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Discussion

Measurements of the solidification process of resins from cantilever beams resonances

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ABSTRACT

In this work, we introduce a technique to infer elastic and mechanical properties of light-curing resins by using cantilever beams. The methodology includes vibration resonance measurements performed with a fiber optic Fizeau interferometer. As is known, the natural resonance frequency of cantilever beams depends strongly on any variation in its physical properties and geometry. Following this idea, square shaped solid aluminum beams with a short transverse deep crack drilled near its fixed end were studied. The slot was filled with photo-curing resins and resonance frequency was monitored as polymerization proceeded. In order to track resonance peaks, we adopted a simple electromagnetic actuator to force the beam into oscillations of variable frequencies. Beams were scanned periodically around its natural resonance as photo-curing was carried out. Due to the small vibrations amplitude present at the free end of beams (tens of microns typically), we used a Fizeau interferometric fiber optic sensor placed near the free end. Its extremely high sensitivity and resolution are its outstanding features, yielding a non-invasive sensor that ensures natural evolution and distortionless amplitude and frequency measurements. Results show that liquid resin in the slot did not produce changes on beam resonance prior to curing. On the other hand, photo-polymerization partially recovered original properties of the beam in a few tens of seconds, suggesting that vitrification of resins is completely achieved while photoreaction is still occurring. Moreover, additional information of volumetric shrinkage of polymers can be extracted from these measurements. In summary, this powerful and simple technique enables to evaluate the static resonance of beams as well as polymer shrinkage and solidification time evolution in one single measurement.

1. Introduction

Devices based on cantilever beams have an enormous number of applications; as transducers they can be employed for mass, temperature and inertia measurements. In the automotive industry they are used in mems development and as accelerometers, filters and resonators for telecommunication and, in scientific area, they are mainly employed in atomic force microscopy (AFM) [1,2].

Fundamental frequency for a solid beam with one fixed point and uniformly distributed weight (including beam weight itself) can be calculated as [3]

$$f_0 = \frac{3.52}{2\pi}\sqrt{\frac{EI}{\delta l^4}} \tag{1}$$

with $\boldsymbol{E}$ being elasticity modulus, $\boldsymbol{I}$ the areal moment of inertia, $\boldsymbol{\delta}$ and $\boldsymbol{l}$ the mass per unit length and beam length respectively. For a given aluminum beam sized of 12.2 cm length this frequency must be equal to 385.3 Hz.

It is widely known that any change in physical parameters will modify its natural frequency [4–9]; indeed, this technique is already used extensively for predictive control, and monitoring big structures such as bridges, buildings and dams.

Several methods have been utilized for the resonance measurements of beams: strain-gauges, laser interferometry, high speed imaging processing, and even fiber optic Bragg gratins (FBG) [7,10,11]. In the last decade fiber optic sensors (FOS) have been undoubtedly positioned as one of the best alternatives in scientific and industrial applications because of their excellent performance and accurate results [12,13]. In particular, the Fizeau interferometer FOS is a kind of sensor that forms an interferometric cavity between its probe tip and a high reflective surface like aluminum, allowing distance measurements related with

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changing of its length cavity. Interferometric FOS provide a great versatility, reliable spatial resolution and, due to their non-invasive nature, allow free evolution of dynamic variables, such as amplitude and frequency resonance vibrations.

On the other hand, UV cured resins are widely used in decorative and protective coatings and magnetic and optical disks [14,15]. Moreover, photocured resin-based dental composites are commonly used for dental restoration because of their good properties and final appearance. However, contraction of the polymer-based materials yields a disadvantage for this application because the resulting shrinkage stresses can lead to failure of the interfacial bonds, which results in marginal leakage, premature failure of the restoration, and in some cases micro-cracking of the tooth [16]. Due to these reasons, studies of polymerization kinetics and processes related to UV curing are systematically performed in order to minimize this problem.

