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A Novel Approach for Characterizing Epoxy Mold Compound High Temperature Swelling

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***Abstract*—In this paper, a novel method for measuring high-temperature material swelling of an epoxy mold compound is explored. This work attempts to extend the swelling characterization beyond the capability of typical commercial tools. The approach uses a high-pressure chamber to maintain a saturated liquid environment for the specimen. Digital image correlation is used to measure in-situ strain change in the specimen due to hygro-thermal expansion. The results show moisture-induced swelling increases with temperature and is significant compared with thermal expansion. Results are compared against other measurement methods and published data. This has yielded good fundamental learnings for the novel concept and identified areas for future work.**

***Keywords*—Digital image correlation, moisture, hygroscopic swelling, polymer, epoxy mold compound**

Introduction

Today's electronic nonhermetic packaging technology is largely made up of polymer-based materials, such as epoxies, solder resist, and dielectric resin. These materials have many desirable cost and manufacturing advantages. However, they are also hydrophilic and inadvertently absorb moisture from the environment, causing hygroscopic swelling, reduced glass transition temperature (T_g), lowered elastic modulus, and reduced fracture strength and adhesion properties. This leads to package reliability and manufacturing process issues, particularly in high-density flip chip packages [1, 2].

Absorbed moisture diffuses and condenses deep within the microgaps of polymeric materials and along interfaces between adjacent material layers. Then, it desorbs rapidly when subject to high-temperature reflow conditions. However, due to the rapid temperature ramp, much of this moisture is entrapped within the package due to the presence of hydrophobic materials, like silicon die and copper planes, limiting desorption escape paths. As the package reaches peak reflow temperature, the trapped moisture causes additional mechanical swelling stress due to vapor pressure build-up, and swelling mismatch with nonpolymeric materials. This can lead to interfacial delamination or pop-corn type failures [3]. Even if cracks do not occur and the package is initially electrically functional, latent defects are still a risk. Hygro-swelling stress is caused by differential strain between polymeric material which swell and adjacent hydrophobic materials that do not. Bake has long been used as a method to reset the manufacturer's exposure time conditions of a package; however, it does not restore delamination or adhesive strength degradation. Therefore, understanding material moisture characteristics is critically needed to mitigate the risk of moisture-induced failures through better material selection and modeling capabilities.

The coefficient of hygroscopic swelling (CHS) is one of the most important properties when studying electronic packaging failure mechanisms. It is a measure of change in material hygro-strain over the moisture concentration. The concept is analogous to the coefficient of thermal expansion (CTE). Hygro-stress can be calculated similarly to thermo-mechanical stresses. In place of temperature and CTE, moisture concentration and CHS are substituted. It has been reported that swelling-induced strain can be even higher than thermally-induced strain [4] under certain conditions.

Despite this, moisture-induced stress has been mostly ignored as there are very few reports on material high-temperature CHS available in the literature or material supplier data sheets. This is due to the lack of efficient measuring methods and technical knowledge in this field [5]. It is challenging to measure high-temperature swelling because of rapid moisture evaporation during the measurement. Commercially available tools, like dynamic mechanical analyzer with relative humidity accessory (DMA-RH) [5, 6], have a limited temperature capability of up to 85°C, whereas the typical lead-free solder reflow process can reach up to 260°C. Higher temperature measurements are technically challenging. Higher pressure would be required to keep moisture in a liquid state at elevated temperature which is simply not feasible with today's standard tools. A more popular approach uses standard thermomechanical analyzer (TMA) equipment to measure the strain of a saturated sample as it is heated up. Rapid desorption causes nonuniform moisture distribution. Hence, an averaged strain is assumed. An additional moisture mass correction postprocessing analysis is required to compensate for the moisture loss. This method reportedly tends to overestimate the CHS [2, 4]. Also, some studies suggest avoiding the desorption-based approach due to potentially irreversible hygroscopic swelling characteristics in certain materials [7]. Another method that has been tried is Moiré Interferometry (MI) [8, 9], which has good accuracy and repeatability. However, it has inherent limitations as the corrosion-resistant grating that is replicated on the sample surface can result in measurement errors, particularly with thin specimens. Furthermore, all of these are

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single-temperature measurements, which makes them extremely tedious and time consuming to characterize for a range of temperatures. A more recent technique is digital image correlation (DIC) [10], which uses paint instead of a grating layer and does not impose significant stiffness on the specimen. The DIC quick scanning approach was developed to measure a range of temperatures with one test [11, 12]. However, like the TMA method, it is still based on desorption and has the same assumptions and limitations mentioned previously.

