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Characterization of epoxy based highly filled die attach materials in microelectronics

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Abstract

In semiconductor technologies thermally and electrically conductive adhesives are widely used to attach the die to the substrate. Focus of this work are Isotropic Conductive Adhesives (ICA) with a high amount of electrically conductive filler particles and the characterization of such materials.

One of the challenges of the characterization of die attach materials is to obtain a stable material response, since the material is highly sensitive to the applied curing and temperature loading conditions. This leads to significant changes in the material properties due to post curing and aging caused by decomposition. Viscoelastic material properties from samples with different curing conditions were determined by dynamic mechanical analysis (DMA) in order to evaluate the material model for thermomechanical simulation in a wide temperature range.

The stresses in a package are predominantly driven by the mismatch of the coefficients of thermal expansion (CTE) of the involved materials. Thermomechanical analysis (TMA) was used to measure the orthotropic CTE of die attach materials.

Additional material parameters for thermomechanical simulation such as Poisson's ratio are mostly assumed. In this paper Poisson's ratio measurements at different temperatures are evaluated by Digital Image Correlation (DIC).

The verification of the material model using validation samples like bulk samples are included and the results are discussed.

1. Changes of the material properties of the glue die attach

Virtual prototyping for product development using cost efficient simulation method is already established. However simulation requires a comprehensive understanding of the behavior of the involved materials.

A package comprises a variety of involved materials. Moreover the assembly of the package contains several process steps using different temperature conditions as a loading situation.

One of the assembly steps is the curing of the glue die attach material (DA) after 'pick and place' of the die. Typical curing temperatures during DA are in the range of 150°C to 200°C. Afterwards the glue die attach goes through subsequent steps like wire bonding typically done above 200°C or molding and post-mold curing at 175°C. Each of these assembly process steps have an effect on the material properties of the glue die attach.

As a consequence the material characterization should be done either on samples cut out of real packages or on samples treated with the same temperature condition as in the assembly process. Because the temperature profiles depend on package type and material combinations, a large effort is required to do a temperature profile specific die attach characterization.

The following investigation shows the effect of the process temperature on the thermomechanical properties of the glue die attach. It involves the effects on the temperature and time dependent Young's modulus and on the CTE.

Die attach characterization and (independent) validation experiments were performed using bulk specimen.

2. Dynamic mechanical analysis

The quality of the measurement is highly dependent on the sample preparation. The material is not homogeneous due to the filler particles and contains voids even after being exposed to vacuum. Therefore the measurement results can vary from sample to sample in a range of up to ten percent.

The used measurement equipment was the Q800 testing machine from TA Instruments. To obtain the viscoelastic material properties a frequency sweep measurement was performed. Therefore the material response of a dynamic (sinusoidal) load with oscillating amplitude of a small strain in the linear viscoelastic region at different frequencies was recorded while the temperature is ramped continuously from the starting temperature of -60°C up to 220°C.

The heating run was repeated five times and the measurement at 1Hz was used to investigate the change of the temperature dependent Young's modulus. The results are shown in Fig. 1.

The matrix of the glue die attach is a polymer material. At temperatures below the glass transition temperature T_G the material is stiff. As the temperature passes the transition region the material stiffness drops. The material is getting stiffer after sustaining an additional heating run and the largest difference was

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observed between the first and the second heating run. The material change between the fourth and fifth heating run was smaller but still detectable.

