

Experimental and Numerical Analysis of Hygroscopic Behavior of a Polymer Material

Wael Mokhtar¹, John Witte²
Grand Valley State University, Allendale, MI, 49401
mokhtarw@gvsu.edu, john.m.witte@gmail.com

Abstract

An initial validation study was performed for the purpose of establishing a process for determining the coefficients for diffusion and hygroscopic expansion of a commercially available nylon material, which are necessary for performing numerical simulations of hygroscopic behavior. The study goals were to validate the experimental procedure and analysis tools for which to support the evaluation of hygroscopic strain in materials that are intended to be used in an automotive application. A literature review was performed regarding the analogy between thermal and moisture sorption problems for the purpose of justifying the usage of commercial thermal Finite Element Analysis (FEA) software in simulating moisture sorption. Both an analytical and finite element model were created utilizing the experimentally determined material properties and were found to predict experimental values of moisture uptake at 240 hours with 1.70% and 2.07% discrepancy, respectively. The finite element model was able to predict hygroscopic strain with 5.4% discrepancy. The majority of the error was associated with the large sample thickness requiring a longer exposure time than was allowed for the experiment, and the resolution of the available measurement instrumentation. A longer study should be performed on thinner material samples to further validate the model.

Introduction

A literature review has indicated that the study of hygroscopic mismatch stresses which occur between polymer and other substrate materials has been widely researched for application in the electronics industry. However, when polymer components are used in mechanism applications, dimensional variation due to hygroscopic effects may also be of concern as design performance may be affected. A literature review did not identify a documented process which describes the complete process for obtaining material properties and using them to simulate transient dimensional variation for arbitrary shapes in mechanisms as a result of hygroscopic effects.

As an effort to improve the reliability of mechanism designs composed of various polymer components, the transient response of hygroscopic effects must be further understood. For example, extreme relative humidity and temperature changes observed in automotive interiors may affect the sliding fit between two components that was initially found acceptable before dimensional variation due to hygroscopic effects took place. A detailed cross-section of a sliding joint composed of two dissimilar polymer materials is shown in Figure 1. The amount of

¹ Ph.D, Associate Professor of Mechanical Engineering

² Graduate Student at GVSU and Product Engineer, Gill Industries, Inc.

interference may vary to the point that Bump Squeak and Rattle (BSR) or too high of efforts are observed when hygroscopic effects are considered.

Mechanisms of this type may extend across multiple vehicle platforms and be produced well in excess of 1 million units per year. This results in a large number of vehicles being operated in a wide variety of environmental conditions where the mechanism must still meet the same functional requirements. Improved simulation capabilities will allow for a better understanding of how various changing conditions may affect the operation of the mechanism.

Typical automotive specifications may require a mechanism to function at a temperature range of -20°C to 60°C and a relative humidity as high as 95% at 38°C . If the moisture diffusion is to be understood at the extreme temperature and humidity conditions specified for a new design, new experimental data specifically related to the design requirements must be taken in order to quantify the necessary material properties. These values are not typically published for general use in simulation.

Some steady state material data regarding moisture uptake are published as a result of testing per ISO62:2008 and ASTM D570. The measured values which are typically published following these experimental guidelines have limited use in the context of this study because they are only valid at the temperature and humidity condition at which the data was taken. Additionally, the maximum moisture uptake at a specific condition is typically the only value given. In order to simulate hygroscopic effects additional data is required including the diffusion coefficient and the relationship between the change in moisture content and subsequent strain.

In addition to the requirement to experimentally determine diffusion properties, the ISO and ASTM testing specifications do not provide guidelines for quantifying the strain associated with the moisture uptake as they tend to focus on quantifying the material properties which are only associated with the diffusion process. A literature review must be performed in order to understand what proposed methodologies are available for quantifying hygroscopic strain and relating this value to moisture uptake.

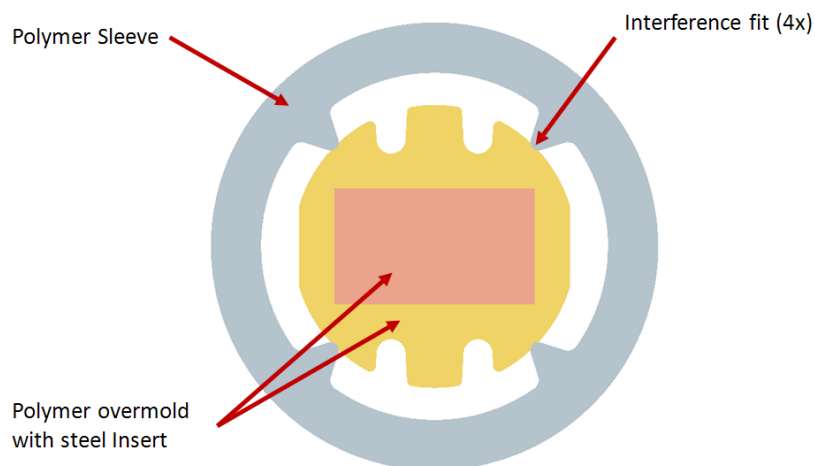


Figure 1: Section view of sliding fit between sleeve and insert

The proposed research will seek to better understand the material properties of a particular polymer material for the purpose of hygroscopic simulation. The water submersion test procedure will be utilized because it avoids the requirement for costly environmental test equipment and greatly reduces the time required to run the experiment and obtain material data. Water submersion conditions typically result in the largest potential moisture uptake, or saturated moisture condition, when compared to ambient conditions. Therefore, the water submersion method also results in the most severe deformation at a saturated moisture condition. Methodology utilized in this study may be repeated with access to environmental test equipment if a simulation at ambient conditions was determined necessary in future studies.

