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Environmental Effects on the Dimensions of SL5195 resin

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Abstract

The effect of humidity, temperature and temperature ramping rate on the dimensional changes of the photo-cured Stereolithography resin SL5195 was investigated. It was found that moisture absorption by the SL resin is a very slow process at the ambient temperatures. Varying relative humidity (RH) between 20% and 90% in the environment only produced slight changes in the sample dimensions during the time period investigated. Increasing environment temperature caused a significant increase in the sample dimensions through thermal expansion along with accelerated moisture absorption at 50% or higher RH. Increasing the temperature ramping rate reduces the moisture absorption during the thermal cycles.

Keywords: Resin, Water Absorption, Rapid prototyping, Stereolithography

Introduction

Stereolithography (SL) is one of the most widely utilized rapid prototyping (RP) processes. SL parts have not only been used as prototype models, but also found increased uses in various rapid tooling (RT) applications. They can be used as injection moulds directly (Hilton and Jacobs, 2000) or after a thin layer of metals being deposited on to the parts (Yeung et al., 1996). In the KeltoolTM (Phelan, 1997) process, the SL parts are used as master patterns for the production of solid metal tools. The SL parts have also been used as the patterns for investment casting (Dzugan and Yancey, 1997) and ceramic mould casting (Liu et al., 2001) for the fabrication of solid metal parts. In some of the RT applications, such as in the KeltoolTM and ceramic mould casting processes, the SL parts only encounter ambient temperatures. For the injection mould applications, on the other hand, the SL parts are exposed to different thermal cycles. The Quickcast patterns in investment casting process have to be heated to high temperatures and burned off to form the casting mould cavity. Such temperature changes will significantly affect the dimensions of the SL parts, and hence the accuracy and functionality of the moulds or parts derived from these parts. Furthermore, it has long been known that industrial plastics, in particular the epoxy-based polymers, tend to absorb water from the environment under high humidity conditions or in water (Allen, 1979; Brewis et al., 1983; Apicella et al., 1981a; Apicella et al., 1981b; Apicella et al., 1982; Fedors 1980; Gardner and Martin, 1980; Martin and Gardner, 1981; Woodham and Pinkston, 1970). Excessive water absorption by polymers can cause severe degradation of the

mechanical properties and also lead to dimensional changes in the polymers or the polymer based composites (Apicella et al., 1982; Brewis et al., 1983; Gardner and Martin, 1980; Martin and Gardner, 1981; Woodham and Pinkston, 1970). Recently, some research work has also been conducted on the effect of humidity on the properties of the photo-cured resins under different humidity environments (Peng and Wimpenny 1996; Winmill et al., 1998). Peng et al. (1996) reported that the prolonged exposure of the SL5180 resin to a high humidity environment at the ambient temperature caused significant degradations in tensile strength, accompanied by gains in weight and dimensions. Winmill et al. (1998) investigated the water absorption behaviour of the Somos[®] 7110 and Somos[®] 8110 photo-cured resins under different humidity environments at 21 °C. Their results also demonstrated noticeable dimensional and weight changes for the specimens soaked in water for 7 weeks. The combination of different temperature and humidity conditions encountered by the SL parts in various rapid tooling applications mentioned above could result in pronounced dimensional changes that could in turn have significant impacts on the accuracy and functionality of the tools or parts produced from the SL parts. A clear understanding of the responses of the cured SL resin to various environmental conditions is critical for the proper use and handling of the SL parts. In the current study, the dimensional changes of the SL5195 photo-cured resin under different temperature and humidity conditions were investigated.

