TAMPEREEN TEKNILLINEN YLIOPISTO
TAMPERE UNIVERSITY OF TECHNOLOGY

Kati Kokko

Reliability of ACA Joints with Conformal Coatings in Harsh Environments

Julkaisu 913 • Publication 913

Tampere 2010

Tampereen teknillinen yliopisto. Julkaisu 913
Tampere University of Technology. Publication 913

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Reliability of ACA Joints with Conformal Coatings in Harsh Environments

Thesis for the degree of Doctor of Science in Technology to be presented with due permission for public examination and criticism in Tietotalo Building, Auditorium TB109, at Tampere University of Technology, on the 1st of October 2010, at 12 noon.

Tampereen teknillinen yliopisto - Tampere University of Technology
Tampere 2010

ISBN 978-952-15-2438-7 (printed)
ISBN 978-952-15-2446-2 (PDF)
ISSN 1459-2045

TO MY FAMILY

To love someone deeply gives you strength.
Being loved by someone deeply gives you courage.
–Lao Tzu

ABSTRACT

The use of electronics is spreading to a variety of new applications. Electronics are facing different environments and reliability needs to be maintained. In harsh environments, the use of conformal coatings protects the electronics and improves their reliability. However, the performance of conformal coatings in different environments and applications needs to be ascertained.

This thesis reports the use of conformal coating materials and structures to protect anisotropically conductive adhesive joined flip chips against different environments, the main focus being their use in implantable electronics. With packaging technologies, the implant may be miniaturized, and the technologies used need to be proven to be extremely reliable. The environment inside the human body is very demanding, and thus thorough studies need to be conducted to ensure the protection capabilities of the conformal coatings and the reliability of the devices. In this thesis, the reliability testing of anisotropically conductive adhesive joined flip chips on FR-4 and PI substrates was studied in constant humidity tests, temperature cycling tests, salt spray tests, and hydrolysis tests using epoxy, parylene C and epoxy-parylene C conformal coatings. The test results were analyzed mainly in terms of the selection of the failure criterion, different failure modes and mechanisms. Conformal coating materials affect the failure modes in those tests and even though the failure mechanism remained the same in them, the stresses were not generated in the same way with coating causing delaminations in different interfaces as without coating. The results confirmed the overwhelming protection capability of parylene C against moisture and impurities. To conclude, electronics miniaturization has considerable potential for medical electronics and reliable solutions can be made small and flexible if needed.

ACKNOWLEDGEMENTS

This work was carried out at the Department of Electronics of the Faculty of Computing and Electrical Engineering at Tampere University of Technology during the period 2003-2010.

I wish to thank my supervisor, Professor Lauri Kettunen, for his guidance and support for finalizing this thesis. I also wish to thank my former instructor, Adjunct Professor Pekka Heino, for his guidance and the freedom he gave me during my doctoral studies. I am very grateful to my co-authors Laura Frisk, D.Sc. (Tech.), Hanna Harjunpää M.Sc., Anniina Parviainen M.Sc., Anna-Maija Haltia M.Sc., and Professor Minna Kellomäki, all at Tampere University of Technology.

During my doctoral studies I worked in the Electronics Packaging and Reliability Group, and I wish to thank all its members for their help and support during this process. Especially, I wish to thank Laura Frisk and Erja Sipilä Lic.Sc. (Tech.), for the many fruitful discussions and encouragement, and Janne Kiilunen M.Sc., for the help with test equipment.

I owe a debt of gratitude to my M.Sc. thesis supervisor, Professor Eero Ristolainen (†2005), who gave me the opportunity and support for doctoral studies. Without him this work would never have even been started.

This thesis was supported financially by the Academy of Finland, the Nokia Foundation, the Ulla Tuominen Foundation, the Tuula and Yrjö Neuvo Fund, the Jenny and Antti Wihuri Fund, and the Emil Aaltonen Fund, whose support is here gratefully acknowledged.

I wish to thank my friends, mother-in-law Pipsa, Eeva, Anna, John and Ossi, for their kind support and help. I am greatly indebted to my parents, Ritva and Kalevi (†18.06.2010), and my sister, Kirsi, for all the love and support during the years. We recently walked through hard times together and continue our journey honouring the memory of my father. Together we will be strong.

Most of all, I own my heartfelt thanks to my husband Timo, and our sons Viljami and Aleksi for being there for me and giving me unconditional love during this process. Without your support this work would not have been finished.

Tampere, August 2010

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LIST OF PUBLICATIONS

This thesis consists of an extended summary and the following publications:

I Kokko, K., Frisk, L. and Heino, P. "Thermal cycling of ACF joined flip chips on FR-4 and PI substrates with parylene C coating", Soldering & Surface Mount Technology, Vol. 22, No. 3, 2010, pp. 42-48.

II Kokko, K., Parviainen, A. and Frisk, L. "Corrosion protection of anisotropically conductive adhesive joined flip chips", Microelectronics Reliability, Vol. 50, No. 8, 2010, pp. 1152-1158.

III Kokko K., Harjunpää H., Haltia A-M., Heino P., Kellomäki M. "Effects of conformal coating on anisotropically conductive adhesive joints; a medical perspective", Soldering & Surface Mount Technology, Vol. 21, No. 4, 2009, pp. 4-11.

IV Kokko K., Harjunpää H., Heino P., Kellomäki M. "Influence of medical sterilization on ACA flip chip joints using conformal coating", Microelectronics Reliability, Vol. 49, No. 1, 2009, pp. 92-98.

V Kokko K., Harjunpää H., Heino P., Kellomäki M. "Composite coating structure in an implantable electronic device", Soldering & Surface Mount Technology, Vol. 21, No. 3, 2009, pp. 24-29.

VI Kokko K., Harjunpää H., Heino P., Kellomäki M. "Hydrolysis testing of ACF joined flip chip components with conformal coating", Proceedings of the 17th European Microelectronics and Packaging Conference & Exhibition (EMPC), Rimini, Italy, June 15th- 18th, 2009.

AUTHOR'S CONTRIBUTION

Publication I, "Thermal cycling of ACF joined flip chips on FR-4 and PI substrates with parylene C coating", was contributed by the author together with the co-authors as follows: the tests were planned with the help of Dr. Frisk, the test samples were assembled, the conformal coating processed, the testing carried out, and the results analyzed by the author. The SEM analysis was conducted at the Department of Materials Science at Tampere University of Technology under the supervision of the author. The author wrote the manuscript, and the co-authors commented and improved the text.

Publication II, "Corrosion protection of anisotropically conductive adhesive joined flip chips", was contributed by the author together with the co-authors as follows. The tests were planned with the help of Dr. Frisk, the test samples were assembled, and the testing carried out by the author with the help of A. Parviainen. The conformal coating was processed and the results were analyzed by the author. The SEM and EDS analysis was conducted at the Department of Materials Science at Tampere University of Technology under the supervision of the author. The author wrote the manuscript, and the co-authors commented and improved the text.

Publication III, "Effects of conformal coating on anisotropically conductive adhesive joints; a medical perspective", was contributed to by the author together with the co-authors as follows. The tests were planned, the test samples assembled, the testing carried out, and the results analyzed by the author. The conformal coatings were processed by H. Harjunpää, A-M. Haltia and the author. The SEM analysis was conducted at the Department of Materials Science at Tampere University of Technology under the supervision of the author. The author wrote the manuscript, and the co-authors commented and improved the text.

Publication IV, "Influence of medical sterilization on ACA flip chip joints using conformal coating", was contributed by the author together with the co-authors as follows. The tests were planned, the test samples assembled, the testing carried out, and the results analyzed by the author. The conformal coatings were processed, and the medical sterilization implemented by H. Harjunpää and the author. The DSC analysis was conducted at the Department of Biomedical Engineering at Tampere University of Technology under the supervision of the author with H. Harjunpää. The SEM analysis was conducted at the Department of Materials Science at Tampere University of Technology under the supervision of the author. The author wrote the manuscript, and the co-authors commented and improved the text.

Publication V, "Composite coating structure in an implantable electronic device", was contributed by the author together with the co-authors as follows. The tests were planned, the test samples assembled, the testing carried out, and the results analyzed by the author. The conformal coatings were processed, and the medical sterilization implemented by H. Harjunpää and the author. The SEM analysis was conducted at the Department of Materials Science at Tampere University of Technology under the supervision of the author. The author wrote the manuscript, and the co-authors commented and improved the text.

Publication VI, "Hydrolysis testing of ACF joined flip chip components with conformal coating", was contributed by the author together with the co-authors as follows. The tests were planned, the test samples assembled, and the results analyzed by the author. The testing was carried out at the Department of Biomedical Engineering at Tampere University of Technology under the supervision of H. Harjunpää and the author. The conformal coatings were processed and the medical sterilization implemented by H. Harjunpää and the author. The SEM analysis was conducted at the Department of Materials Science at Tampere University of Technology under the supervision of the author. The author wrote the manuscript.

LIST OF ABBREVIATIONS AND SYMBOLS

3-D	Three Dimensional
ACA	Anisotropically Conductive Adhesive
ACF	Anisotropically Conductive Film
ACP	Anisotropically Conductive Paste
ALT	Accelerated Life Testing
ASCF	Artificial Cerebrospinal Fluid
CNT	Carbon Nanotube
CTE	Coefficient of Thermal Expansion
DC	Direct Current
DRAM	Dynamic Random Access Memory
DSC	Differential Scanning Calorimeter
ECA	Electrically Conductive Adhesive
FDA	Food and Drug Administration
FR-4	Grade of substrate material
IC	Integrated Circuit
ICA	Isotropically Conductive Adhesive
I/O	Input/Output
MCM	Multi Chip Module
MEMS	Micro Electro Mechanical System
MVTR	Moisture Vapour Transmission Rate
NCA	Non-Conductive Adhesive
PBS	Phosphate Buffered Saline
PDA	Personal Digital Assistant
RH	Relative Humidity

SIP	System-In-Package
SOC	System-On-Chip
SOP	System-On-Package
T_g	Glass transition temperature
USP	United States Pharmacopeia

1 INTRODUCTION

The science of today is the technology of tomorrow.
–Edward Teller

In the world today electronics are almost everywhere. People become used to the idea of intelligent devices and applications that make their lives easier. Electronics is a part of everyday life through mobile phones, laptops, PDAs, etc. [Tum04a]. Furthermore, electronics have spread to a variety of areas ranging from applications in agriculture and the food industry to personal body area networks [Bae06][Wan06]. These diverse application areas and environments of use for the electronics pose serious challenges to the functioning of electronics. To fulfil all the requirements given to electronics, they need to be unnoticeable and reliable, in both software and hardware. The enabling technology for this, among the software and system technology, is the packaging of electronics [Tum01].

Miniaturization of electronics is one key issue when electronics needs to be smaller in size, have higher performance, and perform with increased functionality [Ris02]. There are many different miniaturization opportunities available today, and the most sophisticated ones can be roughly divided into four groups: system-on-chip (SOC), multichip module (MCM), system-in-package (SIP), and system-on-package (SOP) [Tum04b]. The future of SOC seems rather complicated, since the integration of analog with digital electronics and flash and DRAM memories into a single IC results in enormously complicated processes [Pat06]. The other miniaturization methods mentioned earlier consider more or less the packaging of multiple ICs in the same package. Furthermore, SIP and SOP enable the use of 3-D packaging and vertical stacking of ICs.

To achieve that level of miniaturization, inside the SOP or SIP package space saving solutions need to be chosen. One method of saving space is to use a flip chip technique for interconnection. In this case all the interconnections are under the chip, and no extra area is needed other than the area of the chip [Lau95]. Furthermore, the different solutions in 3-D packaging enable a high level of miniaturization and different techniques are introduced in references [Har05][Kaw08][Kha08][Min06][Yam03]. Miniature and space saving electronics also include substrate materials and flexible solutions in which electronics can bend along the other structures of the device [Bos09][Car09][Dea07], or can be folded to fit the package [Hei09][Kal05]. This dissertation uses flip chip technology as a miniaturization technique, and flexible substrates are considered as an alternative for the traditional, glass-fibre reinforced, FR-4 substrate.

In order to achieve reliable and durable electronics against harsh environments, the shielding needs to be considered. Conformal coatings are used to protect electronic devices from moisture, corrosion, and from other terrestrial environments such as a salt atmosphere, abrasion from particles, dust and blowing sand, handling, ozone, fungi, bioorganisms, cleaning solvents, and chemicals [Lic03]. Conformal coatings are typically applied to printed circuit boards as thin layers covering the whole board or a part of it. They are generally classified according to the molecular structure of their polymer backbone, and they can be divided into five groups: acrylic, epoxy, silicone, urethane, and parylene [Zha00]. Traditionally conformal coatings have been used in automotive, avionics, and military electronics, but their use is spreading to other application areas as the use of electronics is likewise spreading. One of the problems in the more extensive use of conformal coatings in different application areas is the lack of performance and reliability results [Hun06].

In medical devices, the packaging of electronics is one of the key issues. The biocompatibility, functional interfaces, reliability, modelling, testing, calibration, and integration are all important factors of the packaging of microelectro mechanical systems used in medical applications (BioMEMS) [Sal06] and in the packaging of all medical electronic devices. Hermetic sealing of medical devices is commonly used and a metal casing around the device ensures the protection of the electronics inside. However, in different applications hard metal casing is not always the best choice for protection and packaging [Sey09]. Thin and flexible coatings may be preferable to the more traditional choices. Biocompatibility is important for medical devices when safety and reliability is considered. Biocompatibility and packaging complement one another, and are important to the success of future medical devices [Sal06].

1.1 Objectives and scope of the thesis

Reliability of an electronic device is crucial and in certain applications may be improved with conformal coatings. In this work the effects of conformal coatings on anisotropically conductive adhesive (ACA) joined flip chips were studied. The ACA joined flip chips with conformal coatings were tested extensively in different accelerated life tests, including thermal cycling, salt spray, constant temperature and humidity, and hydrolysis testing. The failure mechanisms of the joints were studied, and compared with non-coated test samples. The application area in the research was mainly concerned with medical electronics, where the electronic devices could be used inside the human body. The demands for electronics protection are high in these applications and the usage environment of such is corrosive, even though thermally stable. Furthermore, the durability of ACA joined flip chips coated with conformal coatings against medical sterilization was studied. Both rigid and flexible substrates were used, and several conformal coating materials and combinations were selected.

The aim of the work was to ascertain whether the conformal, organic coatings could even partly substitute for the hard metal casings in the shielding of electronics in harsh environments. The reliability of the joints in different atmospheres was studied and the effect of conformal coatings on reliability was evaluated. Furthermore, failure criteria are a function of the specific applications, and the effects of different selections were compared. The failure modes differed according to the conformal coating used and thus the failure criteria chosen affected the reliability results obtained.

1.2 Structure of the thesis

This thesis consists of an extended summary followed by six publications. The extended summary is divided into 5 chapters providing relevant background on the topic and presenting the main results. Chapter 1 gives a general introduction to the need for miniaturization of electronics and conformal coatings in electronics, and Chapter 2 introduces the flip chip technology concentrating on the ACA technology. In Chapter 3 the conformal coatings and coating methods are presented and the materials used in this work are discussed in more detail. Chapter 4 deals with the reliability of the ACA flip chip joints with conformal coatings specializing in the field of medical electronics. In this chapter failure criteria, failure mode, and failure mechanisms are discussed. In Chapter 5 the final conclusions and a summary of the publications are presented.

2 FLIP CHIP JOINING USING ANISOTROPIC CONDUCTIVE ADHESIVES

No great discovery was ever made without a bold guess.
–Isaac Newton

A chip in which the active area or I/O side is facing the substrate is called a flip chip [Lau95]. It can be mounted on the substrate with various interconnect materials and methods, generally divided into solder joining and adhesive joining. Adhesives have many advantages over solders, including environmental properties, mild processing conditions, fewer processing steps, and fine pitch capability [Li06]. However, adhesives still have some limitations, such as lower electrical and thermal conductivity compared to solder joints, conductivity fatigue in reliability tests, limited current carrying capability, metal migration fatigue in reliability and high voltage tests, and poor impact strength [Li06]. Although adhesives have many advantages over solders and the research is active, adhesives cannot replace solders in every application.

In adhesive joining electrically conductive adhesives (ECAs) are used to form the conductive path from the chip to the substrate. Furthermore, non-conductive adhesives (NCAs) can be used with stud bumps forming direct physical contact. Different ECAs are available consisting of an organic binder matrix and metal fillers [Li06]. They can be divided according to their conductive filler loading level into isotropically conductive adhesives (ICAs) and anisotropically conductive adhesives (ACAs) [Li06]. ICAs conduct equally in all directions, and the percolation threshold has been passed [Lic05]. Percolation theory states that for electrical conductivity in a polymer a minimum critical volume of filler particles is required in order for each filler particle to be in contact with two other particles [Kir73][Lic05]. At the percolation threshold, the resistivity of the adhesive drops abruptly and thus starts to conduct isotropically. In ACAs, the volume of conductive particles is low, and the percolation threshold has not been passed. Therefore the adhesive matrix remains non-conductive. In this work, ACAs were used for interconnections, and they are discussed in more detail in the following sections. The reason why ACAs have been selected as the interconnection method is their future promise and possible environmental friendliness. Furthermore, the mild processing conditions and fine pitch capability are good properties in demanding applications.

