ARTICLE

Complementary EIS/FTIR study of the degradation of adhesives in electronic packaging

Michael Schneider | Uta Gierth | Lenka Simunkova | Paul Gierth | Lars Rebenklau

Fraunhofer IKTS, Dresden, Germany

Correspondence
Michael Schneider, Fraunhofer IKTS, Dresden Winterbergstr. 28, 01277 Dresden, Germany.
Email: Michael.Schneider@ikts.fraunhofer.de

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Abstract
Adhesives are widely used in electronic packaging and are of vital importance in the reliability of electronic systems. A deep understanding of the degradation mechanism of adhesives under corrosion load plays a key role in lifetime prediction. Unfortunately, most of the common reliability tests are destructive. The present approach combines the nondestructive methods, electrochemical impedance spectroscopy and the Fourier-transform infrared spectroscopy, as powerful tools in a complementary manner to describe the degradation mechanism and kinetics of two epoxy-based adhesives, which are commonly used in electronic packaging. It is demonstrated that the application quality is the dominating impact on the optimization of the lifetime.

KEYWORDS
adhesives, degradation, EIS, electronic packaging, FTIR

1 | INTRODUCTION

All electronic devices, regardless of their application and function, are based on a combination of different materials. In particular, metallic and polymers are the most used materials in all cases, so that interfaces between these material groups are unavoidable. Mass products like printed boards have protective polymer films (solder stops) on their copper surface,[1] flex boards consisting of embedded copper foils in a polymeric layer,[2] the usage of conductive adhesives can be an alternative interconnection technology for mounting electronic components[3] and component packages or complete module packages can be based on molding all metallic surfaces in protective polymers.[4,5] The polymers for most of these applications are mainly based on epoxies.

The reliability and lifetime of electronic packaging consequently depend on the degradation behavior of the polymers under loading scenarios during the industrial application and are mainly determined by the strength of the interface of the adhesion.[6] According to the environment of the application, the loading scenario can be very complex and an interplay of various factors (e.g., moisture, temperature, pressure, pollutions, UV-radiation etc.). Nowadays, a number of short-time corrosion tests are available to prove the reliability of electronic packaging devices under various corrosion loading (neutral salt spray test, VDA-alternating climate test, cataplasma test[3]). A common test to study failure mechanisms in electronic packaging is the pressure cooker test (PC test).[7] Zhang et al.[8] studied the hydro swelling of polymer resin after the test using gravimetric methods and infrared spectroscopy. The problem with all these tests is the timely acceleration by intensifying the test conditions, which frequently implies overloading inclusive of the risk of the irreversible change of degradation mechanism.
and kinetics. Consequently, the differentiation of individual degradation effects is hardly possible. Usually, the tests are completed by mechanical proof, for example, lap shear tests (according to Reference [9]) or shear tests (according to Reference [10]). Unfortunately, all these test cases are destructive tests. Additionally, corrosion tests only allow a snapshot in time at the interruption or end of a test scenario. Monitoring of the degradation or corrosion process, respectively, is virtually impossible. Lam et al. [6] presented a stress-assisted hydrogenation model to describe the kinetics of degradation of adhesive joints in electronic packaging, which demonstrates the aforementioned complexity. They conclude that the time of failure particularly depends on the water uptake of the polymer. The water uptake associated with the hygroscopic swelling of polymers in electronic packaging is also the main subject in the publication of Ardebili et al. [11]. However, the authors exclusively used nonelectrochemical methods to determine the swelling process. One of the few approaches which is related to electrochemistry is a work of Verdingovas et al. [12], which investigated the leakage current as a sensitive signal for ion contamination and the condensation of water films on printed circuit boards (PCBs). The fact that electrochemical techniques are scarcely used to investigate the reliability of adhesives in electronic packaging is a surprise because electrochemical measurement techniques are well-established and successfully applied in corrosion research of protective coatings. Notably, electrochemical impedance spectroscopy (EIS) has been developed as a vital tool to study the degradation of polymer coatings over more than 20 years. [13–20] A number of experiences can be analogously applied to the problems of degradation of epoxy molds and adhesives in the field of electronic packaging. Lab experiments by using electrochemical methods usually reveal a better understanding of the fundamental mechanism of the degradation of adhesives and polymers. In the case of small excitation methods like EIS, the methods are nondestructive and can be supplemented by various material diagnostic techniques, for example, Fourier-transform infrared spectroscopy (FTIR). [21] FTIR is a well-established method to identify polymer groups or monitor the curing process or water uptake. [22] Currently, a number of publications combine EIS and FTIR to study corrosion or degradation of protective coatings simultaneously. [19,23,24] However, the combination of both techniques is fairly elaborate regarding sample preparation. In the present work, the authors combine the EIS and FTIR, two nondestructive methods, in separate ways to study the water uptake and the adhesive degradation depending on the immersion time in sodium nitrate solution during the exposition. The complementary application of both these methods allows the elucidation of the trigger of degradation and the description of the kinetics of water uptake as the prerequisite processes of the ongoing degradation process and finally component failure. Additionally, both techniques are used to evaluate the state of degradation before and after the complex corrosion load by using the PC test.