This work introduces a novel approach to measuring high-temperature hygroscopic swelling of polymeric materials. It keeps the specimen in a saturated state using a custom high-pressure chamber with a glass port that allows continuous DIC strain monitoring. The objective of this paper is to (1) present a novel approach for measuring hygroscopic swelling of a mold compound, over a range of temperatures up to >200°C; (2) report the CHS of the mold compound up to elevated temperature and compare its significance to thermal expansion; (3) validate the accuracy of the proposed method by comparing the results against other methods, analytical solutions, and published data.

CHS is defined as the ratio of the ultimate hygro-strain over the percentage total weight gain in the sample saturated with moisture as shown in eq. (1). In other words, it is the relative change in length per 1% weight moisture absorbed.

$$\beta = \frac{\varepsilon_\theta}{C} \tag{1}$$

where β is the CHS (strain%/moisture%), ε_h is the hygroscopic strain in percentage, and C is the moisture content percentage.

Methodology

The proposed method is a novel approach for obtaining swelling strain at elevated temperatures and addresses some of the aforementioned limitations. At the heart of the experimental setup, is an enclosed pressure chamber that keeps moisture in a liquid state at elevated temperature >100°C. As the chamber is heated up, the expansion of water increases the internal pressure since the volume is fixed, driving the boiling point well beyond 100°C. The sealed chamber has a glass port at the bottom which allows for a DIC camera to capture the swelling strain of the specimen throughout the test. The experiment setup is illustrated in Fig. 1. It is expected that the hygro-swelling can be measured together with the thermo-mechanical strain. Unlike existing methods, the goal and challenge is to conduct the swelling measurements up to over 200°C while maintaining the specimen in a constant moisture condition.

Two-dimensional, or 2-D DIC scanning used here is a well-established noncontact full-field optical deformation measurement technique, in which in-plane deformation is determined by comparing changes in digital images before and after deformation, with the resulting strain calculated [13].

Experiments

The following experiments were conducted to determine the material moisture concentration, thermal expansion, room temperature hygroscopic swelling, and high-temperature hygro-thermal strain. These are necessary to work out the material CHS in eq. (1).

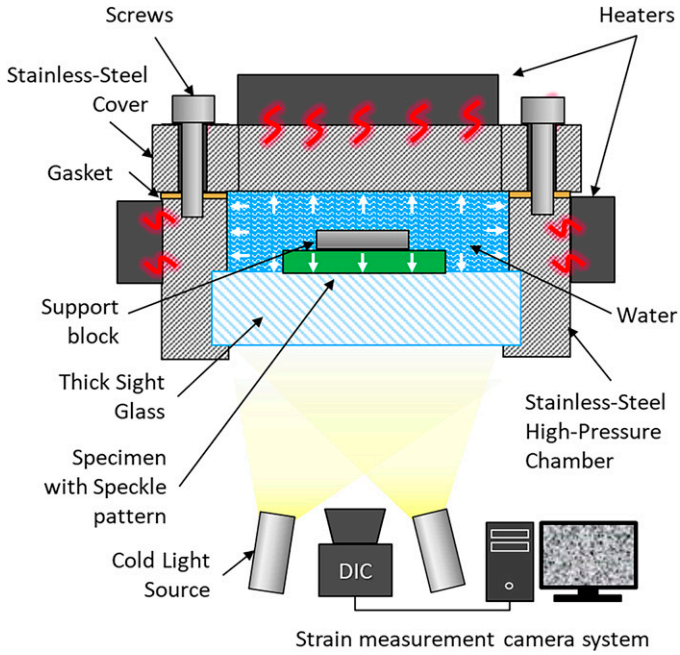

Fig. 1. High-temperature hygroscopic swelling chamber experiment concept setup.

A. Sample Preparation

A postcured homogenous epoxy mold compound (EMC) used in electronic packaging, with 83% filler weight, was chosen for this study. Mold compound makes up one of the highest polymer content within a package. Specimens measuring $8 \times 40 \times 0.35$ mm were cut out from molded flat copper strips using a laser, then dried in an oven at 125°C for 6 hours to remove any initial moisture. Thin specimens were used to reduce the moisture saturation time, improve temperature uniformity, and allow for one-dimensional (1-D) diffusion to be assumed since the thickness is much smaller than the in-plane dimensions.