2.1 The structure of anisotropic conductive adhesives

With ACAs the loading level of conductive fillers is below the percolation threshold and thus prevents conduction in the adhesive matrix. Conduction is achieved in a vertical direction when the chip is connected to the substrate. Figure 1 shows a schematic illustration of an ACA joined chip. The

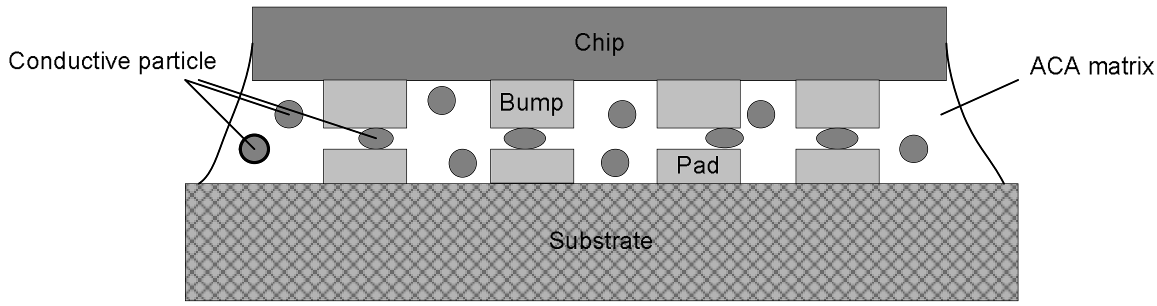


Figure 1 Schematic illustration of ACA joined flip chip.

conductive path has formed after the interconnection, when conductive particles have been trapped between the bumps and the pads.

Both thermosetting and thermoplastic materials are used as an adhesive matrix [Li06][Lin08]. Thermosetting material forms a three-dimensional cross-linked structure during curing, and it transforms into a rigid solid [Li06][Lin08]. Thermosetting adhesives include epoxies, silicones and cyanate esters, and the advantage of using them lies in their ability to maintain strength at high temperatures, good chemical and corrosion resistance, and low cost [Li06][Lin08]. Thermoplastic adhesives are rigid materials at temperatures below the glass transition temperature (T_g), but above T_g the polymers exhibit flow characteristics [Li06][Lin08]. Phenolic epoxy and maleimide acrylic preimidized polyimide are examples of thermoplastic adhesives, and the principal advantage of such adhesives is the relative ease of reworking [Li06][Lin08].

Conductivity of the adhesive is enabled with conductive particles added to the polymer matrix. In ACAs the conductive particles were first carbon (C) fibers and were then replaced by solder balls and after that with nickel (Ni) balls [Asa95]. The Ni particles were coated with gold (Au) to reduce the contact resistance [Asa95]. The development of conductive particles went on to polymer balls coated with Ni and Au [Asa95][Lin08]. These polymer balls were further improved by coating the balls with insulating resin, which is broken only under pressure [Asa95]. This improves the insulating property of the ACA in the substrate plane. As history repeats itself, the solder balls in ACA solutions have been reintroduced, and are under investigation by some research groups [Eom08a][Eom08b][Ver06][Win09]. The introduction of carbon nanotubes (CNT) as the conductive fillers in conductive adhesives is also under study [Wu09].

In this work two different ACAs from H&SHighTech Corp. were used in the studies. The main properties of the ACAs used are collected in Table 1. The values shown in the table were measured by the ACAs manufacturers. In both adhesives, non-conductive filler particles were added to the adhesive matrix. The filler particle used was silicon dioxide (SiO_2), and the diameter of the particles was 0.8 μm. The function of the particles in the adhesive matrix was to decrease the coefficient of thermal expansion (CTE).

Table 1 The main properties of the ACAs used in this work.

	Adhesive A	Adhesive B
Publication	I, III – VI	II
Adhesive type	Epoxy based thermoset	Epoxy based thermoset
Thickness /μm	40	40
Conductive particle, diameter /μm	Au coated polymer ball, 5	Au coated Ni, 8
T_g /°C	112	113
CTE (below T_g) / ppm/°C	40	39
CTE (above T_g) /ppm/°C	561	552
Moisture absorption /wt% (85°C/85%RH/500 h)	1.9	2.1

2.2 The assembly process

The assembly process of ACA interconnection includes dispensing, alignment, and bonding. ACAs are available in two distinct forms, paste and film. They can be denoted as anisotropic conductive paste (ACP) and anisotropic conductive film (ACF). Pastes can be printed by screen or stencil, or dispensed with a syringe [Liu07]. Films are supplied by manufacturers as a reel [Liu07] and they can be laminated on substrates using pre-bonding values recommended by the adhesive manufacturers. The pre-bonding values are temperature, time and pressure applied to the adhesive film during pre-bonding. After the adhesive dispensing and possible pre-bonding, the chip needs to be carefully aligned, since the ACA interconnections do not have self-alignment [Liu07]. Poor alignment may influence the pressure distribution and decrease the contact area for electrical interconnection [Liu07]. The next step in the ACA assembly process is the actual bonding process where both mechanical and electrical contacts are formed. During that process heat and pressure are applied to the chip for a certain amount of time. Figure 2 shows the process steps of an ACF assembly process. A good ACA flip chip joint should be stress or strain free and should have a strong inter-atomic bond [Lai96]. These depend on the curing temperature, heating rate and time, alignment, and pressure on a flip chip during bonding [Lai96].

In this work, the chips were connected using ACFs. The pre-bonding step was made with the flip chip bonder also used for the final bonding. The bonder used was a Toray FC-1000 semi-automatic flip chip bonder. The alignment accuracy of the machine was ± 5μm and the bonding parameters were selected from those recommended by the adhesive manufacturer. Table 2 collects the bonding parameters used in this work.

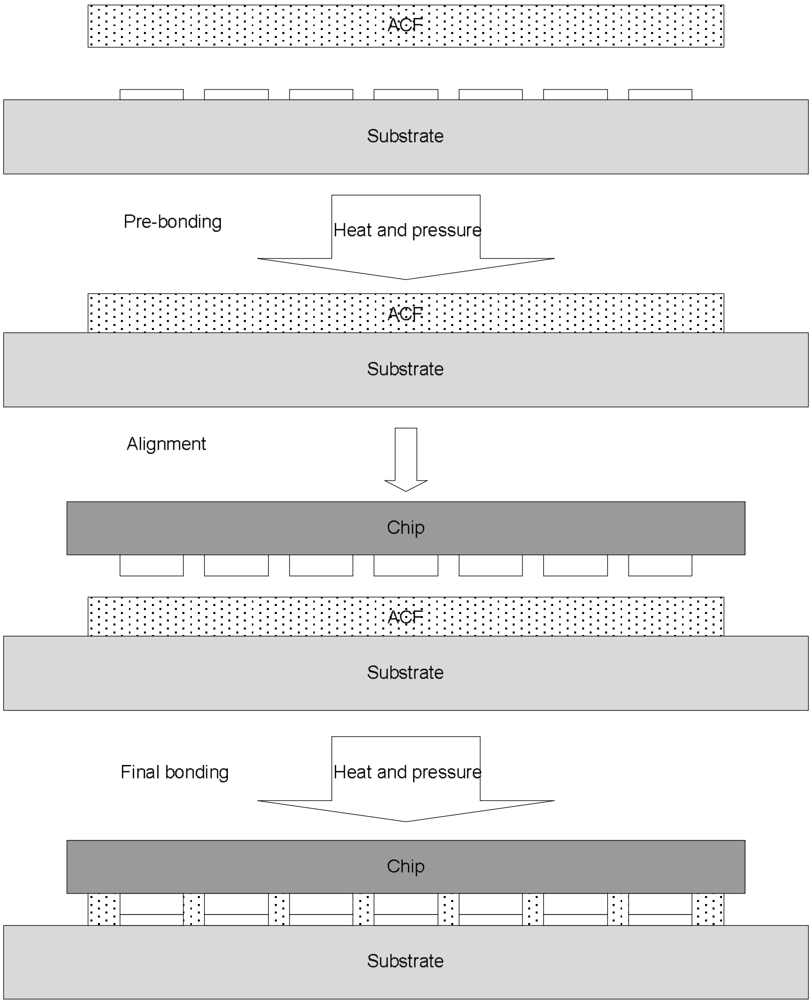


Figure 2 Schematic of the ACF joining process.

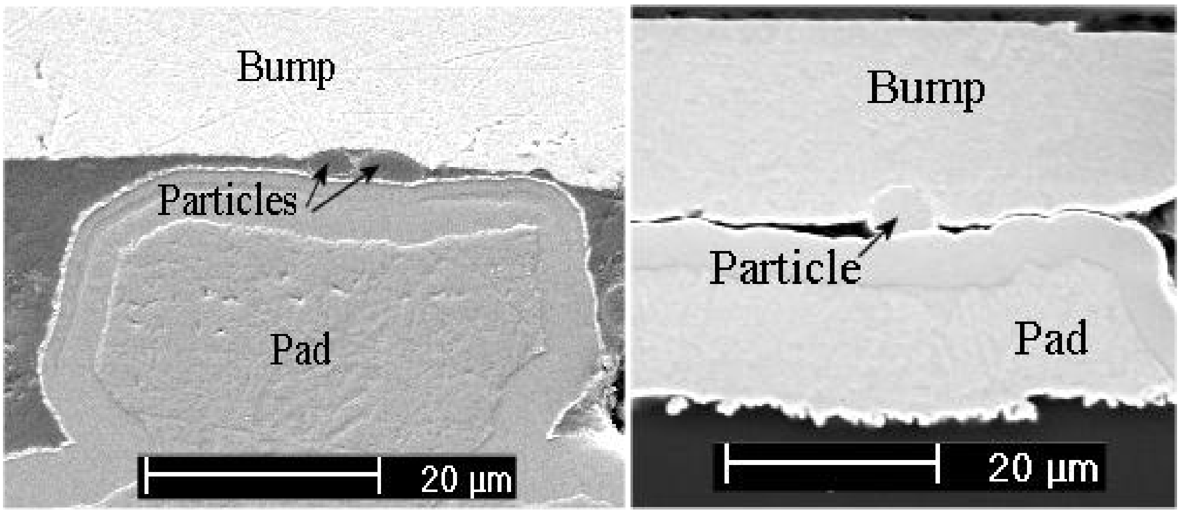
Table 2 The bonding parameters used in this work.

	Temperature /°C	Time /s	Pressure /MPa	Publication
Pre-bonding	100	5	1	I-VI
Final bonding I	210	25	80	I,II
Final bonding II	210	25	110	III,V
Final bonding III	210	25	136	IV,VI

The process parameters of an ACA assembly are temperature, pressure load, tacking time, and bonding time. Bonding quality is affected by bonding and curing temperature and time, temperature ramp rate, alignment accuracy, pressure value, pressure distribution and pressure application rate, bump height and uniformity, and board planarity and stiffness of the contact interfaces [Li06].

2.2.1 Pressure

Bonding force is critical in the electric performance of the ACA [Liu98]. The pressure is applied to the chip to press the bumps through the adhesive and to form a contact to the pads on the substrate, as presented in Figure 2. The conductive particles are of a ball shape before die bonding, and are trapped and deformed between the bumps and pads [Lin08]. The particle deformation can increase the contact area and thus the electrical conductivity [Kwo06b]. If an external insulation layer is on the conductive particles, it is damaged and the interior metal layer is exposed [Lin08]. With rigid particles (e.g. Ni), particle deformation is very limited and the contacts are created through indentation of particles into the metallization [Hu97]. Figure 3 shows the two different types of contacts made by these two particle alternatives. In Figure 3a the Au coated polymer particles are deformed and the contact is formed. In Figure 3b the rigid Ni particles penetrate the softer bump material (Cu) and the contact is formed.



(a) (b)

Figure 3 Interconnection using (a) deforming Au coated polymer balls, and (b) rigid Ni particles as conductive particles.

The bonding pressure needs to be selected carefully depending on the bump and conductive particle material. The adhesive manufacturer may provide recommendations, but there is a wide range of possible pressures and it needs to be evaluated which bonding pressure is preferable for the solution. Too low bonding pressure yields to high contact resistance [Cha02b]. If the applied pressure is too high, the metal coated polymer ball may crack causing the metal cover to crack also breaking the conductive path [Cha02b]. The ideal situation to deform particles is when the conductive particle is squashed until just before the metallic layers begin to break [Cha02b]. At that point the contact area is at its largest and contact resistance at its lowest. It is proposed that the d_1/d_2 degree from 1.2 to 2 gives the best combination of a low resistance value and a long-term reliability [Sep99].

The bonding pressure affects the amount of conductive particles trapped between the contacts in the chip and substrate. During bonding the viscosity of the ACA resin decreases and, depending on the bonding pressure, the flow along the bumps and bump spaces affects the number of conductive particles that remain trapped between the contacts [Yim07]. On the other hand the number of trapped particles influences the bonding force needed to achieve adequate deformation of the particles [Kwo06b].

2.2.2 Temperature

The bonding temperature during the bonding process determines the degree of curing of the ACA. The temperature used with a specified bonding time determines the degree of cure and the speed of the curing reaction [Che02a]. During the thermal curing process of epoxy based adhesive, higher temperature initiates and accelerates the cross-linking reaction by providing higher activation energy, thus resulting in a higher degree of cure [Udd04]. The higher degree of cure results in stronger chemical bonding at the adhesive interface and the adhesion strength also improves [Udd04]. However, the thermal stability performance of the ACA differs with different bonding temperatures, and if the ACA is cured at higher temperatures the thermal stability is decreased [Tan04]. It is suggested that this is caused by the thermal oxidation of the epoxy matrix of the ACA during bonding process [Tan04]. The researchers found networks scissoring of the adhesive matrix during bonding process at elevated temperatures [Tan04].

The physical and mechanical properties of the ACAs are also affected by the curing temperature. When the same degree of cure is achieved using different curing temperatures and times, the effect of bonding temperature can be evaluated. Lower curing temperature leads to denser and more homogeneous networks in cured ACFs, and they have higher modulus, higher glass transition temperature, lower coefficient of thermal expansion and less water barrier properties [Hwa08a]. It has been found that the glass transition temperature increases with the increase of the cross linking density and decreases with the increase of water absorption [Cha03].

The number of conductive particles trapped between bumps and pads is influenced by the curing rate of the ACA and the bonding temperature [Udd04]. During the early stages of the bonding process the ACA becomes soft and rubbery and the conductive particles are able to move within the ACA. When the curing process proceeds the ACA becomes hardened and the mobility of the conductive particles is lost. When higher bonding temperatures are used, higher curing degree is achieved in a shorter time, and the ACA becomes stiffer with higher modulus [Udd04]. The conductive particles recover a little after the bonding process, but the high contact area of the deformed particles remains stable after that process [Udd04]. In ACA joints with low curing degree, the number of conductive particles in the joint may even diminish during the thermal ageing and due to this the contact resistance increases [Riz05].

2.2.3 Time

The bonding time affects the degree of curing. In adhesive bonding the process time is relatively long and the degree of cure also depends on the bonding temperature [Che02a]. The bonding time could be decreased by increasing the bonding temperature, but if too high processing temperatures are used, the assembly itself may be impaired or the thermal stability decreased as discussed earlier. To become cost-effective the cycle time of the bonding process needs to be reduced and still maintain at low enough temperature. One option to reduce the bonding time of a single chip is to divide the bonding process into two phases, the first one occurring under pressure and the post-curing step in the normal reflow process [Sep03].

2.3 Factors affecting ACA joint reliability

The reliability of ACA joints is affected by numerous factors. Both the adhesive itself and the bonding parameters affect joint reliability [Cai09]. The conductive particle material used affects the reliability of ACF joints. It has been revealed that Au-coated polymer balls were more reliable than Ni particles when contact resistance and peel strength were studied after constant temperature, constant temperature with humidity, and temperature cycling tests [Yim99].

Different bonding parameters can lead to a shift of contact resistance of ACA joints and the decrease of adhesive strengths during the various environmental tests [Cai09]. These environmental tests include thermal cycling testing, constant temperature with humidity testing, and constant temperature testing. In thermal cycling testing the CTE mismatch between silicon chips and ACAs cause failures. Moisture affects the reliability of the ACA, and since ACA matrix absorbs moisture efficiently, the matrix swells causing tensile stress [Yin06].

The contact resistance of ACA joints is prone to instability over time, especially in high temperature and humidity conditions [Cha02a]. Compressive forces are generated to the ACA joint by the cure shrinkage of the adhesive and by the external bonding pressure during the manufacture [Kri98]. Both the cohesive strength within the adhesive and the adhesion of the joint must be sufficient to maintain this compressive force. The thermal expansion and the swelling of the adhesive due to moisture and mechanical stress from the environment will all try to diminish this compressive force [Kri98]. These cause increases in contact resistances and the performance of the ACA joints is impaired.