Literature Review

As previously mentioned, a number of studies have been performed which pertain to application in the electronics industry. A large amount of effort has been committed to attaining the ability to predict hygroscopic mismatch stresses between various components in an integrated circuit board. Hsiang-Chen Hsu et al. [1] evaluated the coefficient of moisture expansion by means of Thermal Gravitational Analysis (TGA) and Thermal Mechanical Analysis (TMA) in polymeric packaging materials used in complimentary metal-oxide-semiconductor (CMOS) image sensor image packages (CIS). The overall transient moisture distribution was simulated utilizing commercial FEA software. Moiré Interferometry (MI) was also used to compare the experimental deformation to finite element simulation. X.J. Fa and S.W.R. Lee utilized Moiré interferometry (MI) as a method for analyzing hygroscopic strain. MI was also used by Yoon et al. [2] who proposed a technique for analyzing the combined hygroscopic and thermo-mechanical effect with FEA based simulations and experimental validation. Zhang et al. [3] performed hygro-thermo-mechanical modeling and testing for an electronics reliability application. Dynamic Mechanical Analyzer (DMA), TMA, and TGA techniques were used to determine material properties. Weide-Zaage et al. [4] studied moisture diffusion in printed circuit boards. Concentration distribution was compared based on FEA simulations, taking into consideration the effects of copper layers acting as a barrier to diffusion. Hu et al. [5] studied the effect of thermal and hygroscopic stresses on delamination in Light Emitting Diode (LED) packages.

M.H. Shirangi and B. Michel proposed a method for evaluating non-Fickian, or dual stage behavior in polymer materials [6]. M.H. Shirangi and B. Michel also proposed that swelling of the polymer matrix can theoretically increase the available empty volume in a polymer the material allowing a greater maximum amount of moisture to be absorbed, which results in non-Fickian behavior. Non-Fickian behavior was also studied by Celik et al. [7] with regards to electronic packages.

X.J. Fan and S.W.R. Lee found that saturated moisture concentration was a measure of the moisture content under given temperature and humidity conditions [6]. C_{sat} in the studied polymers was found to be linearly proportional to the ambient relative humidity. For most polymer materials, C_{sat} was found to be temperature independent below the glass transition

temperature. Above the glass transition temperature, the C_{sat} for the studied samples showed strong dependency on temperature.

Onur Sayman [8] presented an analytical approach to performing a general stress analysis for thick or thin multi-layered composite cylinders under hygrothermal loading. The composite epoxy-glass fiber materials studied showed different mechanical properties in the principal material directions. Analytical calculations were compared to FEA simulations. Derrien and Gilormini [9] were able to show that a composite may have a nonlinear sorption isotherm even though the polymer matrix has a linear one, as a result of the internal stresses induced by swelling. The composite reinforcing material is assumed to not absorb moisture and consequently not swell. As a result, the composite material will tend to absorb less moisture than a comparable sample composed purely of the matrix material for two reasons. First, the reinforcing material takes up a component of the volume and does not absorb moisture. Second, the reinforcements induce internal stresses that restrict the potential absorption of the matrix material. LI et al. [10] presented an analytical model for effective mass diffusivity in composites. Square and hexagonal arrays of cylindrical filler materials were considered. The models allowed for the prediction of mass diffusivity based on geometry and fiber volume fraction.

Yu [11] presented a study of moisture diffusion in polymeric packaging materials at high temperatures. A comparison of conventional testing technique TGA was compared with Gas Chromatography/Mass Spectrometry (GC/MS) as well as Karl Fisher Titration (KFT) on molding compound, underfill, and die attach materials used in the electronics industry. Results obtained using TGA were found to be affected by the outgassing of solvent at high temperatures where Karl Fischer Titration was found to be more reliable in this application.

Many studies made use of TMA / TGA for experimental procedures. Alternative measurement methods were used in studies performed by M.H. Shirangi and B. Michel [6] studied the diffusion of moisture in plastic encapsulated devices. Samples were dry baked for 24 hours at 125°C and then exposed to 85° C / 85% relative humidity. The test samples consisted of molded discs 100 mm in diameter and 2 mm thick. Wei et al [12] studied the hygroscopic dimensional changes in resin-matrix composites during water sorption/desorption cycles. Disk shaped specimens with a 15 mm diameter and 2 mm thickness were prepared according to ISO 4049. Poenninger and Defoort [13] described a test facility for the measurement of the length variation of polymer composites. Commercial laser interferometers with a resolution of 10 nm working under vacuum conditions were found to yield a total accuracy of 0.1 μm. The length variation was determined by using two laser interferometers.

Field Problems and Formulation of Thermal Moisture Analogy

Moisture diffusion is considered a field problem. Other examples of engineering field problems include torsion of a non-circular section, ideal irrotational fluid flow, seepage, heat transfer and electrostatic potential [14]. The general field equation which may be used to describe these types of problems is given as:

$$D_x \frac{\partial^2 \phi}{\partial x^2} + D_y \frac{\partial^2 \phi}{\partial y^2} - A\phi + B = 0 \quad [1]$$

where ϕ is the field variable and D_x , D_y , A , and B represent physical properties which are specific to the field problem being analyzed. In a three-dimensional thermal problem with no internal heat generation case, the transient heat conduction through a material may be modeled with the following [1]:

$$\frac{\partial T}{\partial t} = \alpha_T \left(\frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2} \right) \quad [2]$$

where T is the absolute temperature, x , y , z are the spacial coordinates, t is the time, and α_T is the thermal diffusivity.