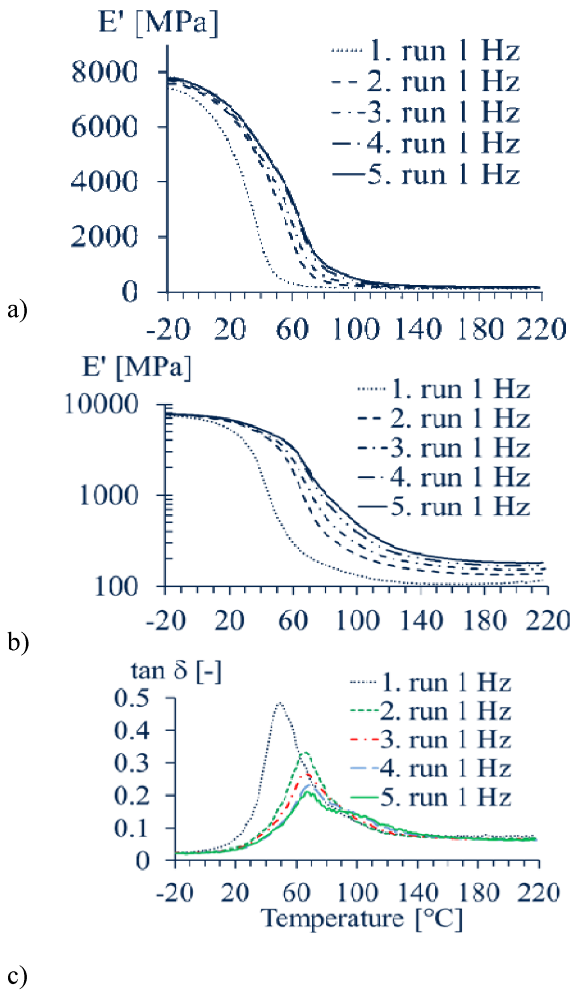


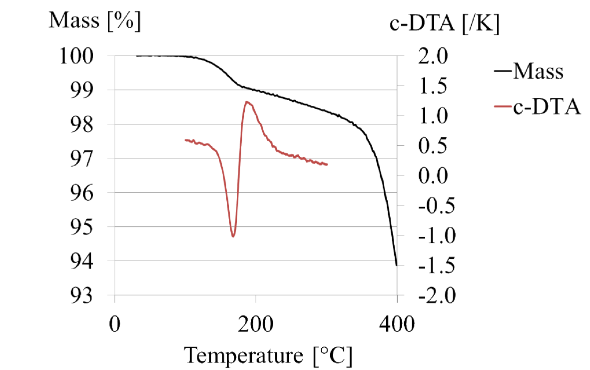
Figure 1 a) Storage modulus E' over temperature during several heating runs b) Young's modulus E' shown on logarithmic scale to compare the rubbery modulus during several heating runs c) Loss factor tanδ' over temperature during several heating runs

The increase of the rubbery modulus is shown in Fig.1b.

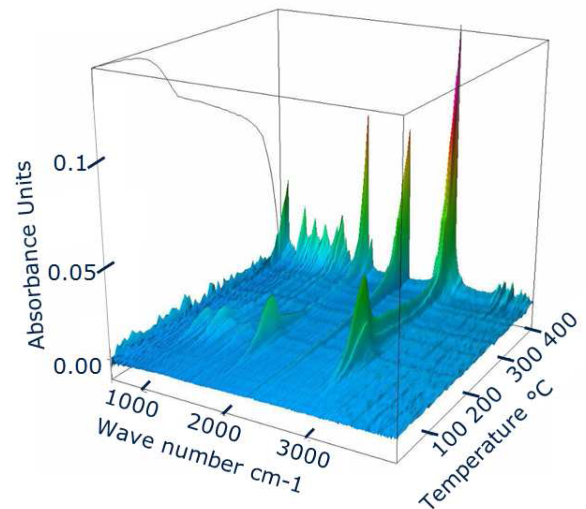
The peak of the loss factor tan δ shows the T_G around 50°C after curing the sample according to the data sheet (s. Fig. 1c). In the second run T_G was shifted to a higher temperature of about 67°C. Exposing the sample to further runs does not lead to an additional shift of T_G. However, already the second run shows not only a clear T_G peak but also an additional shoulder between the temperatures of 70°C up to 110°C.