Experimental

The samples used were coupons made of SL5195 photo-cured epoxy resin and fabricated using an SLA5000 machine. The coupons have a rectangular cross section with dimensions of 150 mm in length, 12.5 mm in thickness and 25.4 mm in width. The experiments were conducted in an environmental chamber with controlled humidity and temperature. The experimental set up is schematically shown in Figure 1. The dimensional changes over time under different environmental conditions were continuously recorded using a data acquisition system. For the investigation of the effects of temperature on the dimensional changes, the samples were held at 40 °C, 60 °C, 80 °C, and 100 °C for 24 hours with the relative humidity in the environment being fixed at 50%. The temperature ramping rate was 50 °C/hr for both heating up and cooling down stages. The effect of the temperature ramping rate was also investigated by heating the samples from ambient temperature to 100 °C from 5 °C/hr to 200 °C/hr. The samples were held at 100 °C for 2 hours and then cooled down to 20 °C at the same rate as in the heating up stage. The humidity of the environment was kept at 50% RH during the whole thermal cycle. The effect of humidity level was studied by varying the relative humidity of the environment from 20% RH to 90% RH in a number of steps at 20 °C and 30 °C. In addition, a comparison of dimensional changes was also made at 100 °C at 50% and 90% RH. Before the tests, the samples were kept at ambient environment for 3-4 weeks with a

temperature of 23 °C and relative humidity around 50%. For each test, the samples were first conditioned at 20 °C for 24 hrs for the sample to reach thermal equilibrium with the environment in the environmental chamber. Weight changes were obtained by subtracting the final weight of the samples at the end of each thermal cycle from the initial weight. Relative displacement of the coupons is calculated by dividing the length change at any given time with the initial length.

Take in Figure 1.

Results

Effect of temperature on dimensional changes

The dimensional changes of the specimens during thermal cycles with different soaking temperatures are shown in Figure 2. The relative displacements of all the specimens increased rapidly with increasing temperature at the heating up stage, mainly due to the thermal expansion of the SL resins. A reverse dimensional change was observed on the cooling down stage of the curves for the same reason. At the soaking temperatures, the overall dimensions expanded with soaking time, but the rate and the magnitude of the changes varies with the soaking temperature. At 40 °C and 60 °C, the length of the specimens reached a constant value at first, and then increased very slowly over time. At 80 °C and 100 °C, the length of the specimens increased constantly with time, and higher temperature produced faster rate of dimensional increases. After being cooled down to the ambient temperature, the samples soaked at 40 °C returned to the

original dimensions, while the samples exposed to 60 °C, 80 °C and 100 °C showed a retained increase in length.

Take in Figures 2&3

Figure 3 shows the total changes of the relative displacement occurred at the soaking temperatures alone and the net changes of the relative displacement after the samples are cooled down from the soaking temperature to 20 °C. Soaking at 40 °C for 24 hours produced little changes in net length and in the total displacement at the soaking temperature. When the soaking temperature is increased further, both the total displacement at the soaking temperature and the net displacement at the end of each thermal cycle increased linearly with the soaking temperature. Figure 4 shows the residual weight changes of the samples as a function of soaking temperature. When the soaking temperature increased from 40 °C to 100 °C, the residual weight change of the samples also increased linearly. The increase in weight is directly attributed to the absorption of water by the samples. The total amount of moisture absorbed and entrapped in the samples is strongly affected by the temperature of the surrounding environment. Clearly, when the accuracy of the SL parts needs to be maintained, the parts have to be stored and handled below 40 °C.

Take in Figures 4&5

Effect of humidity level

The dimensional changes of the SL specimens as a function of humidity level and time at 20 °C and 30 °C are shown in Figure 5. At both temperatures, the samples showed relatively large initial changes in dimensions during the conditioning stage. At 20 °C, the initial relative displacement was about -0.03%. This was caused primarily by the thermal contraction as a result of the temperature drop from the ambient temperature 23 °C to 20 °C, and was consistent with the amount of shrinkage calculated from the thermal expansion coefficient of $1.08 \times 10^{-4} \text{ mm}/(\text{mm} \cdot ^\circ\text{C})$ provided by 3D Systems and the 3 °C of temperature change. For similar reasons, the initial increase in dimensions for the specimen tested at 30 °C was the result of the slight temperature increase in the sample after it was placed into the environmental chamber. At 20 °C, however, the dimensional changes of the sample were hardly detectable when the humidity is varied from 20% RH to 90% RH and kept at each level for 8 hours. Only a very slight increase in dimensions could be observed at around 90% RH, and the dimensional changes were reversed when the humidity level was decreased from 90% RH to 20% RH. With prolonged holding for up to 7000 minutes at 20% RH, the dimension of the sample continue to decrease, also at a very slow rate, indicating the loss of moisture from the SL resin at the RH levels of 20% or lower. At 30 °C, the dimensional changes of the sample showed a similar trend. A very small peak was observed at 90% RH, and the dimensions returned to its initial levels when the humidity was decreased from 90% RH back to 20% RH. These results showed that, although moisture absorption does

occur at the ambient temperatures in a high humidity environment, at such temperatures the absorption process is slow and seems to be reversible.

