

DTG coating Ormocer[®]-T

for

Temperature Sensing Applications

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1 INTRODUCTION

The success of FBGS's Draw Tower Gratings (DTGs) is largely due to the Ormocer^{®1} coating. The Ormocer[®] (ORganic MOdified CERamic) material is selected from a group of hybrid polymers, which gives our optical fiber an excellent strain transfer, a wide operating temperature range, and the ease of processing and handling. More information about our choice to use Ormocer[®] as a superior coating for our DTGs can be found on our website: <u>http://www.fbgs.com/technology/why-we-have-chosen-to-use-an-ormocercoating/</u>.

Our original Ormocer[®] coating has been proven to work exceptionally well in a broad range of applications. Its excellent adhesion to glass and its hardness have shown to be crucial for proper strain sensing. Nevertheless, these positive properties can have an adverse effect for temperature sensing applications. The optical fiber in this case is often mounted in a strain-free configuration (free-hanging optical fiber) leading to an increased influence of coating effects, particularly when harder coatings such as Ormocer[®] are being applied.

A well-known example of such a coating effect is due to the hygroscopicity of the coating material. Just like many other polymers, Ormocer[®] is known to be hygroscopic and will consequently expand volumetrically during exposure to humidity. Using a strain-free configuration, the force from the coating induced by the temperature changes and acting on the outer surface of the optical fiber will become predominant and can be picked up by the inscribed Fiber Bragg Grating (FBG) element. This effect is even further enhanced when the coating is relatively stiffer. A softer coating could equally be hygroscopic but its softness will impede the coating to transfer the force and to strain the optical fiber.

The above example nicely illustrates the ambiguity of our Ormocer[®] coating: the same property enabling strain sensing applications complicates temperature sensing measurements. Consequently, there is a clear need to work with a dedicated "temperature sensing coating", while maintaining the compatibility and the unique advantages of the Ormocer[®] material.

Ormocer[®]-T coating is specifically designed for temperature measurement applications. It maintains its excellent adhesion to glass but clearly differentiates itself from the well-established Ormocer[®] coating as being much softer, which makes it the preferred match for temperature sensing applications. We will show in the next paragraphs how this coating enables more precise temperature measurements resulting in an improvement in the accuracy of temperature measurement by a factor 3 to 4.

2 ORMOCER[®] VS. ORMOCER[®]-T

The main difference between the Ormocer[®] and Ormocer[®]-T coating types is the stiffness. Generally speaking, Ormocer[®]-T is relatively much softer than Ormocer[®], for all temperatures (> 0°C). The stiffness of a material is generally quantified using its elastic modulus or Young's modulus. This parameter relates mechanical stress to strain and is temperature dependent. To illustrate the difference in stiffness between both coating types, the Young's modulus of both materials is depicted in Figure 1.

Figure 1 clearly shows that the Young's modulus of Ormocer[®]-T is smaller compared to the standard Ormocer[®] for temperatures above 0°C. At 0°C, the stiffness of both coating types is nearly identical. However, when exposed to higher temperatures, the stiffness of the Ormocer[®]-T is steeply reduced, in contrast to the Ormocer[®] coating, which only gradually declines.

¹ Ormocer® is a Trademark of Fraunhofer-Gesellschaft zur Förderung der angewandten Forschung e.V.





Figure 1: Young's modulus as a function of temperature for Ormocer® and Ormocer®-T.

3 HYGROSCOPIC EFFECT

As discussed in the introduction, an important challenge related to temperature sensing using FBG based sensors, is to minimize the effect of changes in Relative Humidity (RH), which inevitably are linked to changes in temperature. Particularly for stiffer coatings such as Ormocer[®] or Polyimide, where temperature measurements can easily be jeopardized by changes in humidity level leading to a reduced accuracy in temperature sensing. However, this effect can be mitigated though the use of the Ormocer[®]-T coating, being much softer and therefore reducing the FBG sensitivity to humidity changes.

To verify this reduction in sensitivity, a controlled experiment with real-time parameter monitoring was carried out. Several samples of both coating types (Ormocer[®] and Ormocer[®]-T) were exposed to different levels of RH at constant temperature (20°C), while mounted in a climate chamber in a free-hanging configuration. The measured values of reference temperature (T) and RH during this 20 hours experiment are shown in Figure 2. The associated wavelength shifts for the different samples are shown in Figure 3 are a measure of the strain transfer from the coating to the fiber.