Diffusion of moisture in polymer materials obeying Fick's Second Law is described by the following equation:

$$\frac{\partial C}{\partial t} = D \left(\frac{\partial^2 C}{\partial x^2} + \frac{\partial^2 C}{\partial y^2} + \frac{\partial^2 C}{\partial z^2} \right) \quad [3]$$

where C is the moisture concentration and D is the moisture diffusivity. While equation [5] is adequate for a single material analysis, moisture problems often require the consideration of multi-material interfaces. Because the concentration potential is not identical between materials, a wetness field variable is utilized to avoid differential difficulty and to allow for a single equation to describe the problem:

$$\frac{\partial w}{\partial t} = D \left(\frac{\partial^2 w}{\partial x^2} + \frac{\partial^2 w}{\partial y^2} + \frac{\partial^2 w}{\partial z^2} \right) \quad [4]$$

where w is the wetness defined as:

$$w = \frac{C}{C_{sat}} \quad [5]$$

where C is the moisture concentration at a particular point and C_{sat} is the saturated moisture concentration of the material under the specific conditions being analyzed.

Because moisture diffusion through a material may be described as mathematically similar to thermal diffusion, the correspondence of material properties which are relevant when considering these behaviors may be highlighted. A comparison of various physical properties is shown in Table 1:

Table 1: Corresponding material properties for thermal moisture analogy

Property	Thermal Analysis	Hygroscopic Analysis
Primary Variable	T	Wetness, w
Density	ρ	1
Thermal Conductivity	k	$D \cdot C_{sat}$
Specific Heat	c	C_{sat}
Coefficient of Thermal Expansion	α_{te}	$\beta \cdot C_{sat}$

In a thermal analysis as described by equation [2], the primary variable is temperature which represents the potential for heat transfer whenever a thermal gradient exists. The coefficient of thermal expansion describes the linear relationship between material temperature change from a starting reference point and the resulting relative strain.

Comparatively, in a hygroscopic analysis the primary variable is the wetness. Equation [4] describes the behavior of moisture sorption. Although the true driving potential through a material is the moisture concentration gradient, the absolute moisture absorption potential at a specific humidity and temperature condition is not the same for all materials. Therefore, to allow for a multi-material analysis the moisture concentration is normalized by the absolute moisture potential. In a hygroscopic analysis, the density has no physical meaning so a value of 1 is used in the field equation. This is in contrast with a thermal analysis where all parts in an assembly have equal potential to reach the external temperature.

Because the wetness parameter is used in a typical analysis, the diffusivity must be multiplied by the saturated moisture concentration of the material at the external temperature and humidity conditions of interest. This calculated value is comparable to the thermal conductivity through a material. The saturated moisture concentration is also used in place of the specific heat for material, which essentially means that the diffusivity is the only factor driving the rate of moisture diffusion. The amount of hygroscopic strain is generally assumed to be linearly proportional to the moisture concentration of a material and may be expressed as:

$$\varepsilon_h = \beta C \quad [6]$$

where ε_h is the hygroscopic strain, β is the coefficient of hygroscopic strain, and C is the moisture concentration.

Fickian versus Non-Fickian Diffusion

Yu [11] presented the four models which are shown in Figure 2 regarding the characteristics of moisture absorption, Fickian, Sigmoidal, Two Stage, and Case II. ISO 62:2008(E) describes procedures for verifying Fickian absorption, but the key recognizable characteristics may include by the following:

1. Mass of absorbed moisture versus time^{1/2} is initially linear.
2. As time increases, the absorption curve levels to a saturated level of moisture content.
3. A plot of M_t/M_{sat} versus $t^{1/2}/L$ is identical for different values of material thickness.

Sigmoidal sorption may occur when a material that would typically display Fickian behavior is exposed to a variable surface concentration. This indicates that the actual transport through the material is Fickian, but the initial curve is not linear due to the slow establishment of a constant boundary condition at the surface of the material. Two stage sorption consists of two distinct segments, the first composed of fast Fickian absorption, the second is composed of slow non-Fickian absorption. Instead of leveling off at the completion of the Fickian absorption, the curve continues to show increased moisture absorption with time. This behavior is typically

attributed to diffusion-relaxation resulting in increased moisture holding capacity of the material. Case II sorption is essentially a linear model which may be accompanied by a significant amount of material swelling.

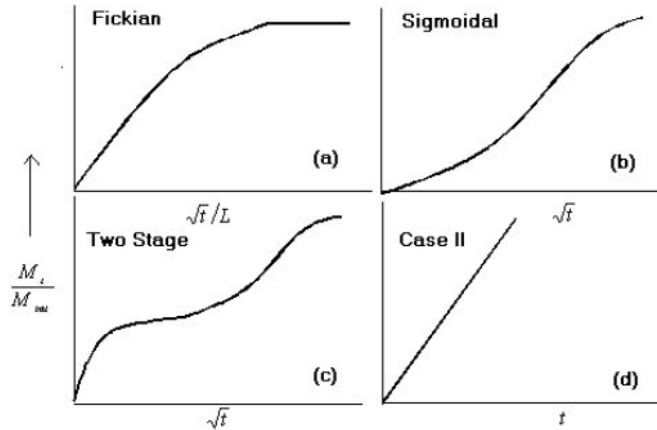


Figure 2: Characteristic curves for (a) Fickian, (b) Sigmoidal, (c) Two Stage, and (d) Case II [9]

1D Moisture Diffusion

For a one-dimensional case involving a single material, equation [6] may be reduced to:

$$\frac{\partial c}{\partial t} = D \frac{\partial^2 c}{\partial x^2} \quad [7]$$

Figure 3 describes a one-dimensional case where a material of thickness L is exposed to a moisture concentration at the boundaries $X = L/2$ and $X = -L/2$.

