Characterization of preform raw materials for high power fiber lasers using LID absorption measurement technique

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Abstract

Two different concepts are introduced and compared to measure for the first time residual absorption in pure and doped fused silica fiber preform raw materials at 940 nm and 1550nm directly by means of the laser induced deflection (LID) technique in order to analyze the minimal achievable attenuation in high power fiber lasers based on the doped fused silica raw materials.

It is found, that attaching a thin disk sample of the preform raw material to a practically non-absorbing substrate at the wavelength of investigation is the best measurement concept with respect to material consumption, absolute calibration and impact of the strong scattering in the doped raw materials on the measurement.

For several Yb doped laser active raw materials the initial absorption value at 1550 nm (ranging from less than 50 db/km to about 1800 dB/km) is compared to the total loss achieved for the manufactured fiber at 1200 nm, 1315 nm and 1600 nm, respectively. For some of the chosen materials the fiber loss is very comparable to residual raw material absorption indicating that the initial absorption is the dominant loss mechanism in the manufactured fiber. In contrast, for some fibers the total loss exceeds the values of the raw material absorption which allows the conclusion that additional loss mechanism like scattering, stress, geometrically fluctuation and micro or macro bending contributes to the fiber attenuation.

Keywords: High power fiber laser, fiber preform, laser active materials, absorption

1. INTRODUCTION

High power fiber lasers, operating in the near infrared and with output powers in the kilowatt regime, play an important role in nowadays laser micromachining applications. In order to achieve high laser efficiencies and to allow very high laser power values the fiber losses need to be kept to a very low level (~ 10-20 dB/km) that long fiber lengths (~ 30-40 m) can be efficiently used. By the use of longer fiber lengths, the doping level of the material can be reduced to avoid photodarkening.

Typically, characterization of laser active fibers starts by optical inspection of the fiber performs before entering the drawing process to characterize e.g. the interface quality between core and cladding structures. Unfortunately, the results of the visual inspection and the determined fiber attenuation don't always coincide since losses in optical fibers result from many mechanisms like absorption, scattering (interface between core and cladding structures, defects and material inhomogeneities), stress, geometrically fluctuation and micro or macro bending.

Since manufacturing of preforms is time consuming and costly due to the requirement of many complex process steps, a pre-characterization of the laser active raw materials is desired to avoid labor-intensive fiber production currently required to characterize the attenuation and quality of the core material. Furthermore, it is difficult to distinguish between the different losses only on the basis of fiber attenuation measurement.

Unfortunately, spectrometric evaluation of Yb-doped silica raw materials practically only detects the enhanced scattering accounting for the dominant loss in the raw materials. This scattering, however, is strongly downscaled in the manufactured fiber and becomes a non-critical loss part. On the other hand, residual absorption will play a major role for the final fiber attenuation but is virtually not detectable in the spectrometric characterization of the raw material. Furthermore, the optical path in Yb-doped bulk silica rods (∅ ~ 15 mm, l = 150 mm) is not long enough to determine the expected fiber attenuation volume with the needed accuracy. Therefore, the possible suitability of laser active raw materials is so far only investigated once the fiber is manufactured.

In this work we report on two different concepts to measure the residual absorption in raw materials for high power fiber lasers directly by applying the laser induced deflection (LID) technique which has previously demonstrated its high sensitivity for measuring very low bulk and coating absorptions [1-3].

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2. EXPERIMENTAL METHODS

The LID measuring principle (Figure 1), one amongst many photothermal absorption techniques, has been introduced previously and applied commonly to measure residual absorption in bulk materials and thin films [1-3].

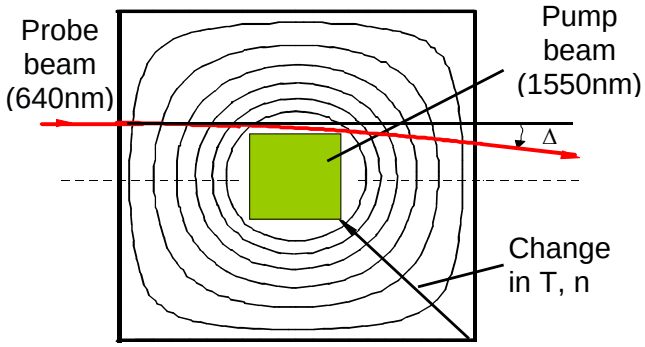

Figure 1: Sketch of the LID measurement principle including calculated isolines of temperature and refractive index for fused silica as well as the probe beam propagation