The change of the material properties is caused by post-crosslinking processes in the polymer material accompanied by irreversible structure changes of the macromolecular lattice and chemical changes [1]. Thermal aging of the material could be caused by oxidation of the crosslinked network [1] but this would take place on a larger time scale. However, the increase of the rubbery modulus as shown in Fig. 1b is an indication for aging mechanism or further crosslinking. Also the shoulder in the loss factor curve in Fig. 1c indicates different chain lengths of the cross-linked polymer as a result of e.g. decomposition or additional crosslinking.

Thermal gravimetric analysis (TGA) together with Fourier transformed infrared spectroscopy (FTIR) was used to detect decomposition or evidence of degradation. By means of TGA the change in weight – mass loss – due to a temperature increase from room temperature up to 400°C was measured.



a)



b)

Figure 2a) Thermal gravimetric analysis (TGA) and differential thermal analysis (DTA) of the DA b) Fourier transformed infrared spectroscopy (FTIR)

The curing temperature of the material is 160°C. The weight loss between room temperature and 160°C is around 1% due to outgassing and curing. Increasing the temperature further to 400°C an additional weight loss of 5% was detected. At 200°C the presence of carbon

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dioxide with a wavelength of the spectral band around 2350 cm^{-1} [2] is an evidence of degradation mechanism as a result of decomposition.

Glue die attach is highly sensitive to temperature loading. It is not possible to clearly distinguish between the end of post-linking and the start of decomposition and degradation.

During package assembly the process step with the highest impact on the material properties of the die attach glue is the post-mold curing process (PMC) of the molding compound (MC), where the glue is exposed several hours to a constant high temperature. To assess the change in material properties of the glue due to this process step the viscoelastic material properties were determined with and without PMC.

The Young's modulus and the CTE were compared for a sample cured according to the data sheet recommendations and a sample exposed additionally to the post-mold curing temperature conditions. The result is shown in Fig. 3.

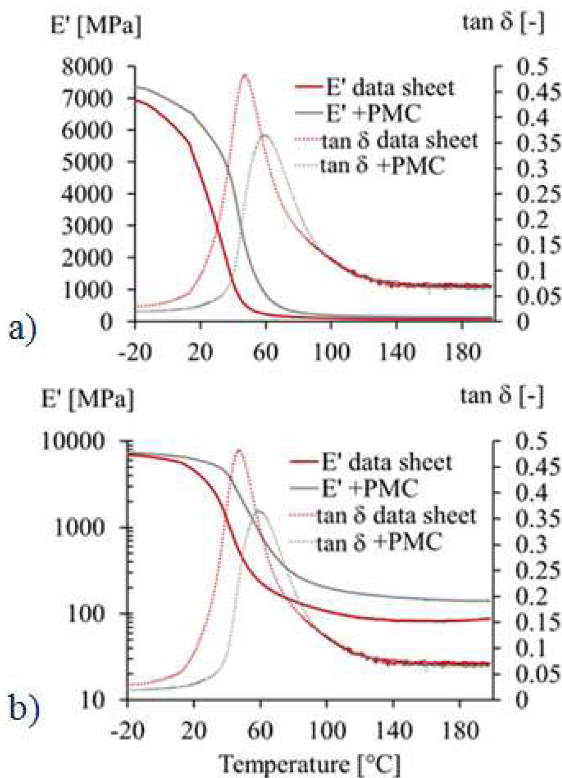

Figure 3a) Comparison of the storage modulus E' and the loss factor from a sample cured according to data sheet recommendations and a sample with an additional PMC b.) Young's modulus E' shown on logarithmic scale to compare the rubbery modulus with and without additional PMC

The glassy modulus of the samples is increased significantly by the additional post-mold curing, e.g. at 0°C from 6300 MPa to 6977 MPa (about 10%).