During the polymerization reaction, the viscous monomer matrix is transformed into a rigid material. Physical properties such as hardness and bulk volume of the polymerized composite are related to the conversion rate (quantity of reacted versus unreacted material) which depends on composition as well as on parameters. Knowledge about the polymerization evolution, including the solidification process of the material and stress build-up, is important with the aim of analyzing a complete model.

Numerous techniques have been employed for photocuring polymer studies. Most of them have measured shrinkage produced during the photoirradiation by using mercury dilatometers, density changes before and after polymerization, laser beam scanning, laser interferometry, etc. [17–23].

FOS have already shown promising results for monitoring stress build-up and shrinkage of dental cements [24]. Also, cantilever deflection was used, for example, to study stress development in UV curing of multifunctional acrylate and methacrylate coatings [25].

In this work, we monitored both the solidification and shrinkage temporal evolution of BisGMA resins by employing a cantilever beam excited periodically with a sweeping signal, while the photocuring reaction proceeds. To do this task, we have drilled a transverse deep crack near the fixed extreme of the beam. Under these conditions the natural frequency is expected to be higher than the measured frequency for the undamaged beam. This experimental fact is due to the weakening generated in the beam. Hence, for constant amplitude excitation, a lowering of the resonance frequency and overall stiffness must be exhibited while at the same time, higher resonance amplitude is produced. When the slot is filled with a sample material, and photopolymerization is carried out, beam properties would change with its original stiffness returning partially. By tracking vibration amplitude through curing evolution, it is possible to find out the time needed to achieve solidification of the resin.

In addition, polymeric shrinkage developed during photoreaction produces a deflection on the beam that reduces the cavity. This phenomenon may be employed as an alternative way for the evaluation of degree of resin contraction through amplification provided by the end of the bar [26,27]. This simple technique enables simultaneous evaluation of the temporal evolution of polymer shrinkage and solidification process as well as natural resonance frequency in one single measurement.

2. Materials and methods

A square shaped solid aluminum bar of side 0.94 cm and length 12.2 cm was used in cantilever configuration as shown in Fig. 1. A 5 mm deep crack was drilled with a leaf of 1 mm width near the fixed end of a cantilever. The beam oscillations were produced by means of a 10-mm-diameter, 25 turns, 0.2 mm gage copper wire coil, attached to the free end of the beam. The solenoid weight was negligible compared with the beam weight, and was placed close to a permanent magnet, in a way such that its magnetic field was uniform and constant in density compared with solenoid dimensions.

With this classic electromagnetic scheme, a proportional magnetic force will develop in the beam if the coil is excited with variable current signal. For this experiment, coil current was sinusoidal shaped and frequency was swept with a peak value of 300 mA. At any time, the current amplitude injected to the solenoid was kept constant and distortionless, in order to avoid spurious variations on all measures.

The oscillation amplitude generated with this arrangement resulted to be about few tenths of microns, and was detected easily by means of Fizeau interferometer FOS fixed at 1.4 cm from the free end. The distance of the probe tip from the beam was about 2 ± 0.2 mm. Beam geometry and Fizeau FOS placement are shown in Fig. 2, together with additional relevant data. Detailed analysis of Fizeau sensor performance and signal post-processing are described in more detail in Refs. [26–28].

To perform resonance measurement of beams, the signal generator (HP33120A) was tuned manually until maximum amplitude was verified in all cases, and in each frequency step its corresponding acquisition data for posterior processing was acquired.