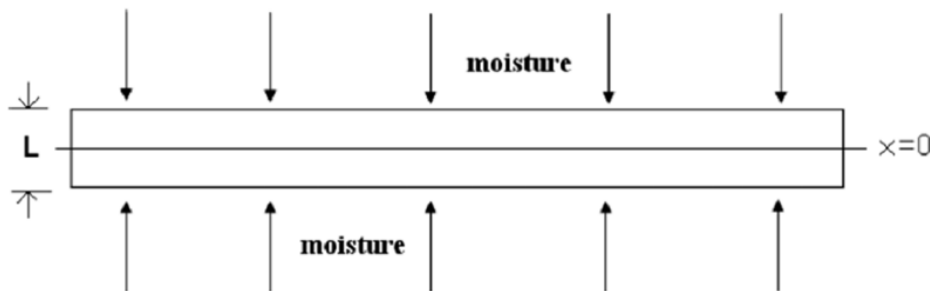


Figure 3: One-dimensional moisture diffusion in an isotropic material [1]

The initial and boundary conditions for the problem may be described as:

Initial Condition:

$$C = 0, \text{ for } -\frac{L}{2} \leq x \leq \frac{L}{2} \quad [8a]$$

Boundary Condition 1:

$$\frac{\partial c}{\partial x} = 0, \text{ at } x = 0, t \geq 0 \quad [8b]$$

Boundary Condition 2:

$$C = C_{\infty}, \text{ at } x = \frac{L}{2} \text{ and } x = -\frac{L}{2}, t \geq 0 \quad [8c]$$

Using Laplace transformation method and separation variable method, equation [9] may be expressed as:

$$C(x, t) = C_{\infty} - \frac{4C_{\infty}}{\pi} \sum_{n=0}^{\infty} e^{\left(\frac{-D(2n+1)^2\pi^2 t}{L^2}\right)} \cos\left(\frac{(2n+1)\pi x}{L}\right) \quad [9]$$

where the moisture concentration at any particular depth of the on-dimensional moisture diffusion model may be determined. Integrating equation [9] yields:

$$\frac{M_t}{M_{\infty}} = 1 - \sum_{n=0}^{\infty} \frac{8}{(2n+1)^2\pi^2} e^{\left(\frac{-D(2n+1)^2\pi^2 t}{L^2}\right)} \quad [10]$$

where the absolute weight gain of the sample may be calculated as a function of time. The use of 20 summands is generally considered sufficient [15].

Determination of Diffusion Coefficient (ISO)

ISO 62:2008(E) and ASTM D570 address the required practices to conduct a controlled experiment for the purpose of evaluating moisture absorption in polymer materials. The standards each have a slightly different description of the requirements for test sample size and experimental procedures. A brief summary is included but the standards themselves should be reviewed prior to conducting sample testing.

Two applicable dimensionless numbers may be used to fit empirical data with ideal Fickian diffusion. The dimensionless values are given by:

$$\varphi = \frac{D\pi^2 t}{d^2} \quad [11]$$

where φ is the dimensionless value, D is the diffusion coefficient, t is the current time, and d is the sample thickness. The second dimensionless value is the wetness calculated with equation [5]. The dimensionless values for ideal Fickian diffusion are listed in ISO62:2008(E). These values may also be utilized as described in the ISO standard to validate the agreement of the experimentally measured material behavior with Fick's diffusion laws.

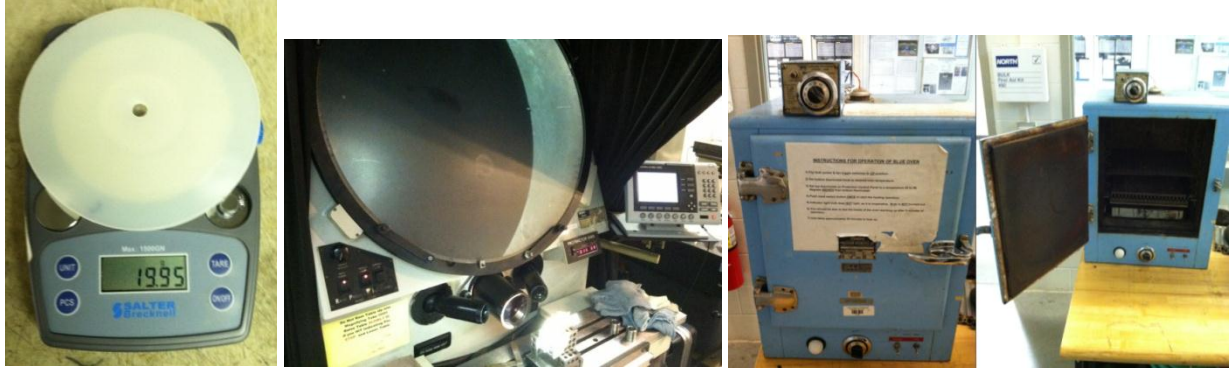
Experimental Method

Apparatus

Scale: Salter Brecknell, 100 g capacity, 0.05 g resolution (Figure 6a)

Optical Comparator: Rank Precision Industries Quadra Chek 200 (Figure 6b)

Oven: Insulated Oven with Circulating Fan (Figure 6c)