2 EXPERIMENTAL

2.1 Sample preparation

The substrates were commercially available FR4 PCB single-side plated with a 35 μm copper layer and standard ENIG (electroless nickel immersion gold)-finishes according to IPC standard 4552 with electroless nickel of 3–5 μm and immersion gold of 0.05 μm thickness (Multi Circuit Boards Ltd.). All metallic substrates were precleaned by wet wiping the surfaces with ethanol followed by drying. The adhesives were coated by screen printing using a 200 mesh and 40-micron wire diameter screen, with a 25-micron emulsion coating from a Koenen High Tech Screen. The process was done using an EKRA Microtronic 2 Screen Printer with a 75 shore squeegee. The squeegee was pressed at 1.5 bar on the samples with a 500 μm screen standoff and squeegee speed of 10 mm/s during the screen printing process.

The epoxies were selected based on different applications in the electronic packaging industry. EP 653-TD (Polytec, further on named as PT) is a highly reliable epoxy for dam and fill applications on PCB. ESL 242-SB (Electro Science Lab, further on named as ESL) is a mineral-filled adhesive and surface protection coating for electrical boards like coated alumina oxides or PCB.

A conventional ventilated box oven (Heraeus, Germany) has been used for all drying and curing steps. All temperature steps have been initially chosen according to datasheet indications and later been modified for further identification of degradation mechanisms. PT was finally cured for 45 min at 125°C and ESL was finally cured for 105 min at 125°C. Other curing conditions were investigated, but will be not part of this study. Therefore, the samples are referred to hereinafter as PT45 and ESL105.

2.2 Corrosion single load

The samples were immersed in an electrochemical 3-electrode flat cell containing an aerated 0.01 M NaNO₃ solution (σ ≈ 1.2 mS/cm; pH 7.5; at ambient temperature) acting as a working electrode. A saturated calomel electrode KE11 (Sensortechnik Meinsberg) and a platinum mesh act as a reference electrode and counter electrode, respectively. The cell was coupled with a
computer-controlled and impedance-enabled potentiostat PGU-IMP-Micro (IPS Ingenieurbüro P. Schrems). The behavior of the samples were monitored by discontinuous EIS over the whole time of immersion. The impedance measurements were carried out at open-circuit potentials or rather 0 mV using a sinus potential of 20 mV peak-to-peak between $10^4$ and $10^{-2}$ Hz.

2.3 | Corrosion multiple load

The combination of humidity (100% RH), temperature (121°C) and pressure (2 bar) was realized by the so-called PC test, according to DIN EN 60749-33[7] using an autoclave Wolf LaM-3-20-MCS-J. The samples were investigated with EIS before in 0.01 M NaNO₃ with the aforementioned measurement parameters before and after PC test.