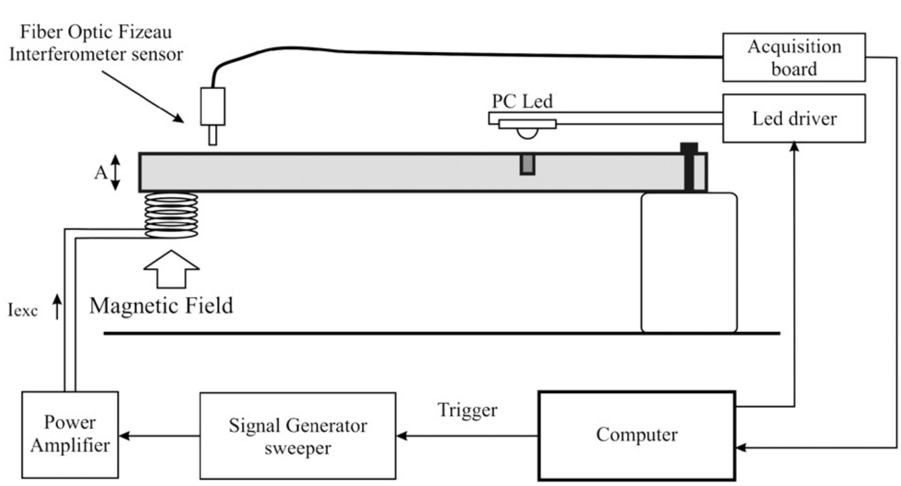

Fig. 1. Vibration measurement setup.

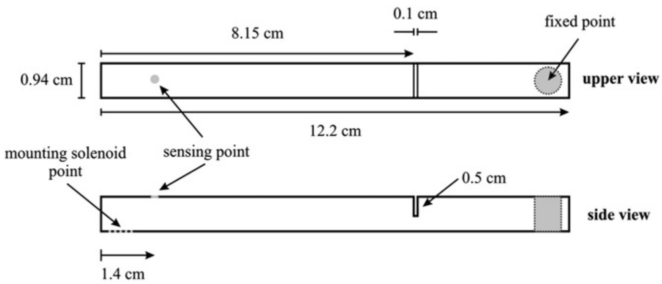

Fig. 2. Geometrical details of the cantilever beam.

For tracking resonance changes, frequency sweepings were made with the same signal generator by varying the frequency of the sinusoidal solenoid current signal periodically. In order to synchronize every acquisition per sweeping, a personal computer (PC) was configured as the master controller of all events. Frequency sweeping was programmed from 200 to 500 Hz, at the linear mode and with a time sweeping of 4 s (75 Hz/s).

This excitation scheme provides a single and feasible method to allocate frequency resonance dynamically with its time evolution. By following the resonance peak as the curing reaction proceeds, it is possible to determine the time at which the solidification state is reached. Quality factor of the beam is relevant; indeed the entire technique depends on it. However, a higher value could have a negative impact on measurements, because excessive oscillation amplitude would require a longer time to vanish, widening the system response.

For this work, BISGMA/TEGDMA resin was used with 1% camphorquinone (CQ) and dimethylaminoethylmethacrylate (DMAEMA) used as the photoinitiator system. Light curing was carried out by 10 min of continuous irradiation with a high power LED centered at 465 nm (LUXEON LUMILED–3 W), controlled with a regulated current source (LD500–Thorlabs) at 500 mA. Maximum optical density delivered to sample resins was approximately 30 mW/cm^2 measured with a calibrated silicon power meter (Ophir PD200). Sensor probe was aligned orthogonally to the beam and centered with respect to beam width, in order to minimize light modulation issues [29]. The electrical signal proportional to interference pattern was amplified and acquired on a PC data acquisition board with software specifically written to obtain maximum versatility and performance. Post-processing of measurements simply consisted in the allocation of peaks within every scanning cycle, to find out the corresponding resonance frequency.

3. Experimental results

Natural resonances of beams under different conditions are discussed firstly. As illustrated in Fig. 3, whether the deep crack drilled in the cantilever is empty or filled with uncured resin, it has the same response to excitation. Although this may be regarded as an obvious conclusion, it ensures that liquid state resin does not modify mechanical properties of the drilled beam. As expected, fully cured resin shows that natural resonance of beams is returned very closely to the undamaged beam case. Both experimental facts jointly with the lowered amplitude of resonance peak suggest that solidification of resin within the deep crack partially restores the effect of the damage.