Figure 6 shows the dimensional changes of the samples in different humidity environments during a thermal cycle in which the samples were heated from room temperature to 100 °C and held at the temperature for 2 hours before being cooled down to room temperature, with a heating and cooling rate of 50 °C /hr. By comparing the two curves for the samples tested at 50% and 90% RH, it can be observed that at temperatures below 50 °C, the different RH levels did not produce clear difference in the dimensional changes, and the two curves overlapped. At above 50 °C, however, the two curves departed from each other and the difference between the two became greater with increasing temperature. In addition, the dimensional changes occurred during soaking at 100 °C was 0.32% under 90% RH, almost 1.7 times as great as the 0.19% observed under 50% RH. As is also shown in Figure 8, the weight gain at 90% RH is 0.51 g, as compared with the 0.12 g at 50% RH. These results show that the moisture absorption of the SL resin depends on the RH levels, and temperature plays a critical role in the moisture absorption dynamics.

Take in Figures 6 &7

Effect of temperature ramping rate

The effect of temperature ramping rate in the thermal cycles on dimensional changes is shown in Figure 7. At any given temperature on the heating portion

of the curves, the total dimensional changes decreased with increasing temperature ramping rate from 5 °C/hr to 200 °C/hr. The reverse is true for the cooling portion of the curves. There are two factors that contributed to these results. The first one is the lack of temperature equilibrium between the sample and the environment. The second one is the total time the sample is soaked to the environment. Because the SL resins are poor thermal conductors, it takes time for the whole sample to reach the temperature equilibrium with the surrounding environment. During the heating up stage, the sample temperature will be lower than the environment or the set temperature. The degree of difference is dependent on the temperature ramping rate. Lower ramping rates allow the sample temperature to better approach that of the environment. As the ramping rate goes up, the available time for heat transfer is reduced and the temperature deviation between the sample and the environment will increase accordingly. At the heating up stage of the thermal cycle the temperature delay will be reflected as an apparent reduction in the dimensional changes as the temperature ramping rate goes up. This effect disappears once the temperature of the sample has caught up with that of the environment, and is not expected to lead to weight changes of the samples. On the other hand, higher ramping rate also gives the samples shorter time to expose to the high temperature environment. For example, the total ramping up and ramping down time between 20 °C to 100 °C will be 32 hours at a ramping rate of 5 °C/hr, while that for a ramping rate of 200 °C/hr will be only 0.8 hours. This kind of time difference affects the amount of moisture absorption and hence the final weight gain.

Figure 8 shows the residual weight and dimensional changes at the end of the thermal cycles as a function of the temperature ramping rate. Also included in the figure are the data for the samples tested at 90% RH and 50 °C/hr ramping rate. Both the residual weight gain and length changes decreased slightly with increasing ramping rate. This could be attributed to the different exposure time each sample had experienced as a result of the different ramping rate.

Take in Figures 8&9

Effect of multiple cycles

Figure 9 shows the dimensional changes of a specimen as a function of time while the sample was cycled between 20 °C and 100 °C for five times. Conditioning the sample at 20 °C and 50% RH for 72 hours before the cycles produced no detectable changes in sample dimensions. During the thermal cycles, the total displacement of the sample increased steadily after each cycle, reflected by the height increase of the peaks. The residual displacement at the end of each temperature cycle also increased gradually with the number of the cycles, indicating that more moisture absorption by the SL resin in each cycle.