2.4 | Shear test

Long time stability of material interfaces is typically investigated by shear force measurements of interface samples. This characterization was performed with the adaptation of MIL-STD-883 method 2019.9. Shear force samples were prepared by the separation of pcb boards into $2 \times 2$ mm shear force samples. The metallic sides of these samples were picked and placed into undried adhesives using an Expert Pick and Placer (Essemtec AG). Every characterization sample consists of eight measurement joints. The drying and curing of the samples has been performed following the previously given conditions. The shear test itself was carried out using a Dage Series 4000 Pull-Shear-Tester (Nordson Corporation) at a shear speed of 400 µm/s and a shear height of 200 µm above the adhesive surface. Test results consist of the maximum shear force to destroy the joining sample and the optical inspection of the fracture modes.

2.5 | FTIR spectroscopy

FTIR spectroscopy in attenuated total reflection (ATR) with diamond as an internal reflection element mode was complementarily applied to the EIS investigation before and after the corrosion load experiments. The spectrometer used was a Thermo Fisher Scientific™ Nicolet™ iS™10 (Thermo Fisher Scientific Inc.) with a range of wave numbers between 500 and $4,000 \text{ cm}^{-1}$ and a resolution of $8 \text{ cm}^{-1}$. The incidence angles amounted to 45°, and 52 scans per measurement were carried out. The spectrometer was housed in a glove box under a nitrogen-purged atmosphere and a relative humidity of about 2%.

3 | RESULTS AND DISCUSSION

3.1 | Shear tests

As discussed in Section 1, the shear test is a very common method in the electronic packaging sector. Therefore, the following discussion of results should be based on it. Figure 1 shows the results of the PC test for PT45 and ESL105. Surprisingly, the values of shear strength of the samples show a larger spread before exposition in the PC test in comparison with the measured values after the test. After the PC test, the shear strength is strongly decreased for both materials but more pronounced for ESL105 than PT45 and amounts to approximately 5% of the initial value.

3.2 | Corrosion single load

The single load was carried out as an immersion test with simultaneous EIS monitoring for both model approaches of various polymer adhesives (PT45 and ESL105), as described in Section 2. In the interest of clarity, Figure 2 shows a selection of recorded spectra representing the

![Figure 1](https://wileyonlinelibrary.com)
development of the impedance behavior of PT45 over the time of immersion up to 300 days. The magnitude of the impedance is exclusively characterized by a family of lines with a slope of $\frac{d \log(Z)}{d \log(f)} \cong -1$. The phase shift of almost $-90^\circ$ confirms the dominant capacitive behavior of the sample over almost the whole range of measured frequencies. Only at low frequencies does the phase shift deviate from $-90^\circ$ and tends to a smaller shift due to the increasing influence of resistive behavior. The frequency where the resistive influence first appears increases with ongoing time of immersion. The impedance is outstandingly high and amounts to $10^5 \Omega \cdot \text{cm}^2$ at $10^4$ Hz and reaches nearly $10^{11}$ at $10^{-2}$ Hz. Altogether, the results suggest a high corrosion protection and less degradation susceptibility.

Such behavior is commonly explained by an equivalent circuit illustrated in Figure 3. $R_{el}$ represents the electrolyte resistance whereas the resistance $R_{adh}$ and the capacitance $C_{adh}$, respectively, characterize the adhesive polymer. In this case, the adhesive is assumed as free of pre-existing defects. $R_{el}$ is not visible in the frequency range of the measured spectra, but can be assumed to $\approx 2 \, \text{k} \Omega \cdot \text{cm}^2$ based on the electrolyte conductivity and the geometric design of the cell assembly.

In a very practical manner, some researchers have reduced the equivalent circuit to a single capacitance element. Indeed, the development of the capacitance of the adhesive is the most interesting parameter because it reflects the water uptake of the polymer during the immersion time. In the present work, the authors likewise calculate $C_{adh}$ from the imaginary part ($Z_{im}$) of the complex impedance at 1 kHz according to the following equation:

$$C_{adh} = \frac{1}{-j2\pi f Z_{im}}. \quad (1)$$

The amount of water uptake is calculated by using the well-known Brasher–Kingsbury equation as follows:

$$X_V = \frac{100 \times \lg(C_f/C_0)}{\lg 80}, \quad (2)$$

where $X_V$ represents the water uptake in vol%, $C_f$ and $C_0$ the capacitance of the polymer at the time of measurement and at the beginning of immersion, respectively (Figure 4).