These plots of resonance were build processing interferograms for every single excitation frequency. One of these typical signals is shown in Fig. 4. Note that the number of interference fringes per cycle gives the amplitude of each vibration and the period makes it possible to know the corresponding frequency.

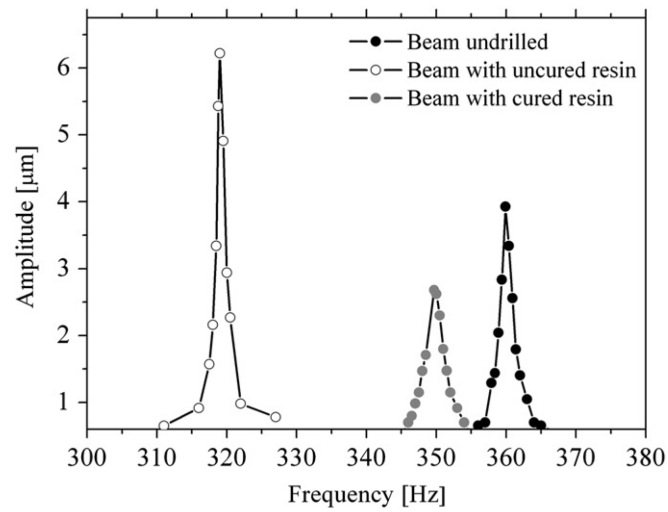

Fig. 3. Resonance curves for undamaged beam (black solid circles), fully filled uncured resin (clear circles), and beam with cured resin (gray solid circles). Noticeable changes are seen between different states. Specifically the restoration to original condition is remarkable (slightly better than 75%).

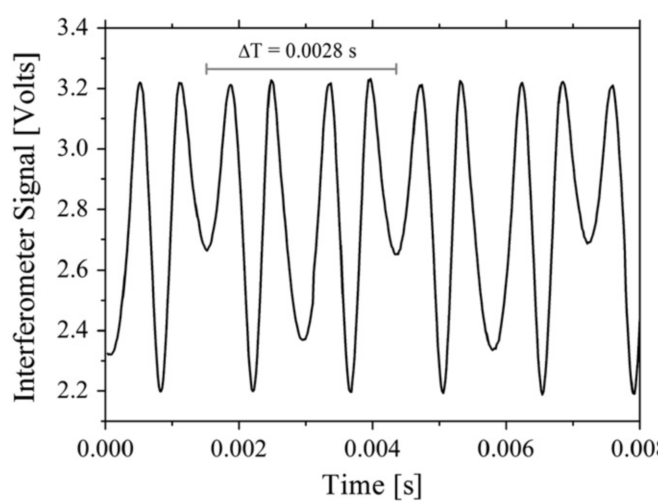

Fig. 4. Interferogram signal corresponding to oscillation of the beam with uncured resin excited at nearly 328 Hz.

Fig. 5 explains graphically the tracking method designed for this work. The saw-tooth-like signal in (b) shows the variation profile of the frequency rate of the sinusoidal excitation current injected to the solenoid. This is the way beams are forced to oscillate periodically through curing time. The upper part (a) is the output signal from the Fizeau FOS that reveals clearly the resonance condition. As can be seen, the passage by the natural resonance is certainly noticeable. Considering dimensions and properties of employed beams, no phase shift occurs between vibration amplitudes and excitation frequencies. Thus, once the 4-s time window is established, the frequency at which the peak appears is simply calculated from the sweeping ramp.

The resonance evolution of the beam based on precedent comments can be seen in Fig. 7. This curve shows how resonance develops from nearly 320 Hz to 350 Hz on cantilever beam, whereas the photocuring reaction is performed, according to values presented in Fig. 3.