The T_G according to the peak of the loss factor is shifted from 49°C to 58°C. Therefore the largest difference between the storage modulus of the material cured according to data sheet recommendations and with additional post-mold curing can be found between 40°C and 70°C, e.g. at 40°C the storage modulus is increased by about 245% due to the additional PMC. Also the rubbery modulus is increased, e.g. at 100°C from 107 MPa to 201 MPa (about 88%).

3. Thermal mechanical analysis

The change in specimen length resulting from a temperature increase was detected using the measurement equipment TMA Q400 testing machine from TA Instruments.

For the CTE measurement only the second run was used to evaluate the coefficient of thermal expansion (CTE) [3]. The CTE is calculated from the measurement results as the ratio of thermal strain and the temperature difference. Thereby the slope of the dimensional change below T_G is referred to as CTE_1 and above T_G as CTE_2.

Fig. 4a shows the cross section in a package by means of scanning electron microscopy (SEM) and Fig. 4b a magnification in the region of the glue die attach. The material is highly filled with silver flakes.

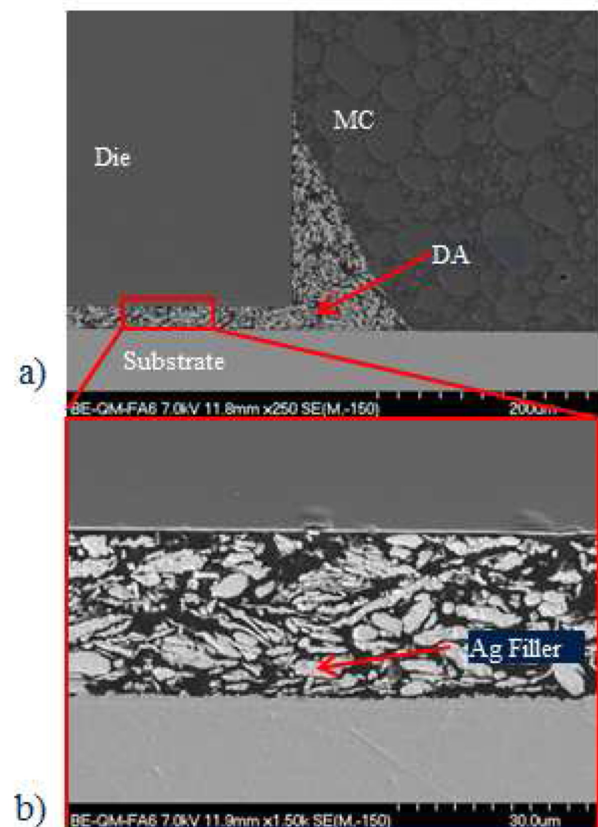

Figure 4a) SEM-image of a cross section of a package b) Magnification of the silver filled epoxy based glue DA layer

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As a result of the assembly process of dispensing the die attach material and placing the die, the silver filler particles below the die area are oriented in a preferred horizontal direction (s. Fig. 4b).

The DA layer in a package has a typical thickness of several 10µm. However, the measurements of the CTE were performed on bulk samples with a thickness being one order of magnitude larger. They were produced in a squeegee process.

The cross section from the side (cross section) and from the top (top down) were characterized by means of SEM on local scale and the filler particles were colored red for better recognition.

The result is shown in Fig. 5. Also in the artificial bulk samples a preferred orientation of the silver flakes was observed, like previously shown for the package. The top down view on the bulk sample shows that the filler particles are randomly distributed and oriented (s. Fig. 5b).

To assess the impact of this anisotropic orientation of filler particles the measurement of the CTE was repeated in different directions, in-plane and out-of-plane.