The results are shown in Figure 4. The capacitance shows a rapid rise within a short time after the immersion of the sample. It is assumed that the electrolyte penetrates into tiny pores or imperfect cured local sites on the outer site of the adhesive. In this early stage, the water uptake amounts to approximately 2.5%. This is followed by very slow diffusion of water into the depth of
the adhesive, which is indicated by a very slow increase of the capacitance over most of the time of immersion up to 7 months. After this time, the total uptake of water is about 4% of the volume.

In contradiction to PT45 is the behavior of ESL105. Figure 5 shows likewise selected spectra representing the development of the impedance over the time of immersion up to 66 days. In comparison with the spectra of PT45 shown in Figure 1, the differences of the behavior of ESL105 are clearly visible.

Already at the beginning of immersion, a significant influence of the ohmic behavior of the adhesive is visible in the low frequency range (between $10^{-1}$ and $10^{-2}$ Hz) and the magnitude of impedance at $10^{-2}$ Hz is almost two orders of magnitude lower in comparison with the PT45 sample. According to the literature regarding the corrosion protection, PT45 can be classified as “excellent” whereas ESL105 is only “good.”\[27\] With ongoing immersion time, the course of the spectra of the ESL105 sample differs completely from the shape of graphs presented in Figure 1. A second-time constant $\tau = R \times C$ appears, and according to the literature, the authors assume an equivalent circuit as shown in Figure 6.

The equivalent circuit consists of $R_{el}$ in series with an $R_{pore} C_{adh}$ element whereby $R_{pore}$ is, in turn, in series with a further RC element, which consists of the polarization resistance $R_p$ and the double layer capacitance $C_{dl}$. Such an equivalent circuit is frequently used to interpret the corrosion protection performance of polymer coatings (see inter alia\[26,29,30\]). This circuit is especially suitable if the coating or adhesive has application defects (pores, blisters) or weakly cured paths inside and, therefore, are often called a “pore model.”\[13,31\] Again, $R_{el}$ is not visible in the frequency range of the measured spectra but can be calculated, and due to the similar experimental design, it is identical to the aforementioned results of PT45. The capacitance behavior expressed in the high-frequency range ($\geq 10^3$ Hz) can also be used to evaluate the water uptake according to Equations (1) and (2). Figure 7 shows the development of the capacitance and the water uptake over the time of immersion up to 66 days.

The water uptake increases very rapidly within the first hours up to approximately 4% of the volume. Between 1 and 6 days, the diffusion of the electrolyte into the adhesive follows a $\sqrt{t}$ law similar to the behavior of electrolyte diffusion into PT45 but after only 8 days, the penetration velocity increases again and the content of water within the additive amounts to approximately

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**FIGURE 4** Development of the capacitance at 1 kHz and the resultant water uptake calculated based on the electrochemical impedance spectroscopy shown in Figure 2

**FIGURE 5** Bode plot of the electrochemical impedance spectra measured on ESL105 in 0.01 M NaNO$_3$, (a) magnitude and (b) phase shift (the lines represents the results of the fit routine based on the equivalent circuit shown in Figure 6, whereby constant phase elements were used instead of capacitances (C)) [Color figure can be viewed at wileyonlinelibrary.com]
8 vol% and shows an accelerating increase to about 20% after 20 days. Simultaneously, a second time constant appears in the impedance spectra. The development of the second time constant at lower frequencies identifies the influence of the defects in the adhesive coating, which locally results in a fast transport of electrolyte species along weak paths in the polymer, and consequently, the delamination of the adhesive coating characterized by the appearance of a double-layer capacitance $C_{dl}$. The delaminated or defect area $A_{del}$ at a distinct time $t$ can be estimated by the following equation:\(^{(3)}\):

$$A_{del} = \frac{C_{dl}}{C_{dl0}} \times 100\%$$

where $C_{dl0}$ represents the theoretical capacitance assumed to be 25 µF/cm². The calculated delaminated area of the immersed ESL105 sample amounts to <0.01% after 6 days and increases via 1% (8 days) to 7% after 20 days. This example likewise elucidated the advantage of the EIS, which allows a detailed description of the mechanism and the kinetics of the interface processes.\(^{(32)}\)