Analyzing the complete interferometric curve in Fig. 6, it is possible to recognize a complex signal formed by the superposition of two main phenomena: on one hand, all consecutive resonance measurement is due to frequency scanning. This signal

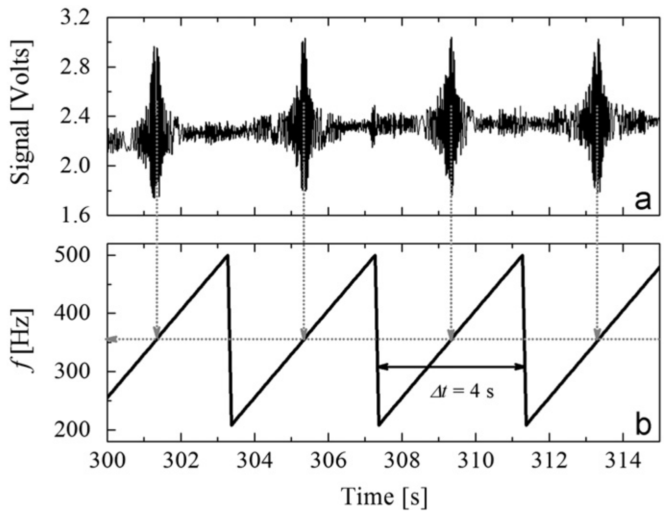

Fig. 5. Details of the sweeping technique: (a) signal evolution through experimental time windows; and (b) frequency sweeping versus time.

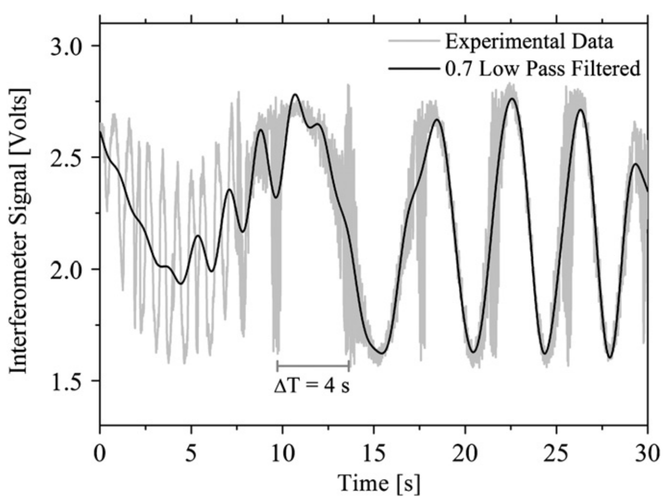

Fig. 6. Details of the full sensor signal with a low pass filtered version (black solid line) to separate the effect of passage through the resonance (high frequency signal of 4 s period). Sensor located at 1.4 cm from the free end.

of higher frequency has a period of 4 s and when properly treated gives rise to the resonance curve as a function of time.

On the other hand, a very low frequency term (solid black line in Fig. 6) is another interferogram caused by the approach of the beam to the sensor probe tip, as a consequence of volumetric shrinkage of resin into the deep crack, that tends to bend the bar progressively reducing the interference cavity. More details are shown in Fig. 8. Moreover, polymeric shrinkage curve obtained from this low frequency term processing agrees with those obtained with our Fizeau FOS with different experimental arrangements [26]. However, to prove this, another simple experiment was conducted by curing the same resin sample, under the same experimental conditions, but without the frequency sweeping signal. The resulting plot for this measurement can be seen in Fig. 9, which obviously does not contain the high frequency component characteristic of the resonance passage. A comparison between this experiment and the standard one with resonance scanning is consistent and the result is displayed in Fig. 10. This latter effect must not be taken as a simple observation; it denotes a typical shrinkage profile that is in