The laser irradiation of a pump (excitation) beam induces absorption and subsequently a temperature gradient between irradiated and non-irradiated parts of the material. The temperature dependent refractive index transforms the temperature profile into a refractive index profile ("thermal lens") that is used to deflect a probe (test) beam by the angle Δ while passing the sample perpendicularly to a pump (excitation) laser beam. In order to quantify the deflection data a calibration of the setup is required. As previously described [2,3], homogeneous surface absorption and bulk absorption can be simulated by electrically heated samples of the same material and geometry. Using the electrical calibration the absorbed mean laser power within the thin film is calculated and divided by the applied mean laser power to achieve absolute absorption values. It needs to be mentioned, that all photothermal or calorimetric absorption measurement techniques only detect that part of the absorbed photon energy that is transferred into phonon energy. Therefore, all absorption data represent a lower absorption limit that needs to be taken into account when an energy balance is considered.
Laser based LID absorption measurements at raw materials for fiber lasers have been performed using a compact LID setup shown in Figure 2. The investigated samples have been prepared by powder sinter technology [4].

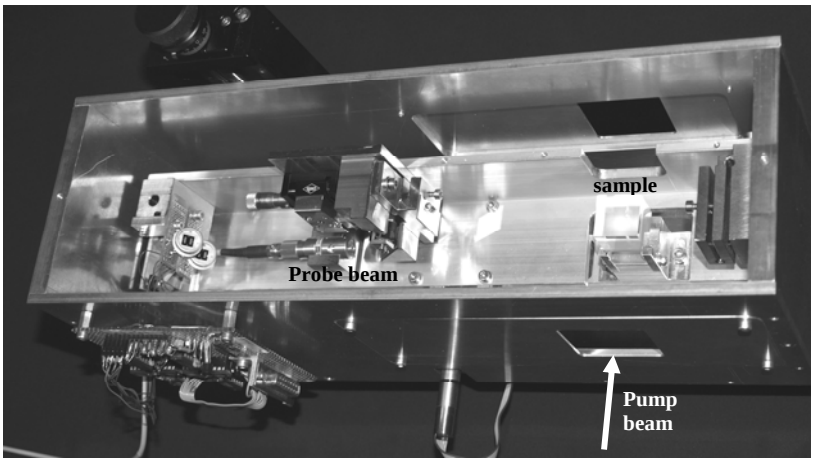

Figure 2: Photograph of the compact LID setup applied to direct absorption measurements

A cw Er-doped fiber laser running at 1550 nm (for pure and Yb doped fused silica samples) and a cw diode laser running at 946 nm (for pure fused silica sample only) have been used as pump (excitation) source. For both lasers the beam profile is shaped to a homogeneous spatial profile (top hat) of ∅~5 mm (1550 nm) and 4x4 mm^2 (946 nm), respectively.

The applied laser power ranged between 3 and 6 W. Two bulk absorption measurement concepts have been applied resulting in different sample geometries and electrical calibrations. For the first concept fully polished rectangular samples of 15x15x10 mm^3 geometry were taken and standard bulk absorption measurement strategy [3] was applied. For electrical calibration a hole was been drilled centrally into the samples after laser measurements and a resistor was placed inside. The calibration value was then obtained by applying defined electrical power to simulate the effect of pump laser absorption. For the second concept thin disk samples of ∅ = 10…25 mm and 1mm thickness were prepared. In order to allow the probe beam passing as indicated in Figure 1, the thin disk samples have been attached optically to a 20x20x20 mm^3 cube made of pure fused silica. Thus, the probe beams are deflected by the thermal lens generated by heat transfer from the thin disk sample to the attached cube. This concept coincides in general with the strategy for surface/coating absorption measurements [3]. Therefore, equivalent calibration has been applied by equipping calibration samples a flat heater mounted at the front surface.
After fiber manufacturing the total fiber loss has been measured at the common wavelength of 1200 nm used to characterize Yb doped fused silica for fiber lasers with respect to residual losses in the silica network using commercial spectrometers (First system: Lambda 900 by Perkin Elmer; Second system: Monochromator HR-250 by Jobin Yvon, Detectors: Si-diode and PbS-detector) and cut back attenuation measurements [5]. In addition, measurements at 1315 nm and 1600 nm have been performed. Due to the lack of powerful lasers in the wavelength region around 1200 nm, the absorption data obtained at 1550 nm are taken for comparison to the fiber attenuation values.

3. EXPERIMENTAL RESULTS AND DISCUSSIONS

3.1 Concept using rectangular samples (15x15x10 mm^3)

Prior to the measurement of Yb doped fused silica at 1550 nm the concept was tested and proven at 946 nm using pure silica material (HSQ 100 by Heraeus) with negligible scattering. Using LID technique an absorption value of (114±8) dB/km was obtained for a rectangular sample. For three fibers made of HSQ 100 total losses of 108 dB/km, 107 dB/km and 105 dB/km were measured. The very good agreement of the data reveals that in principle the absorption loss in the fiber can be predicted by characterizing raw materials of rectangular geometry and the accompanied electrical calibration.
Figure 3 shows two investigated Yb doped fused silica raw material samples which have been modified for calibration after laser based absorption measurements. The photograph of the sample on the right hand side of Figure 3 indicates that the investigated material partially exhibits strongly enhanced scattering compared to pure fused silica material.

