

High NA fibers — a comparison of optical, thermal and mechanical properties of ultra low index coated fibers and air clad MOFs

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Abstract

Fibers with numerical aperture of about 0.45 and higher are interesting for power transmission, remote sensing or for laser fibers with high pump efficiency. To achieve this goal we followed two ways of fiber designing. On the one hand we prepared step index fibers with different ultra low refractive index polymer coating (fluorinated acrylate, silicon based coatings, silica based hybrid glass coatings). We investigated and reviewed thermal stability (TG) of the coatings and the optical (spectral attenuation, NA, macrobending loss), and mechanical (tensile strength) properties of different polymer coated fibers. On the other hand microstructured optical fibers (MOF) offer new possibilities to expand the polymer material limited NA span. We fabricated different solid core air clad MOF with maximum NA = 0.55. Despite of the obvious advantages of these holey fibers (no material absorption in air holes, high refractive index contrast 1.45:1) some specific properties have to be considered critically. The essential small bridge widths cause deficits in mechanical stability.

Keywords: High NA fiber, PCF, fiber, loss, tensile strength, coating

1. Introduction

High NA fibers are interesting for power as well as for signal transmission or remote sensing. Because of the limits in silica dopance (and the resulting narrowness of refractive index changing) there are pursued two concepts for passive light guides: polymer clad silica fibers (PCS) with pure silica core and low index polymer coating as cladding [1, 2] and so called air clad fibers with a holey (“air”) ring around a silica core [3]. Whereas in the first case the transmission properties of the fiber are strongly influenced by material absorption effects of the coating polymer material by evanescent field absorption air clad fibers allow in principle to depress the transmission loss level to the material based limit of the silica core. On the other hand, there is a growing demand for low index materials with a high thermal stability. The goals are fiber lasers with a high out power in the kW range [4, 5, 6].

2. Preparation of fibers

All fibers were drawn in a laboratory scale drawing device. The typically used preforms as core material for solid fibers was Heraeus Suprasil F 300, characterized by a low OH concentration (< 1 wt.ppm). The drawn fibers had a diameter of $125 \pm 0.5 \mu\text{m}$.

The drawing speed was about $10 \text{ m}\cdot\text{min}^{-1}$. The fibers were coated with different low index coatings (table 1) using a gravity coating system. We used splitted dies with nearly cylindrical nozzles. For different coating thickness the die cross section diameters were changed between $160 \mu\text{m}$ and $345 \mu\text{m}$ (figures 1). The applied coating thickness s was approximated according to [7] with :

$$s = \sqrt{\frac{R^2 - a^2}{2 \ln\left(\frac{R}{a}\right)}} - a \quad (1)$$

s : coating thickness
 $2R$: coating die diameter
 $2a$: fiber diameter

Table 1 Investigated coatings

coating	sort	company	curing
RT601	silicone	Wacker Silicones	thermal
HG-Li-3	hybrid material *	Hybrid Glass Technologies	thermal
HG-Li-V-T	hybrid material *	Hybrid Glass Technologies	thermal
Teflon AF	fluorinated hydrocarbon	DuPont Polymers	thermal
PC370	acrylate	Luvantix Co., LTD	UV
PC373	acrylate	Luvantix Co., LTD	UV
CRI37	acrylate	Crystal Research Inc.	UV
1D3-63	acrylate	DSM	UV

* The novel low refractive index cladding materials were synthesized by a well-controlled, cost effective sol-gel process [8]. Briefly the one step synthesis of the heat curable FHG (Fluorinated Hybrid Glass) involves prehydrolysis and condensation of silica precursor-TEOS with alkoxy silane bearing methyl groups and silane bearing perfluoro-alkyl groups (FAS). The product of such reaction, when stripped off solvents, constitutes low viscosity, clear liquid, that harden quickly upon heat exposure. Synthesis of the UV curable FHG is carried in a similar way

with a difference that one more silane bearing either vinyl or acrylate groups is introduced to the formulation. (By varying the amount of FAS the refractive index of the FHG can be in the range 1.37-1.44)