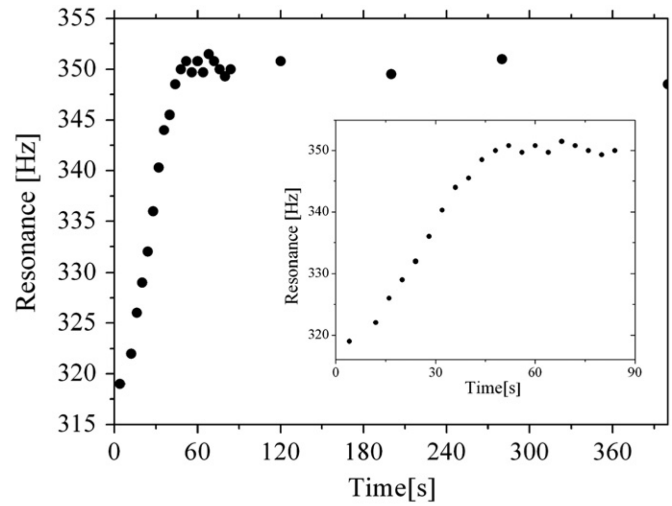

Fig. 7. Resonance frequency evolution for a typical curing reaction. Inset shows a detail of the same curve for the first 90 s, where most of the change occurs.

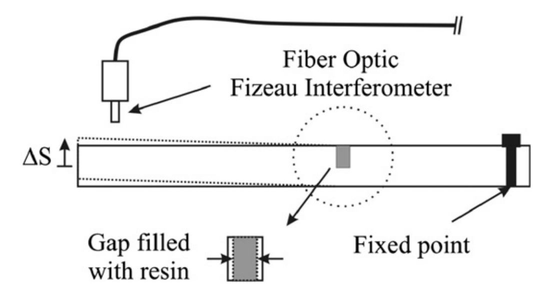

Fig. 8. Deflection mechanically amplified by cantilever beam length due to resin volumetric shrinkage in the deep crack.

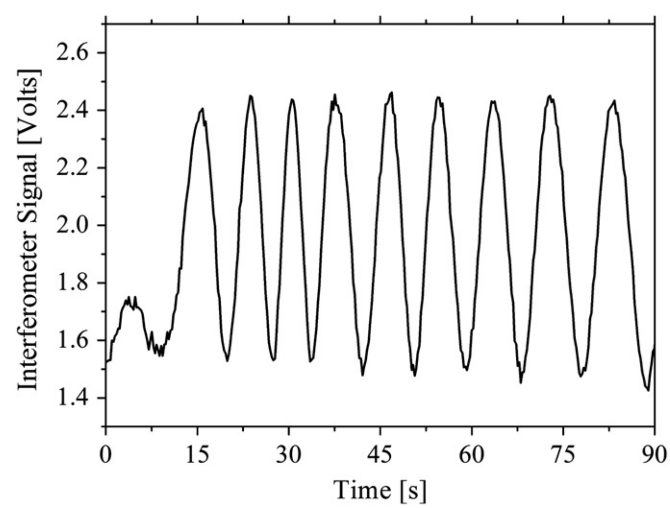

Fig. 9. Interferogram corresponding to curing without sweeping frequency, denoting the bending effect on the beam due to volumetric shrinkage inside deep crack.

agreement with previously published results obtained using other techniques [25].

Finally, to illustrate the ability of this method, Fig. 11 shows complimentary results obtained from a cantilever beam with the same geometry, same electromagnetic excitation scheme, same resin, but with 6 cm length. For this particular case, the resonance frequency change varies from 950 Hz for the uncured resin to about 1100 Hz for cured resin. Previous resonance frequency of

