from 1.5 to 5 μm indicating that the scattering loss due to crystallites and bubbles is dominant. The tensile strength has been measured on the same fiber and estimated to be about 60 MPa.

It is concluded that this first fiber has a poor optical quality as expected but that the multiphonon losses were effectively shifted towards the 5 μm region.

4. Purification of Yb and In compounds

The most dangerous absorbing impurities associated with Yb^{3+} is Nd^{3+} with an absorption peak at 2.55 μm . Extraction technics using organic esters have been proved to be efficient (Nd^{3+} is non detectable by ICP-plasma).

Indium based materials are purified as InCl_3 by sublimation. InCl_3 has a high vapor pressure compared to Fe, Ni, Co, Cu chlorides.

This report can be concluded in a positive way because we have demonstrated during the last five months the possibility of fibering the BIZYT glass. The first experiments were very optimistic and we are conscient that the first parameter we have to optimize is the optical quality of the preform.

Our program is now to adjust two glasses with Δn of about 1 % compatible in expansion and resistance to crystallization. Cl/F, Ba/Na, Th/rare-earth substitutions will be tried to reach this goal. We hope to decrease in a near future the scattering loss.

Figure - Section of a BIZYT fluoride fiber, $\varnothing = 140 \mu\text{m}$, with a PTFE jacket