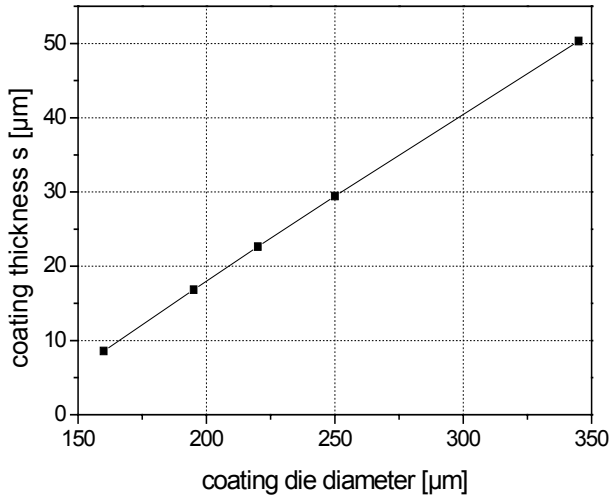


Figure 1 Dependence of the coating thickness from coating die cross section, calculated from (1); squares: used die dimensions (160, 195, 220, 250 and 345 µm)

Thermal curable coatings were treated in a inline tube furnace at a maximum temperature of about 400 °C and a dwell time of about 3 s. UV curing polymers were polymerized in a FUSION UV Curing System™ F450T with B-bulb. The lamp power density was varied between 60 W·cm⁻¹ and 120 W·cm⁻¹. The maximum total curing power was 1200 W. All curing procedures were processed in nitrogen atmosphere (18 slm).

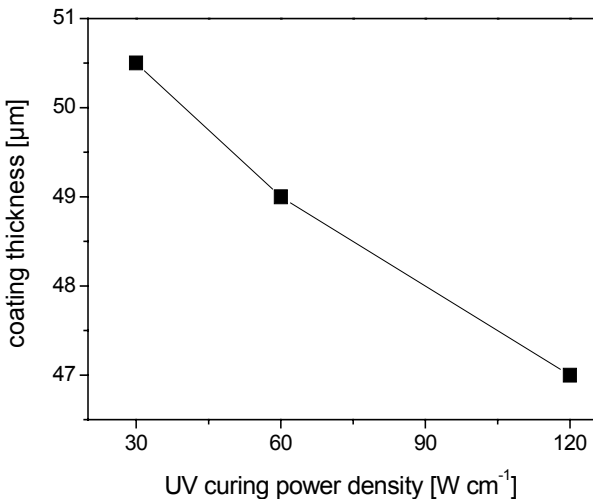


Figure 2 Influence of the UV curing power intensity to the coating thickness (Low index acrylate coating: UVF PC 373; Luvantix)

With increasing the UV lamp power typically decreases the thickness of the cured polymer coating. Associate with this effect increases the sensible coating hardness and coating yellowing (figure 2).

Air clad fibers were prepared by stack and draw method [9]. The preforms consist of a central silica rod surrounded by exact adjusted capillaries in an outer cladding jacketing tube. The drawing procedure was carried out in a differential pressurizing regime. Its parameters were changed to manufacture air clad fibers with different air fraction in the holey ring. The air clad fibers were coated with single layer UV Acrylate (n @ 1,300 nm: 1.51, thickness: 50 µm).

Typical fiber cross sections of high NA polymer coated and air clad fibers are shown in the micrographs below (figures 3 and 4):

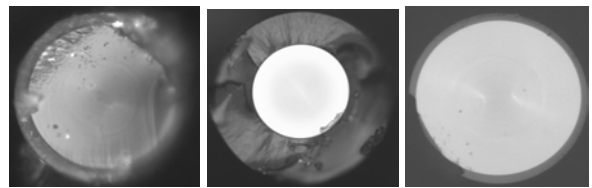


Figure 3 Cross section micrograph of a Teflon AF, silicone and hybrid material coated fiber