(a)

(b)

(c)

Figure 6: (a) Salter Brecknell digital scale, (b) Rank Precision optical comparator, and (c) drying oven

Procedure

1. Cut 4 96.8 mm diameter disks from a common sheet of 2.32 mm thick material.
2. Burnish indicator markings immediately across from each other to allow for the diameter to be repeatedly measured between the same points at each new time period.
3. Dry bake at 125° C for 24 hours.
4. Fill insulated container with 23° C distilled or reverse osmosis purified water.
5. Obtain initial diameter measurements for each of the four samples.
6. Obtain cumulative initial mass of four samples.
7. Submerge samples in water for 24 hours.
8. Remove samples and remove surface water. Measure cumulative mass and individual diameter.
9. Repeat immersion and measure mass and diameter at 24, 48, 96, 192, and 240 hours. Circulate water every 24 hours.

Results

A CAD model of the disc samples was created based on the experimental sample measurements with a thickness and diameter of 2.32 and 96.8 mm, respectively. A 6.5 mm center hole was also included in the CAD model to match the samples. The center hole was a result of the manufacturing process used to create the discs from nylon sheet. A 0.4 mm hex mesh was created with radial and cylindrical divisions oriented to best match the expected direction of moisture absorption and dimensional variation as shown in Figure 7.

Figure 8 shows the imposed boundary condition of zero radial moisture flow through the surface around the circumference of the sample, based on the simplified assumption that the vast majority of diffusion would occur through the large opposing faces of the thin sample in a 1 dimensional manner. The simulation was set to run a total of 240 hours of exposure to moisture with 2000 sub steps, or 0.12 hour increments. The simulated wetness and corresponding directional deformation are shown in Figures 9 and 10, respectively. The total deformation may be correlated to the experimentally measured diametrical change.



Figure 7: Meshed geometry

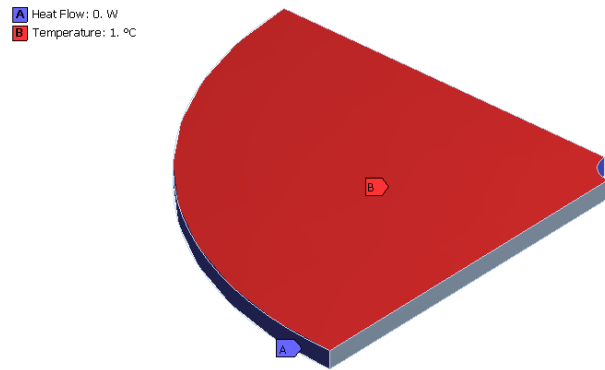


Figure 8: Boundary conditions

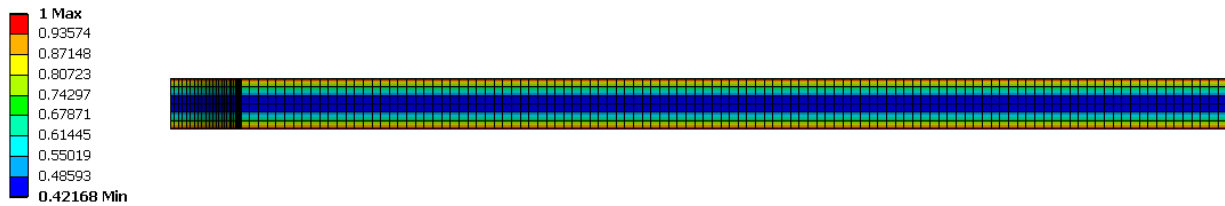


Figure 9: Wetness at 240 hours

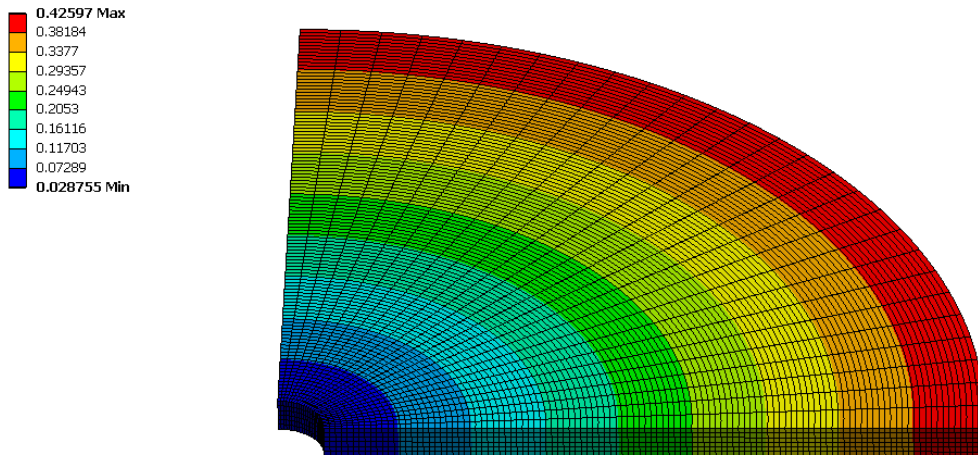


Figure 10: Total $\frac{1}{4}$ deformation for the $\frac{1}{4}$ symmetry model at 240 hours

The purpose of the experiment was to provide initial validation of the proposed procedures for experimentally determining the values of D , β , and obtaining good correlation with the FEA model. Based on these criteria, a time period of 240 hours of moisture exposure was allotted for the experiment with the understanding that full saturation of the 2.32 mm thick samples may not be reached. The initial experiment would provide an indication of whether the sample dimensions were appropriate or if a different material thickness or diameter should be used in future experiments to obtain full saturation in a reasonable amount of time. The experiment would also demonstrate that the commercial FEA software could appropriately be adapted based on the thermal moisture analogy.