The bonding pressure affects the reliability of the adhesive joint. With temperature cycling testing, the lower bonding pressure yields to improved reliability of the ACA joints [Sep99][Yim98]. However, the substrate, bump, pad and particle material used affect the reliability behaviour of the ACA joint. With thinner, more flexible substrates the high bonding pressure is not as detrimental as it is with rigid substrates [Fri06a]. Also, after high temperature and humidity aging test the reliability is reduced, while the contact resistance is increased [Che06]. The contact resistance deterioration using higher bonding pressures may have been caused by elastic stress relief [Che06].

The materials used to form the contact also affect ACA joint reliability. With different substrate materials reliability can be altered, and the quality of the substrates also plays an important role in reliability. The different bump and pad geometries affect the reliability, as discussed in reference [Liu07]. The overetching of the pads on substrates is one issue that affects ACA joint reliability. If the pads are overetched the contact resistance is higher and the contact area is lower, causing lower reliability. Moreover, the use of high bonding pressure may cause the sinking of the pads into the substrate, causing the conductive particles not to deform enough [Liu07]. This can be overcome by using a larger pad area than the bump area [Liu07]. An example of overetching is seen in Figure 4, where a SEM image is presented. As can be seen, the pads on the substrate are much thinner than the bumps on the chip. Moreover, the shape of the pads reveals that the original width of the pads has been wider, and due to the fabrication process the pads have been overetched. In this work overetching of the pads was found in the substrates in the studies performed for Publications III-VI.

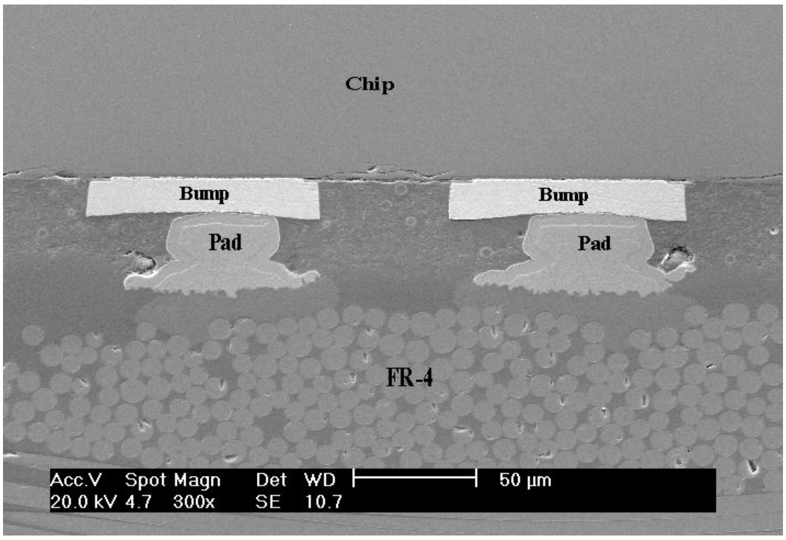


Figure 4 Micrograph of an interconnection showing overetching of the pads.

3 CONFORMAL COATINGS IN ELECTRONICS

Necessity is the mother of invention.
–Plato

To achieve reliable electronics in varying and harsh environments protective coatings can be applied to electronic circuits, and these are commonly known as conformal coatings [Hun06]. The two key functions of conformal coatings are environmental protection and electrical insulation. Conformal coating materials are used in electronics to protect the electronics from moisture, handling, ionic contaminants and particles [Lic03]. More extensively conformal coatings may also cover the photoresists, solder masks, and other coatings with special functions used in electronics manufacturing. However, these coatings are beyond the scope of this thesis.

There is very little information available to predict the lifetime of a product with different conformal coatings in different environments. This thesis extends the knowledge of the effects of conformal coatings on the reliability of flip chip joints.

3.1 Requirements and functions of conformal coatings

Conformal coatings have certain requirements that need to be fulfilled, so that they work as intended. The basic requirement is adhesion to all conformal coatings in every application [Mis05]. There are also other requirements for conformal coatings whose importance depends on the application in which they are used.

3.1.1 Adhesion

Adhesion is needed in every conformally coated system in order to ensure reliable functioning of the coating and protection of the covered components. Adhesion of the conformal coating must be good both initially and during the operation and lifetime of the hardware [Lic03]. Adhesion depends both on the type of coating material and the condition of the surface to which the coating is applied. To achieve good adhesion to a surface, the surface needs to be clean. No residues from fingerprints or fluxes are allowed, and thus thorough cleaning is needed before the coating process.

Adhesion is a function of the ability of the liquid coating to wet the surface, and it can be measured in terms of a wetting angle [Hau91]. The adsorption or thermodynamic theory of adhesion introduces dependence between the wetting angle and the surface tensions [Jan10]. The wetting angle is defined in a drop of liquid on a solid surface as the angle between the tangent plane and the surface of the liquid and the tangent plane and the surface of the solid [Sro01]. It can be calculated from Young's equation:

$$\gamma_{SV} = \gamma_{SL} + \gamma_{LV} cos\theta \qquad (1)$$

Where γ_{SV} is the surface tension from solid to vapour, γ_{SL} is the surface tension from solid to liquid, γ_{LV} the surface tension from liquid to vapour, and θ is the wetting angle [Jan10]. Figure 5 presents the wetting angle. The smaller the wetting angle the better the adhesion achieved.

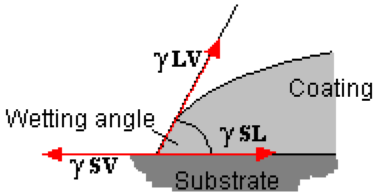


Figure 5 Wetting angle.

Wettability and adhesion of the surface to the coating can be improved either by altering the substrate or the coating. Solid surfaces can be physically or chemically treated to convert monoatomic or molecular surface layers to polar structures [Lic03]. Uv or ozone surface treatment may also be used, and anodizing or applying a chemical conversion coating may improve the adhesion of metal surfaces [Lic03]. Surfactants, polar solvents, and diluents may be added to coatings to reduce their surface tension and thus improve their wetting properties [Lic03]. Primers may also be used as adhesion promoters, silanes being the most commonly used ones. This is because they form a molecular bridge between the substrate surface and the coating, thus improving the adhesion [Lic03]. This is formed by the hydrolysis of silane with the –OH groups on the substrate surface [Jan10]. A polysiloxane network is then covalently bonded to the substrate surface and the coating material can easily react with it [Jan10].

3.1.2 Other requirements

In applications where conformal coatings are used, there are several requirements of the coatings. Even though they depend on the application, there are similarities among the requirements. They include low moisture vapour transmission rate (MVTR) and low water absorption to ensure good water barrier properties [Lic03]. In addition, no occurrence of delamination, blistering, flaking, or chemical decomposition should be detected during aging and high purity material to diminish variations in material properties [Lic03]. The coating material should also be non-nutrient and exhibit low out gassing properties [Lic03]. CTE should match the CTEs of the materials to be coated in order to minimize the stresses. High tensile stress may result in cracking of the coating and is thus not preferred [Reu98]. Corrosion protection properties to a variety of metals used in electronics circuits are needed if the intended environment is corrosive [Reu98]. Electrical properties of the coating material should be clarified and if used in such an environment, radiation should not degrade the coating material [Lic03]. As well as meeting all these requirements the coating material and the manufacturing process should still remain cost effective [Eva08] thus in some applications costs are not the delimiting factor.

3.1.3 Environmental protection

The most common conformal coating protects against moisture, and since no polymer is completely impermeable to water vapour, a given quantity of water will always remain in the polymer structure [Sup07]. Varying degrees of moisture protection are achievable with polymer coatings. The level of protection depends on the molecular structure of the polymer, the degree of cure or polymerization, the amount of impurities, and the degree of adhesion to the surface. When the appropriate coating material has been selected, the material's permeability to moisture, ions, or gases must be known, to ensure adequate protection. Low moisture permeability and absorption are two parameters that define this. Moisture permeability denotes the transport of liquids or vapours through a material [Wel99]. It may occur either through micro cracks and/or pinholes or permeation through the polymer structure [Lic03]. Moisture absorption denotes the uptake of liquids or vapours by a material [Wel99]. It is reported as the percent of water absorbed at a given temperature in a given period of time.

Moisture in electronic packages promotes metal ion migration. Metal migration, especially silver migration, occurs between closely spaced conductor lines [Lic03]. The term electrochemical migration used in the phenomenon describes the way electronic shorting occurs. Electrochemical migration occurs when ions flow through an electrolyte from one conductor to another that is of a different electrical potential [Tom00]. This flow of positively charged metallic ions between two conductors can lead to dendrites being formed on the cathode and subsequent short circuiting [Tom00]. It results in reduction of insulation resistance, an increase in current leakage, and eventually catastrophic shorting [Lic03]. With conformal coatings the extent of metal migration can be reduced.

Corrosion protection is another important function of conformal coating. The most common ways of corrosion are direct chemical corrosion and galvanic corrosion [Lic03]. In direct chemical corrosion the metallic material reacts with its environment. Metal corrosion occurs as a result of chemical reactions between the metal, M, and moisture. A simple anodic oxidation, corrosion, reaction is of the form

$$yM + xH_2O \rightarrow M_yO_x + aH_2 \uparrow + bH^+ + be^- \tag{2}$$

where a + b = x [Ose96]. This anodic oxidation reaction is accompanied by an equivalent cathodic reaction, usually reduction of H_2O or O_2 [Ose96]:

$$2H_2O + 2e^- \rightarrow H_2 \uparrow + 2(OH)^- \tag{3}$$

$$O_2 + 2H_2O + 4e^- \rightarrow 4(OH)^-. \tag{4}$$

These three equations (2)-(4) show that the transfer of electrons is required for corrosion to proceed. In preventing corrosion, it is therefore vital that the current flow between the anode and cathode is minimized. Electrolytic corrosion processes in electronic devices proceed at a rate that depends on the electrochemical kinetics of the corroding metal surface or by the rate of charge transfer between the two electrodes [Ose96].

In electronic device packages, different metals are used, and when dissimilar metals are in contact, galvanic corrosion may occur. These two metals may be either contacted directly or through a conductive solution. A built-in potential develops across the two metals and therefore even without externally applied potential, electrolytic corrosion may occur [Ose96]. Anode and cathode are formed so that the more noble metal becomes the cathode and the less noble the anode. A galvanic series of metals and alloys in different environments have been brought together to predict the risk of galvanic corrosion between different materials [Tul95]. The galvanic series are organized such that the further away the two metals are in the series the more likely is corrosion to occur.

Coatings also protect the electronics from handling, both from physical abuse and ionic residues, e.g. fingerprints. Coating enhances the protection of the solder joints, wire bonds, and fragile components. However, it is also possible that the coating stresses the interconnections and decreases the reliability due to high CTE etc. [Lic03]. If the application is subjected to excessive handling, the coating also needs to have a high degree of abrasion resistance.

Resistance to micro-organisms is a vital property of a coating, especially in humid, tropical, or semi-tropical environments, or in applications where electronics are in contact with the soil. Microbial growth can decompose polymers, degrading both their physical and electrical properties. It can induce corrosion of metals and bimetallic combinations, and it can alter the surface properties of plastics increasing roughness and friction. Most synthetic polymers are non-nutrients and thus they are inherently resistant to micro-organisms [Lic03]. This is why organic coatings have been used to protect otherwise nutrient substrates.

3.1.4 Electrical insulation

One of the important functions of the coating material is electrical insulation. This is expressed in terms of insulation resistance which is defined by volume resistivity, surface resistivity and dielectric strength. The electrical insulation is affected by the composition, purity, and structure of the coating and the environment also plays an important role. Humidity and other contaminants change the resistance of the material and hence the insulation properties. The resistivity also depends on the degree of cure, and variation of resistivity with temperature is important to take into account.

Insulation resistance (IR) is the ratio of applied voltage to the total current between two electrodes in contact with a specific material. It is directly proportional to the length and inversely proportional to the area of the specimen. Formula 4 gives the equation

$$IR = \rho l/A \quad (5)$$

where ρ = resistivity, Ωcm, l = length, cm, A = area, cm^2 [Cal03]. Volume resistivity is the resistance of a cube of bulk dielectric material one centimetre per side expressed as Ωcm. Surface resistance is the resistance between two electrodes on the surface of an insulating material expressed in Ω/□. Dielectric strength is the voltage gradient at which dielectric failure of the insulating material occurs under specific conditions.

3.2 Conformal coating materials

Conformal coatings are generally classified according to the molecular structure of their polymer backbone. Five major conformal coating types are prevalent, and these include epoxy, silicone, acrylic, polyurethane, and parylene [Zha00]. In this thesis, epoxy and parylene C conformal coating materials were used, but all the aforementioned major conformal coating types are introduced next.

3.2.1 Epoxy

Epoxy resins are used widely in electronics, mostly because of their many good properties, including low cost, ease of processing, and excellent thermal, electrical, mechanical, and moisture barrier properties [Lic03]. However, these properties vary a lot depending on the formulation. Epoxies in general have many good properties, but there are three properties that make them useful as electronics coating materials [Won95]:

- Excellent adhesion to most substrates without the need for primers and the sustainability of the adhesion under harsh environments
- Excellent resistance to moisture, salt spray, organic solvents, and other chemicals
- Good electrical characteristics and stability of these parameters over a range of humidity and elevated temperature conditions.

Adhesion is associated with the extremely polar, surface-active nature of the epoxy molecules that form both strong chemical bonds and mechanical interlocking with the substrate. Hydrogen bonding is possible due to ether linkages and their available electron pairs in the epoxy resin.

Water absorption of epoxies has been studied and it has been reported that after 24 hours of immersion it was in a range of 0.1 to 0.5 % [Lic03]. In boiling water water absorption after two hours of immersion was from 0.13 to 2.3 % [Lic03]. In Publication II water absorption of FR-4 substrate material was measured. FR-4 is based on epoxy laminate with glass fibre reinforcement. The results were in the range mentioned above.

Electrical properties of the epoxies are good, and remain fairly stable in different environments, though the properties change due to the varying environments. Insulation resistance depends on the moisture and temperature, and the stability of epoxy can be improved by purifying the epoxy or using alternative syntheses. Dielectric constants for epoxies are low, from 3 to 6 at room temperature and they are slightly frequency dependent. Dielectric strength is high, ranging from 300 to 500 volts/mil for 0.125 inch thick samples.

Epoxies consist of a strained three-member epoxy ring, also called oxirane ring or ethoxyline group [McA85]. The basic structure is illustrated in Figure 6. This epoxy ring is highly reactive, and it can react with different substrates which gives the resins their versatility. Different curing agents give insoluble and intractable thermoset polymers. The properties of cured polymer can be affected by including other constituents in the composition, e.g. fillers, solvents, diluents, plasticizers, and accelerators [McA85].

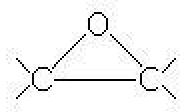


Figure 6 Basic structure of epoxy.

In this work, specifically in Publications III – VI, epoxy was used as a conformal coating material. Epoxy was chosen because of its suitable properties and its extensive use. The epoxy used in this work was biocompatible and non-toxic, and it complies with United States Pharmacopeia (USP) Class VI biocompatibility standards for medical devices and implantation applications. The main properties of the epoxy coating material used are compared with parylene C and presented in Table 3.

Table 3 Main properties of the conformal coatings used in this work.

Property	Parylene C	Epoxy
T_g / °C	87-97	60
CTE (below T_g)/ppm/°C	40-50	60-90
Moisture absorption /wt%	0.01-0.06	0.08-0.15
Water permeability/g/m^2 over 24h	3-4	28-37

3.2.2 Silicone

Silicones are compounds which may be very different in nature; they range from soft gels to hard coatings. Silicones are one of the purest polymers, enabling their use in high reliability applications. The key properties of silicone coatings are listed below [Lic03]:

- Excellent moisture resistance
- Excellent electrical properties in a wide range of both temperature and frequencies
- Low and high temperature stability.

The electrical properties of silicones are superior; for example, the dielectric constant at room temperature and 100 Hz is from 2.8 to 3.8. Environment has only little effect on the electrical properties, and some of the values may even decrease with increasing temperature [Lic03]. The temperature stability is due to the silicon-oxygen backbone structure. Silicones can be safely used up to 180°C for long periods of time [Lic03]. The major drawbacks of silicones have been relatively high cost and low tensile and tear strengths.

Silicone contains a repeating silicon-oxygen backbone and has organic groups attached to a significant portion of the silicon atoms by silicon-carbon bonds [Har85]. This backbone is referred to as siloxane [Won95]. The general structure is illustrated in Figure 7. These R1 and R2 groups may be the same or different organic groups. These organic groups are methyl, hydrogen, chlorine alkoxy, acyloxy, or alkylamino, etc. [Har85]. Silicones also include fillers, additives and solvents.

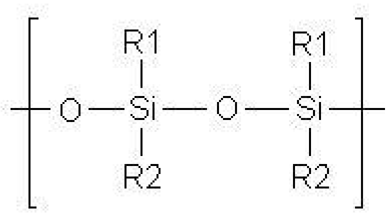


Figure 7 General structure of a linear silicone polymer.