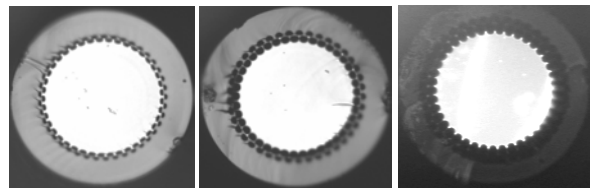


Figure 4 Micrographs of a air clad fibers with different air fractions (left to right: NA: 0.15; 0.40; 0.55)

3. Optical behavior of High NA polymer clad fibers

3.1. Transmission properties

The attenuation behavior of low index coated silica fibers is influenced by material effects of silica core, the absorption behavior of the polymer and the quality of the silica polymer interface. The purity (typical OH content: <0.2 wt.ppm) and the homogeneity of the used F300 silica material allows a loss level in the dB km⁻¹ in the NIR spectral range. However, the background loss of the polymer clad fibers is significantly influenced by the interaction of fiber silica surface with the polymer and the polymer properties itself. Low index silicone coated fibers with equal cross section parameters, equal material components vary more then one order of magnitude, depend on the pre-curing conditions of the coating material and the coating thickness (figure 5).

Apparently the originate of the different loss levels is based on an unfavorable influence of already polymerized parts of the silicone on the coating behavior during the drawing procedure. Obviously the inhomogeneous degree of pre-polymerization (beginning of gelification) causes in an inhomogeneous distribution of mechanical tension in lateral and circumferential direction of the complete cured fiber coating. The consequences are tension fluctuation based

(micro-)bending losses in considerable scale. On the other hand a very thin coating layer (10 μm) of such a weak coating material like silicon elastomer is not able to protect the fiber surface safely from environmental impacts. The consequence is a slightly increased background loss related to the 53 μm thick adequate coated fiber (figure 5).

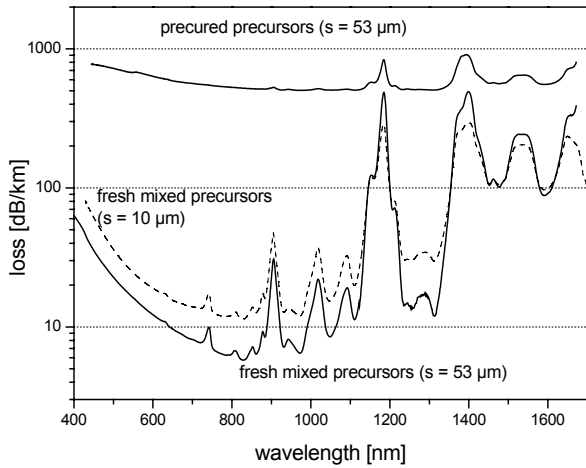


Figure 5 Loss spectra of silicone elastomer coated fibers based on fresh mixed (cooled by 10 °C) and precured (ca. 1 h at 30 °C) silicone components

Also UV polymer coated fibers are influenced in their transmission windows significantly by the curing conditions. Namely highly NA coatings, e.g. fluorinated acrylates, require a high UV power density for a sufficient polymerization degree, i.e. a manageable coating abrasiveness. The consequences are shifts in the spectral transmission window of the fiber (figure 6).

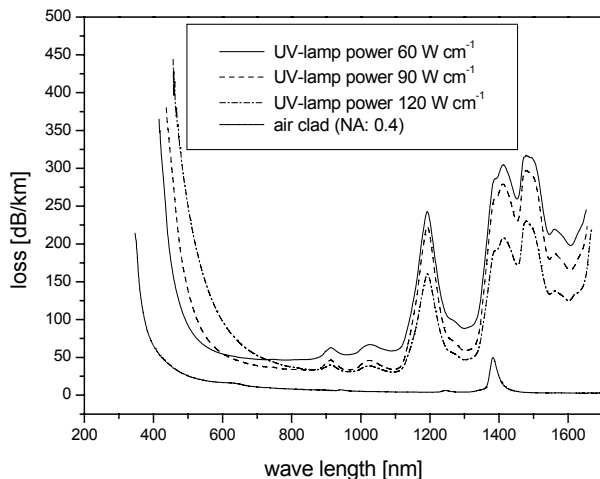


Figure 6 Loss spectra of low index UV-acrylate coated F300 fibers (DSM 1D3-63), the coating was cured with different UV lamp power density (The loss graph of the air clad fiber is pictured to reveal coating absorption effects.)