One side of all strain measurement samples was sprayed white with black speckles on their ends using high temperature-resistant paint, then allowed to cure in an oven at 100°C. The speckled pattern is necessary for DIC strain measurements. Fig. 2 shows an image of the speckle painted specimen.

B. Moisture Absorption and Concentration

From eq. (1), the moisture concentration C within the material is needed for the computation of CHS. This can be obtained through direct measurements from an absorption test or analytically through moisture diffusion modeling. For the

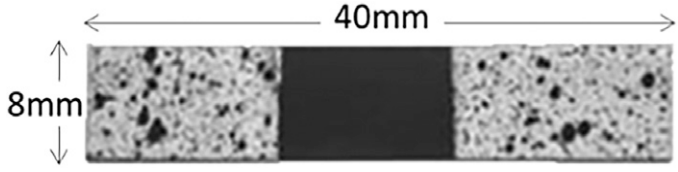

Fig. 2. Mold compound specimen sprayed with high-temperature speckled paint.

latter, saturated moisture concentration (C_{sat}) and diffusivity (D) need to be determined.

The absorption test involves dried specimens immersed in a beaker of distilled water and maintained at a room temperature of approximately 20°C. Specimen weight was measured using an analytical balance with 0.01 mg sensitivity. Specimens were carefully removed from the beaker, and excess surface water was wiped off with a dry cloth, weighed, then put back in the beaker. This process was repeated periodically until the specimen weight remained unchanged.

The weight of the sample is taken as the wet weight. The moisture weight gain, M_t was determined by:

$$M_t(\text{wt}\%) = \frac{m_t - m_0}{m_0} \times 100\% \quad (2)$$

where m_t is the specimen weight at time t and m_0 is the initial specimen dry weight.

For validation, C_{sat} was also determined analytically from the material diffusivity D. A thermal gravimetric analyzer (TGA) was used to measure absorption at different temperatures 30, 60, and 80°C.

For thin isotropic samples used in this experiment, moisture absorption at constant temperature can be described by Fick's law, where the solution for 1D diffusion can be analytically expressed as below [6].

$$\frac{M_t}{M_\infty} = 1 - \frac{8}{\pi^2}\sum_{0}^{\infty}\frac{1}{(2n-1)^2}\exp\left[-\frac{(2n+1)^2\pi^2 D}{h^2}t\right] \quad (3)$$

where M_t is the change in sample weight at time t due to moisture absorption ($M_\infty > 0$); or desorption ($M_\infty < 0$); D is diffusivity and h is the sample thickness.

The temperature dependency of diffusivity D can be described by Arrhenius equation below [11] .

$$D = D_0.e^{\left(-\frac{E_D}{RT}\right)} \quad (4)$$

Eq. (4) can be rewritten as:

$$\ln D = \ln D_0 - \frac{E_D}{RT} \quad (5)$$

where D_0 is a constant pre-exponential coefficient, E_D is the activation energy, R is the universal gas constant, and T is the absolute temperature.

The diffusivity D at these temperatures was determined by non-linear regression fitting Fick's 1D diffusion model eq. (3) to each of the measurements.

C. Thermal Expansion

Thermal expansion characteristics need to be determined to extract the hygro-strain component ε_h, from the combined hygro-thermal measured strain. Hygro-strain is used to determine the CHS from eq. (1).

A set of dried speckle painted specimens were placed in the dry chamber (setup shown in Fig. 1 except without any distilled water), against the sight glass, while the temperature was increased from room temperature approximately 20 to 250°C. Thermocouples were mounted on the chamber wall and a dummy sample as a means of monitoring the temperature of the sample during the test. Thermal expansion was measured using 2-D DIC. For validation purposes, thermal expansion was also measured using a TMA system operated in expansion mode.

D. Hygro-thermal Expansion

Another set of dried speckled specimens were immersed in the chamber filled with distilled water at room temperature approximately 20°C, as shown in Fig. 1. Hygro-swelling strain changes are measured using DIC until full saturation is achieved. Then temperature is steadily ramped up to approximately 220°C with the heaters while strain is continuously recorded in 2 s internvals. A reference hygrophobic material specimen with known thermal expansion characteristics is also placed in the chamber and simultaneously measured to cancel out any noise from measurements. A piece of copper was used as the reference specimen. For swelling strain validation, the DMA-RH test was conducted at 30, 60, and 80°C using the approach in He [6].