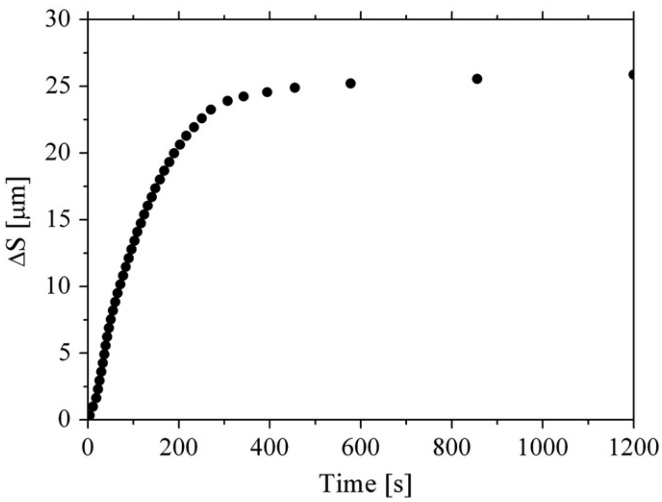

Fig. 10. Resin contraction effect, measured from experimental data shown in Fig. 9.

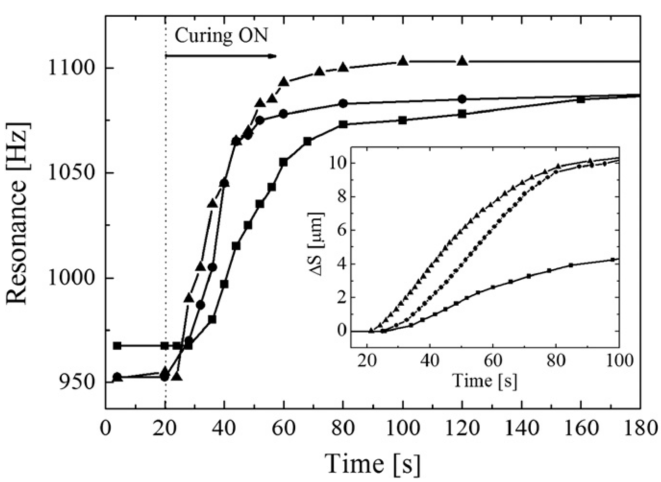

Fig. 11. Another case in which resonance was measured for the same resin formulation but irradiated at 100% (triangles), 50% (circles) and 20% (squares) of the maximum power curing source. In this case, a cantilever of 6 cm length was used with a similar excitation scheme. Inset: resin contraction and its effect of bending measured at 0.7 cm from the free end at 100%, 50%, and 20% of maximum power employed in this work, derived by processing the low frequency signal of the entire interferograms shown in the main graph.

undamaged beam was 1213 Hz. In addition, the dependence of curing light source power applied to resin was assessed, and it was 100%, 50% and 20% of the maximum power allowable. As expected, no significant differences were registered as the power intensity was varied. In contrast, there is some dependence on cantilever bending effect because photocuring reaction status is related with volumetric shrinkage. It may be clear that this bending effect on the beam will depend on this parameter.

On the other hand, solidification process in these kinds of resins takes no longer than a minute regardless of polymerization dynamics and can be deduced indirectly from the cantilever bending (ΔS). This is an important fact, because vitrification of resin into the deep crack does not change significantly with power curing light intensity.

4. Conclusions

In this work, we presented a novel application that employed a cantilever beam forced into oscillations measured by means of a Fizeau FOS and vibration analysis techniques. Liquid to solid-state evolution of photocuring materials placed in the deep crack made in beams can be analyzed. From experimental results, it has been found that the curing process of the resin restores partially the effect of the damage and original elastic properties of the beam. Thermal influence, occurring jointly within the curing process itself and demonstrated before [30], appears to be negligible in this proposed scheme due to the great cooling provided by aluminum beam. In this way, heating processes can be reduced and so better shrinkage estimation can be done, because the observable phenomenon is the total contraction without distortion, amplified by the length of the bar. In turn, the frequency shift (related to the phase changes produced in the resin), along with evolution as a function of time reveals that the rigidity is reached before the entire curing of the resin proceeds. In addition, a nearly stable resonance frequency is reached for 20% of the total curing time. This simple technique allows for the measurement of the solidification state as a function of time, static resonance frequencies and polymerization shrinkage degree of resins deduced indirectly from the cantilever bending of the tip in one single experiment.

Acknowledgments

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