With increasing the UV lamp power density the short wave length edge of the fiber is red shifted, whereas the evanescent field absorption acrylate bands (e.g. 1.2 μm ; 1.47 μm) are not changing significantly. Figure 6 shows, that the intensity of edge shifting is influenced by the polymer coating specifics (e.g. polymer absorption and scattering effects)

However, the edge shifting is also caused as a feature of the fiber silica core. By change from silica F300 (<0.2 ppm OH) to F100 (approx. 1200 ppm OH) the 100 dBkm^{-1} wavelength pass amount decreased (figure 7). By processing the same drawing and coating procedures (UV acrylate 1D3-63) with both silica materials leads the short wavelength edge behavior is slightly different with varying the UV curing lamp power. In total prevails the coating effect on the red shift of this edge with the UV curing power.

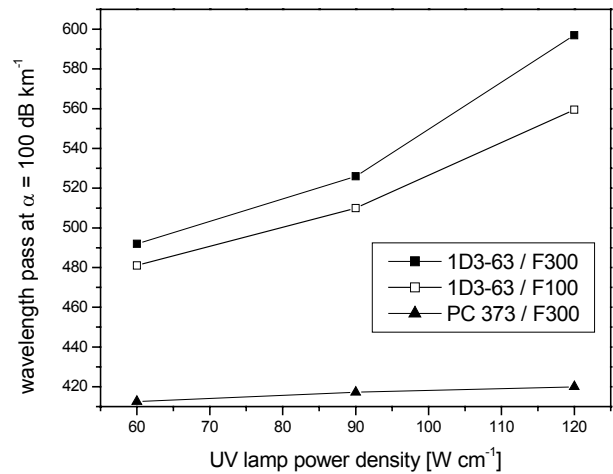


Figure 7 Influence of UV curing power on short wavelength loss (wavelength at loss level 100 dB km^{-1})

For a great variety of low index polymer coated F300 fibers (diameter 125 μm) we investigated the background loss at 800 nm (table 2). We could not find a relationship between the numerical aperture and background loss. For a comparison of the background loss of polymer coated and air clad fibers two microstructured fibers are also listed in the table (cross section of the air clad fibers see figure 4, left and middle).

Table 2 Different low index polymer coated fibers and the corresponding NA and background loss

Coating	thickness [μm]	NA	loss @ 800 nm [$\text{dB}\cdot\text{km}^{-1}$]
RT 601	53	0.358	6.5
RT 601	10	0.358	12.2
HG-Li-3	26.5	0.470	17.6
HG-Li-V-T (low speed drawing)	5.5	0.490	25.2
HG-Li-V-T (high speed drawing)	6.5	0.500	26.3
Teflon AF (single layer)	5	0.606	8.9

Teflon AF (double layer)	10	0.606	10.7
PC 370	50	0.459	10.5
PC 373	52	0.447	7.64
CRI 37	56	0.459	57.2
1D3-63	52	0.340	34.1
Air clad fiber	36 *	0.22 **	15.7
Air clad fiber	36 *	0.40 **	8.1

* The air clad fiber is coated with the high index acrylate DSM 3471
The coating has no optical function.