The guidelines for ISO62:2008(E) immersion at 23° method was approximately followed as this allowed for a much more rapid rate of moisture absorption when compared to exposure to atmospheric conditions. Dimensional data was captured for five individual samples. Due to the limitations of measurement resolution and maximum mass for the available scale, cumulative mass was measured for samples 1-4 and the average value was used for the determination of hygroscopic parameters. A single larger sample may have been used to better work with the scale, but this would have limited the ability to measure dimensional variation due equipment limitations and resulted in greater complications in developing measurement fixtures to reduce error due to warping of the samples.

Reverse osmosis water with a total dissolved solids (TDS) content of 9 was used for sample immersion. For the duration of the experiment, the air temperature was observed to fluctuate between 23.2° and 25.3° C. The water temperature was assumed to have fluctuated relatively less than the air temperature due as a result of the large volume of water which was used, the relatively higher specific heat of water, and the fact that the water was stored inside an insulated container. The samples were dry baked at 125° C for 24 hours prior to immersion.

Per ISO 62:2008(E), the percent mass change versus time was plotted on a logarithmic scale to determine t_{70} , or the point at which 70% of saturation had been reached. As indicated by Figure 26, the material did not appear to have reached the 70% saturation point as the logarithmic plot of the mass change was still relatively linear. The dimensionless empirical method was used to approximate this value for the time period of interest relative to this experiment.

The values for D and an assumed C_{sat} that resulted in an appropriate correlation with ideal Fickian behavior are shown in Table 5 and plotted against the theoretically exact Fickian dimensionless values in Figure 11. For comparison, a diffusion coefficient was also determined on the basis of assuming the published value of 8.5% saturated moisture content. As can be seen in Figure 11, the assumed values fit the ideal Fickian curve for the time period of interest relatively well, although the curvature of the experimental data would be expected to significantly deviate as the material continued to absorb moisture. Both sets of values were subsequently utilized in the finite element simulation of the experiment.

The experimentally determined values for D and C_{sat} were converted to m/kg/hr for appropriate agreement with the requirements for defining material properties in the FEA

software. The cross-referenced relationship for the basis of the values shown in Table 2 is given in Table 1.

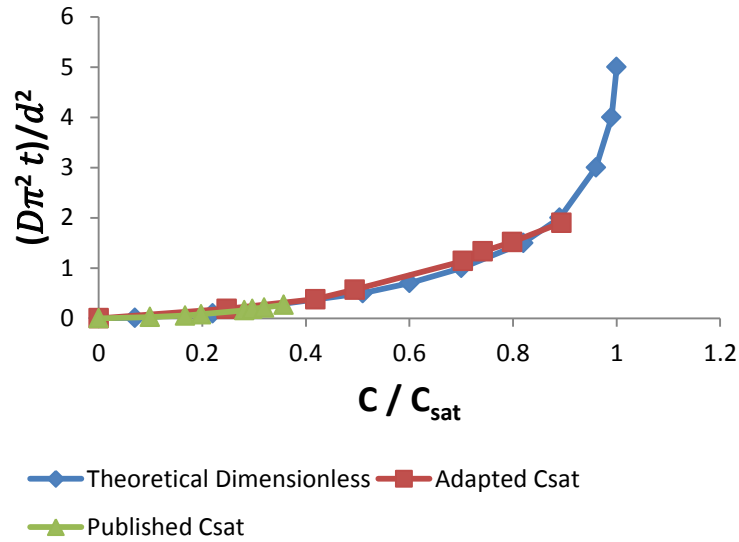


Figure 11: Dimensionless comparison of ideal Fickian behavior versus experimental data for the time period of interest

Table 2: Experimentally determined values for D and C_{sat}

Parameter	Short Term Value	Published C_{sat}
D (mm ² /s)	1.2×10^{-6}	1.7×10^{-7}
C_{sat}	3.4%	8.5%

The samples had a tendency to warp as they expanded, so they were forced to a flat state with gage blocks and a light clamping force prior to measurement in the optical comparator. The samples followed subjectively similar trends, which served as evidence that the dimensional measurement error was adequately controlled for the experiment. The hygroscopic strain versus average moisture concentration was plotted for the purpose of determining β and is shown in Figure 12. A linear approximation was fitted to the data resulting in a value of approximately 0.2595 mm³/mg for β . The data showed good correlation to a linear approximation of β with an R^2 value of 0.9849.

As shown in Figure 30, the simulated moisture content initially overestimated the experimental values. This discrepancy may be associated with numerical error in the finite element model, or the fact that the values for β and C_{sat} were approximated before the samples were allowed to reach saturation.

The published saturated moisture gain for the nylon 6/6 material under similar conditions was 8.5%, while the value used to approximate the data for the duration of the experiment was 3.4%. If the experiment were allowed to run to full saturation, it is anticipated that a very different diffusion coefficient and saturated moisture content would have been required to match

the dimensionless data. Because it was not guaranteed that all moisture was removed from the samples by repetitively drying and checking sample mass prior to beginning the absorption process, it could not be guaranteed that the experiment would show an 8.5% uptake at saturation. The final absorption percentage is a relative value and requires that all moisture has been removed from the sample if the published value is expected to be experimentally obtained.