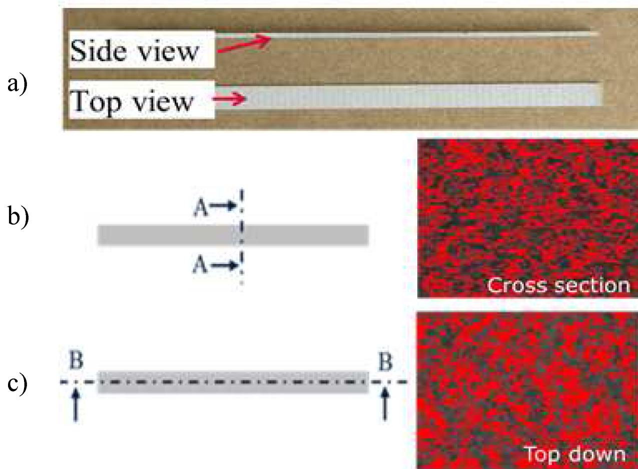

Figure 5a) Original image from the bulk samples in side view and top view b) SEM-image of a cross section on an artificial test sample (cross section)c) SEM-image from a grinded sample from the top (top down)

The comparison of the thermal strain over a temperature range from -50°C up to 250°C for the in-plane measurement doesn't show a significant difference between the samples cured according to data sheet recommendations and the samples exposed to an additional PMC temperature load (s. Fig. 6).

CTE_1 for the sample cured regarding data sheet is around 46 ppm/°C, the sample with the additional PMC thermal loading has a CTE_1 around 44 ppm/°C. The CTE_2 from the data sheet cured sample is around 65 ppm/°C comparable with the CTE_2 of the sample exposed to PMC with around 68 ppm/°C. The out-of-plane CTE of the differently treated samples are also comparable.

The CTE_1 out-of-plane for the data sheet sample and PMC sample are 104 ppm/°C and 85 ppm/°C, respectively, which are in the same range. Also the CTE_2 is in the same order of magnitude with 230 ppm/°C for the sample prepared after data sheet recommendation as for the sample additionally exposed to PMC with 265 ppm/°C.

However, there is a significant difference between the thermal expansion in-plane and out-of-plane of about factor four above T_G. One possible explanation for the significant difference between in-plane and out-of-plane is that the filler particles suppress the epoxy matrix in its thermal expansion in the in-plane direction which will be compensated in the out-of-plane direction.

Despite this significant difference, the supplier data sheets usually only provide the isotropic thermal expansion of the conductive DA material.

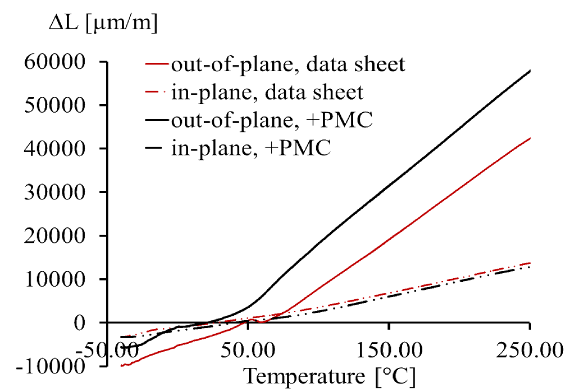

Figure 6) Dimensional change over temperature

4. Poisson's ratio calculation

Instead of shear and volumetric experiments, uniaxial experiments are often used to provide material data for simulation [1]. These uniaxial experiments require the Poisson´s ratio for the calculation of the shear and bulk modulus, which is often an assumed value based on educated guesses.

The Poisson's ratio ν is defined as the negative ratio of transverse and longitudinal strain in uniaxial tensile experiment.

In this paper the Poisson's ratio was assessed performing tensile experiments and subsequently analyzing the deformation of the samples using Digital Image Correlation DIC [3, 4, 5]. DIC is a non-destructive measurement method to obtain the surface deformation field of a loaded sample based on pattern recognition algorithm [6].

The testing equipment for the tensile experiments was the Gabo Eplexor 500 machine from Netzsch Gabo Instruments GmbH. The used tele centric stingray camera 201b and lens allows capturing images with 8bit intensity resolution and 1628X1236 spatial resolutions. The required detectable displacement should be in a range of 4 µm (1 Pixel) to obtain the correct displacement field.