3.2.3 Acrylics

Acrylics are widely used coating materials to protect printed wiring boards. They have been used due to the many benefits they offer including low cost, excellent electrical insulation properties, moisture protection, and ease of repair [Lic03]. Acrylics have low resistance to strong acids and solvents. This enables reworkability, but in some demanding environments acrylics cannot be used. Acrylic resins are derived from acrylic acid-esters and their derivatives [Har04].

There are many different acrylic solutions available, and even in homemade electronics acrylic varnishes are used to protect electronics. The selection of suitable conformal coating material includes the determination of operating temperature range and other use environments properties. The solvent or chemical resistance needed must be ascertained, and the needed rework possibilities, desirable cure time, application area, and if any standards need to be fulfilled.

3.2.4 Polyurethane

Polyurethanes were the first polymers used to protect printed circuit boards. They offer good moisture, fungus, abrasion, solvent and chemical resistance as coatings [Pec99]. They have good adhesion, low shrinkage, flexibility, and elasticity [Pec99]. Rework is hard due to the high chemical resistance, however, polyurethanes can be softened and penetrated with a hot solder iron, which enables the reworking of a coated device [Lic03] [Pec99]. Many polyurethane solutions are hazardous to health since they contain isocyanate groups [Pec99]. These groups can be modified by co-reacting them with a polyol to reduce the number of free isocyanate groups and lower the toxicity [Lic03]. Polyurethanes include materials that incorporate the carbamate functional group as well as other functional groups, such as ester, ether, amide and urea [Bac85]. The basic structure of polyurethane is presented in Figure 8, where R denotes the different functional groups.

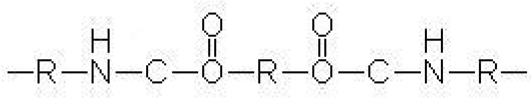


Figure 8 Basic polyurethane structure.

3.2.5 Parylene

Union Carbide developed a simplified process for preparing p-polyxylylenes and gave a trade name to their high-purity p-polyxylylenes, namely parylene. Parylenes have long been used in high reliability electronics applications, and there are still many new areas where parylenes can be used due to their many good properties. Parylene forms extremely thin, pinhole-free, uniform, and high purity conformal coatings due to its deposition technique. It has many good properties for advanced electronics packaging which include [Lic03][Ton93]:

- Room temperature deposition
- Uniform coating
- Excellent adhesion to most surfaces
- Excellent moisture resistance
- Pure material, requires no hardener or catalyst. No by-products are released during polymerization
- Good electrical properties.

Due to the vapour phase polymerization deposition technique, parylenes have many good properties. The deposition takes place at room temperature and since parylene deposits from a gaseous diradical state to a solid state, it remains pure with no need for hardeners or catalysts [Ton93]. Furthermore, it forms a uniform coating throughout the sample due to the deposition technique. Figure 9 shows an example of the uniformity of parylene coating. Moisture resistance is reported to be excellent with parylenes and any moisture that permeates will not degrade the polymer or contribute to chemical corrosion due to the purity of parylene [Ton93]. Because of the adhesion moisture will enter and exit without becoming trapped beneath the coating [Lic03]. In Publication II the water absorption of the parylene C coated FR-4 substrate was measured, and it was seen that moisture absorption was as presented in Table 3.

Parylenes are highly resistant to all chemicals and organic solvents. Rework and repair are therefore difficult, but on the other hand parylenes are excellent protectors against solvents and chemicals. The electrical properties are good, among the best of the polymers [Lic03]. The dielectric breakdown voltage and insulation resistance are both good, and in both cases material purity plays an important role in these properties. Parylenes are thermally stable in vacuum and in inert atmospheres up to 200 °C, but in air up to 115 °C. Parylenes start to oxidize above that temperature, and the oxidation starts from the weakest links between the aromatic groups [Lic03].

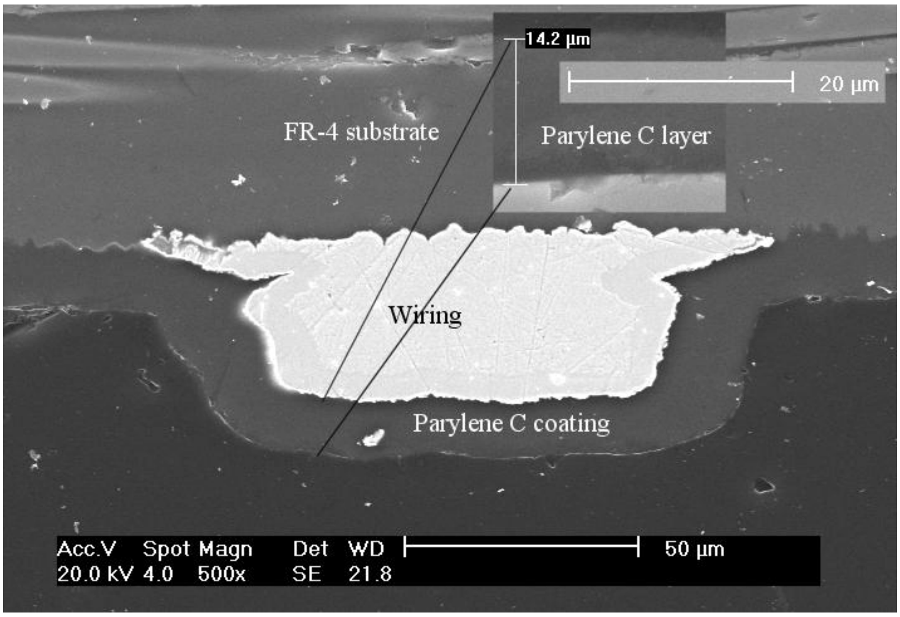


Figure 9 Micrograph of parylene coating on a wire on a substrate. The parylene forms uniform coating throughout the sample. (Publication VI)

Parylene consists of a carbon hydrogen backbone, as can be seen in Figure 10. Originally there were three different parylene types which differed only in the number of chlorine atoms in phenyl groups. Parylene N does not contain any chlorine atoms, parylene C contains one chlorine atom, and parylene D contains two chlorine atoms per phenyl group [Lic03]. Another parylene group, parylene HT, has been introduced and contains fluorine atoms in lieu of hydrogen atoms in the aliphatic portion of the molecule. Parylene forms long-chain, high-molecular-weight linear polymers. In this thesis parylene C was used in the studies in Publications I – VI. The main properties of parylene C are presented in Table 3.

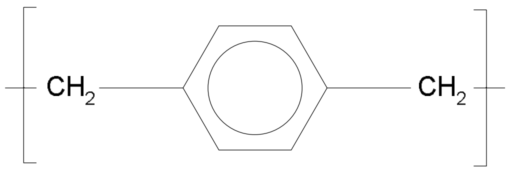


Figure 10 Basic composition of poly-p-xylylene.

3.3 Conformal coating methods

Coatings can be made using many different methods. The whole manufacturing process of the coating includes possible masking of the parts that are not to be coated, cleaning of the surface, and preparation of the surface and coating material [Wor75]. After preparations the coating is applied, then drying and curing of the coating takes place, the mask is removed if masking was used, and inspection and rework are done if needed [Lic03]. Masking is needed if there are parts in the electronic device that are not to be coated, for example connectors. Cleaning and preparing the substrate or device before coating is important to ensure sufficient adhesion of the coating material to the substrate. If the adhesion needs to be improved, several methods may be used including the use of primers. In this study, with parylene C, Silane was used as a primer to enhance the adhesion of the parylene C especially to a silicon chip. After application of the coating material, the curing can be done in many ways. Heat curing is the most used method, but ultraviolet light, moisture curing, microwave curing, or e-beam curing can also be used [Lic03]. Next, the most frequently used coating methods are introduced.

3.3.1 Spray coating

Organic coatings are often applied using spray coating. This can be done in many different ways; a compressed-air vaporization method being the most common way. The other techniques include airless pressure spray, hot flame spray, hot vapour impelled spray, electrostatic spray, dry powder resin spray, and the ubiquitous aerosol can spray [Lic03]. To ensure good quality of the coating, it may need to be sprayed on several different planes to ensure complete coverage [Lic03]. The viscosity of the diluted coating should be low enough to prevent filleting or bridging of the components, which may cause stresses [Lic03]. To prevent or minimize pinholes or pores that penetrate the coating, multiple coats of the same material are applied on the sample [Lic03]. Spray coating is fast, low cost, and adaptable to various shapes and sizes. On the other hand there is material loss from overspray, difficulties in obtaining complete coverage on complex parts and in achieving uniform thickness, and there are solvent emission issues.

Spray coating is also a much used method in applying conformal coatings to homemade electronics. The basic coating materials, such as acrylics and polyurethanes, are available in spraying cans. The conformal coating material can then be applied easily without any special equipment. However, if more reliable coating is needed, better equipment should be used.

3.3.2 Dip coating

Dip coating is a simple coating method, where samples are coated by dipping them in a tank full of coating material. The film thickness is controlled by viscosity, flow, and the rate of withdrawal of the sample [Har02]. Dip coating is a low-cost method, and it has good coverage even of complex parts. On the other hand the viscosity and pot life of the coating bath must be controlled, and the bath may be contaminated [Lic03]. The rate of withdrawal is critical in the process and masking needs to be done if there are parts that should be left uncoated [Lic03]. The coating also forms fatty edges on the lower parts of samples, when the coating film thickness differs from top to bottom. This is called a *wedge effect* [Har02]. In this work the epoxy coatings for samples in Publications III – V were applied using dip coating.

3.3.3 Vapour deposition

Vapour deposition can be employed only to very few polymer types. P-polyxylylene (parylene) is the only polymer that practically deposits with vapour deposition [Lic03]. The coating process is presented in Figure 11. In this technique, parylene deposits from a gaseous diradical state to a solid state on the surface of the sample. The raw material, di-para-xylylene is a white powder that is applied in the coating system to the sublimation furnace (Figure 11). The vaporization of the di-para-xylylene occurs at approximately at 150 °C. The next step is pyrolysis, where the gaseous dimer dissociates into gaseous diradicals (Figure 11). These diradicals then polymerize on any surface they contact forming long-chain linear polymers (Figure 11).

With vapour deposition ultra-thin pinhole-free films can be applied, excellent coverage over complex high-density modules is achieved, and high purity coating is achieved. On the other hand the technique is specific to parylene coatings, masking and rework are difficult, and special deposition equipment is required.

3.4 Special requirements for medical applications

Electronic devices that are used as medical applications and especially implants have many requirements and pose many challenges. Biocompatibility and an implant package are among the challenges that need to be solved when implantable devices are developed [Zho05]. Biomaterials are used in the interface between the living tissue and the device, making the implant biocompatible. Not all the materials used inside the implant are biocompatible, and thus the coating material has great reliability requirements [Mia05][Zho05]. The coating may not fail during the lifetime of the implant, since this could cause a leakage of toxic materials to the body or the leakage may cause the failure of electronic devices inside the implant.

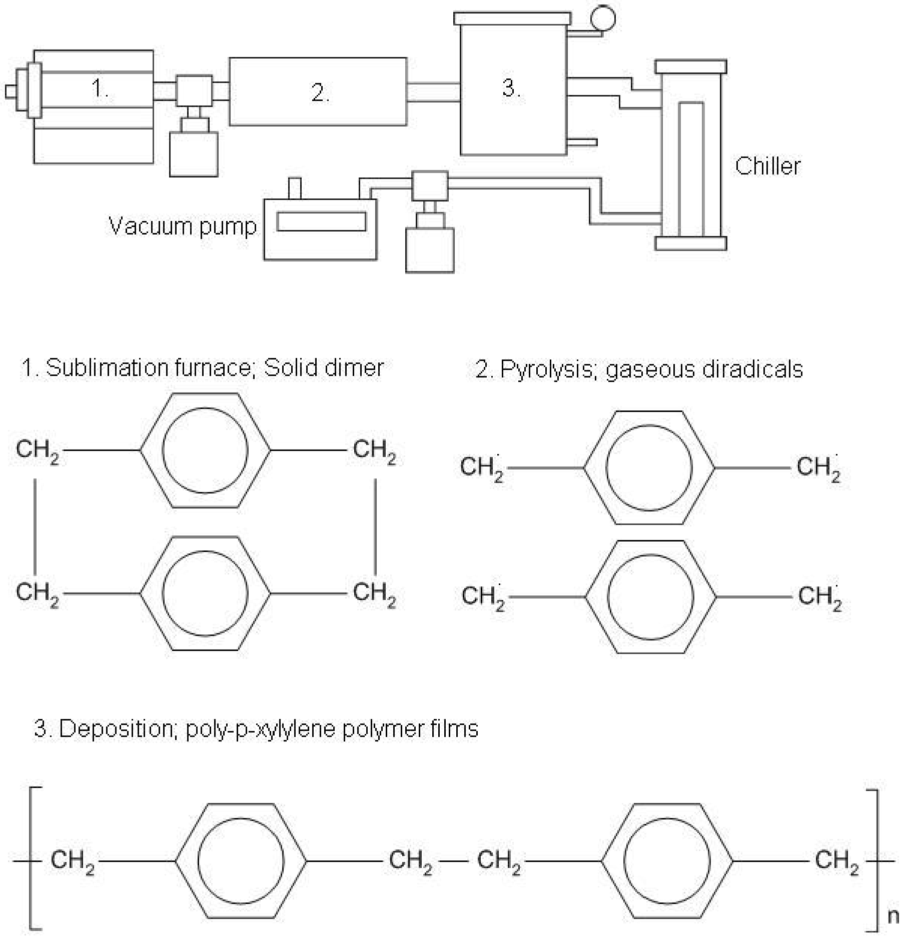


Figure 11 Parylene deposition system. Modified from references [Lic03][Son09].

Medical applications have very stringent requirements for coatings. In the United States, under the Federal Food, Drug, and Cosmetic Act (FDCA), the United States Pharmacopeia (USP) and National Formulary (NF) are recognized as official compendia which regulate medical devices in general. The Food and Drug Administration (FDA) is a part of the U.S. Department of Health and Human Services, and regulates food, drugs, medical devices, biologics, animal feed and drugs, cosmetics, radiation-emitting products and combination products [Sal06]. Even though these organizations operate in the United States, the regulations are obeyed worldwide. Medical devices are sorted into Classes I – III, which determines the requirements for a general device type [FDA10][Sal06]. These classes group medical devices depending on the level of control necessary to ensure the safety and effectiveness of the device [FDA10]. Legally recognized product standards have been established by the United States Pharmacopeia (USP), which is a voluntary, non-for-profit organization promoting public safety [Sal06].

The materials selected for a medical device should be on the basis of chemical, toxicological, physical, electrical, morphological, and mechanical properties most suitable for the task [Sal06]. Biological evaluation is only required if the intended use of the device requires direct or indirect contact with the body.

However, if the material used already has USP classification or FDA approval, the evaluation process is not needed in its full extent. The requirements for the materials include high purity and inertness of the coating material, biocompatibility and biostability, integrity and uniformly thin and low weight [Lic03]. Additionally, the coating must ensure the full functionality of the device which meets the requirements given for the conformal coating protection in general.

Ensuring that all the requirements for medical applications are met, typically metal or ceramic casings are used [Mey01][Mia05]. Flexible parts of the devices are coated with some polymer materials tested for their biocompatibility [Mey01][Mia05]. However, polymers have been used for cases when the required life-expectancy has been relatively short [Kaz04]. When the requirements of the shielding materials are considered, the expected life time of the product should be known. [May01] lists some important rules for the construction of implantable stimulators which have a lifetime of more than 10 years [May01]. The rules include the importance of biocompatibility, the mechanical design of the implant, which has to be small in size and lighter in weight and must not present corners or edges [May01]. Furthermore, all electrical conductors carrying direct current (DC) have to be sealed hermetically to avoid electrochemical corrosion and also when different metals are used electrochemically equivalent materials are preferred due to corrosion [May01]. Additionally the moisture uptake of the polymers needs to be taken into account e.g. if conductive adhesives are used.

Medical devices in contact with body fluids need to be sterilized prior to use. Sterilization of healthcare products and materials is a specialized process, implying complete inactivation of all viable forms of life or reproduction [Rog05]. Even though the definition of sterilization implies that all viable micro-organisms will be destroyed during sterilization, there is no single method that is compatible for all healthcare products [Rog05]. Sterilization methods range from heat to lethal chemicals and physical processes [Lar96]. Heat can be used to sterilize devices, and either moisture or dry heat may be used. Dry heat requires high temperatures and long periods of time, and is thus not useful in many electronics applications [Lar96]. Autoclave sterilization uses pressurized steam with a pressure of 103.42 kPa (15 psi), a temperature of 121 °C and exposure time of 20 minutes [Lar96]. The most commonly used gas sterilization method is ethylene oxide. This reacts with many chemicals, e.g. alcohols, amines, organic acids, and amines [Lar96]. Radiation sterilization can be achieved either using electromagnetic radiation or particle radiation. Electromagnetic radiation may be either ionizing or nonionizing. It is done using microwaves, ultraviolet, gamma-rays, or x-rays [Lar96].

