

## Thermo-Mechanical Properties Of An Epoxy Molding Compound In Pressure Cooker Environment

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**Keywords:** Epoxy Molding Compound, Creep, Moisture Effect

### Abstract

*Semi-conductor devices are mostly encapsulated by epoxy molding compound (EMC) materials. During encapsulation stresses are generated due to the curing of the epoxy. Moreover, additional stresses will build up during cooling down from molding to ambient temperature caused by the differences in the coefficient of thermal expansion. These residual stresses add up to the stresses generated during mechanical loading and may lead to product failure.*

*The viscoelastic properties of the encapsulation material (epoxy) depend highly on temperature, moisture and degree of cure. In case that moisture sensitivity was included in these researches the highest temperature had to be limited to 100 °C (In practice even to about 85 °C). This limitation is restrictive for the application of the obtained thermo-mechanical properties data in reliability studies.*

*The present research focused on the thermo-mechanical properties of the EMCs in harsh environment, i.e. Temp>100 °C & RH≈100%. For the present harsh environment study a special moisture-temperature-pressure chamber i.e. Pressure Vessel (PV) with a highly accurate tensile tester is designed and developed. The functionality and performance of the setup is assessed measuring the viscoelastic creep compliance of the EMC in dry condition supported by a measurement in a Dynamic Mechanical Analyzer, DMA Q800 TA instrument Co. Applying the Time-Temperature Superposing principle, the viscoelastic master-curves and related shift factors are also extracted. Finally the viscoelastic behavior of the EMC for the harsh environment is determined and the effect of the moisture is considered.*

### 1 Introduction

It is well known that polymers and polymer-based composites show strong temperature and time dependent behaviour. These viscoelastic properties are highly dependent on temperature, degree of cure and moisture. The first two factors have been reported in our previous works [1, 2] while concerning the issues for epoxy molding compounds. In [1] the thermo-mechanical properties of a series of epoxy resins were studied. It was shown that the filler decreases the cure shrinkage and thermal contraction of epoxy resins. However it increases the modulus below and above the glass transition temperature. Moreover in [2], the changes in viscoelastic properties during cure for commercial molding compounds is monitored. Using a specially developed shear tool, a full cure dependent visco-elastic model for the shear modulus of these materials was extracted.

Studies showed that moisture in any form causes swelling and degradation in polymer composites. This factor can also (and generally does) affect the mechanical and physical properties of the polymers and composites, as described in many studies [3–6].

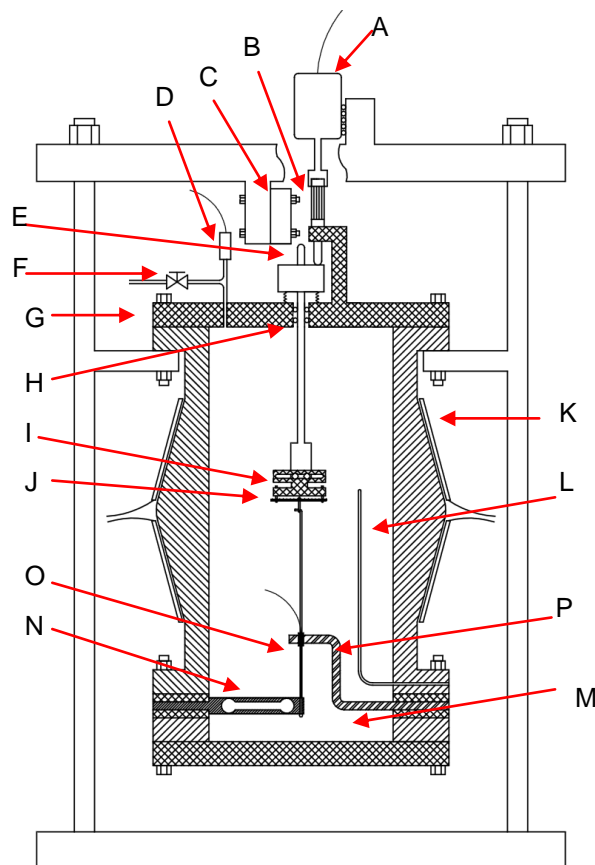
The present research focuses on the viscoelastic properties of EMCs in steam at elevated pressure conditions. For the present harsh environment study a special steam chamber (Pressure Vessel) with a highly accurate tensile setup is designed and tested. The functionality and performance of the setup is assessed measuring the viscoelastic creep compliance of an EMC in dry condition and comparing that with creep measurements using a commercial Dynamic Mechanical Analyzer. Applying the Time–Temperature Superposing principle, the viscoelastic creep compliance master curves and related shift factors are extracted and compared. As the next step, the mechanical properties of the EMC are determined for the pressurized steam environment and the effect of the moisture is quantified.

