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Monitoring of solidification induced strains in two resins used in photofabrication

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ABSTRACT

In this work, measurements of in situ solidification and post-fabrication strains in two commercial stereolithography photopolymers are presented. The photopolymers examined in the present investigation are a multifunctional acrylate-based resin and an organic–inorganic polymer known as ORMOCER®, which have been used in single and two-photon stereolithography, respectively. The resulted solidification strain build up is monitored via the use of an embedded Fibre Bragg grating (FBG) sensor during photopolymers stepwise exposure to UV-light irradiation. Analysis of the recorded spectra demonstrate the development of high consolidation strains in both UV cured photopolymers as well as a significant increase of their magnitude after post-fabrication thermal treatment at 75 °C. Based on the strain data from the utilized optical sensor, information on the degree of cure for one of the two photopolymers is also provided.

1. Introduction

Photofabrication of functional components via the utilisation of advanced rapid prototyping systems has comprised a highly challenging task for the past decade. The technologies employed for the creation of such entities include techniques like stereolithography, three-dimensional printing, direct deposition etc. Concurrent technological evolution of micro-fabrication systems as well as custom engineered materials has lead research work into a whole new range of topics and applications [1,2]. Many, well documented [3,4], approaches utilising the aforementioned technologies rely upon the utilisation of prototype multifunctional materials within the context of specially developed rapid fabrication system apparatuses [5–10].

In UV-activated polymerisation techniques the photopolymer absorbs energy by selective energy exposure. Processing parameters such laser power and scanning speed during fabrication, resin characteristics such as resin formulations, photosensitivity rate and degree of cure as well as followed post-cure methods are very important in the final properties of the built structures. During photo-polymerisation the polymer shrinks upon changing from liquid to solid. This results to cure-induced strains, which cause internal stresses, in the cured material. Volume shrinkage of the resin is one of the major causes leading to poor accuracy of the fabricated parts [11]. Several studies have been reported aiming at investigating the curing characteristics of photopolymers used in classical lithographic processes [12,13]. For instance, Salmoria et al. [14] have investigated the cure process and thermal degradation of a liquid and cured photopolymer used in the stereolithography process. On the other hand, few works have been reported on the UV based solidification phenomena of micro-fabrication suitable photopolymers. Nguyen et al. [5] have investigated the rate of polymerisation and conversion at different temperatures and intensities of UV light of an acrylate-based micro-fabrication photopolymer using Photo Differential Scanning Calorimetry (PDSC). Furthermore, Lee and Cho [15] have reported on the micro-stereolithography photopolymer solidification patterns for various laser beam exposure conditions.

Experimental techniques capable of monitoring the curing shrinkage and mechanical changes during the polymerisation process are increasingly attracting attention. In recent years, the use of optical Fibre Bragg grating (FBG) sensors has been considered as one of the most effective methods for in-process monitoring, as well as post-process determination of the micro curing strain state induced to the material. Apart from being inexpensive and electromagnetic interference resistant, fibre–optic sensors are extremely small possessing unique embedding characteristics in relation to a variety of host polymeric as well as composite materials. The fact that the mechanical behaviour of the glass fibre that contains the grating is not affected, leads to the conclusion that the technique is well suited for the determination of non-uniform axial stains development due to the shrinkage of the resin during polymerisation. Scientific works have been reported that use Fibre Bragg grating (FBG) sensors for simultaneous monitoring of the fabrication strain and the cure temperature in epoxy resins and fibre reinforced composites [16–18].

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This work investigates the photopolymerisation induced shrinkage strains in two photopolymers commonly used in micro-fabrication. A fibre–optic sensor is utilized for the effective determination of the resulted shrinkage strains throughout the consolidation process upon exposure to UV light of specific irradiation intensity. This paper reports on the experimental results of the recorded FBG spectra and the evolution of cure-induced shrinkage strains at the end of selected UV exposure times and after an applied thermal cycle. Finally, the FBG wavelength data were used to calculate the resulted degree of conversion allowing a better understanding of the polymerisation efficiency of one of the two photopolymers during the applied light exposure.

2. Photocurable resins employed

The first resin was the acrylate-based photopolymer by Micro Resist Technology [19] which has been widely used in the past for single photon micro-stereolithography [20,21]. The material's main three components are a sensitizer dye, an amine co-initiator and a multifunctional acrylate monomer. This specific material has been customized in order to achieve high performance in optical applications and demonstrates high transparency as well in the visible and near infrared bandwidth [6].

The second resin is the ORMOCER® – Ormocomp by Micro Resist Technology [19]. In general, ORMOCERs® consists of a highly crosslinkable organic network [22] as well as inorganic components which lead to high optical quality. Both resins have been used in two-photon polymerisation [5,10]. Several studies and experimental works [4] have been conducted incorporating ORMOCER® leading to the creation of a variety of components and structures. It has to be noted that the material's biocompatibility in addition to its polymerisation effectiveness in the micro-scale contribute towards the continuous extension of micro as well as nano-scale research including applications in the medical sector [23].