Even though the adhesive connected chips are not in direct contact with the body fluids, they are still part of the medical device to be implanted. Therefore the adhesive interconnections need to be tested so that they withstand the sterilization conditions and do not degrade due to sterilization [Lic05]. Publication IV discusses the effects of gamma sterilization to the ACF joints with different conformal coatings protecting the device.

A medical sterilization method needs to be chosen according to the application. In medical devices where electronics are present sterilization methods utilizing high temperatures cannot be used. Ethylene oxide is one method used, but the drawback of this is the toxic residuals left on the surface of the device. Radiation sterilization is an effective method, but ionizing radiation can harm electronic components. It is important to verify which radiation to use in electronic applications.

4 RELIABILITY OF ACA JOINTS WITH CONFORMAL COATINGS

All truths are easy to understand once they are discovered;
the point is to discover them.
–Galileo Galilei

Reliability has been defined as “the probability that a system will perform its intended task under operating conditions for a specified period of time” [Mee98a]. When electronic component reliability is studied, both reliability physics and reliability statistics need to be considered [Jen95]. Reliability physics deals with failure mechanisms, the occurrence of failures and factors influencing them [Jen95]. Reliability statistics tries to establish lifetime patterns and life time models according to reliability results [Jen95]. Both of these are valuable but they need to be tied together to accomplish reliable lifetime distributions and predictions of the lifetimes. This study concentrates on the reliability physics of ACA joints protected by a conformal coating, but the statistical methods have also been taken into account when analyzing the results.

Reliability models have been developed to predict the reliability of electronic devices as a function of the application environment and operating conditions [Pec97]. Before using a reliability model, the evaluation criteria of the reliability model need to be established [Pec97]. When the evaluation criteria have been established, the environmental stresses, design parameters, manufacturing processes, failure modes and mechanisms, and statistical distributions need to be assessed [Pec97]. The statistical methods most commonly used in reliability of electronics are normal, log-normal, exponential, and Weibull distributions [Jen95]. The most used distribution in electronics reliability analysis is Weibull, and in this work too, Weibull distribution is used to analyze the results. Weibull distribution describes either increasing or decreasing failure rates simply and is thus used for product life analyses [Nel04]. In Weibull distribution the cumulative distribution function is as follows

$$F(t) = 1 - \exp\left[-\left(\frac{t}{\alpha}\right)^{\beta}\right] \tag{6}$$

Where β is the shape parameter, and α the scale parameter [Ohr98]. α can also be called the characteristic life, which is the 63.2th percentile of the variable. α has the same unit as t, for example hours, months, cycles, etc. [Nel04]. β is unitless and may also be called the “slope” parameter describing the slope of the lines in Weibull plots [Nel04].

The failure criterion used in different studies affects the reliability results obtained. It is important to decide which is considered a failure in each application [Liu02]. The different failure criteria are usually analyzed separately, but reliability models under different failure criteria are also introduced [Liu05]. However, usually only one failure criterion is used in published reliability studies, e.g. in references [Fri05][Hwa08a][Mis02]. In this work the effects of different failure criteria are further discussed.

4.1 Accelerated life testing

Life testing is carried out to obtain information about the lifetime properties of the components or devices. Such testing is usually time-consuming and accelerated life testing (ALT) is then considered. Accelerated life testing consists of a variety of test methods for shortening the life of products or hastening the degradation of their performance [Nel04]. In ALT there are three different ways of accelerating the reliability test; increasing the use-rate of the product, increasing the aging-rate of the product or increasing the level of stress [Mee98a]. When ALTs are conducted, careful design and interpretation of the results is needed to avoid the pitfalls of accelerated testing [Mee98b][Suh02].

Sometimes ALTs may hasten failure modes or mechanisms that are different from those that could occur in the field [Suh02]. New failure modes may result from a fundamental change in the mechanism causing the material or component to fail [Mee98b]. These failures should be well understood so as to avoid erroneous conclusions. Another risk in ALT is the uncertainty of the statistical estimates made based on the reliability results [Mee98b]. If the decisions are based on point estimates alone, it can be misleading [Mee98b]. Multiple factors affect the degradation of electronic devices in use conditions. When ALT is conducted, it is hard to accelerate the time-scales of all of these factors [Mee98b]. Therefore the relationships of these variables and degradation and components life are important to determine when ALT should be used [Mee98b][Suh02]. Failure mechanisms occurring in ALTs might easily mask one another, especially if ALT is conducted only at certain temperatures [Mee98b]. The results need to be analyzed carefully to reveal such masking and another test should the conducted if needed. Yet another pitfall is a situation where ALT results in bimodal distribution of failures [Suh02]. Infant mortality failures may occur concurrently with the anticipated failures [Suh02]. Again, masking may occur and the interpretation of the results needs to be done with care. ALTs are often used to compare different products or materials, etc. to decide which is most reliable in field conditions. The problem is that it is difficult to justify the assumptions simply on the basis of the ALT results [Mee98b]. More importantly the failure mechanisms of the different products need to be explained to make the assumptions [Mee98b]. When ALTs are designed, the deceleration of the accelerating variables also needs to be considered [Mee98b]. For example, high temperature alone may decelerate the failures that occur due to moisture.

In many cases accelerated life tests endeavor to simulate real conditions in actual use [Nel04]. The only difference should be the high level of overstress variable; however, in many cases they differ much from real life operating conditions [Mee98b][Nel04]. Nevertheless, the tests may still be useful, since it can be assumed that products of materials that perform well in ALT also perform well in the field [Nel04]. Thus it is useful to distinguish test reliability from field reliability [Nel04]. By using ALT acceleration factors can be established from the reliability data. With acceleration factor, the field reliability of a tested product can be estimated. Acceleration factors can be company tradition, or they can be estimated from reliability data using both accelerated life and typical use life of the product [Nel04].

Before ALT, a test plan needs to be in place. It is really important to carefully plan the tests, since if the test has a good plan the conclusions are usually easy to draw without complicated data analyses [Nel04]. First of all, the tests and stress levels need to be decided, likewise the test length. The number of specimens, or sample size, also needs to be planned beforehand. Often the sample size is limited by the availability of specimens. In this work the test plans are introduced in each publication.

4.1.1 Constant humidity test

Constant humidity testing is performed to evaluate the reliability of electronic devices in humid environments. The test used in this work was the 85/85 test, where the temperature and relative humidity were 85 °C and 85 % respectively [Jed97]. According to the Jedec standard (22-A101-B), the temperature should be between tolerances ± 2 °C and humidity between tolerances ± 5 % [Jed97]. With this testing method the effect of humidity can be accelerated using elevated temperature. In this work constant humidity testing was conducted in Publications III – V according to the abovementioned Jedec standard.

4.1.2 Temperature cycling test

Temperature cycling testing is used to evaluate the mechanical stresses induced by temperature changes. Electronic devices are subjected to different temperatures during their life span and the temperature changes may cause failures. Jedec standard JESD22-A104C is used to test components and solder interconnections [Jed05]. Even though this standard was specially developed for solder joints, the same kind of testing has been used with electrically conductive adhesive joints as seen, for example, in references [Asc97][Fri06b][Hwa08b][Kim07][Liu99]. The standard specifies many different test conditions which can be used, the most commonly used ones being -40 °C to 125 °C [Fri06b][Kim07][Liu99], -55 °C to 150 °C [Hwa08b] and -40 °C to 85 °C [Asc97]. In this work in thermal cycling reliability of conformally coated ACF joined flip chips were studied Publication I and thermal cycle from -40 °C to 85 °C was used.

4.1.3 Salt spray test

Salt spray testing is used to determine the resistance of solid state devices to corrosion [Jed04]. According to Jedec standard JESD22-A107B, the testing conditions inside the chamber should be 35 °C with tolerances of +3 °C / -0 °C and the sodium chloride (NaCl) concentration in the solution used is 50 g/l ± 5 g/l which equals 5 % concentration of NaCl in deionized water [Jed04]. Publication

II reported the corrosion resistance of ACF joints with and without conformal coating.

4.1.4 Test evaluation methods

Firstly it is important to decide which variable to monitor in order to obtain reliable data from the samples. When fully working devices are tested, the device can be run during testing to detect any malfunctioning. However, the use of test vehicles instead of fully working devices greatly simplifies the test setup and wiring [Ste02]. When test vehicles are used for testing purposes, they must, however, correspond to real parts in every way other than the functionality [Ste02]. In test structures, when interconnections are studied, the interconnection resistance is monitored to detect failures. A daisy chain test structure is a much used method to track interconnection resistances [Mis02][Saa10][Woj00]. In the daisy chain structure, a conductive path is formed through each interconnection with wires on and between chip and substrate. Figure 12 presents the idea behind daisy chain test structure. The drawback of such a measurement system is that the resistances of the measurement wires are also included in the measurement.

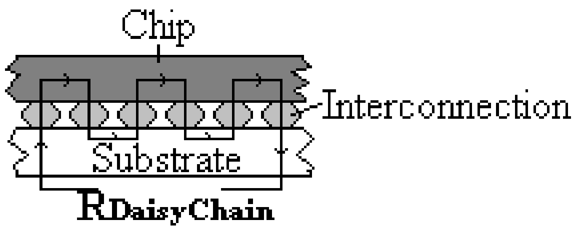


Figure 12 Daisy chain test structure.

Real time measurements during accelerated life testing give exact information on sample behaviour during testing. Another way of monitoring test samples is to measure the samples at fixed time intervals by taking the samples from the testing chamber for measurements. However, with this system the behaviour of the sample during changes in testing environment is not seen. As discussed later, the failure modes in different tests and with different test structures varies and these are hard to detect without real time monitoring of the samples.

The measurement system used in this work is shown in Figure 13. The source voltage (V_s) was 5 V and the precision resistor (R_{ref}) was 1 kΩ. Each test sample had its own measurement channel and the voltage across the samples daisy chain (V_{mes}) was measured. The daisy chain resistance of the sample could then be calculated using the following formula:

$$R_{DaisyChain} = \frac{V_{mes} x R_{ref}}{V_s - V_{mes}} \quad (7)$$

This measurement system was used in all accelerated tests made in this work. The voltage was measured in humidity tests every 60 seconds (Publications III –

V), in temperature cycling test every 20 seconds (Publication I) and in salt spray test every 10 seconds (Publication II).

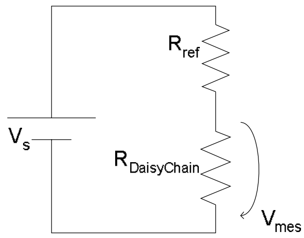


Figure 13 Measurement system.

4.2 *In vitro* testing

In medical applications *in vitro* testing is an important part of the development process. Before devices are ready to use, they need to be extensively tested for their reliability and biocompatibility. Reliability testing intended for electronics may also be performed for medical devices, but they also need to be tested under conditions simulating the real body environment [Chl05][Wil98]. *In vitro* testing is used to test the materials and devices for their biocompatibility in a simulated atmosphere. *In vitro* is defined as 'studies performed outside of a living organism' [Sal06]. Different solutions can be used to simulate the body conditions. The simplest solution is to use salt water or saline (0.9 % NaCl) [Bes06][Lo07], but a more accurate atmosphere is achieved using special buffer solutions. Examples of these solutions are Ringer Fluid [Chl05], phosphate buffered saline (PBS) [Rub10], artificial blood plasma [Bes06], and artificial cerebrospinal fluid (ASCF) [Bes06].

In this work *in vitro* testing was carried out in Publication VI, where samples were tested in PBS solution at 37 °C for ten months. This test was not accelerated and thus it took a long time to test the samples. Reference [Rub10] presents a study where *in vitro* testing was accelerated by using elevated temperatures. They used in PBS at temperatures of 37 °C, 60 °C and 85 °C [Rub10]. They stated that for polymers, if the aging is accelerated due to Arrhenius behaviour, 60 °C is the upper temperature limit. The Arrhenius life-temperature relationship describes the degradation of a product due to chemical reactions or metal diffusion over a temperature range [Nel04]. The rate at which the degradation occurs in Arrhenius behaviour can be calculated according to

$$Rate = Ae^{-\frac{\Delta G^*}{RT}} \qquad (8)$$

where A is a constant, ΔG^* is the activation free energy, T is the absolute temperature and R is the gas constant (= 8.3145 J / K mol) [Ohr98]. Due to this equation, the Arrhenius model can be extended to cover a range of temperatures. The use of 85 °C has been justified by the fact that this temperature has commonly been used in the moisture testing of electronic devices [Rub10].

However, at 85 °C the samples were sensitive to salt ions when stored in PBS [Rub10]. Another study using elevated temperatures with PBS soaking is reported in reference [Sta03]. In that study 85 °C and 95 °C temperatures were used and an attempt was also made to extrapolate the lifetime using the Arrhenius relationship [Sta03]. However, the testing did not yield the anticipated results and the acceleration was not considered a success.

After *in vitro* testing, *in vivo* testing is conducted. *In vivo* is defined as 'studies performed inside of a living organism' [Sal06]. This usually refers to animal testing. Before *in vivo* testing is authorized, extensive *in vitro* testing needs to be completed. During *in vivo* testing tissue inflammation and foreign body response occurs after short periods of implantation [Vad10]. The tissue is damaged in an implantation procedure and because of the presence of the device. The body responds naturally to wounds or implanted devices by fibrotically confining the device and thus preventing it from interacting with the surrounding tissue [Vad10]. This causes a cellular encapsulation of the implant device formed by proteins and cells in a thickness range of 10-100 μm, causing degradation in sensor performance, but gives long-term stability of the sensing elements [Vad10].

In this work the test samples were measured after fixed time intervals during *in vitro* testing. Publication VI describes this in more detail. Monitoring of the samples during testing has the same principles as with ALT. The use of real time measurement is not so critical in tests where the testing is not accelerated. The phenomena in the structures can be seen even with fixed interval monitoring.

4.3 Failure modes and criteria

After environmental testing the reliability data needs to be analyzed. First of all, the purpose of the data analysis needs to be stated [Nel04]. In electronics reliability analysis the failure percentages and characteristic life are often useful parameters to distinguish between two materials or products. When ALT is used to estimate the actual field life, acceleration factors need to be calculated. Reliability data analysis is usually fitted to a reliability model to gain the desired information from the analysis. In this work, the reliability results are presented using Weibull distributions in Publications I – II, and failure percentages in Publications III – VI. In the following paragraphs the reliability results are discussed in more detail. The reliability data analysis in the publications has been continued to compare the various conformal coating materials and the effects of conformal coating materials in general. Next, the reliability data analysis is continued by evaluating different failure criteria and discussing the various failure modes relating to the failure criteria.

Failure criteria need to be selected according to the field of application. For example many digital inputs can withstand an increase of several hundred ohm in joint resistance without any changes in electrical performance [Ste02]. On the other hand, drivers in power devices may suffer dramatically even from resistance increases of several tenths of ohms [Ste02]. Therefore failure criteria are set on different base in these applications. The failure modes and criteria both affect the reliability results and with some failure modes the effect of the criteria is not so substantial. However, when devices are tested to find the root cause of the failure or the failure mechanism, it is often beneficial to have higher failure criteria. Then, the failure mechanism is more visible in the sample and the analysis is easier. One option is to tighten the failure criterion to obtain accurate reliability data, and then continue testing to facilitate the failure mechanism analysis. If this is done, attention should be paid to the possible masking of the failure mechanisms. It is important that the reliability results and the failure mechanisms match.

In this study, the failure criterion used in Publication I was the duplication of the daisy chain resistance and in Publications II – VI the increase of the daisy chain resistance to a ten times higher value. This criterion is quite strict, and thus the effect of different failure criteria is discussed here at greater depth. However, when the failure criterion is defined, the effect of the measurement system and its variations needs to be ascertained. Furthermore, in these publications the aim has been to analyze the failure mechanisms, when it is advantageous that the samples have failed properly.

4.3.1 Constant humidity test

In a constant humidity test, the tightening of the failure criteria affected the results substantially, especially with certain failure modes. The reliability results obtained in the studies reported in Publication III are here further discussed with different failure criteria. The test lots discussed here are a non-coated test lot, an epoxy coated test lot, and a parylene C coated test lot. The failure criteria selected for further study here are the following: a 10% increase in daisy chain resistance, a 20% increase in daisy chain resistance and a 10% increase in daisy chain resistance with peaks ignored. The last failure criterion where the peaks have been excluded means that the sample is considered to have failed when the resistance value is permanently increased by 10% from the original value. This was chosen as a criterion since in some test lots the daisy chain resistance first shows short peaks at temperature changes or at the lower temperature limit, as discussed in Publication III.