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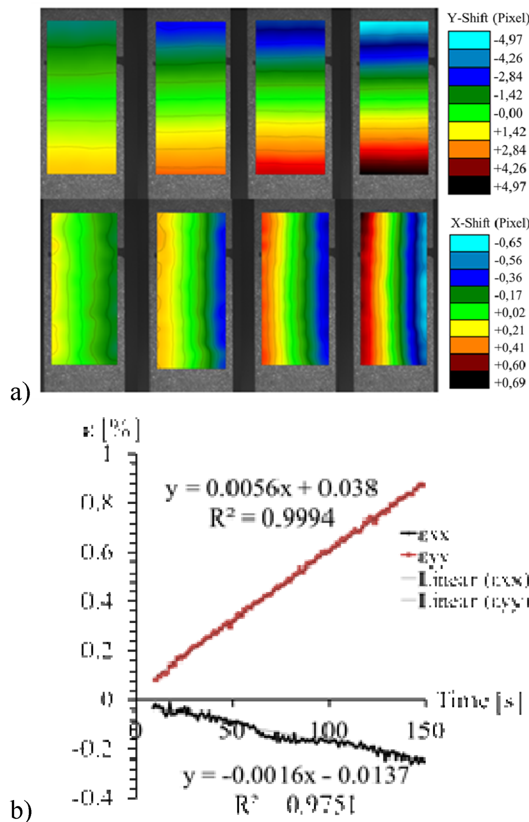

Figure 7a) Displacement field in y- and x-direction at e.g. -10°C (Sample 11) b) Strain ε_{xx} and ε_{yy} over time

Measurements were performed at 5 different temperatures. The strain in y- and x-direction was calculated based on a displacement driven experiment (s. Fig. 7a and Fig. 7b). To ensure the linear viscoelastic region the measurement was only evaluated for an appropriately limited strain. After reaching the temperature equilibrium at a certain measurement temperature the deformation during the experiment was monitored taking 1 image per second.

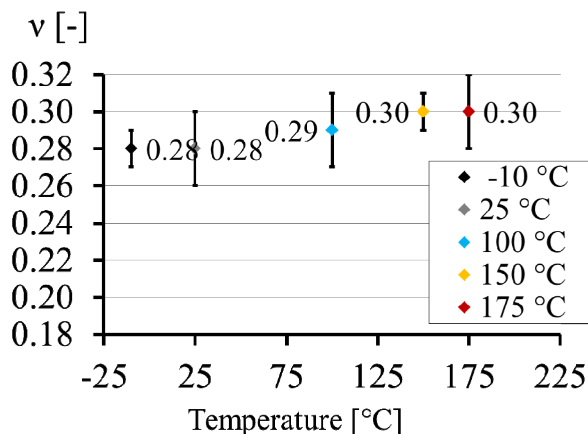

Figure 8) Temperature dependent Poisson' ratio

The matrix of the DA is an epoxy material, which has a Poisson's ratio around 0.35 below T_G and 0.5 above T_G [7, 8]. However, the material is highly filled with silver.

Based on an estimation using the rule of mixture [9] the Poisson's ratio should be 0.35 below T_G and up to 0.44 above T_G.

The preliminary results of the measurements as shown in Fig. 8 do not confirm the expectation and even justify the importance of a temperature dependent measurement method for the Poisson's ratio. The measurement was performed on at least 9 samples for each temperature group. The arithmetic mean value with the standard deviation is shown in Fig. 8.

The measured values below T_G are lower than the expected value. Moreover the measured Poisson's ratio above T_G does not show the expected rise towards the incompressible value of 0.5, but only a slight increase to a value of 0.3.

The reason behind is not yet fully understood and will be investigated further. Voids in the samples could be one possible reason. However, also in a package a certain amount of voids in the glue layer cannot be fully avoided. Therefore the impact of voids cannot be neglected.