## **2. Design of the setup**

A special steam chamber is developed in order to measure the viscoelastic properties of the EMC in pressurized steam. The simplified schematic diagram of this setup is shown in Figure 1. The steam chamber with tensile setup initially contains 0.5 liter water. This water converts to steam while heating up the setup. The initial air in the system is removed by a pressure release valve in top of the vessel such that the chamber always contains water vapor at 100% humidity. For temperatures above 100 °C this corresponds to a higher pressure, a relation which is known from standard thermodynamics.

The designed and developed pressure vessel consists of a thick walled steel housing and a loading section. The loading section includes a 3-Point Bending set up (I, J), force transducer (N) and displacement measurement part. (B, C in Figure 1)

The moving set up consists of a controllable micrometer (B), a step motor with holding fixture (A) and a shaft (E). The controllable micrometer is positioned on top (=outside) of the vessel in the relatively cool area and is driven by a programmable stepping motor. The movable shaft enters the upper flange plate through a hole with appropriate pressure seal. The upper side of the shaft is clamped by the guiding fixture, moving the shaft vertically. The movement of the shaft is captured by the displacement transducer (Laser sensor) mounted outside of the vessel.



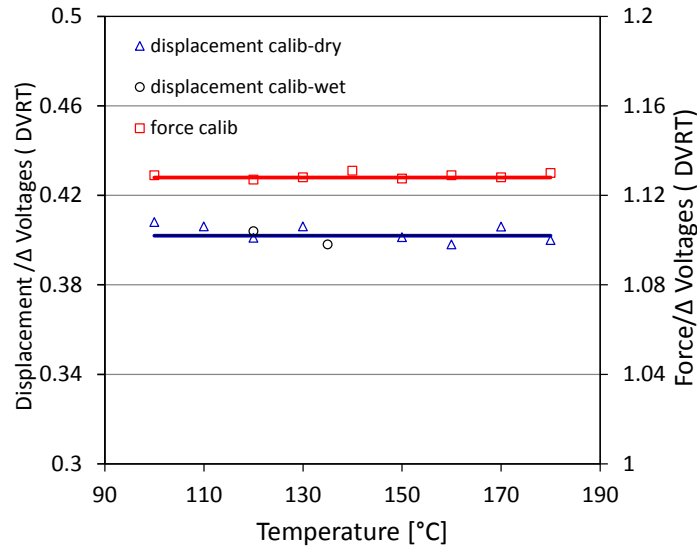
**Figure 1.** Schematic drawing of Pressure Vessel apparatus (not to scale). A: programmable stepping motor with guiding fixture; B: controllable micrometer; C: displacement transducer (laser sensor); D: pressure transducer; E: shaft; F: pressure release valve; G: top flange; H: pressure seal; I and J: 3PB setup with sample; K: heating elements; L: thermocouple; M: water reservoir; N: force transducer (loadcell); O: DVRT; P: DVRT holding fixture.

As Figure 1 shows the 3PB setup along with the sample are positioned at the lower part of the movable shaft. The 3PB setup can be used for specimen with various dimensions.

A rigid rod connects the sample to the load cell positioned on the lower flange plate inside the vessel. The load cell consists of a hollow beam (N) of which the deflection is measured using a Differential Variable Reluctance Transducer (O in Figure 1).

For a 3PB experiment the sample is loaded by moving the shaft upwards. The applied load and displacement of the load cell are monitored by recording the voltage differences of the DVRT. These voltages are converted to load and displacement of the DVRT using a calibration curve. The calibration data is obtained by applying known loads and displacement to the load cell at different temperatures, ranging from 100-180°C. The force and displacement calibration factors turn out to be 1.13 N/Volt and 0.406 mm/Volt (at 130 °C) and are slightly temperature depended (Figure 2). Calibration curves in wet conditions are much more involved and were performed at only two temperatures (120 °C and 135 °C). The results are included in Figure 2 as the open circles and show that the calibration constants is not affected by moisture.

The load cell is designed for relatively low forces. Therefore the beam of the load cell has a non-negligible displacement which must be considered. The displacement of the sample is defined as the difference between the shaft and loadcell displacement.