Figure 14 presents the results of the non-coated test lot with three different failure criteria. As can be seen, when the peaks are ignored, the result is much better than if the failure is detected in the first peak where the resistance value exceeds the 10 % limit. This reveals that in this test the non-coated test lot had a failure mode that showed first peaks in the resistance value prior to permanent changes in the resistance value. Figure 15 shows one example of such a test sample where short peaks in the resistance occur. In this particular sample only the 10 % increase is seen. In this sample the resistance value of the sample varied somewhat all the time, which is partly caused by the real time measurement system. It is important to distinguish between real changes in measured variables and errors caused by the measurement system. The selection of a failure criterion sets limits to the errors that the measurement system has caused. In some applications peaks can be ignored, while in some other cases one short peak can already cause fatal errors in the system [Ste02].

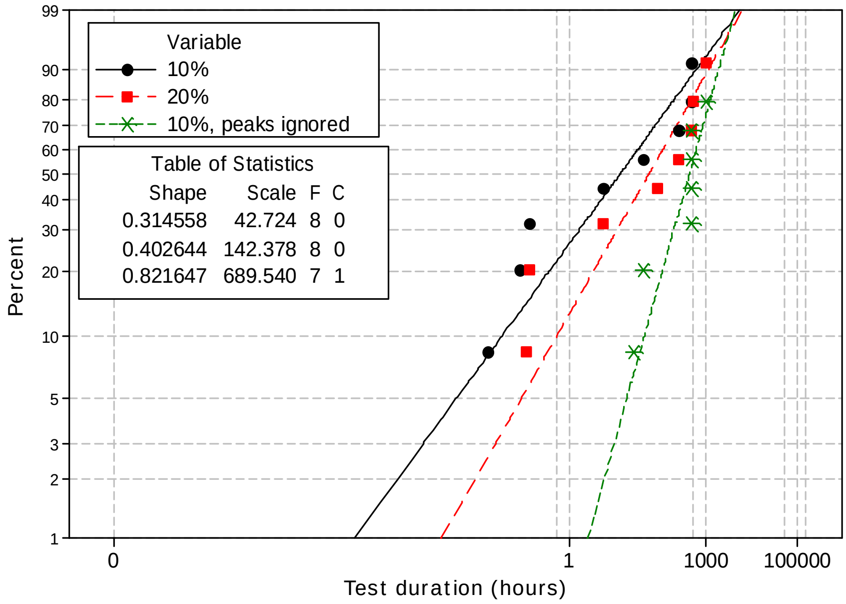


Figure 14 Weibull distribution of the 85/85 test non-coated test samples showing results with three different failure criteria.

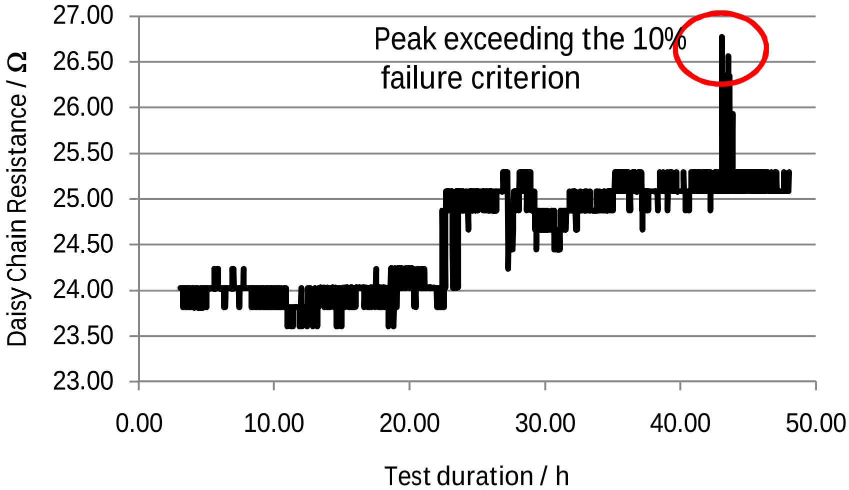


Figure 15 Behaviour of one test sample from a non-coated test lot showing peak in resistance value.

The reliability results of the epoxy-coated test lot did not change notably when the failure criterion was changed. This can be seen in Figure 16, where the Weibull distribution is presented. The failure mode of this test lot was different than in the non-coated test lot, and here an abrupt change in the daisy chain resistance occurred, as can be seen in Figure 17. When the failure mode is an abrupt change, the different failure criteria do not show marked differences in reliability results.

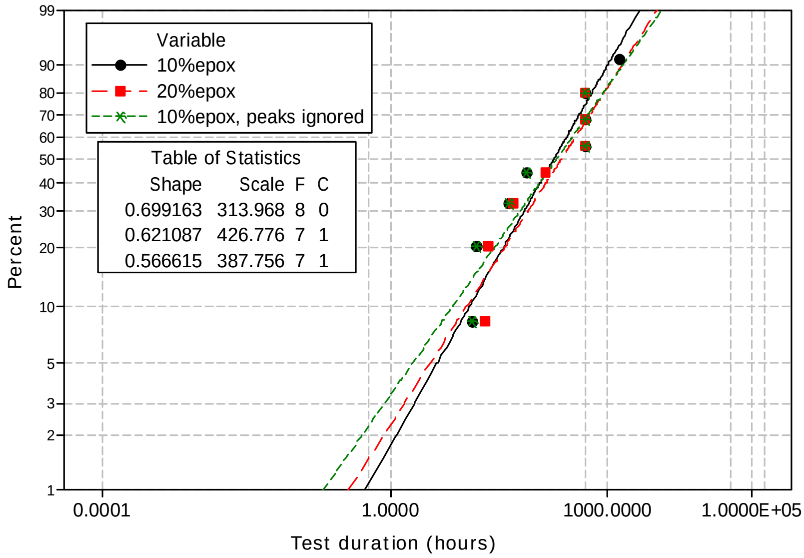


Figure 16 Weibull distribution of the 85/85 test epoxy coated test samples showing results with three different failure criteria.

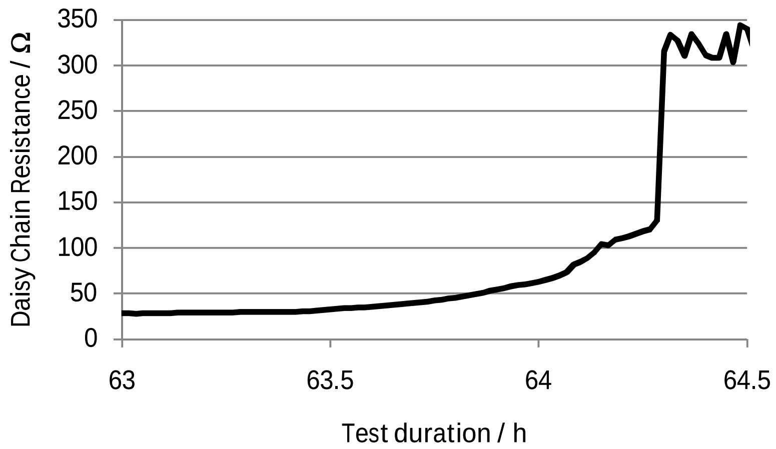


Figure 17 Behaviour of the daisy chain resistance in epoxy coated test lot showing abrupt change in the value.

The parylene C coated test lot ignoring the peaks did not change the time of the failure, as can be seen in Figure 18. The results with a 10 % increase in daisy chain resistance and a 10 % increase in daisy chain resistance with peaks ignored yielded similar results. The dominant change was changing the failure criterion from a 10 % to a 20 % increase in daisy chain resistance. This leads to the conclusion that the failure mode was a regularly increasing resistance value. This is also seen in Figure 19, where the daisy chain resistance of one test sample is presented while failing. In this case the failure mode is simple, since no peaks occur. In failure modes like this the effect of different failure criteria may be marked. It is important to decide according to the application and meaning of the reliability tests which failure criteria to use.

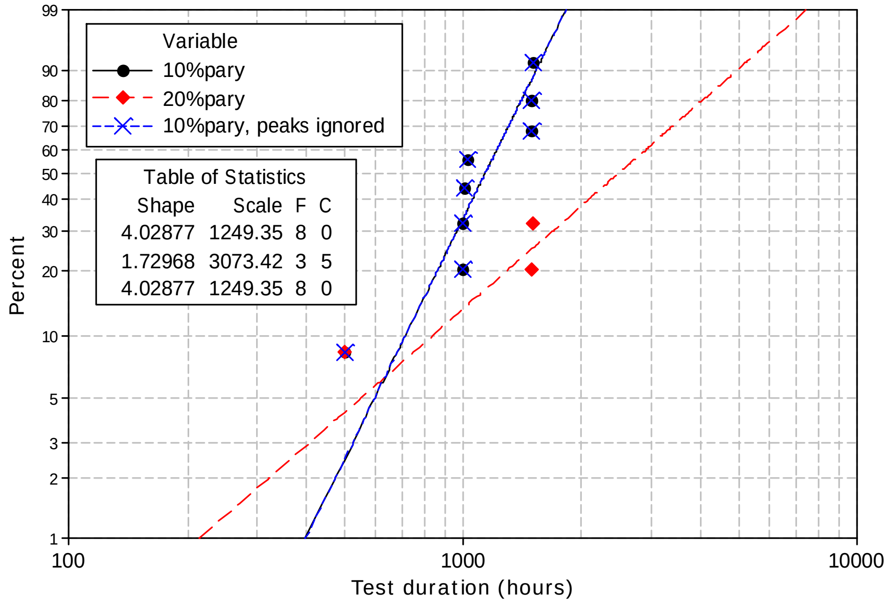


Figure 18 Weibull distribution of the 85/85 test parylene C coated test samples showing results with three different failure criteria.

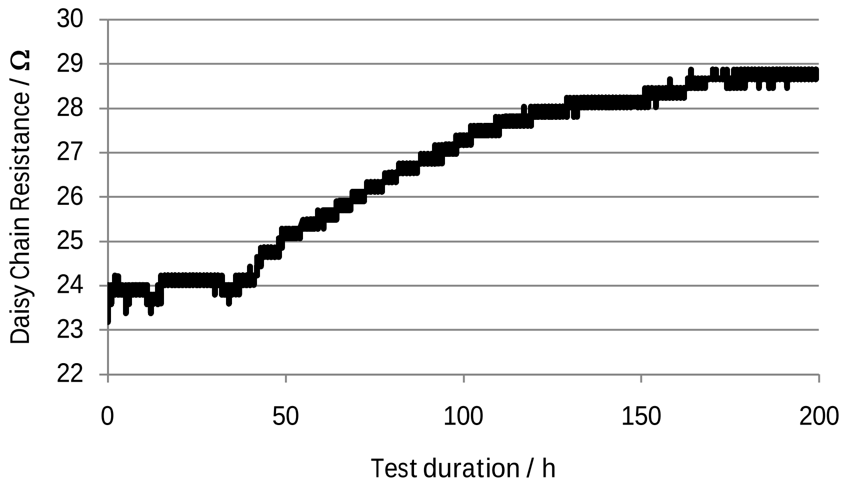


Figure 19 Behaviour of the daisy chain resistance in parylene C coated test lot showing regular change in the value.

4.3.2 Temperature cycling test

Temperature cycling test results were analyzed in Publication I using failure criteria of duplication of the daisy chain resistance. As described in the publication, failure mode in temperature cycling testing is usually seen first at either of the temperature extremes or when the temperature changes. When measurements after a fixed number of cycles are used this behaviour cannot be seen and the reliability results might be rather different. Resistance fluctuation of ACF joined flip chips during temperature cycling testing has been reported, for example, in references [Fri05][Fri06b][Sep02].

With FR-4 substrates both non-coated and parylene C coated test samples failed first during temperature changes or at the lower temperature limit. This is seen in Figure 20, where the fluctuation of the measured voltage at different temperatures is presented. If the measurements had not been conducted continuously, the results of this experiment would have been different. This has been explained in Figure 21 using two different failure criteria. The figure shows the results both for non-coated samples (FR-4 test lot, Figure 21a) and parylene C coated samples (FR-4P test lot, Figure 21b). The first failure criterion was the same as in Publication I; duplication of the daisy chain resistance or the voltage across the daisy chain resistance. Another failure criterion used was the same, but the peaks that occurred only during thermal cycling were excluded. As expected, the results have been improved somewhat. However, these results with peaks excluded, shown in Figure 21, do not yield results similar to those obtained with measurements made after a fixed number of cycles. These results would be even more optimistic, since these results presented in Figure 21 were taken right after the sample showed first failure at room temperature. However, the next measurement point when using a measurement after a fixed number of cycles would have been somewhat later than this point.

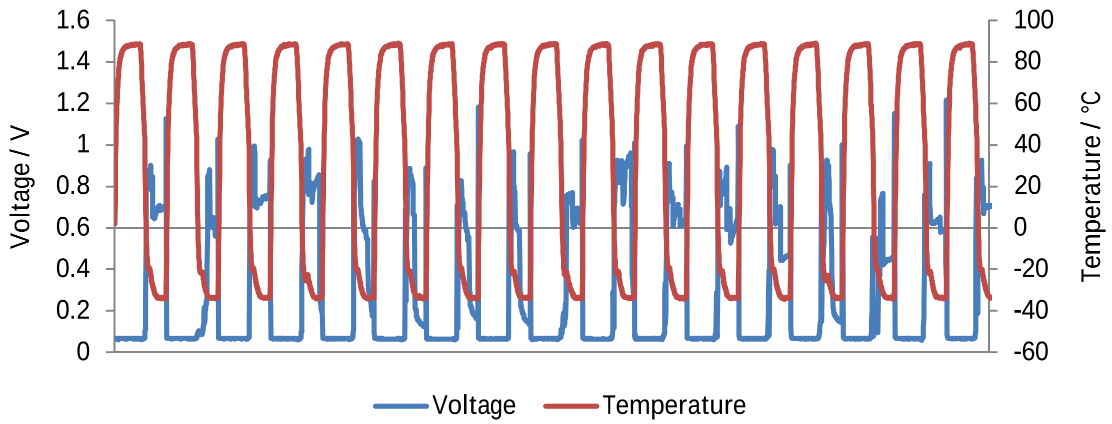
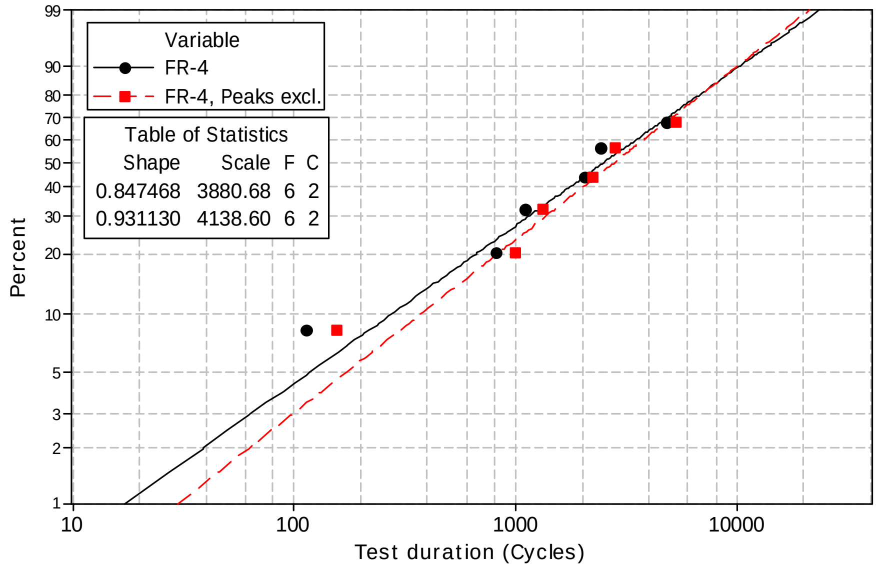


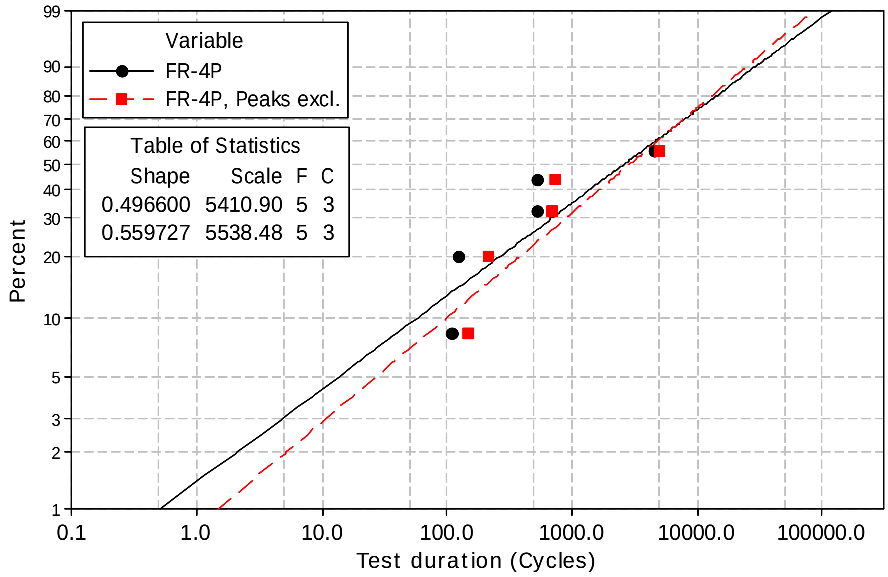
Figure 20 Fluctuation of the measured voltage due to test temperature.