** The NA is due to the refractive index difference of the silica core and the air filled capillary ring.

3.2. Macro bending loss

The bending loss is an important parameter in technical fiber practice. The investigated influence of bending radius on additional loss is shown in figures 8 and 9. The losses has been investigated by laying a fiber in one loop and measuring of the transmitted light intensity. For a better comparison the intensity difference in the loop length has been calculated to one meter length. It is clearly seen that the fiber with silicone coating (coating: RT601, NA: 0,358 @ $\lambda = 1,0 \mu\text{m}$) show the strongest influence of the bending radius on the additional fiber loss. However, probably it is a matter of an addition of several effects. The investigated fibers had lengths of about two meters, possibly to short for a total mode mixture. So the influence of the number of modes (depending on fiber diameter and NA) on the loss is to large. Another effect is the coating thickness. For the investigation of bending loss of the silicone coated fiber a sample with a $10 \mu\text{m}$ layer was used. In this case the influence of micro bending losses can superpose the macro bending loss effects.

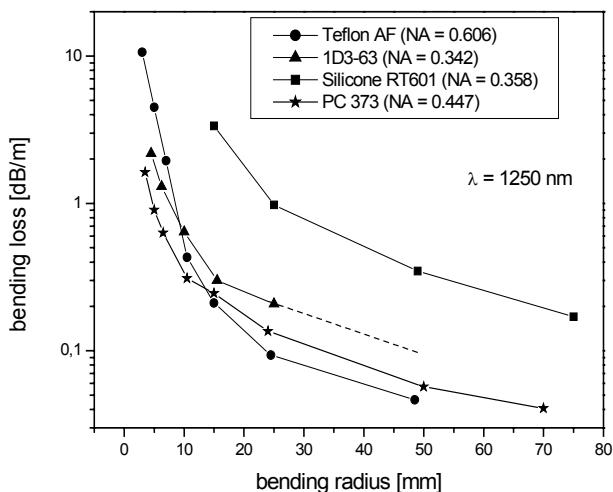


Figure 8 Effect of bending radius on additional loss (NA given in the inset has been measured at $\lambda = 1.0 \mu\text{m}$)

The air clad fibers exhibit an expected loss behavior. The strongest influence at small bending radii possesses the fiber with the lowest numerical aperture (figure 9).

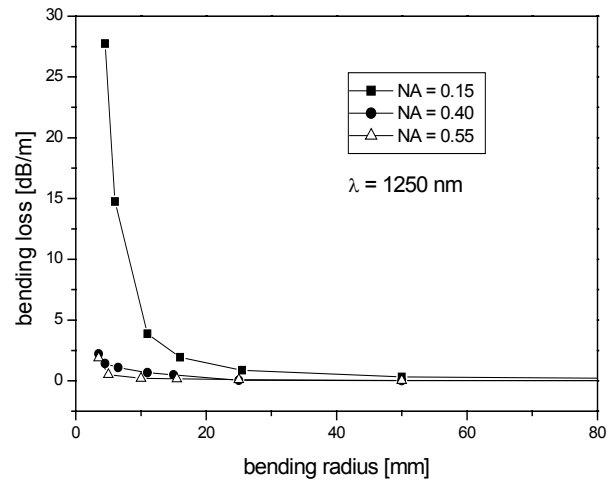


Figure 9 Bending loss in dependence on bending radius for air clad fibers with different NA (loss @ $\lambda = 1250 \text{ nm}$)

The NA of air clad fibers is finally strong influenced by the bridge width and length. Issa et. al. [10] calculated the numerical aperture for air clad fibers in a perpendicular arrangement of bridges for the multimode core. The investigation of fibers with an air clad in a design with a non perpendicular arrangement (figure 10, no. 3) makes it quite complicated, and the results become inconsistent (figure 11). Another fact is a formation of a second hole ring during fiber drawing (in dependence on the method of preform construction) with a further influence on numerical aperture. In principle there is an increasing NA with decreasing bridge widths, but with a strong dependence on the hole arrangement.