**Figure 2.** Force and displacement calibration data for the loadcell at different temperatures in dry and wet environment. Symbols: measured data, Full line: linear approximation.

The chamber of the pressure vessel includes three parts: top, middle and lower. The lower section contains the water reservoir and is made from a bolted flange with flange plate. This section also comprises the loadcell and DVRT. The middle section has two removable glass windows for sample mounting and observation. Furthermore, this section is equipped with heating elements and thermocouple. The top part is made up from a bolted flange with flange plate on which the motion part with laser sensor is installed. This section also entails the pressure transducer measuring the inside steam pressure.

### 3 Experiments

#### 3.1 Material

A commercial epoxy molding compound (MP8000, Nitto Co.) was molded into strips of 38×5×2mm by NXP Semiconductor Co., Nijmegen.

#### 3.2 Mechanical properties in dry environment

As a start, the newly constructed apparatus was tested by doing creep measurement in dry condition. The extracted mechanical properties are then compared to the creep data obtained from a commercially available apparatus (Dynamic Mechanical Analyzer, DMA Q800, TA Instruments Co.) in 3 point bending mode with the same sample size of 20×2×0.5 mm. The experiments cover the temperature range of 100-190 °C. The compliances versus time graphs of both measurements are plotted in Figure 3 and Figure 4.

Time–temperature superposition (TTS) [7] was applied for both sets of measurements. The creep compliance master curves were constructed by horizontal shifting of the compliance curves on the logarithmic frequency scale by a shift factor  $a_T$ , using  $T = 160$  °C as the reference temperature. The reduced time is defined as the creep time multiplied by a shift factor  $a_T$ , i.e.  $t_{red} = a_T t$ . The respective values for the shift functions are shown in Figure 5. At higher temperatures the curves could be fitted to the so-called WLF-equation, Eq.1 [8], whereas below the so-called switching temperature,  $T_c$ , an Arrhenius model was used, Eq.2. The extracted fitting parameters are shown in Figure 5.

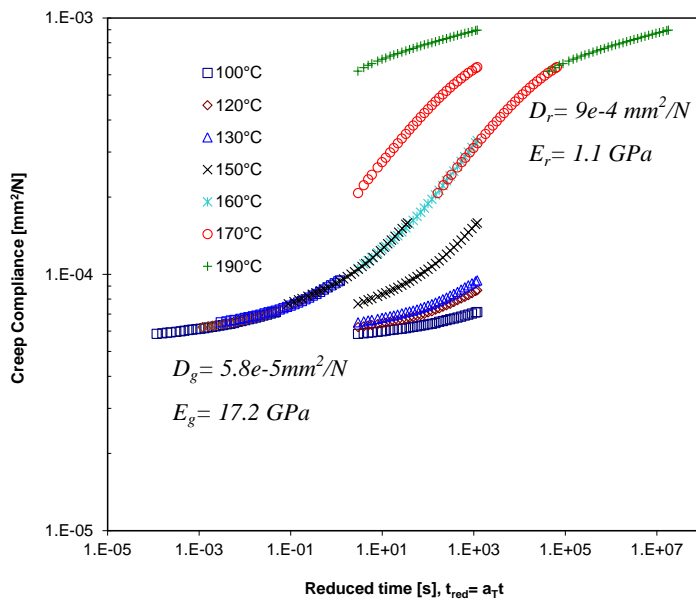
$$\log a_T^{WLF} = \frac{C_1(T - T_{ref})}{C_2 + T - T_{ref}} \quad T \geq T_c \quad \text{Eq.1}$$

$$\log a_T^{Arrh} = \frac{-H}{2.30R} \left( \frac{1}{T} - \frac{1}{T_0} \right) \quad T \leq T_c \quad \text{Eq.2}$$

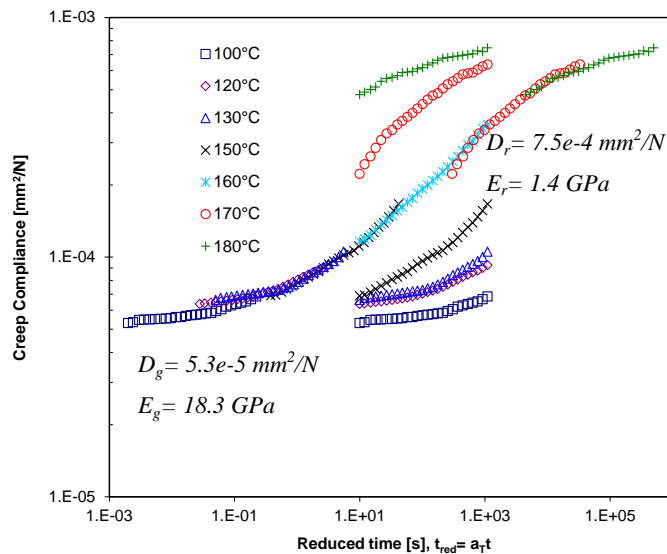
here  $H$  and  $R$  denote the activation energy and gas constant respectively.  $C_1$ ,  $C_2$ , and  $T_0$  are the fitting parameters.