As shown in Table 4, the final moisture uptake as predicted by the FEA model at 240 hours was approximately 576 mg of water versus the experimental value of 588 mg, resulting in a 2.1% discrepancy. As subjectively shown in Figure 30, this error was relatively consistent throughout the simulation. The moisture concentration at the first time step was calculated at 98 mg which may simply be a result of mathematical error in the numerical model. The mesh was refined one additional step to a 0.25 mm reference size, but the error remained in the summation. It was also possible that the lack of precision instruments when gathering experimental data or the fact that the sample was not allowed to reach full saturation before calculating the necessary coefficients also contributed to this error.

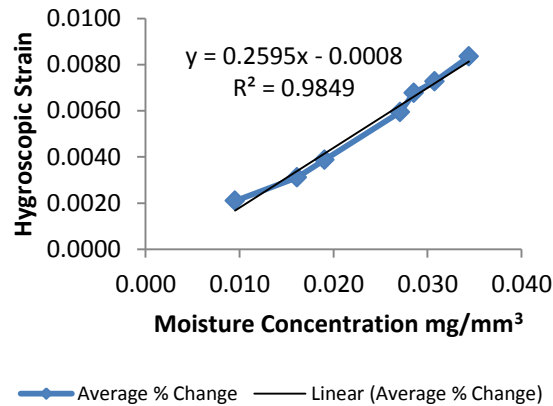


Figure 12: Linear approximation of hygroscopic strain versus moisture concentration for purpose of determining β

The moisture uptake following ideal Fickian diffusion as calculated with equation [10] is also plotted in Figure 13, using the same diffusion coefficient and saturated moisture content values that were used in the FEA model. For the thin sample, moisture concentration was considered to be uniform for the entire sample thickness in order to obtain an approximation of total moisture uptake. As shown in Table 4, these values only deviated by 1.70% from the experimentally obtained results, thus providing additional evidence that the discrepancy in the FEA model was likely associated with numerical error and not the material parameters.

A comparison between the FEA models and experimentally determined diametrical expansion is shown in Table 5. The FEA model which was formulated with the short term data basis data predicted a radial deformation of 0.426 mm, or a corresponding diametrical change of 0.852 mm. The experimental average for all five samples was 0.808 mm resulting in a simulation discrepancy of 5.4%. Experimental data was utilized to calculate the coefficient of hygroscopic expansion but the actual value entered into the FEA software is factored with the saturated moisture content. Because the saturated moisture content associated with the short term basis

model was an estimate to get the data to match the dimensionless model, the error in expansion may also be associated with the experiment not being run to full saturation in addition to numerical error.

As shown in Table 5, the model run based on the 8.5% published saturated content basis coefficients actually showed larger error at the 240 hour time of interest. The initial numerical error was also significantly larger, which may be attributed a diffusion coefficient which was found to be one order of magnitude less than the short term basis model. As 10^{-8} is a relatively small value for the commercial software it was possible that rounding error contributed to the discrepancy. This could be remedied by utilizing a time scale more appropriate for the 8.5% published basis model which would allow for a larger numerical value for the diffusion coefficient. It should also be noted that the relative size of this numerical error at the beginning of the simulation would be less evident if the analysis was run to a full saturation of 8.5%.

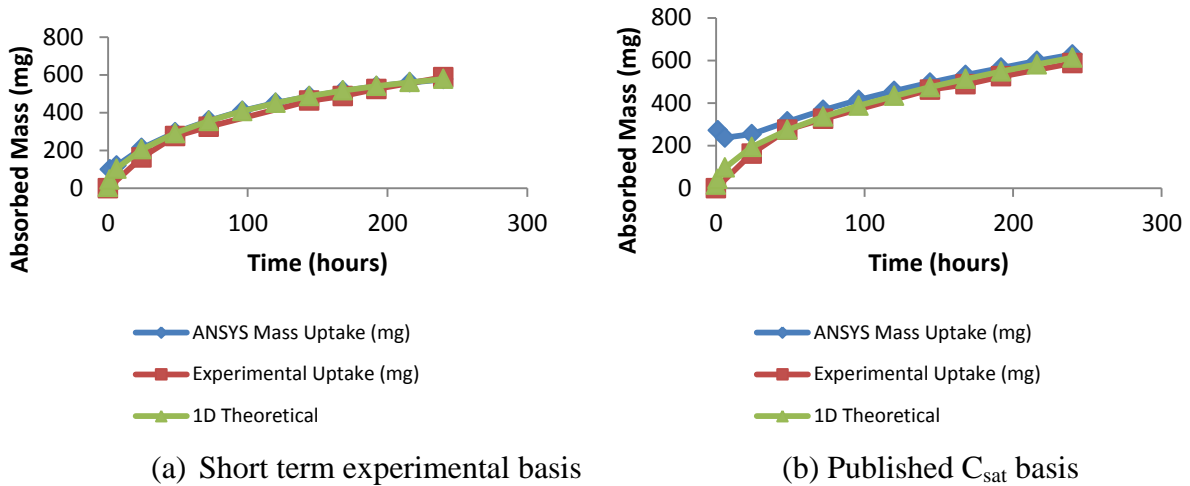


Figure 13: (a) Simulated moisture content versus experimental values for short term experimental basis and for (b) published C_{sat} basis

Table 4: Experimental, FEA, and 1D theoretical moisture uptake at 240 Hours

Method	Short Term Experimental		Published C_{sat} Basis	
	Moisture Mass at 240 h (mg)	Discrepancy from Experimental	Moisture Mass at 240 h (mg)	Discrepancy from Experimental
Experimental	588	N/A	Same	N/A
FEA	576	-2.07%	628	6.80%
1D Theoretical	578	-1.70%	613	4.21%