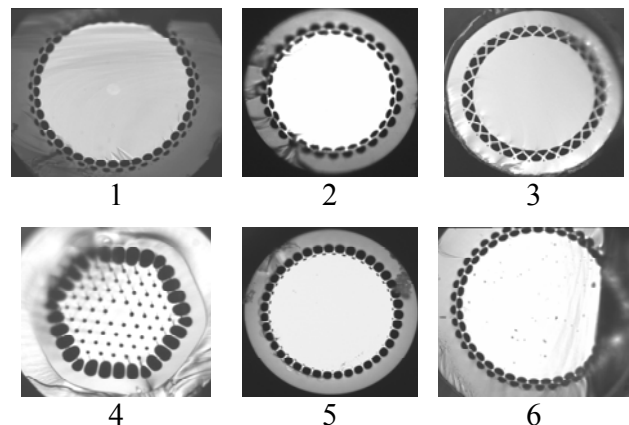


Figure 10 Cross sections of air clad fibers The numbers are relating to figure 11

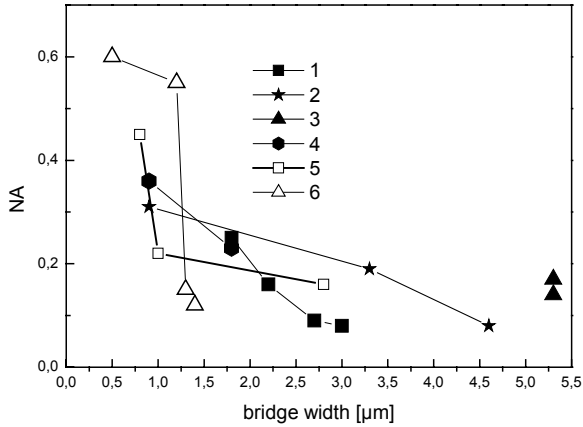


Figure 11 Numerical aperture in dependence on the bridge width for air clad fibers with different hole arrangements (The numbers correspond to the cross section numbers in figure 10)

4. Mechanical properties

The mechanical behavior was investigated by dynamic tensile proof testing with an elongation velocity of $30 \text{ mm}\cdot\text{min}^{-1}$ and calculated by Weibull analysis. In contrast to standard acrylate coating the plurality of different polymers shows a wide disperse tensile behavior (table 3).

Table 3 Tensile strength and Weibull parameter of different polymer coated fibers

Coating	thickness [μm]	tensile strength [N]	m-parameter
silicone RT 601	53	20.07	26.5
HG-Li-V-T (low speed drawing)	5.5	7.91	5.91
HG-Li-V-T (high speed drawing)	6.5	6.87	11.24
Teflon AF (single layer)	5	9.63	2.03
Teflon AF (double layer)	10	17.82	4.78
PC 373	52	37.64	2.39
CRI 37	56	19.63	3.73
1D3-63	52	64.89	138.0

With the exception of the fiber coated with 1D3-63 all other investigated fibers show a quite low tensile strength and a low Weibull modulus. There are different reasons for this behavior, i) the hybrid material coatings as well as the Teflon AF are only applicable as thin film coatings, ii) silicon for example is a coating with a low adhesiveness to the fiber surface, iii) cured PC373 and CRI37 have shown small embedded impurities like bubbles caused by the coating process or solvent residuals.

Typically the tensile strength increases with the coating thickness (see figure 12).

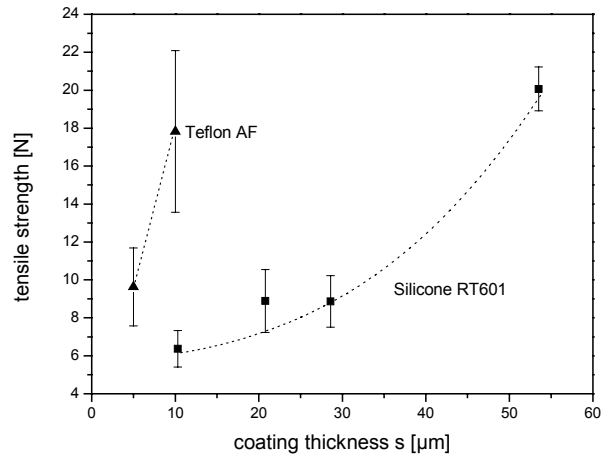


Figure 12 Influence of coating thickness on tensile strength of two types of polymer coated fibers

The mechanical strength of air clad fiber is discussed in a previous publication [11]. We have shown that tensile strength values of air clad fibers are lower than that of unstructured pure polymer (standard high index acrylate) coated fibers and strongly influenced by the thickness of the outer cladding. However, in a direct comparison air clad fibers possess a similar or higher tensile strength than the here investigated polymer coated fibers.