After 66 days, the shape of the impedance spectrum changes again and a third-time constant becomes apparent. Now, the sample shows massive delamination as well as local destruction of the adhesive and undermining corrosion of the ENIG metallization layer. The evaluation of water uptake has already reached their limitation. Consequently, the immersion experiment was terminated after about 2 months.

The aforementioned investigations were complemented by the FTIR investigation of the adhesive in the initial state and after the immersion experiment. Figures 8 and 9 show the spectra of PT45 and ESL105, respectively. For better impressiveness, the spectra are split into the range between 2,400 and 3,800 cm\(^{-1}\) (Figures 8a and 9a) and 600 and 2,400 cm\(^{-1}\) (Figures 8b and 9b). Comparing the initial stage of both adhesives, the fingerprint range of the ATR spectra differs in the number and intensity of the bands, thus characterizing the specific composition of the adhesives. Although both adhesives are epoxy-based polymers, the accurate chemical formula is subject to corporate secrecy by the producers and not known in detail. Therefore, the author's interpretation is based on the spectra of well-known epoxy systems. More interesting regarding this study, is the fact that in both cases, the spectra before and after the immersion experiment appear unchanged. Neither have bands disappeared nor have new bands appeared. Furthermore, significant band shifts or variations of intensities can be ruled out in the fingerprint range. In the upper frequency range, the integral intensity of the broad band at about 3,400 cm\(^{-1}\) representing $\nu$-OH stretching vibration increases for both materials after exposition in NaNO\(_3\). This confirms the water uptake measured by EIS, as demonstrated in Figures 4 and 7. Thereby, it appears that ESL105 even shows a significant $\nu$-OH band even in the initial stage. In contrast, the $\nu$-OH band of PT45 before the experiment is only indicated. It might be that ESL105 uptakes a small amount of water during the curing process or the curing condition did not lead to a complete volatilization of water during the processing of adhesion, respectively. The authors have renounced a further quantitative analysis of band intensities due to the experimental limitations, for example, no guarantee of exactly identical contact pressure of the samples during ATR measurements. In case of ESL105, the samples before and after immersion were the same but not the very same. However, the unchanged fingerprint range (Figures 8b and 9b) suggests that the molecular structure of the adhesives is not significantly influenced by the impact of the immersion test.
In the case of PT45, this result could be expected regarding the EIS. The almost unaltered FTIR spectra of ESL105 after the immersion test is in contrast to the expectation after EIS measurements. However, it confirms the assumption of the dominating tracking of the delamination process by penetration of electrolyte via pre‐existent defects after application of the adhesive and not by hydro swelling and/or intrinsic chemical degradation of the adhesive themselves.

### 3.3 Corrosion multiple load

To realize multiple load scenarios as typical for corrosion, the authors chose the application of the PC test (see Section 2). In contrast to the immersion test, it was only possible to investigate the samples before and after the corrosion load. The results for both adhesives are presented in Figures 10 and 11. Before aging by PC test, the spectra for both additives are similar to the spectra before the beginning of the corrosion single load, which should be expected and shows the reproducibility. After the test, the graphs show a similar shape in comparison with the initial state but the magnitudes decrease over the whole frequency range. The authors evaluated the water uptake considering the change of the capacitive reactance and indeed the more complex load had led to a higher water uptake (PT45 ≈ 11 vol% and ESL105 ≈ 21 vol%, respectively) already after 96 hr. This fact can particularly be explained by the high temperature. The relation between the diffusion coefficient $D$ and the temperature is commonly described by an Arrhenius equation (Equation 4) and $D$ increases exponentially with increasing temperature.