3. Fibre Bragg grating principles

Fibre Bragg gratings are specific regions along the length of an optical fibre's core where spatial periodic (or quasi-periodic) modulation of the refractive index has been conducted [24]. The basic principle of operation of the FBG sensor is based upon the refractive signal's (centre wavelength λ_B of the back-reflected light from the Bragg grating) changes which depend upon the core's effective refractive index (n_{eff}) and the periodicity (Λ) of the grating [25]. This means that Fibre Bragg gratings operate as wavelength selective filters [26]. Their main property is to reflect light transmitted from a broadband source over a narrow wavelength range and at the same time transmit without changing the remaining wavelengths giving a local steep reduction in the transmitted spectrum. Therefore, the Bragg wavelength is expressed by the following formula:

$$\lambda_B = 2n_{eff}\Lambda \tag{1}$$

In principle, the spectral response of an FBG is a function of temperature and strain variations due to the fact that the effective refractive index and optical fibre physical dimensions are affected and therefore subjected to change [27]. The Bragg wavelength shift ($\Delta\lambda_B$) caused by the change of strain ($\Delta\varepsilon$) and change of temperature (ΔT) can be expressed as:

$$\frac{\lambda_B - \lambda_{B0}}{\lambda_{B0}} = \frac{\Delta\lambda_B}{\lambda_{B0}} = (1-p_e)\varepsilon_Z + (\alpha_f + \xi)\Delta T \tag{2}$$

where, λ_{B0} is the Bragg wavelength at a reference state, p_e the strain–optic coefficient of the optical fibre, α_f is the thermal expansion coefficient of the fibre and ξ is the thermo-optic coefficient of the fibre, respectively. A value of $p_e = 0.22$ is reported in the literature [27], determined experimentally by mounting the Bragg grating on a cantilever beam and applying a known loading to the beam while measuring the shift in the Bragg wavelength.

When the FBG sensor in embedded into a host material and both experience temperature changes, the Bragg equation is modified to account for the thermally induced axial strains in the fibre as a result of the mismatch in the coefficients of thermal expansion between the optical fibre (α_f) and the host material (α_m). Therefore, a more generalized equation can be written as:

$$\frac{\lambda_B - \lambda_{B0}}{\lambda_{B0}} = \frac{\Delta\lambda_B}{\lambda_{B0}} = (1-p_e)\varepsilon_z + (1-p_e)(\alpha_m - \alpha_f)\Delta T + (\alpha_f + \xi)\Delta T \tag{3}$$

In the case where the embedded sensor and the host material are not subjected to any temperature variations, Eq. (3) can be further simplified to the following form:

$$\frac{\lambda_B - \lambda_{B0}}{\lambda_{B0}} = \frac{\Delta\lambda_B}{\lambda_{B0}} = (1-p_e)\varepsilon_z \tag{4}$$

Several studies have been reported on the determination of non-uniform strain along a FBG [17,26,28,29]. In order for the local Bragg wavelength distribution $\lambda_B(z)$ to be expressed as a function of the position, (z), along the grating length, a novel Optical Low-Coherence Reflectometry (OLCR) technique has been developed at Ecole Polytechnique Fédéral de Lausanne (EPFL). With this method, it is possible to detect the amplitude and phase of reflected light the FBG sensor's individual gratings. Subsequently, according to the coupled-mode theory, light that propagates through a FBG can be described by coupling between the forward-propagation fundamental guided mode and its backward-propagating counterpart [26,29]. This in effect leads to the introduction of an unique coupling coefficient $q(z)$ which can be derived by the experimental data. Using an inverse scattering algorithm, the phase of the coupling coefficient is determined as a function of position z along the grating. Thus, the local Bragg wavelength $\lambda_B(z)$ can be easily determined without any assumptions on the strain profile. The exact working principles of the technique are well documented elsewhere [30]. Consequently, the complete strain distribution profile along the embedded sensor can be calculated and localized area results within the specimen can be extracted leading to more precise conclusions. When a new optical period distribution, $\lambda_B(z)$, is known, then the corresponding strains can be calculated from Eq. (4) which takes the following form:

$$\frac{\lambda_B(z) - \lambda_{B0}(z)}{\lambda_{B0}(z)} = \frac{\Delta\lambda_B(z)}{\lambda_{B0}(z)} = (1-p_e)\varepsilon_Z(z) \tag{5}$$

4. Experimental details

4.1. Experimental set-up

Specimens of 10 mm × 10 mm × 40 mm (width × height × length) were fabricated at room temperature in a custom made aluminum mould, where its open top surface was exposed to UV radiation (Fig. 1). A specimen length of 40 mm was chosen because in a previous work [17] regarding the embedment of FBG sensors into cylindrical epoxy specimens it is reported that a specimen length of 40 mm is sufficient enough to lead to constant value of strain in a relatively long interval of the specimen's central region. It is also reported that in smaller in length specimens, the strain plateau decreases significantly or does not appear at all. The mould cavity was coated with a thin layer of liquid release agent in order to minimize bonding of the solidified photopolymer to its surfaces.