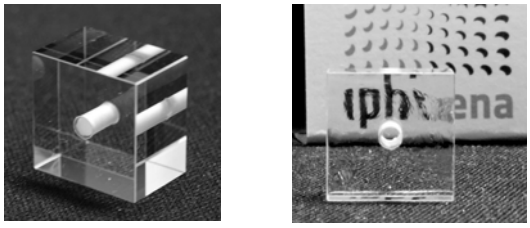

Figure 3: Photographs of investigated samples (geometry 15x15x10 mm^3) taken after preparing for electrical calibration and indicating strong scattering obtained in some samples

Although the strong scattering in the raw material is downscaled during fiber manufacturing, it arises from the measurements that the probe beam guiding becomes critical for these strong scattering raw materials. The reason is illustrated in Figure 4. Figure 4a shows a picture of the two position sensitive detectors (PSD) used to measure the probe beam deflection before inserting the sample into the LID setup. The two probe beams (bright spots) can be easily recognized. After placing a strongly scattering sample in the setup, however, the two spots disappear (Figure 4b). Due to the strong scattering inside the sample the originally small probe beam diameter is significantly enlarged and the PSDs can not distinguish between separated beams any more. As a consequence absorption measurements for this type of sample are not possible.

a) b)

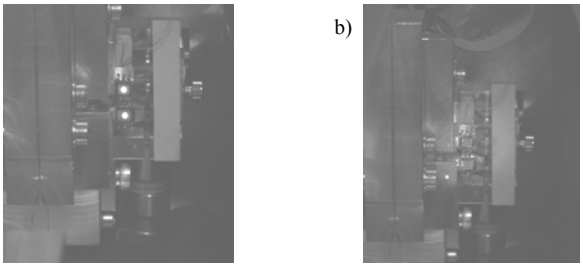

Figure 4: (a) Photograph showing the two probe beams (bright spots) hitting the position sensitive detectors (PSD) before inserting a sample into the LID setup and (b) disappearing probe beam spots after inserting a strongly scattering sample

There are further drawbacks using the sample geometry of this first concept. The geometry requires a relatively large part of raw material slab which cannot be used for fiber manufacturing which is primarily a question of costs. In addition, sample manufacturing is costly since complete surface polishing is required to avoid measurement errors due to reabsorbed scattered light at unpolished surfaces. Since the raw materials are not fully identical with respect to their doping levels they differ from each other in their measurement relevant material parameters (expansion coefficient, heat conductivity, dn/dT). As a result, electrical calibration is necessary for each individual sample, i.e. after laser based measurement every sample needs to be prepared for electrical calibration (hole drilling and resistor insertion). Besides the time consuming procedure the laser based sample measurement cannot be repeated once the sample is prepared for calibration. Finally, strong scattering in the samples prevents to transfer the concept to shorter wavelengths due to the λ^{-4} dependence of scattering and the relatively large sample length of 10 mm.

3.2 Concept using thin disk samples (∅ = 10…25 mm, 1mm thickness)

Bearing in mind the drawbacks of the concept in the previous section, a second sample geometry have been chosen. In comparison to the rectangular samples, an only 1mm thick thin disk sample is strongly preferred with respect to raw material consumption and sample manufacturing. The basic challenge for using this concept was to transfer the LID principle (Figure 1) to thin disk samples which not allow a probe beam passing perpendicular to the pump beam due to the small sample length of only 1mm. To solve this problem, the thin disk has been optically attached to a larger cube (20x20x20 mm^3) made of pure fused silica as shown in Figure 5a. Hence, the probe beams are guided through the fused silica cube and deflected by the thermal lens generated by heat transfer from the thin disk sample to the cube. By this, even strongly scattering thin disk samples do not affect the probe beam guiding which is one main advantage compared to the previous concept. In order to avoid additional LID measuring signals from the cube itself an extremely low absorbing material (F300 by Heraeus) has been chosen. It was proven before the actual investigations that irradiating the cube without attached thin disk yields no LID signal for the given laser power values.

a) b)

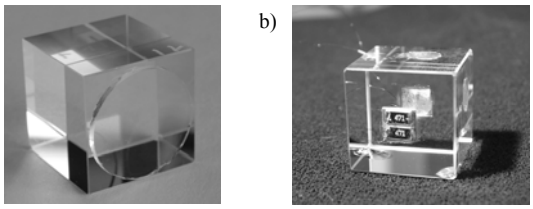

Figure 5: (a) Photograph showing a thin disk sample attached to a 20x20x20 mm^3 fused silica cube and (b) 20x20x20 mm^3 fused silica cube attached with electrical heaters for calibration

