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Modelling the effect of moisture on the depth sensing indentation response of a stereolithography polymer

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ABSTRACT

Stereolithography (SL) resins are highly hygroscopic and their mechanical properties are significantly affected by the level of moisture in the environment. In addition, the load response of these materials is highly time dependent, hence, an appropriate rate dependent constitutive model is required to characterise their mechanical behaviour. In this work, the time dependent mechanical behaviour of an SL resin is investigated under varying humidity conditions using depth sensing indentation (DSI) tests. In the experimental study, a DSI system fitted with a humidity control unit was used to explore the influence of moisture on the mechanical properties of a SL resin. Samples were tested with 33.5%, 53.8%, 75.3%, 84.5% relative humidity (RH) inside the chamber while the temperature was kept constant at 22.5 °C. It was seen that hardness and modulus decreased with increasing absorbed moisture in the resin. Material parameters obtained through bulk tests were used to develop a coupled stress-diffusion finite element model incorporating rate dependent material behaviour. It is proposed that this model can be used in predicting the effect of the environment on the performance of SL manufactured components.

1. Introduction

SL resins can be used to manufacture parts using an approach to manufacturing called rapid manufacturing (RM) or additive manufacturing (AM) [1]. The SL process is one of the main processes of AM for polymers and is considered highly accurate and consistent [2]. The materials used in the SL process are termed photo-polymers because they are cured using ultra-violet (UV) light [3]. The majority of these SL resins are thermosetting polymers, such as epoxies and acrylates, with the addition of a photo-initiator. Currently, the SL process has limited use for producing end-use parts owing to the instability of SL materials at high levels of relative humidity and long term UV degradation [4]. Hence, in order to increase the applications of SL as a manufacturing process, materials more suited to a wide variety of end-use applications must be developed. One of the material aspects that require significant development is the environmental stability of the SL materials post-build.

Moisture absorption in polymers leads to a range of effects, such as plasticization by weakening of the intermolecular interactions among the functional groups of the chains [5,6], de-bonding at filler–matrix interfaces [7–9], structural damage, such as micro-cavities or crazes [10,11], and chemical degradation of the polymer matrix due to hydrolysis and oxidation [10–12]. It can also involve the generation of free radicals or other reactive species which may act as plasticizers or reactants [13,14]. Long-term exposure can involve a decrease in the molecular weight due to chain scission or the breaking of cross-links in the polymer network [15]. Absorbed moisture significantly affects the mechanical properties and glass transition temperature (T_g) of polymeric materials [6,7,9,16,17]. The changes in mechanical property of polymeric components due to moisture absorption can be related to their performance and can be scrutinized by performing tensile tests, shear tests, micro-hardness tests, DSI tests, etc. on samples after moisture conditioning.

SL parts exhibit spatial variations in properties which can be evaluated by using DSI tests [18]. DSI tests produce quantitative measurements of modulus and hardness with nanoscale spatial resolution by monitoring load and depth during indentation with a sharp or blunt indenter [19]. However, the drawback is in interpreting the data, which is complicated by the complex loading conditions and in polymers moisture absorption and time dependent material response add further complexity.

There have been a few studies on the nanoindentation of polymers in a fully immersed environment [20–24], using a liquid cell but, to date, no work has been reported where the effect of varying RH is investigated. In the present work, the time dependent mechanical behaviour of a SL resin is investigated under varying humidity conditions by using a humidity control unit (HCU) to control the environment in a DSI machine. The spatial variations in properties were investigated by testing at different locations

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and depths. Finite element analysis was performed to simulate nanoindentation under varying relative humidities. In order to extract material properties for use in numerical modelling, bulk mechanical tests and moisture absorption tests were performed. A time dependent coupled stress-diffusion model is proposed that can be beneficial in predicting the effect of the environment on the performance of SL manufactured components. The schematic of this research work is summarised in Fig. 1. Details of the experiments and FEA are discussed in Sections 2 and 3 respectively.

2. Experimental methods

2.1. Material preparation

The polymer investigated in the present study is an epoxy based resin, Accura 60, manufactured by 3D Systems (Rock Hill, SC, USA). The samples were manufactured in a flat orientation using an SLA7000 SL machine, also from 3D Systems, with 4 mm thickness. Thereafter, the samples were washed in the chemical solvent tri-propylene glycol monomethyl ether (TPM) and cleaned with methanol. Finally, UV light post-curing was employed for 30 min to stabilise and improve the mechanical properties of the samples. After being subjected to the post manufacture treatments, all the samples were stored in darkness in a dessicator for 20 days to ensure stability before testing.