4.3.3 Salt spray test

Salt spray testing of ACF joined flip chips was reported in Publication II for both non-coated and parylene C coated test lots. The failure criterion in this study was a tenfold voltage across the daisy chain resistance. The samples with parylene C conformal coating did not fail during the 3,000 hour testing. The non-coated test samples failed, and the failure modes were analyzed. As stated in the article, the daisy chain resistances were increasing prior to the failures but the increase was rather fast. This is seen in Figure 22, where the daisy chain resistance of one test sample can be seen. In this test sample the resistance value first shows peaks and the resistance value increases and then fails. The failure criterion for this particular sample was 99Ω. The parylene C coated test samples did not fail and thus the failure modes could not be seen. However, the daisy chain resistances were measured during the test and the values before and after testing are presented Publication II.



(a)



(b)

Figure 21 Reliability results for temperature cycling testing (a) with non-coated and (b) parylene C coated test lots with two different failure criteria.

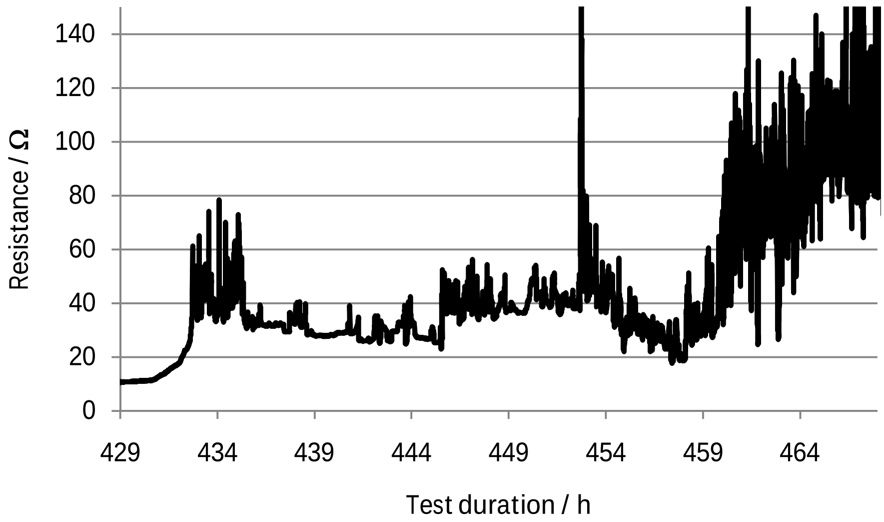


Figure 22 Daisy chain resistance behaviour at failure in salt spray test.

4.4 Failure mechanisms

The conditions and physics relating to the failure process provide an understanding of the mechanisms that cause failure [Mar99]. Typically failure mechanisms in electronic components and devices include corrosion, distortion, fatigue fracture, and wear [Mar99]. These are due to operational and environmental stresses. The environmental stresses were studied here. The stresses applied were temperature and humidity, salt spray, thermal cycling, and hydrolysis solution. The effects of these environments on ACF flip chips were studied and the influences of protective conformal coatings in these environments evaluated. These have been presented at length in the publications and are here discussed further.

Temperature causes failures to electronics in various ways. High temperature accelerates chemical reactions in materials and weakens the insulation properties and might ultimately even cause dielectric breakdown [Mar99]. It also causes outgassing of plastic materials and electromigration in conductors [Mar99]. High temperature causes changes in different material parameters, which alters the behaviour of the whole device at elevated temperatures. This also needs to be considered when ALT is used to obtain reliability data for electronics. At low temperatures, materials become brittle, the water condenses from the materials and ice may form on the surfaces of the device [Mar99]. Chemical reactions are reduced and again material parameters change.

4.4.1 Humid environments

Humidity and moisture are present in almost every operating environment of electronics devices, thus not always causing any reliability challenges. However, the mechanical behaviour of polymer systems is significantly affected by the absorption of atmospheric moisture [Fan09]. Humidity and moisture in electronics packages cause corrosion of materials, decreases insulation properties and changes resistances, etc. [Mar99]. Especially at elevated temperatures, structures with polymer materials, including ACA interconnections, have reliability challenges with moisture [Lin08][Liu07]. Since polymers absorb moisture they degrade and cause changes in the behaviour of polymers [Liu07]. Absorbed moisture may lead to plasticization of the material and affect the mechanical performance of the material. This leads to degradation of T_g and changes in CTE and Young's modulus [Fan09][Shi08]. These changes give rise to swelling stress damage due to internal vapour pressure generating voids or promoting the catastrophic growth of existing voids [Fan09][Liu07]. The polymer matrix expansion due to moisture causes the formation of defects like cracks and delaminations due to mismatches of moisture expansion coefficients of the materials [Lin08][Liu07][Teh05]. Moreover, there are initial delaminations at the ACF interface due to defects or process issues [Mer03]. Moisture has an adverse effect on interfacial adhesion, which may accelerate the delamination by weakening the polymer interfaces within the package [Shi08]. As delamination is the major failure mode in humid environments, the moisture performance of the ACF materials needs to be improved to increase the reliability of the material [Mer03]. Furthermore, the interface adhesion strength is another property to improve, but due to the diversity of the ACF materials and chemistry, the difference in moisture properties could be much more significant [Mer03].

Conformal coatings may be used to enhance the resistance of electronic devices against humid environments. Since conformal coatings are polymeric materials they are subject to the same challenges with moisture as other structures inside electronics packages. However, conformal coatings have some different properties, e.g. the moisture absorption of epoxy whereas used as conformal coating is different than the epoxy based ACF. The most important point is the relationship between the physical aging phenomenon, the coating composition and the long-term durability of organic coatings in practice [Per03]. In this study the performance of organic coatings shielding flip chip components was examined.

In Publications III-V the reliability of ACA joined flip chips was studied in humid environments using 85/85 testing. As previously discussed in 4.3.1 the failure modes were different when non-coated, epoxy coated or parylene C coated test lots were analyzed. However, when the failure analysis was performed, all these ACF interconnections failed similarly and delamination was found to be the root cause of these failures. Figure 23 shows delamination in an interconnection in non-coated test lot. As can be seen, the delamination was between a gold bump and a pad on the substrate. Figures 24 and 25 present delaminations in the interconnections from test lots with epoxy and parylene C coatings respectively. With either coating used as a protective layer, delaminations were also found between chip and bump, as presented in the figures. In other words, the gold bump was delaminated from the chip. This same behaviour was seen with composite coated test samples where epoxy and parylene C were used as bi-layer structure in Publication V. Figure 26 shows the delamination in an interconnection shielded with epoxy-parylene C bi-layer. In this case, too, the delamination occurred either between bump and pad or between chip and bump as the SEM images show.

As the conformal coating formed a covering layer on top of the package, it also stiffened the structure. When temperature and humidity changed, the physical dimensions of the structures and thus ACF swelled due to the moisture absorption, the conformal coating did not swell as much causing stresses to interconnections. This might have also led to different paths for delaminations to proceed. As discussed in Publications III and V, the conformal coating delaminated from the top surface of the silicon chip and from the nickel gold plated copper wirings on the substrate. Delamination was probably caused by the physical ageing of the polymer coating material, which caused the weakening of the adhesion [Per03]. Since the adhesion is least between silicon and gold, these were the first surfaces where delamination was seen. This also implies that some stresses occurred in the coated structure and especially with bi-layer structures these stresses in coatings cause failures. These failures have been discussed in Publication V, and Figure 27 adapted from Publication V shows cracking in epoxy layer under the parylene C layer in the bi-layer structure. In Publication V the reason for this was assumed to be the CTE differences of the materials and the differences in moisture absorption and water permeability. In Publication II the moisture absorption was studied through parylene C coating and it was found that moisture penetrates parylene C coating slowly, at least when immersed into a water tank. This result supports the assumption that polymer matrix expansion of the coating due to moisture causes cracking in bi-layer structures and delamination in single coating structures. Coating delamination has been reported to be one of the main reasons for failure in biomedical implants using different coating materials [Fal09].

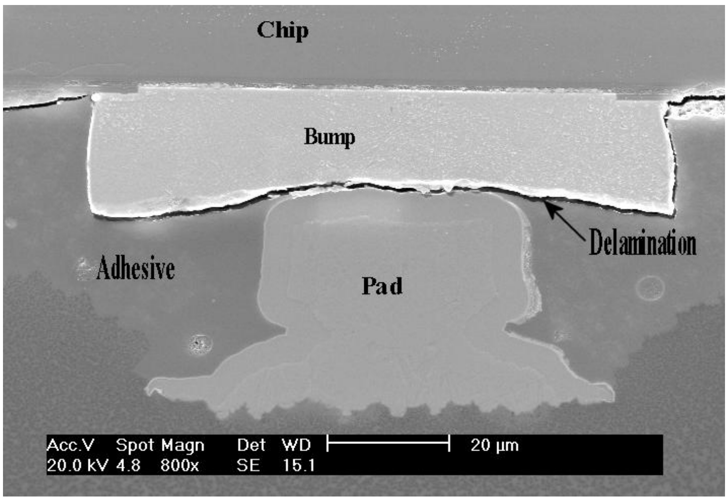


Figure 23 Delamination of non-coated test sample after 85/85 test.

(Publication III)

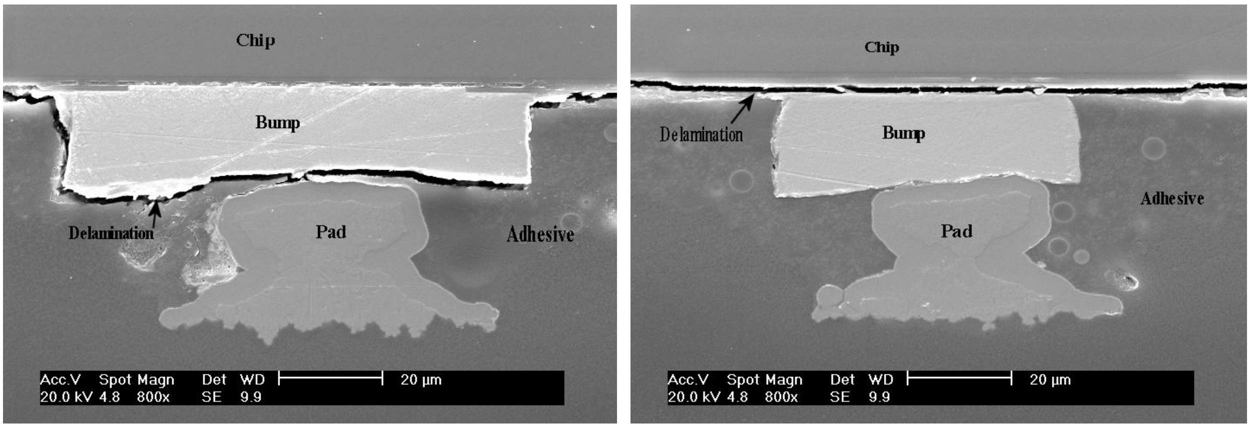


Figure 24 Delaminations in epoxy coated test lot after 85/85 test. (Publication III)

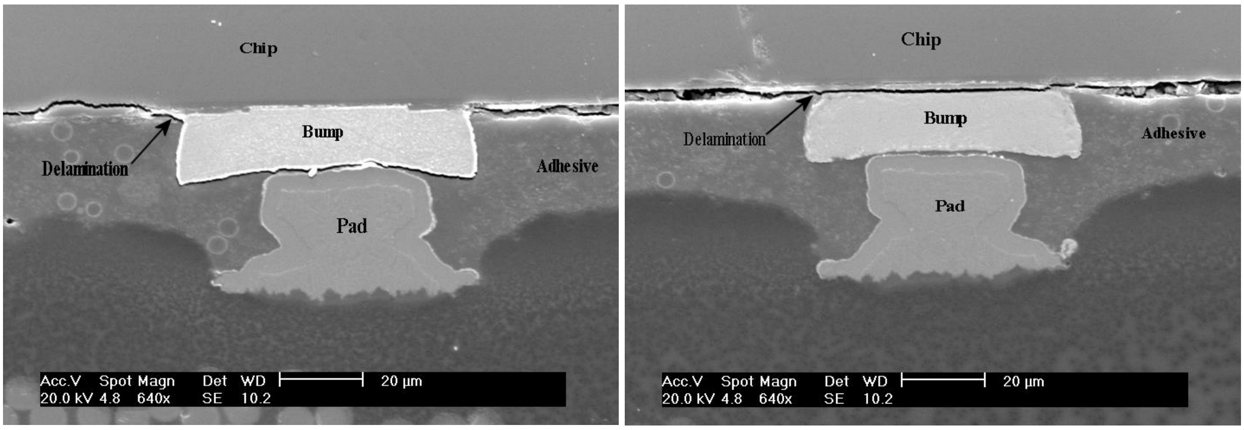


Figure 25 Delaminations in parylene C coated test lot after 85/85 test. (Publication III)

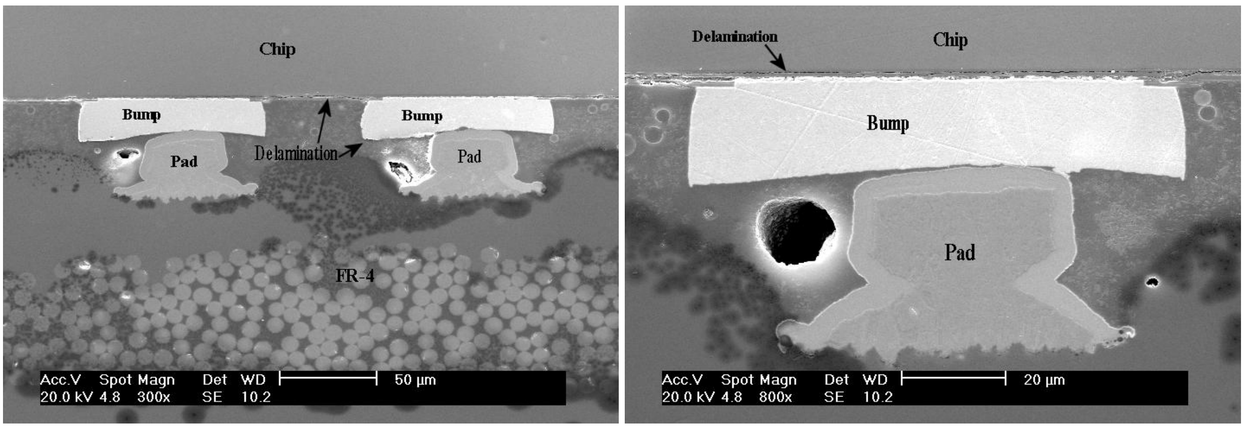


Figure 26 Delaminations in epoxy-parylene C bi-layer coated test lot after 85/85 test.

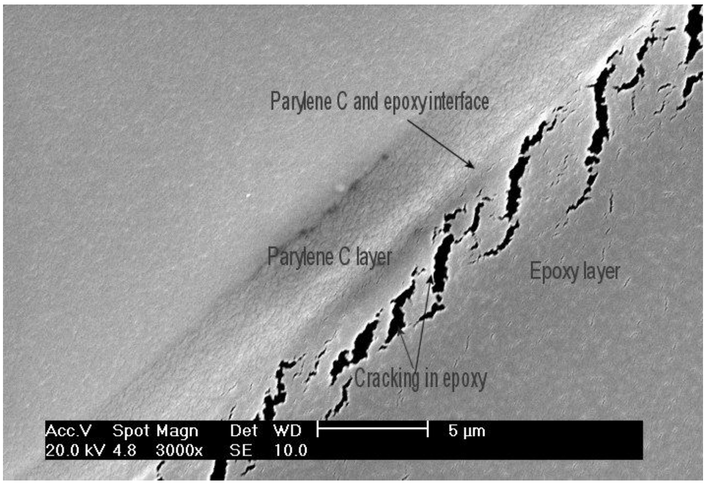


Figure 27 Bi-layer structure of parylene C and epoxy showing cracking in epoxy after 85/85 test. (Publication V)

Sterilization affected the failure mechanisms so that the delamination occurred in sterilized samples only between bump and pad. Figure 28 presents a sterilized ACF interconnection from a test lot having epoxy conformal coating as the protective layer. All the other sterilized test lots showed similar delaminations. Sterilization changed the polymer structure somewhat, as discussed in Publication IV. Both the ACF and the conformal coating were affected by sterilization when gamma sterilization was used to sterilize the medical devices. The effects of gamma sterilization on ACF were reported in Publication IV using differential scanning calorimeter (DSC) analysis. It was revealed that gamma sterilization changed the adhesive matrix. The reason for the changes was assumed to be chain breaks in the matrix [Rog05]. Similar effects in the coating materials are also possible, changing the properties and behaviour of the epoxy coating material. However, parylene C should be resistant to gamma sterilization according to reference [Wol02].