The following steps summarize the procedures used to obtain the hygro-swelling strains and corresponding CHS.

1. Two sets of similar specimens were dried and had their weight measured.
2. Thermal strains were measured on one of the samples as a function of temperature using DIC.
3. The other set was submerged in solution until fully saturated at room temperature. Similarly, hygro-swelling strain is measured using DIC.
4. The chamber and the saturated specimens are then heated while measuring hygro-thermal strain up to high temperatures.
5. The hygroscopic swelling strain curve is obtained by subtracting the thermal strain (step 2) from the hygro-thermal strain (step 4).

Experimentation Results and Validation

A. Moisture Absorption and Concentration

Fig. 3 shows the measured isothermal absorption data and calculated curve for the EMC specimens immersed in distilled water at room temperature. This shows saturated moisture concentration C_{sat} is achieved at approximately 0.3%.

Fig. 4 shows the TGA measurement results for the mold compound, performed at 30, 60, and 80°C for 60% RH condition.

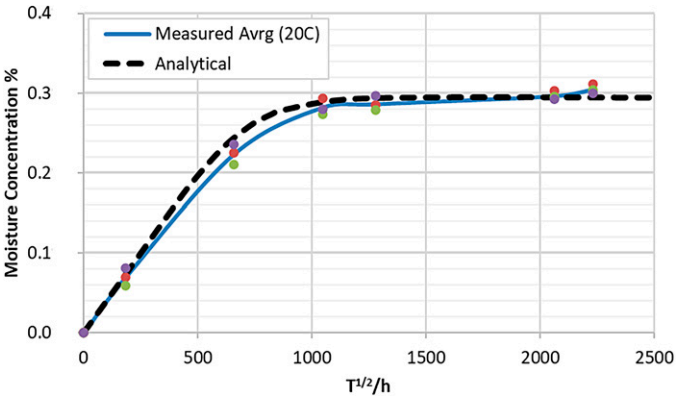

Fig. 3. Water absorption of the mold compound at 20°C.

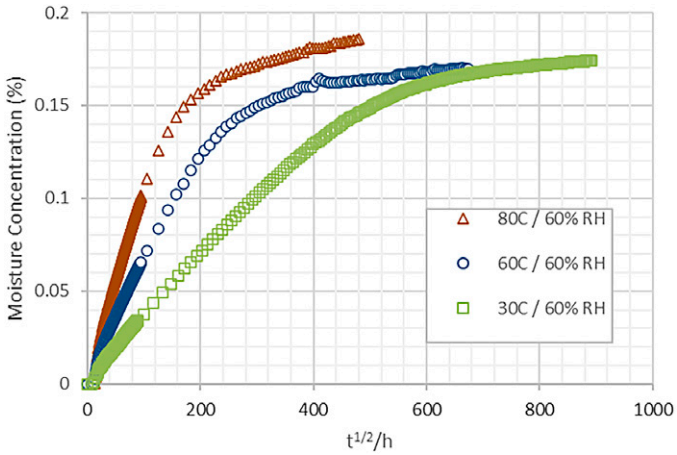

Fig. 4. Moisture absorption for 30, 60, and 80°C at 60%RH.

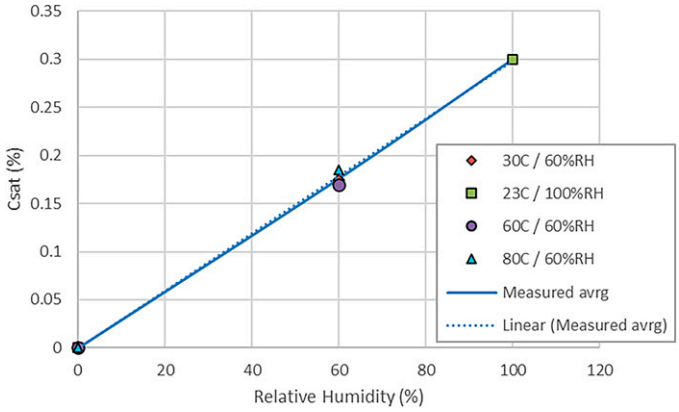

Fig. 5. Saturated moisture concentration of mold at 30, 60, and 80°C in 60% and 100%RH environment, measured with TGA and an analytical balance.