\[
D = D_0 e^{\frac{Q}{RT}},
\]

where $D_0$, $R$, $T$, and $Q$ represent the frequency factor, the gas constant, the absolute temperature, and the activation energy. The influence of the elevated pressure is more

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**FIGURE 8** Fourier‐transform infrared spectra in attenuated total reflection mode of PT45 before and after immersion test in 0.01 M NaNO₃; (a) range of wave number between 3,800 and 2,400 cm⁻¹, (b) range of wave number between 2,400 and 600 cm⁻¹

**FIGURE 9** Fourier‐transform infrared spectra in attenuated total reflection mode of ESL105 before and after immersion test in 0.01 M NaNO₃; (a) range of wavenumber between 3,800 and 2,400 cm⁻¹, (b) range of wavenumber between 2,400 and 600 cm⁻¹
complex. On the one hand, the evaluated pressure decreases the diffusion coefficient, on the other, the evaluated pressure presses the water inside pre-existing pores.

In case of PT45, the water uptake within 96 hr PC test is three times higher in comparison with the immersion test after 7 months. So far, the PC test seems to be only an acceleration of water penetration. Otherwise, in the case of ESL105, the intensification of the corrosion load due to the PC test is also accelerated but in comparison with the immersion test, the acceleration seems not so strictly enhanced, and moreover, does not result in a similar switch in the degradation mechanism as shown in the immersion test of ESL105 after 8 days.

Also, herein, the FTIR spectra can yield additional information in view of possible changes of the molecular structure. The authors compare the spectra of the sample before and after the PC test. The spectra of PT45 (Figure 12) are fairly similar and regarding the inevitable experimental error of the FTIR measurements, the authors assume a steady molecular structure of the adhesive after the PC test. In contrast, the comparison of the spectra before and after the PC test of ESL105 is shown in Figure 13.

The intensity of the broad band around $3,400 \text{ cm}^{-1}$ shows a visible increase after the PC test. Moreover, the so-called fingerprint range ($550$–$1,800 \text{ cm}^{-1}$) shows a significant alteration of the band pattern. This is clearly visible in Figure 14, which illustrates the fingerprint range in lateral magnification. Characteristic bands (marked in Figure 14) have disappeared after the PC test. The authors explain the decreased epoxide and solvent band as an additional curing of the polymer under the conditions of the test. The aromatic band decrease is assumed to be a degradation of the prepolymer. It has to be noted that the curing temperature and the temperature of the PC test are similar. This means that in the case of ESL105, two processes take place which are
contrasting in view of degradation. The complex corrosion load results in a secondary curing, which decreases less crosslinked paths, which act as defects in the initial state, but simultaneously the severe corrosion load enhances the water uptake inherently across the adhesive probably due to the change of the aromatic component of the prepolymer. Consequently, the PC test intervenes in the molecular structure of the adhesive due to the enhanced temperature and therefore adulterates the reality of the application of the adhesive.

4 CONCLUSION

The present work addresses the comparison of two different, epoxy-based adhesives, which are typically applied in electronic packaging and their degradation behavior under single corrosion load (immersion test) and multiple corrosion load (pressure cooker test). The results have shown that the EIS, complementarily used with the infrared spectroscopy, is a powerful tool to describe the kinetics of water uptake as well as the mechanism of degradation in detail. Thereby, both methods are non-destructive tests and allow the study of time-dependent behavior of the samples. The authors monitored the water uptake under a single corrosion load by EIS. It has been shown, that water uptake is the driving force for the degradation of perfectly applied and cured adhesives. However, in this case, the penetration of water through the adhesive is an aging process over long time and does not lead to a significant loss of adhesive properties. Thereby, the difference between the intrinsic properties of both the studied adhesives seems to be only gradual. More important is the application quality, less perfectly applied or cured adhesives tend to enhanced water uptake along preferred pathways ( pores or weakly crosslinked molecules) and result in a localized destruction of the adhesive and an accelerated component failure, which is difficult to predict. In this case, EIS spectra can be a leading indicator to predict the risk of component failure in a timely fashion.

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ORCID

Michael Schneider http://orcid.org/0000-0001-5881-0215

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