Discussion

The process of water absorption by plastics involves the physical adsorption of water molecules on the surfaces, and migration of the water molecules into the body of the plastic materials. Diffusion is believed to be the mechanism for water

molecules migration because the process can be reasonably well described by the classical diffusions laws (Browning, 1978; Shen and Springer, 1976). In general, concentration gradient of the diffusion species, temperature and time are the three major factors that influence the diffusion process. In the case of water absorption in a humid environment, varying the RH level will lead to the change of the density of water adsorbed on the surface of the plastic materials and hence the concentration gradient of water at the surface. This concentration gradient drives water's diffusion. Whether sorption or desorption will occur depends on the hygrothermal history of the samples and the water concentration in the materials compared with that of the equilibrium value at a given condition. Because the samples used in the current study were fabricated and stored in the ambient environment which represent around 20 –25 °C and 50% RH, the humidity level in the samples should be approaching equilibrium with the ambient environment. When the humidity level is increased beyond 50% RH, the samples will absorb more water from the environment, and vice versa (Figure 5,6).

Temperature plays a key role in the water absorption for a number of reasons. First, the equilibrium water content in the epoxy resins is a function of temperature and water vapour pressure of the environment (Apicella et al., 1981a). Increasing temperature will lead to increase in the equilibrium water content of the SL resin, and hence result in greater water absorption tendency. Secondly, the saturation vapour pressure of water increases rapidly with

increasing environment temperature. By definition, the vapour pressure of water will also increase with temperature when the relative humidity level is fixed. The increased vapour pressure at higher temperatures will lead to greater adsorption density of water on the surface. Thirdly, the water absorption rate is a function of the water activity that can be related to the thermal energy of the water molecules. At ambient temperatures, the thermal energy of the water molecules is relatively low. The movement of water molecules in the SLA polymer is difficult. Therefore the water absorption process is very slow at the ambient environment (Figure 4). This is also true even at 40 °C (Figure 2). When the environment temperature is increased at a constant RH level, the higher equilibrium water content in the resin, higher vapour pressure in the environment, and the increased activity of the water molecules, all contribute to the accelerated water absorption, which produces greater dimensional and weight changes (Figure 3, 4).

Varying temperature ramping rate is equivalent to varying the exposure time. Because the distance of diffusion in solids is approximate proportional to the square root of time (Verhoeven, 1975), as compared with the exponential function to temperature, it can be expected that the water absorption rate will only be weakly influenced by the temperature ramping rate or holding time.

Lastly, the dimensional changes caused by thermal expansion can be fairly accurately estimated from the thermal expansion coefficient of the material and

temperature changes. The thermal expansion effect itself need not to be discussed. From the application point of view, however, it should be pointed out that the dimensional changes caused by thermal expansion is much more significant than water absorption in the short term because of the relatively large thermal expansion coefficient of the resin. A few degrees of temperature changes at the ambient temperatures can produce greater dimensional changes than several days of exposure of the specimens to a humid environment (Figure 5). Therefore, for the tooling applications where the SL parts are used as patterns, careful temperature control is needed for maintaining good accuracy. On the other hand, for injection mould applications, the shrinkage of the SL moulds cannot be ignored.

Conclusions

The dimensional change behaviour of the photo-cured SL5195 resin was investigated under different temperature and humidity conditions. At ambient temperatures, the water absorption process is very slow. Varying relative humidity level between 20% and 90% RH at the ambient temperatures only produced very slight dimensional changes. At above the glass transition temperature of the resin, higher RH led to significantly accelerated moisture absorption and much greater dimensional and weight gains. Increasing environmental temperature while keeping the relative humidity constant greatly increased the moisture absorption rate. Increased equilibrium water content in the plastics, greater water vapour pressure in the environment, and higher

thermal energy of the water molecules are believed to contribute to the increased moisture absorption rate at higher temperatures. The slow water absorption rate at the ambient temperatures allows for the SL parts to be stored and handled for a short period of time without causing significant dimensional changes. Higher temperature ramping rate led to reduced moisture absorption and dimensional changes, but the amount of moisture absorbed increased with repeated thermal cycles.

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Fig. 1

fig. 2

fig.3

fig 4.

fig. 5

fig 6.

fig. 7