It can be observed that samples saturate at a roughly similar level, for the same RH. It has been reported that many electronic packaging materials have moisture-saturated concentration levels that are dependent on RH, and somewhat independent of temperature [14], especially those exhibiting Fickian absorption characteristics [15]. Though some studies show moisture concentration could have stronger temperature dependency at higher temperatures, particularly above material T_g [16]. This could be due to reduced enthalpy required for water molecules to move into free volume within the polymer [15].

Fig. 4 also shows that the diffusivity D is higher with increasing temperature, as denoted by the steeper initial gradient for higher temperatures [17]. The diffusivity relationship with temperature can be described by the Arrhenius equation and was discussed in the previous section. The prefactor D_0 and activation energy E_D were determined by fitting the experimental data to eq. (4).

This allowed the analytical determination of the moisture absorption at any temperature. Fig. 3 also shows the analytical isothermal absorption curve for the mold compound specimens in water at 20°C. As can be seen, there is good agreement between the analytical solution and the measured data. This shows that the moisture behavior follows Fick's 1-D diffusion model.

Fig. 5. shows the saturated moisture concentration C_{sat} for various levels of RH. It can be seen that C_{sat} increases proportionally with RH, which is described by Henry's law [6]. C_{sat} for higher temperatures was estimated by extrapolating the TGA measurements [3], since actual measurements were not feasible with this tool. This did not seem to significantly increase the higher temperature C_{sat}.

B. Thermal Expansion

The DIC linear thermal expansion results for the mold compound as a function of temperature are shown in Fig. 6. The TMA data are also superimposed and show good agreement with the DIC data. The glass transition temperature (T_g) is measured to be 150°C, with CTE1 of 11 ppm and CTE2 of 50 ppm. These results demonstrate that the fixture setup and thick sight glass did not have adverse effects on the 2-D DIC system measurements due to potential distortion and fixture thermal expansion at higher temperatures.

C. Hygroscopic Swelling

The hygro-swelling strain of the mold compound when immersed in water and measured using the DIC system at approximately 20°C are shown in Fig. 7. It shows hygroscopic strain reaches saturation at approximately 0.065% swelling. This was found to be about 0.01% lower than the scaled saturated strain measured from the DMA-RH system at 60°C. The different initial diffusion rates, or gradient, are due to different test temperatures. The difference in saturation levels could be partly attributed to the setup time delay between the actual specimen immersion and the start of DIC measurement, when the diffusion rate is highest. Extending the DIC measurement time could also have helped reduce the difference.

The dimensional changes of the specimens under isothermal conditions were also plotted as a function of the percentage weight of water absorbed in Fig. 8. At approximately 0.02% concentration, the swelling began to increase more rapidly. It is hypothesized that the initial unbound moisture mainly fills the micro-voids, or free volume, in the polymer matrix. Hence, there is hardly any initial dimensional change. Above a certain moisture content, after the micro-voids have been filled, water

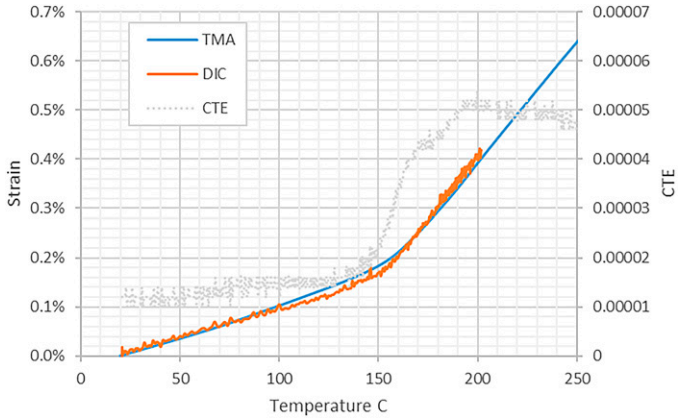

Fig. 6. Thermal strain of mold compound specimen and CTE between 20 and 250°C, measured using TMA and DIC.