Figure 2 and Figure 3 show a clear correlation between the measured RH and the wavelength shift $(\Delta\lambda)$ for the Ormocer[®] coated samples, while there is no significant correlation for the Ormocer[®]-T coated samples.

The RH sensitivity of the Ormocer[®] coated samples is about 3 pm / %RH, which is merely originating from the hygroscopic effect. Water from the air humidity is penetrating the coating and causing it to expand. On the other hand, the wavelength shifts ($\Delta\lambda$) of the Ormocer[®]-T coated samples only show the changes in temperature without any RH influence. The relative softness of the Ormocer[®]-T coating is impeding any strain transfer from the coating to the glass, enabling temperature measurements without any influence of the hygroscopic effect of the fiber coating.





Figure 2: Measured Temperature (T) and Relative Humidity (RH) in the climatic chamber as a function of time (t) during the RH calibration.







4 TEMPERATURE SENSING

A change in temperature will inevitably have an impact on the relative humidity. For the Ormocer[®] coating, this sensitivity to RH adds an extra hysteresis to the temperature calibration data (as seen in Figure 3 – wavelength changes due to humidity changes at constant temperature), which deteriorates the temperature accuracy to 3 to 4°C. The Ormocer[®]-T coating was shown to have a heavily reduced sensitivity to these changes in RH enabling more precise temperature measurements. To visualize these improved temperature sensing capabilities, temperature calibration data for both coating types were measured and compared.

The temperature calibrations consisted of temperature cycles between 0°C and 100°C and were performed on free-hanging samples using both coating types. The results are presented in Figure 4. The sensitivity to RH from the Ormocer[®] coating adds an extra level of hysteresis to the calibration curve while the Ormocer[®]-T coated fiber does not have this effect. **This heavily influences the measurement accuracy for temperature sensing applications, down to 1°C and below.** The improvement in accuracy by a factor 3 to 4 is calculated by comparing (Ormocer[®] vs. Ormocer[®]-T) the residual errors from a quadratic fit and linking these wavelength errors to temperature uncertainties.



Figure 4: Temperature calibration curves for Ormocer[®] (top) and Ormocer[®]-T (bottom) coated samples.



The wavelength response of the Ormocer[®]-T coated samples slightly deviates from the linear interpolation at lower temperatures (< 20°C) and exhibits a bending point around 10°C. Looking back to Figure 1, this effect can be explained by the rapidly increasing Ormocer[®]-T stiffness at lower temperatures. Below 10°C, coating effects (hygroscopicity and thermal expansion) are again coming into play and will contribute to the temperature calibration curve.

5 TEMPERATURE STABILITY

Another key parameter of fiber Bragg gratings is the maximum operating temperature range which is primarily defined by the temperature stability at elevated temperatures, both in terms of Bragg grating reflectivity and wavelength. These parameters are also largely depending on the fiber coating. Hence, the thermal stability of DTGs at elevated temperatures was studied for both coating types.

In a series of tests, the long term stability of the reflectivity and the Bragg wavelength for various DTG samples with both coating types was monitored during exposure to various elevated temperatures. The set temperature was ranging from 100°C to 300°C in steps of 50°C. The period of monitoring for each step was \geq 50 hours. For each measurement 2 samples of each type plus 2 stripped (no coating) samples for reference purposes were included. The reflectivity at elevated temperatures as a function of time was found to be independent of the coating type: there is no influence from the coating contributing to the evolution of the reflectivity at elevated temperatures.

The evolution of the wavelength shift is plotted as a function of time for each coating type and the results of these wavelength stability measurements are presented below in Figure 5, Figure 6, and Figure 7. In order to be able to discriminate between the different temperatures, an offset value was added to the initial wavelength shifts. For the stripped samples (Figure 5), the offset value between 2 successive temperatures is 0.05 nm (50 pm). For the Ormocer[®] and Ormocer[®]-T coated samples (Figure 6 and Figure 7), the shift is 0.1 nm (100 pm). The maximum observed wavelength shifts ($\Delta\lambda_{max}$) are also listed in the graphs corresponding to each temperature setting.



Figure 5: Wavelength stability (Bragg wavelength shift as a function of time) at different elevated temperatures for *DTGs without coating (stripped)*. $\Delta\lambda_{max}$ is the maximum wavelength shift from $\Delta\lambda$ at t = 0 hours.