Table 5: Comparison of simulated diametrical change for disk samples versus experimental average

Method	Diametrical Expansion (mm)	Discrepancy from Experimental
Experimental	0.808	N/A
FEA (Short term basis)	0.852	5.45%
FEA (8.5% Csat basis)	0.933	15.43%

Conclusions

It was found that both the analytical and finite element models showed a reasonable approximation of moisture absorption for the 240 hour time period. The FEA model was found to predict hygroscopic strain with a discrepancy of 5.4%. It was unclear whether the difference in trend between the theoretical modeling and experimental results was due to non-Fickian behavior or if coefficients determined after allowing the material to reach full saturation would have yielded improved accuracy. Regardless, the data obtained from this experiment could certainly be utilized to simulate the approximate dimensional variation in an arbitrary part geometry made of identical material.

It is important to note that when performing such an analysis, the potential warping of relatively thin cross-sections may not necessarily be captured in the model. It was unclear in this experiment if the warping of the disk samples was a result of a certain degree of non-isotropic behavior or whether existing warping of the non-saturated samples was exacerbated during the absorption process. The warping was constrained during the measurement process in this experiment so that it did not skew the one-dimensional measurements. Further study should seek to understand the exact nature of this potential problem.

It should also be noted that quite different values for the saturated moisture content and coefficient of diffusion would likely be found when analyzing data for the material after the experiment was allowed to reach saturation. It was found that the experimental data could easily be fit to ideal Fickian behavior by adjusting the diffusion coefficient when using the published saturated moisture content of 8.5%. The purpose of this study was to fit the curve to the data for the time period of interest (0 to 240 hours) so alternate values were proposed as means of achieving more accurate results for this range.

As the material was assumed to behave in a Fickian manner, there was no reason to doubt that similar levels of discrepancy could be obtained for the complete set of data taken to full saturation if that assumption held true. Running the experiment to full saturation would be beneficial to provide evidence that the published values are accurate and to validate that the drying process was adequate. Additional studies should be performed with more time allotted for the drying process to verify that the samples do not lose any additional mass following subsequent 24 hour iterations in the oven.

References

1. Hsu, Hsiang-Chen, Hui-Yu Lee, Yu-Cha Hsu, and Shen-Li Fu. "Thermo-Hygro-Mechanical Design and Reliability Analysis for CMOS Image Sensor." *Journal of Thermal Stresses* 31:22 Apr. (2008): 917-34.
2. Yoon, Samson, Bongtae Han, Seungmin Cho, and Chang-Soo Jang. "Experimental Verification of Non-Linear Finite Element Analysis for Combined Hygroscopic and Thermo-Mechanical Stresses."
3. Zhang, Xueren, Tong Yan Tee, Hun Shen Ng, Jerome Teyseyre, and Shane Loo, Subodh Mhaisalkar, Fong Kuan Ng, Chwee Teck Lim, Xinyu Du, Eric Bool, Wenhui Zhu, Spencer Chew. "Comprehensive Hygro-thermal-mechanical Modelling and Testing of Stacked Die BGA Module with Molded Underfill." *Electronic Components and Technology Conference* (2005): 196-200.
4. Weide-Zaage, Kirsten, Walter Horaud, and Helene Fremont. "Moisture Diffusion in Printed Circuit Boards: Measurements and Finite Element Simulations." *Microelectronics Reliability* 45 (2005): 1662-67.
5. Hu, Jianzheng, Lianqiao Yang, and Moo Whan Shin. "Mechanism and Thermal Effect of Delamination in Light-Emitting Diode Packages." *Tima Editions / Thermic* (2005): 249-54.
6. Fan, X.J., and E. Suhir, eds. *Moisture Sensitivity of Plastic Packages of IC Devices*. New York: Springer Science + Business Media, 2010.
7. Celik, Emrah, Ibrahim Guven, and Erdogan Madenci. "Experimental and Numerical Characterization of Non-Fickian Moisture Diffusion in Electronic Packages." *Electronic Components and Technology Conference* (2007): 1069-73.
8. Sayman, Onur. "Analysis of Multi-Layered Composite Cylinders under Hygrothermal Loading." *Composites Part A*.36 (2005): 923-33.
9. Derrien, K., and P. Gilormini. "The Effect of Applied Stresses on the Equilibrium Moisture Content in Polymers." *Scripta Materialia* 56 (2007): 297-99.
10. Li, Shoujie, James Lee, and Jose Castro. "Effective Mass Diffusivity in Composites." *Journal of Composite Materials* 36.14 (2002): 1709-24.
11. Yu, Shi. "A Study of Moisture Diffusion in Polymeric Packaging Materials Especially at High Temperatures." *Graduate Thesis* (2002).
12. Wei, Yong-jie, Nick Silikas, Zhen-ting Zhang, and David C. Watts. "Hygroscopic Dimensional Changes of Self-Adhering and New Resin-Matrix Composites During Water Sorption/Desorption Cycles." *Dental Materials* 27 (2011): 259-66.
13. Poenninger, A., and B. Defoort. "Determination of the Coefficient of Moisture Expansion (CME)." *Proceedings of the 9th International Symposium on Materials in a Space Environment* (2003).
14. Madenci, Erdogan, and Ibrahim Guven. *The Finite Element Method and Applications in Engineering Using ANSYS*. New York: Springer Science + Business Media, 2006.
15. ISO. *ISO 62:2008(E)*. International Organization for Standardization, 2008.
16. ASTM. *ASTM D570*. American Society for Testing and Materials, 2010.