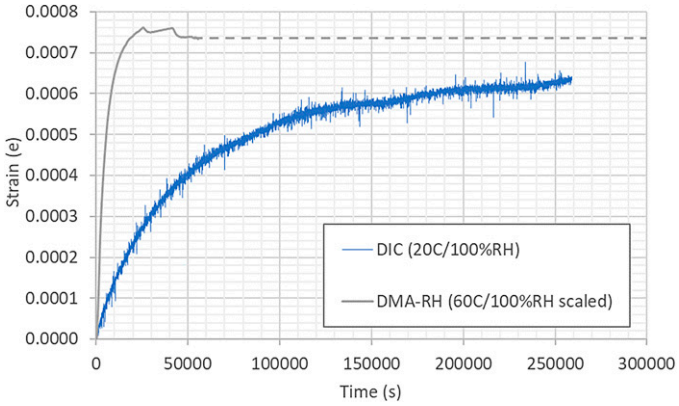

Fig. 7. Moisture absorption induced hygroscopic swelling of mold specimen measured using DIC and DMA-RH.

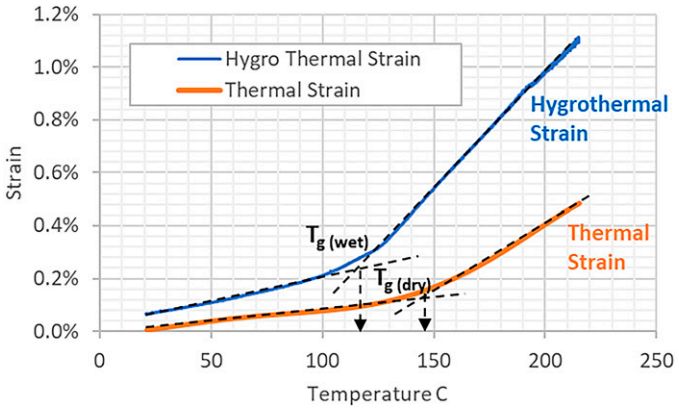

Fig. 9. Hygrothermal and thermal strains as a function of temperature for mold compound specimen measured using DIC.

molecules start to attach and disrupt the polymer chain, causing expansion or swelling of polymer matrix.

The gradient of the swelling strain to moisture concentration is essentially the CHS, which was introduced in the Introduction section. For a given temperature, the strain increases almost proportionally with moisture concentration. It can be observed that the CHS is clearly influenced by temperature, whereby the higher the temperature, the gradient is steeper, and the CHS is higher. From these data, the CHS value for this mold compound was calculated to be 0.2 at 20°C and increased slightly to 0.25 at 80°C.

The saturated specimen hygrothermal expansion, measured through the DIC system, as the temperature is ramped from 20 to approximately 220°C, is shown in Fig. 9. It was observed that with moisture, CTE1 and CTE2 both increased to 16 and 84 ppm respectively, compared with the dry thermal expansion CTE1 and CTE2 of 11 and 50 ppm. This is possibly due to the increase in intersegmental hydrogen bond lengths of polymer chains - and unbound moisture molecules exerting internal vapor pressure [10].

This glass transition temperature T_g, which is the intersection of the two tangents, clearly reduced with moisture absorption. This could be due to the presence of water molecules forming hydrogen bonds with the polymeric chains, increasing the distance and loosening the intersegmental forces between polymer chains, resulting in a lower T_g. This is also known as the plasticizing effect [10, 18].

The Gordon–Taylor equation [19] has been popularly used for estimating the T_g shift of mixtures or copolymers due to a diluent such as water. It is used here to approximate the reduction in T_g due to moisture.

$$T_{g,\,wet} = \frac{\alpha_p c T_{g,\,dry} + \alpha_w (1-c) T_w}{\alpha_p c + \alpha_w (1-c)} \tag{6}$$

In this equation, $T_{g,wet}$ and $T_{g,dry}$ are T_g of the polymer after and before moisture absorption respectively, α_p is the CTE difference between CTE1 and CTE2 of the polymer, c is the moisture content, α_w and T_w are the effective CTE difference and transition temperature of water, respectively. Eq. (6) was applied to the specimen to predict T_g change with respect to the moisture content. The effective CTE of water was assumed at 4,000 ppm/°C, as proposed by [10]. The resultant T_g as a function of the moisture content is plotted in Fig. 10. Interestingly, the analytical T_g delta was found to be 27°C, which is quite close to the experiment measured T_g reduction of approximately 25°C.