Figure 6: Wavelength stability (Bragg wavelength shift as a function of time) at different elevated temperatures for *Ormocer[®]* coated *DTGs*. $\Delta\lambda_{max}$ is the maximum wavelength shift from $\Delta\lambda$ at t = 0 hours.



Figure 7: Wavelength stability (Bragg wavelength shift as a function of time) at different elevated temperatures for *Ormocer®-T coated DTGs*. $\Delta\lambda_{max}$ is the maximum wavelength shift from $\Delta\lambda$ at t = 0 hours.

- For the stripped samples (Figure 5), there are only minor wavelength drops during the first couple of hours, after which the wavelength stays fairly stable.
- For the Ormocer[®] case (Figure 6), relatively large wavelength changes are observed for all tested temperatures, even at the lowest temperatures. At 100°C and 150°C, there is a slow gradual wavelength drop that does not reach full stability after 50 hours. At 200°C, the wavelength drop is more pronounced and definitely not stable at the end. Above 200°C, very irregular wavelength shifts related



to coating degradation start to occur. At 250°C, we can see an initial wavelength drop followed by a wavelength increase. The drop can be interpreted as the accelerated version of what we see at lower temperatures. The wavelength redshift can be explained by the coating starting to come off from the fiber and releasing the initial coating compressing force. At 300°C, this dip occurs more rapidly and more intense, indicating further coating degradation. Coating degradation was also confirmed by visual inspection.

We can conclude that the Ormocer[®] coating is not the ideal candidate for stable wavelength readings at elevated temperatures.

• For the Ormocer[®]-T case (Figure 7), we can see a good resemblance with the stripped DTGs for temperatures up to 200°C. For those temperatures, a small initial wavelength drop can be observed during the first few hours followed by more stable wavelength readings thereafter. Given the relative softness of this coating at higher temperatures, this behavior can also be understood: the coating is too soft to contribute to any wavelength shift. However, at temperatures above 200°C, increased wavelength changes start to occur. At 250°C and 300°C, we can again see a gradual wavelength decrease, resembling the Ormocer[®] coating case at 100°C to 200°C. Visual inspections of the coating after testing at these elevated temperatures reveal similar physical changes as seen with the Ormocer[®] coating.

In conclusion, the Ormocer[®]-T coating is recommended for use for stable wavelength readings up to 200°C, provided that an initial annealing for a few hours is performed.

6 TEMPERATURE SENSING VS. STRAIN SENSING

DTGs with the new Ormocer[®]-T coating is clearly the most attractive solution for temperature sensing applications. However, strain sensing applications generally benefit from a stiffer coating. Nevertheless, further testing on the Ormocer[®]-T coating has shown that **strain sensing, using direct fixation of the optical fiber, is still yielding good results at room temperature. The Ormocer[®]-T coating is however less suited for strain measurements through surface bonding at elevated temperatures. The preferred option in this case would be to use the Ormocer[®] coating. Due to the relative hardness of this coating, an excellent strain transfer to the glass fiber is guaranteed even at elevated temperatures. Furthermore, the described hygroscopic effects caused by the Ormocer[®] coating are not significant in case of bonded DTGs.**

7 CONCLUSIONS

The hygroscopicity of the optical fiber coating typically limits the accuracy of FBG-based temperature sensors. To overcome this challenge, it is recommended to use Ormocer®-T as DTG coating, mainly for temperature sensing applications above 0°C. Using this coating, unwanted strain effects associated with increasing temperatures are not transferred to the optical fiber. Therefore, the accuracy of the temperature measurements is significantly enhanced, down to 1°C and below.

Similar to the Ormocer[®] coating, there is no significant impact of the Ormocer[®]-T coating on the reflectivity of the DTG compared to a stripped one. But unlike the traditional Ormocer[®] coating, Ormocer[®]-T coated samples provide stable wavelength readings up to 200°C.

The Ormocer[®]-T coating is less suited for strain measurements through direct surface bonding at elevated temperatures. The coating gets too soft and unable to properly transfer strain to the optical fiber.

Collectively, it is concluded the Ormocer[®] coating is best suited for strain measurements (using bonded fibers), whereas the Ormocer[®]-T coating is the preferred coating for temperature measurements (using unbonded free fibers).

8 MORE INFORMATION

We are happy to receive your questions, comments and suggestions.

Please feel free to contact us directly through info@fbgs.com.