2.2. Types of experiment

As shown in Fig. 1, three types of experiment were carried out. DSI tests were used to investigate spatially resolved material properties. Gravimetric tests were performed to characterise the moisture uptake behaviour and hence calculate the diffusion constants for use in the FEA. Similarly, bulk tensile and compressive tests and creep tensile tests were carried out to generate the mechanical material properties for use in the FEA.

2.2.1. DSI tests

DSI tests were performed on $50 \times 50 \times 4$ mm samples under 33.5%, 53.8%, 75.3% and 84.5% RH. The NanoTest 600, manufactured by Micro Materials (Wrexham, UK), was used for the experiments. A Berkovich indenter with face angle of 65.3°, giving the same projected area to depth ratio as the Vickers indenter, was used to produce indents. The conditioning of samples inside the machine chamber was carried out by regulating the humidity via a HCU. This unit consists of an ultrasonic humidifier and dehydration unit, which together can be used to set different RH values. The indentation tests were carried out every 24 h for 5 days under these environments. In DSI tests on polymers, many researchers have found a bulge or "nose" effect during the initial portion of unloading as a result of creep, which can lead to errors in the calculation of contact stiffness and contact depth [25–28]. This effect can be minimized in different ways. Most often a dwell/holding time is introduced between the loading and unloading phases [29]. In our present work we have introduced a 300 s dwell time. To compare quantitatively, experimental parameters were kept the same during all the experiments, with 0.5 mN/s loading and unloading rates and a 20 mN maximum load. Five indentations were made 150 μm apart, and the average values of hardness and modulus were calculated using the Oliver and Pharr (OP) method [30]. In this method the initial unloading curve is approximated by a power law, as given in the following equation:

$$P = \alpha(h - h_r)^m \quad (1)$$

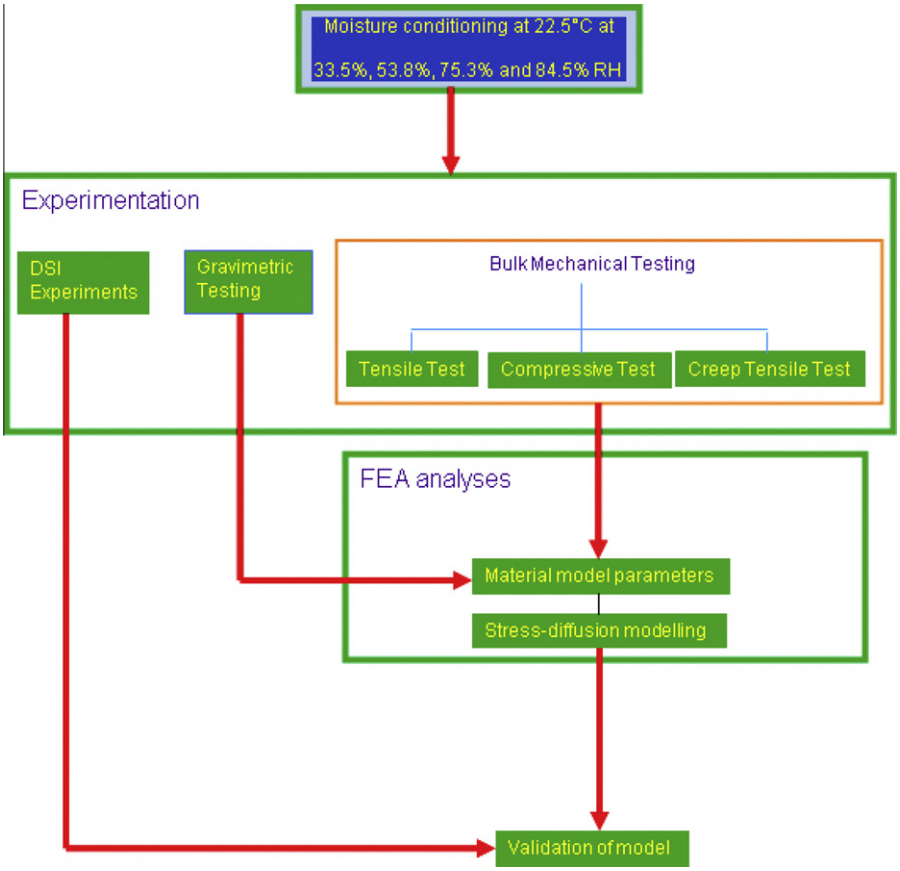

Fig. 1. Schematic of methodology of research.