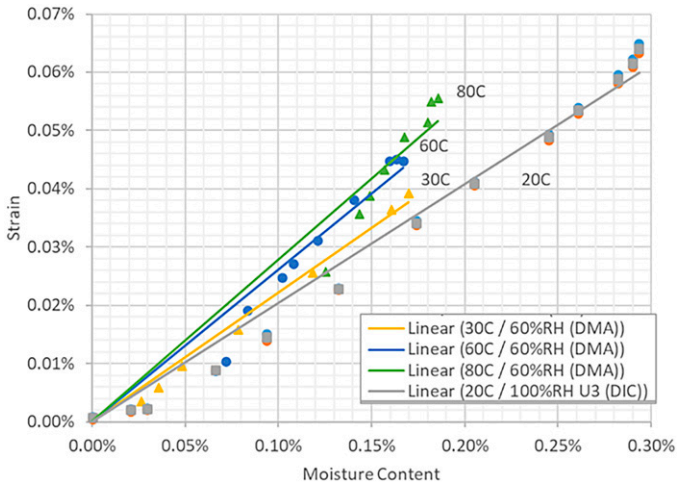

Fig. 8. Hygro-swelling versus moisture concentration of saturated mold compound at 20, 30, 60, and 80°C, measured at 60%RH and 100%RH (for 20°C).

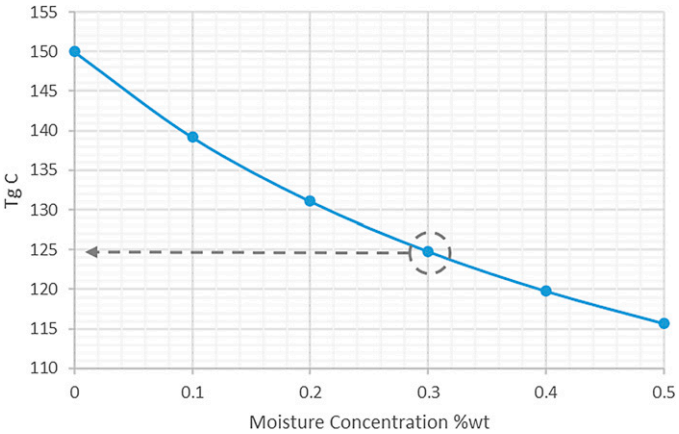

Fig. 10. Analytical glass transition temperature of mold specimen due to moisture absorption based on Gordon–Taylor's equation.

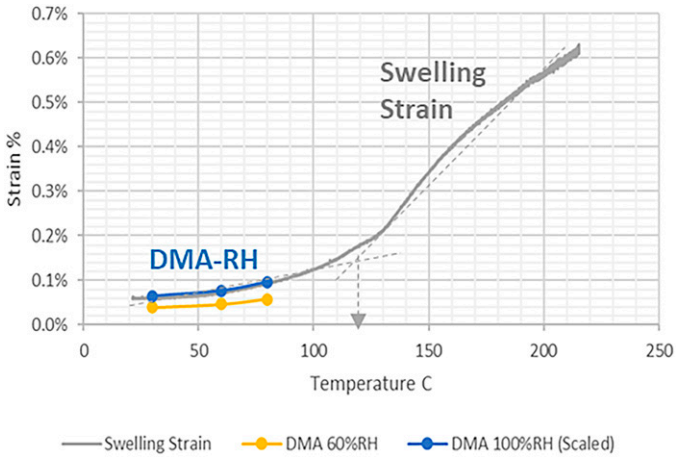

Fig. 11. The hygro-swelling strain of mold compound as a function of temperature up to approximately 220°C measured using DIC along with DMA-RH/TGA results at 30, 60, and 80°C for 100%RH.

While it may not be practical in reality to separate the hygro-swelling strain from the hygro-thermal strain, it is reasonable to assume the swelling strain component can be obtained by subtracting the thermal strain from the hygro-thermal strain. The resultant swelling strain is shown in Fig. 11. The DMA-measured hygro-swelling strains at 30, 60, and 80°C are scaled to 100%RH then superimposed for comparison. The results show good agreement with the swelling strain from DIC measurements. The strain was found to increase with temperature where there is a significant increase in the swelling strain observed between 110 and 130°C which is close to the glass transition temperature when saturated $T_{g(wet)}$. Compared with the thermal strains from Fig. 9, hygro-swelling strains are equal or higher. However, the actual factory ambient environment is usually lower at around 60%RH, and not 100%RH. Hence, the actual swelling strains are also expected to scale lower accordingly.