As Figure 3 and Figure 4 show there is a relatively small difference in glassy and rubbery elastic modulus values. In the viscoelastic region the compliance continuously increased. The obtained master curves appear to be almost identical in the viscoelastic and rubbery region. However differences can be detected in the glassy region which might be attributed to the clamp stiffness of the instrument.

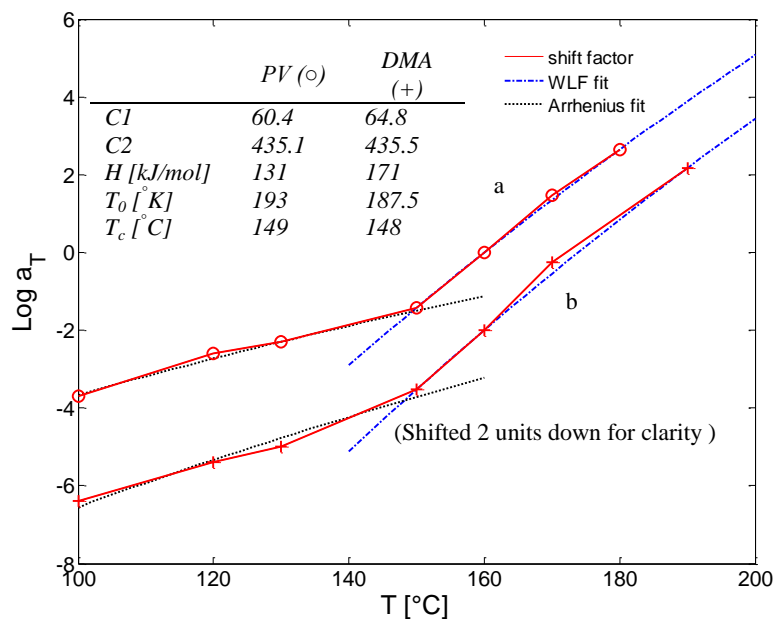
Due to the compliance deviation in the glassy state, small differences in the corresponding shift factors are also observed. However above the switching temperature, the shift factor curves are almost identical. (Figure 5)



**Figure 3.** DMA measurement of the 3PB creep compliance of the EMC sample along with the corresponding master curve at dry environment. Symbols: measured data in different temperatures.  $T_{ref} = 160^\circ\text{C}$ .



**Figure 4.** Pressure vessel measurement of the 3PB creep compliance of the EMC sample along with the corresponding master curve at dry environment. Symbols: measured data at different temperatures.  $T_{ref} = 160^\circ\text{C}$ .



**Figure 5.** Full line: shift factors corresponding to the master curves of Figure 3 and Figure 4, dash lines: WLF and Arrhenius model along with fitting parameter. Symbols: measurement data,  $T_{ref} = 160^\circ\text{C}$ . Curve a and b refer to the pressure vessel and DMA results, respectively.

A separate independent heating scan (not shown) was performed on the DMA at 1 Hz on an EMC sample (size of  $22.6 \times 2 \times 0.5$ ) to have a better access to the glassy and rubbery plateaus values and to establish the glass transition temperature. This resulted in an Elongation glassy modulus of 18 GPa and a rubbery modulus of 1 GPa. The  $T_g$  was determined as  $162^\circ\text{C}$  based on the temperature at which  $E_g$  reduced by a factor 3.