5. Validation approach based on bulk samples

The viscoelastic material model was derived from the DMA frequency sweep measurement. Additional an independent test was performed in order to validate the material model.

For this purpose the relaxation in a tensile experiment was compared to simulation. The model for the simulation is shown in Fig. 9.

The relaxation experiment was performed at five temperatures. The applied strain in the linear viscoelastic region was held for 60 min and the force required holding this constant amount of deformation was recorded over time [3].

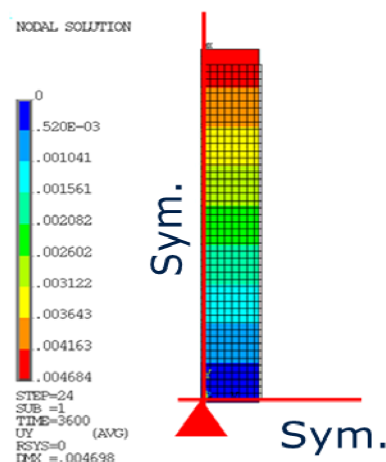

Figure 9) Simulation model for the relaxation experiment (1/8 of real sample)

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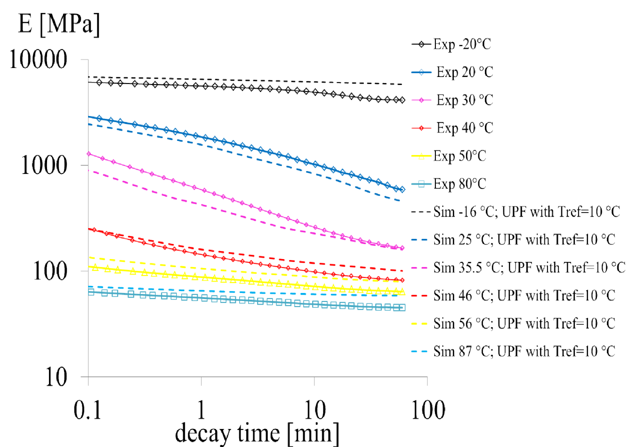

Figure 10) Comparison of the simulation results with the experimental results of the relaxation test

The temperature during the relaxation experiment was measured on the top of the sample surface and used as input for the simulation.

The implementation of the viscoelastic material data in ANSYS® requires the prony series for the master curve and the information how the series was constructed; i.e. the shifting based on the relation between time and temperature response of the material [3, 9, 10]. The used shifting function was a polynomial function, which was implemented as a user defined function [10].

For the epoxy based DA a good agreement between simulation and experimental results could be observed [s. Fig. 10]. The deviation between the simulation and experimental results can be caused by sample to sample variation but can also be due to the temperature loading input in the relaxation experiment.

The relaxation behavior at low and high temperatures as well as in the most important temperature region around T_G was predicted very well by the linear viscoelastic material model used.

6. Conclusions

The material characterization of epoxy based die attaches remains challenging since the material is highly sensitive to the applied curing and temperature loading conditions.

It was shown that different temperature loading conditions lead to significant changes in the material properties resulting from post-curing and aging due to e. g. decomposition. This leads to significant differences between the Young's modulus of the differently treated samples. The comparison of the dimensional change over temperature does not show significant differences.

It was also shown that conductive glue die attaches have an orthotropic CTE due to the preferred horizontal orientation of the silver flakes in the glue layer. The orthotropic CTE of the conductive DA was measured and a significant difference between the in-plane and the out-of-plane CTE was observed.

Moreover, tensile experiments were performed in order to calculate the temperature dependent Poisson's ratio by means of DIC. The measurement results show a deviation between expectation and measured values which is not yet fully understood and needs further investigation.

The linear viscoelastic material model was determined by frequency sweep measurements and verified by performing and simulating a relaxation experiment.

The measurement and the verification experiment were performed on bulk samples. The verification based on a relaxation experiment shows good agreement between simulation and experimental results.

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