5. Thermal stability of fiber coatings

In a silica fiber with a low index coating a part of the launched pump power is coupled into the coating over the evanescent field. Therefore an important criterion for the application of low index coatings in high power fiber laser systems is their thermal stability. For the determination of thermal properties of the coating materials we used a Perkin Elmer TGA7. The coatings were applied as a thin film on a glass substrate and cured for 90 s in a UV curing system with a H-bulb and for 60 min in a curing furnace at $200 \text{ }^\circ\text{C}$, respectively. After curing the coatings were manually reduced to small pieces. During heating with $10 \text{ K}\cdot\text{min}^{-1}$ we measured the weight loss of the polymers (figure 13).

There is a remarkable difference in the thermal stability between UV and thermal curable coating materials. Apparently, UV sensitive groups in the polymer network play an important role in the thermal resistance. HG-Li-T, Teflon AF and the Silicone RT601 possess the highest thermal stability of all investigated coatings. Teflon AF additionally comes with the highest numerical aperture to silica. Unfortunately, it is known as a strong oxygen and water permeable polymer system. In combination with a very low coating thickness of about $5 \text{ } \mu\text{m}$ in a single layer system the long time reliability of this fibers is to low. The thermal stability of silicone RT601 is comparable to that of Teflon AF, but the numerical aperture to silica is 0.358. Therefore, in fiber laser systems with silicone coatings we observe a strong interaction of the pump light with the coating materials. Hybrid glass material is a new class of coatings with a low index (HG-Li-3: 1.3708 @ 1300 nm) and a high thermal stability.

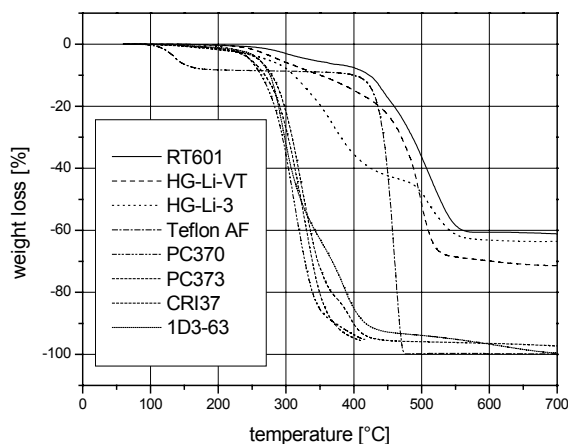


Figure 13 Thermogravimetry of UV and thermal cured low index coating materials, respectively

6. Conclusions

High NA polymer fibers show outstanding characteristics which made them interesting for different applications. The loss spectrum is typically influenced by the specific bands of the organic polymers in result of evanescent field absorption. To depress this effect one can use ultra low index materials (like Teflon) in combination with a low permeable coating as secondary layer. Despite the higher making expenditure another way is the application of air clad fibers. They show no absorption effects and a comparable background loss. A direct correlation between NA and background loss level could not be observed for polymer coated fibers as well as for air clad fibers. For heavily UV curable polymers the short wavelength edge shows a remarkable red shift with the curing illumination intensity. In this case a compromise between mechanical properties (coating hardness) and optical properties (spectral breadth of transmission window) has to be found. The tensile strength of low index polymer coated fibers achieve in particular case the level standard telecom fibers, but typically it is lower. The corrosion attack mechanism could be caused by different chemical groups in the polymer as well by the atmospheric permeability. Because of the complexity of this damage behavior the reasons must be fortified investigated. Because of the auspicious properties of hybrid glass coatings at the current state these coatings are quite promising materials related to an application in high power fiber laser systems.

7. Acknowledgements

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