Based on saturated moisture concentration (Moisture Absorption and Concentration section) and hygro-strain measurements, the mold compound CHS was calculated based on eq. (1). The temperature-dependent CHS is shown in Fig. 12. It can be seen that the CHS values are relatively small (0.2–0.4) below 100°C but rapidly increase above the material T_g to approximately 1.8 at 220°C. The lower temperature range is comparable with the DMA-RH/TGA measurements (Hygroscopic swelling section) of 0.21 at 30°C, and 0.25 at 80°C.

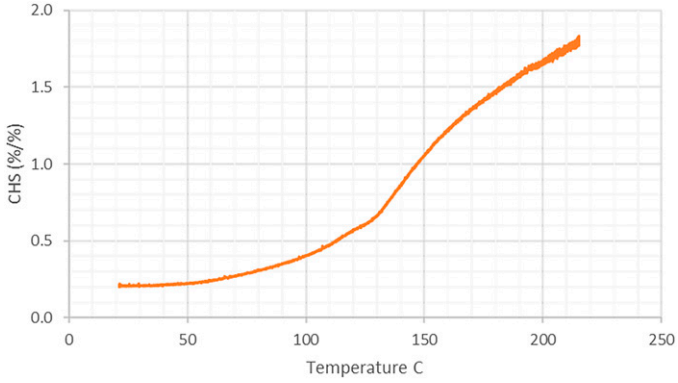

Fig. 12. CHS for mold compound as a function of temperature measured using DIC and the novel high-pressure chamber.

Discussion

1. The thermal strain (550 με) caused by a 50°C increase in the temperature of the mold compound, below T_g, is approximately equivalent to the swelling strain from moisture at just 85°C@60%RH. 60%RH being the typical factory floor environmental condition. Above T_g, the thermal strain (2500 με) caused by a 50°C increase in temperature is similar to the swelling strain at 200°C@60RH. These moisture-induced strain magnitudes are significant, can strongly influence the outcome of analytical analysis, and should be appropriately accounted for.
2. The lower temperature (<100°C) CHS results of 0.2–0.4 from this study is within similar range of other mold compounds from publications [2, 8, 9, 12, 20, 21]. For elevated temperatures, 150 to 220°C, the CHS results of approximately 1.0–1.8 are trending comparatively higher than the few published data of 0.5–1.6 [12, 21]. One possible reason for this is the narrow range of moisture concentration data used for extrapolation in the Moisture Absorption and Concentration section. A follow-up experiment to measure higher-temperature (>100°C) moisture concentration C_{sat} is planned. More details will be shared in a follow-on publication.
3. While this initial EMC testing with the novel method is showing encouraging results, there are still many unknowns and challenges. For example, measuring thinner and lower modulus polymeric materials, such as solder resist, could be problematic due to higher potential for out-of-plane warpage. Tests on other packaging polymeric materials are in-progress.
4. While it is not unusual to use water immersion for moisture absorption tests [17, 22]. Water is known as the "universal solvent." Hence, some materials could inadvertently start to dissolve into water, particularly during prolonged test at high temperatures. This could adversely influence the material swelling characteristics. There is also the potential concern that moisture sorption through water could differ from vapor sorption.
5. While the goal was to ultimately measure swelling strains up to peak reflow temperature of 260°C, the tests were stopped short at approximately 220°C as bubbles were starting to form inside the chamber. Further investigation is needed to address this. To the author's knowledge, there has yet to be any data published or capable test method for such a high temperature.

Conclusions

In this study, a novel method of using a high-pressure chamber and DIC for measuring high-temperature hygroscopic swelling strain was introduced. The feasibility of effectively measuring high temperature hygrothermal strain between 20 and 220°C was demonstrated on a mold compound. The results showed high temperature hygroscopic swelling was significant compared with thermal expansion of the mold compound

material and should be considered when conducting stress analysis. For validation, the $T_{g,wet}$, swelling strain and CHS obtained were compared against analytical solutions, DMA-RH/TGA measurement and other published data. Some potential concerns, limitations and plans for future development work were also discussed.

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