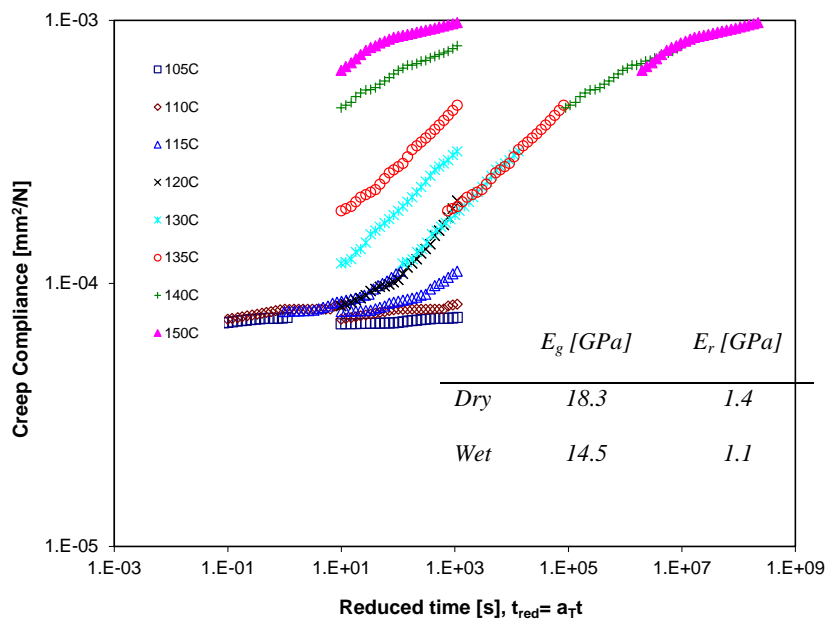
### 3.3 Creep under high pressure steam condition

The viscoelastic creep compliance of the EMC in wet condition was measured using the 3PB set up in the pressure vessel. The sample is placed in the setup and connected to the load cell

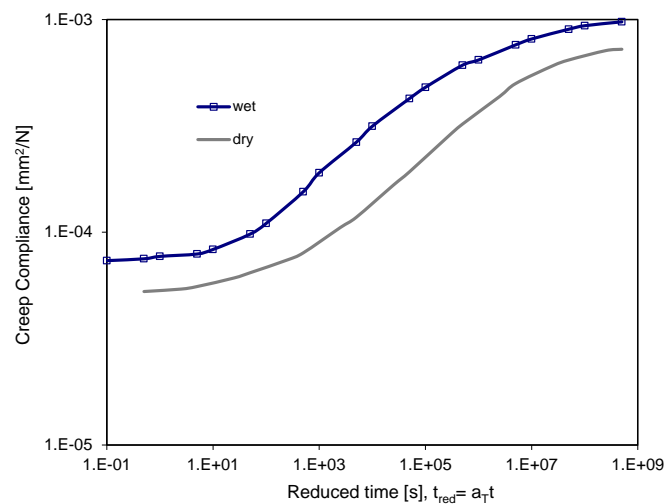
via the lower shaft. The glass window is closed and securely tightened. The temperature of the test section (and water/ steam mixture) is set to the required value.

During the heating up procedure the air/steam mixture was vented through a pressure release valve in top of the vessel. After some hold time at the set temperature the remaining air is negligible and the release valve is closed. It takes about 1 hour to reach the required temperature with steam inside the chamber. Afterwards, a hold time of 3 hours is programmed for the moisture to diffuse in the EMC.

The measured creep compliances are plotted in Figure 6. Applying the time-temperature superposing principle, the related master curve is also extracted assuming  $T_{ref} = 120^\circ\text{C}$ , Figure 6 and Figure 7.



**Figure 6.** EMC elongation creep compliance in wet environment, master curve via TTS principle,  $T_{ref} = 120^\circ\text{C}$ .



**Figure 7.** Creep compliance master curve in dry and wet environment along with the corresponding shift factor,  $T_{ref} = 120^\circ\text{C}$ .

It is known that the process of moisture absorption in polymers is accompanied by swelling which is always associated with changes in the mechanical properties.

As Figure 7 shows the absorbed water decreases the glassy and rubbery elastic moduli by almost 20%. In addition water absorption speeded up the relaxation process. The shapes of the master curves seem to be almost identical in both environments. However the creep occurred almost 40 times faster in moisturized condition and the master curve transferred to the shorter time scale. This phenomenon can be attributed to the  $T_g$  decrease in wet status.

#### 4 Conclusions

In this study the thermo-mechanical properties of an Epoxy molding compound in harsh environment is investigated. A special steam chamber with a highly accurate tensile setup is designed. The performance of the set up in dry condition is verified measuring the creep compliance of the EMC sample. Furthermore, the viscoelastic creep compliance of the EMC in wet environment was extracted and it was observed that steam considerably changes the EMC's mechanical properties.

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