Basically, the thin disk concept can now considered to be equivalent to the measurement strategy for high reflecting mirrors, i.e. absolute calibration is obtained by using a flat heater mounted at the surface of the cube (Figure 5b). Thus, the calibration does not depend on the sample dependent material parameters like expansion coefficient, thermal conductivity and dn/dT. Therefore, only one calibration for the sample set of investigation is needed. Furthermore, in contrast to the previous concept, the thin disk concept allows multiple measurements since calibration and laser based absorption measurement are separated.

Before measuring Yb doped fused silica samples, the thin disk concept was tested by using pure silica (HSQ 100) at the wavelength 1550 nm. An absorption induced attenuation of (31.3±5) dB/km was obtained which is in reasonable agreement with the value (23.5±3) dB/km achieved by traditional bulk absorption measurement using rectangular sample geometry. In addition, the reproducibility was tested including successive attaching and detaching of the thin disk sample. The experiments revealed a reproducibility of better than 5 % indicating that the thin disk attachment is not a major source of a reproducibility error.
After characterizing the thin disk concept a set of Yb doped fused silica samples have been measured before perform and fiber manufacturing. The total fiber loss has been measured conventionally at selected wavelengths using the cut back technique. Table 1 summarizes the obtained data for the investigated samples.

Table 1: Absorption data for different preform raw materials at 1550 nm obtained by LID technique and related total loss values for the manufactured fibers

Slab	Yb_2O_3 [mol%]	Al_2O_3 [mol%]	Absorption @ 1550 nm (LID) [dB/km]	Fiber loss [dB/km]
810	0.15	1	1563.9 ± 80	2800 @ 1200 nm
812	0.15	1	46 ± 7 35 ± 8 44.9 ± 12 34.1 ± 8	~50 @ 1200 nm 19 @ 1600 nm
827	0.05	1	1832.4 ± 95 1774.3 ± 90	2950 @ 1200 nm
831	0.05	1	112.6 ± 12 121.7 ± 11 101.7 ± 8 118.2 ± 26 110.7 ± 15	380 @ 1200 nm ~280 @ 1315 nm ~151 @ 1600 nm

The experimental results indicate that slab 812 exhibits the lowest absorption/loss values and the total fiber loss and the absorption of the raw material are very comparable. Therefore, fiber attenuation is driven by residual absorption and other loss mechanisms are suppressed sufficiently. All other samples show a more or less large gap between the raw material absorption and the total fiber loss. Thus, other losses introduced by the material itself or the perform/fiber manufacturing process play a substantial role for the fiber attenuation. It is pointed out, however, that due to the different wavelengths used for raw material and fiber investigations the comparison might be affected e.g. by remaining OH contents in the samples yielding different amounts of absorption for the chosen wavelengths of investigation.

4. SUMMARY

For the first time to our knowledge, doped raw materials for high power fiber lasers are pre-characterize and selected by measuring directly and absolutely their residual absorption using the laser induced deflection (LID) technique. Two concepts using different sample geometries (rectangular: 15x15x10 mm^3; thin disk: ∅ = 10…25 mm, 1mm thick) have been compared to find to most suitable measurement setup for the investigations. Attaching thin disk samples to a practically non-absorbing substrate is the measurement concept of choice with respect to raw material consumption and calibration. Furthermore, partially strong scattering in the raw materials prevents the use of the rectangular samples.
Experimental results show that in principle the samples can be divided into two groups. For the first, showing the lowest loss values) the obtained total fiber loss is very comparable to the residual absorption found in the doped raw material. This indicates that the fiber attenuation is driven by residual absorption while other loss mechanisms are suppressed sufficiently. Fiber attenuation improvements are therefore only possible when the residual absorption (specific absorbing impurities or imperfections) is further reduced. The second sample group exhibits total fiber losses which for some examples strongly exceed the residual raw material absorption. As a consequence, other effects like scattering in the raw material or the perform/fiber manufacturing process need optimizing.
The experimental findings reveal that raw material characterization for high power fiber laser application is very important to select different materials prior to the subsequent technology steps and for the development of new materials

for fiber optics applications like new glasses for fiber laser or UV-transparent materials. Furthermore, a quantification of the material's absorption contribution to the fiber loss is of great interest for optimizing the purification route, advances in material homogeneity and improving interface adaptation of core and cladding. Although the presented measurements can not always predict the final attenuation of the fiber, they will give a lower fiber attenuation limit that allows preselecting of materials before starting the fiber drawing process.

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