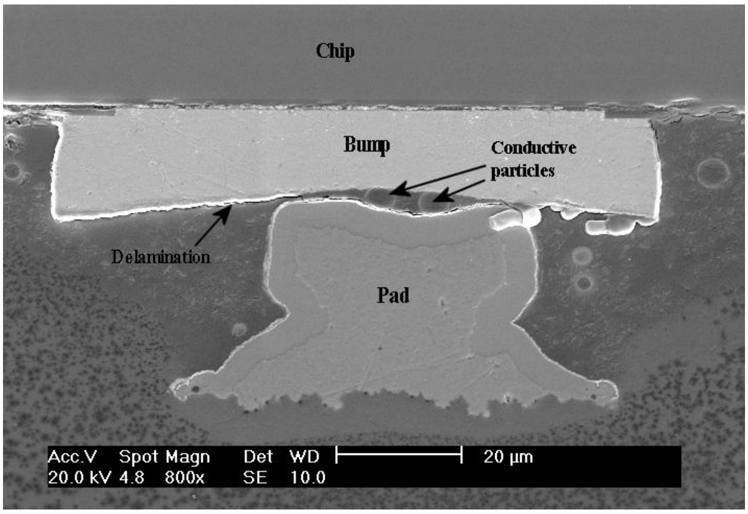


Figure 28 Delamination in epoxy coated sterilized ACF interconnection after 85/85 test. (Publication IV)

4.4.2 Temperature cycling

Thermal cycling or thermal shocks from high to low temperatures or vice versa cause stress relaxation in materials [Mar99]. These are caused by the differences in the CTEs of the different materials used in electronic devices. Interconnections are places where different materials meet and therefore they are subjected to great stresses caused by CTE mismatches [Ali05]. The effect of different in CTEs in the materials used in the package in the interconnections is introduced in Figure 29. The CTE of silicon is 2.5 ppm / °C while for FR-4 it is 12-16 ppm / °C and for polyimide 16 ppm / °C. Furthermore, ACFs have different CTEs depending on the material used and in this study the used ACF had CTE of 40 ppm / °C (Table 1). These differences cause reliability issues in thermal cycling loading. The thermal stresses are already caused to the structure in manufacturing process, where elevated temperatures are used to cure the adhesive. When the assembly is cooled down to room temperature, stresses and strains are created on the structure [Kwo06a]. These internal stresses are relaxed by high temperature exposure above the adhesives' T_g and after that the adhesive's properties are not restored to the as-cured state [Kwo06a]. After this first exposure to elevated temperature the adhesives properties are reversible in temperature cycling [Kwo06a]. Due to temperature changes the whole ACF flip chip assembly is deformed during thermal loading. The ACF material properties have a great effect on the deformation [Kwo06a]. The deformation occurs as warpage of the assembly showing greatest stresses on the corner bumps. At temperatures below the T_g of the adhesive warpage occurs downwards as the substrate contracts more than the chip and the ACF holds these two together [Kwo06a]. At temperatures higher than the T_g of the adhesive mechanical support cannot be maintained and both chip and substrate are free to expand with their inherent CTEs and thus no warpage occurs [Kwo06a]. When conformal coating is added to protect the whole electronics package it renders support to the whole structure below its T_g causing probable differences in assembly warpage and failure mechanisms.

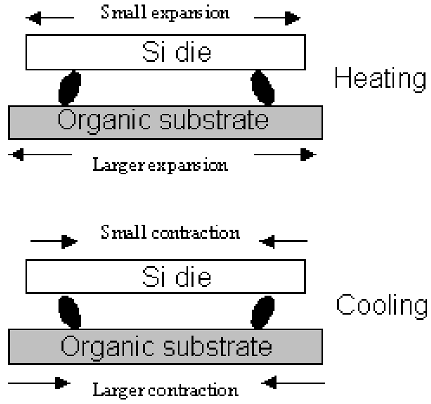


Figure 29 Differences in CTEs causing stresses to interconnections.

In this work, the thermal cycling reliability of conformally coated and non-coated samples was compared in Publication I. The thermal cycling was carried out between temperatures of -40 °C and 85 °C, as mentioned earlier. In that study both FR-4 and PI substrates were studied and PI substrate improved reliability markedly. Failure analysis of the samples with FR-4 substrate again revealed that the failures were due to delamination. When thermal cycling was carried out on the samples, the non-coated test samples showed delamination both between chip and bump, and between bump and pad, as can be seen in Figure 30. With those test samples where parylene C was used as a conformal coating, the delaminations were only seen between bump and pad. This is illustrated in Figure 31. The stresses formed in the structure in constant humidity testing and in temperature cycling testing were different, causing the delaminations to occur on dissimilar interfaces. In constant humidity testing the mechanisms causing the stresses to form in the structure are in the polymer matrix expansion of the coating and the ACA matrix due to moisture, while in thermal cycling testing the delamination is caused by differences in CTEs. The moisture and thermal expansion change both the adhesive and the conformal coating. The CTEs of the ACF and parylene C are both 40 ppm/°C (Table 1 and 2) in this study, diminishing the caused stresses. Moreover, in thermal cycling testing the coating material may decrease the effects of temperature changes to the structure, since the coating material may reduce the thermal conductivity. As with all polymer materials, conformal coating materials do not have good thermal conductivity which may cause challenges in electronics which produce heat when operating. However, in implant applications, heat may not be produced since the implant cannot heat the surrounding environment. The whole structure needs to be designed so that the power consumption is as low as possible.

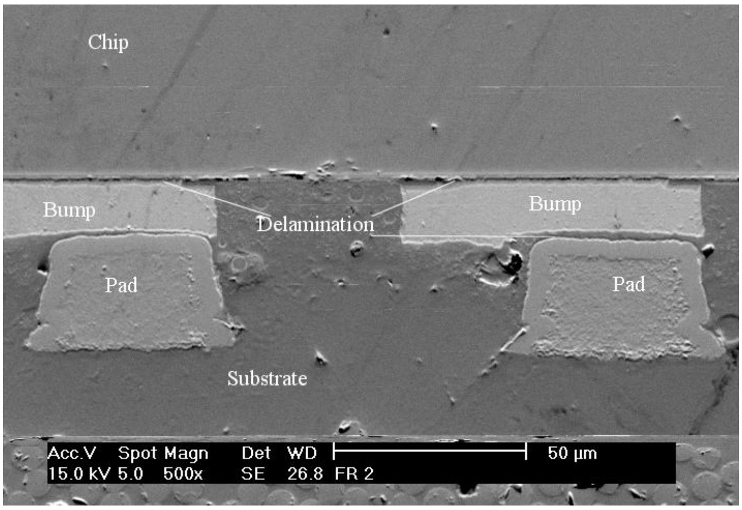


Figure 30 Delamination in non-coated test lot after thermal cycling test. (Publication I)

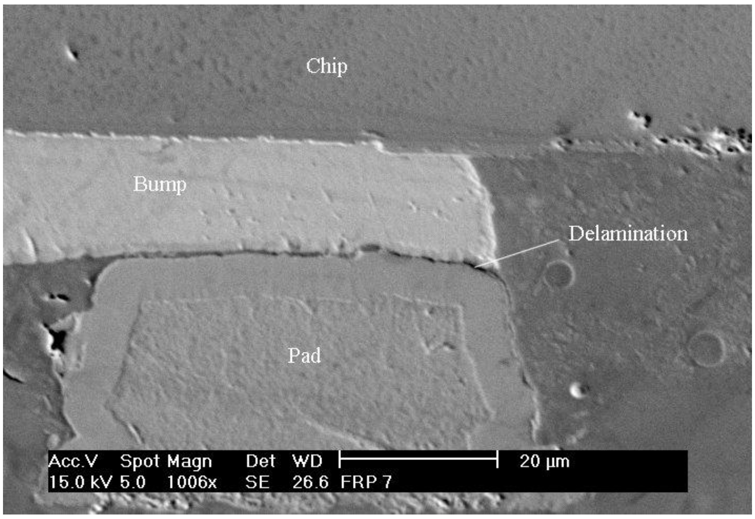


Figure 31 Delamination in parylene C coated test lot after thermal cycling test. (Publication I)

Parylene C conformal coating was studied after thermal cycling testing and it emerged that during testing the adhesion between parylene C and silicon chip was decreased. Delamination was found at the interface between parylene C and silicon. The adhesion of parylene C to the silicon chip was reported in Publication III, where the adhesion of parylene C to the silicon chip was measured using pull-off testing after constant humidity test. Even though the testing was done after exposure to humidity the poor adhesion to silicon could be seen. The adhesion degradation to silicon only after thermal cycling testing can be seen in a micrograph presented in Figure 32, where delamination between parylene C and silicon can be seen. When the delamination entered the interface between parylene C and ACF the delamination weakened since the adhesion of parylene C to the ACF was much better.

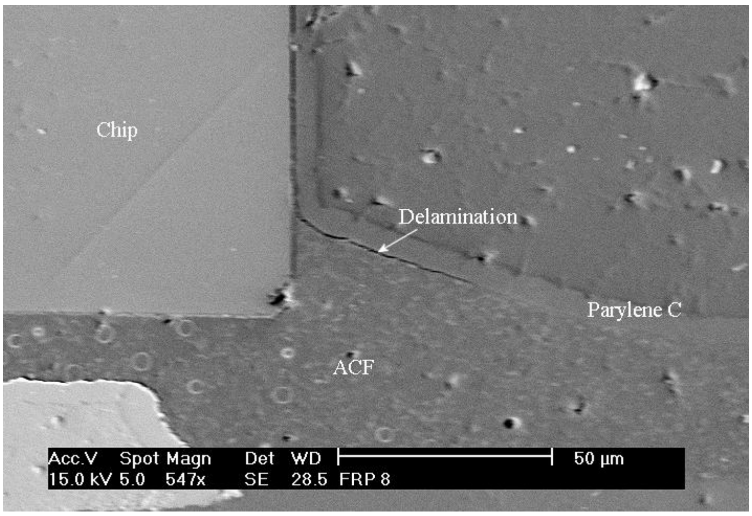


Figure 32 Delamination between chip and parylene C and between parylene C and ACF after thermal cycling test. (Publication I)

4.4.3 Salt spray

Salt spray atmosphere may cause dramatic failures in electronic packages. In such environments the effects of moisture on reliability are exacerbated by salt. It combines with water to form acid/alkaline solutions which damage materials and cause corrosion of metals [Mar99]. Galvanic corrosion is reported as a common form of corrosion due to material combinations inside electronics packages which include gold, nickel, and copper [Amb07]. Failures caused by corrosion can be minimized by selecting appropriate combinations of corrosion resistant materials such as protective coatings [Com86].

Conformal coatings are used to protect electronics from corrosion and in this work, too, parylene C was used to protect ACF joined flip chip components. Parylene C proved to be a good barrier against salt spray atmosphere, as reported in Publication II. Test samples with no coating to protect the ACF joints or substrates suffered from corrosion, which was found to be the root cause of failures in that test lot. An example of a corroded ACF joint is presented in Figure 33. These failure mechanisms and analysis are further discussed in Publication II.

Salt spray testing was performed according to the JEDEC standard introduced in 4.1.3 and in Publication II. When the environment in this test and the environment in hydrolysis testing conducted in Publication VI are compared, it can be seen that the concentration of NaCl is much higher in salt spray testing than in hydrolysis testing. However, in hydrolysis testing there were also other constituents present because of the PBS solution. Furthermore, in the salt spray test the samples were placed in a chamber where the solution containing NaCl was sprayed while in the hydrolysis test the samples were immersed in the solution. This causes differences in the testing environment and the mechanisms by which ions may induce failures. In both tests the parylene C coated test lots had no failures during testing. However, the test samples in the hydrolysis tests had some constituents condensed on the surface of the coating during testing. Figure 34 shows these constituents. The samples having epoxy coating did not show such constituents. The parylene C coated samples after the salt spray test had no constituents on their surfaces.

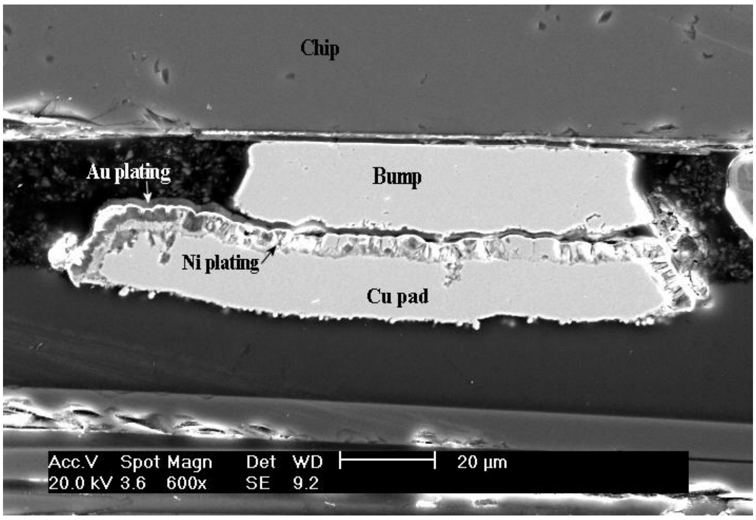


Figure 33 Corrosion of the Ni layer revealed to be the failure mechanism after salt spray testing.

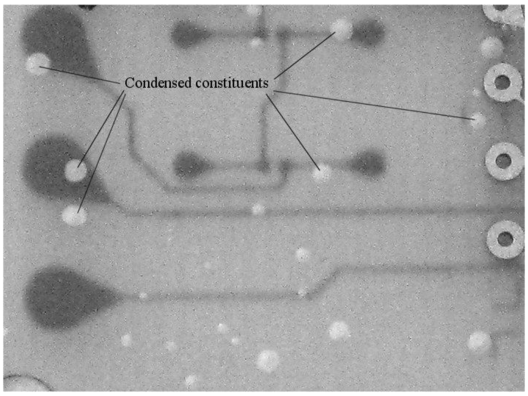


Figure 34 Constituents on parylene C coating after hydrolysis test. (Publication VI)

4.5 Moisture absorption

Fickian laws describe the transport of moisture in polymers or polymer composites. Because moisture cannot penetrate fibres, the behaviour of diffusing moisture in composites is usually affected by the resin properties [Lee93]. The general diffusion law or Fick's second law of diffusion describes the moisture transport by

$$\partial^2 C - \frac{1}{D}\frac{\partial C}{\partial t} = 0 \qquad (9)$$

where C is the moisture concentration in kg/m^3, t is time in s, and D is the diffusion coefficient of the moisture in polymers in m^2/s [Fan09]. The temperature dependence of the diffusion coefficient can be determined using the Arrhenius equation presented in Equation (8). The Diffusion coefficient or diffusivity D can be determined using a best fit curve fitting approach of the experimental weight gain data [Jed08]. A solution for rectangular or square samples is as follows [Jed08]:

$$\frac{M_t}{M_{Sat}} = 1 - \frac{512}{\pi^6} \sum_{l=0}^{\infty} \sum_{m=0}^{\infty} \sum_{n=0}^{\infty} \frac{\exp\,(-D \cdot t \cdot L_{eqv})}{(2l+1)^2(2m+1)^2(2n+1)^2} \tag{10}$$

where $L_{eqv} = \pi^2 \left\{ \left(\frac{2l+1}{x_o}\right)^2 + \left(\frac{2m+1}{y_o}\right)^2 + \left(\frac{2n+1}{z_o}\right)^2 \right\}$

x_0, y_0 and z_0 are the width, length, and thickness of the sample respectively. Weight gain and saturated moisture mass can be determined from the experimental data.

The moisture absorption of FR-4 and parylene C coated FR-4 substrates was studied in Publication II. Rectangular samples were first dried and then immersed in deionized water. The weight gain was measured at fixed time intervals. This was done both at room temperature and in boiling water. Test results were used to approximate the diffusion coefficients with the above mentioned formulas. The analysis was carried out and the diffusion coefficients gained are presented in Table 4 for different test lots. The diffusion coefficient for epoxy found in the literature was from 3 x 10^{-13} to 5 x 10^{-12} m^2/s [Leg09]. In FR-4 structure the presence of glass fibres changes the coefficient from pure epoxy. However, the results for diffusion coefficients are near the range for epoxy and thus they seem reasonable. Parylene C coating improved the water resistance and thus decreased the diffusion coefficient. Furthermore, the diffusion coefficients are dependent on temperature, as can be seen in Table 4.

Table 4 Calculated diffusion coefficients for FR-4 laminates and parylene C coated laminates at room temperature and at 100 °C.

Test lot	Temperature /°C	Diffusion coefficient / m^2/s
FR-4	22	2.1955 x 10^{-12}
FR-4 and parylene C	22	1.2997 x 10^{-13}
FR-4	100	1.4923 x 10^{-11}
FR-4 and parylene C	100	1.2309 x 10^{-11}

The moisture mass gained during testing is presented in Figures 35 and 36 to show the test lots without and with parylene C coating respectively. As can be seen in Table 4 and in the figures, parylene C coating slowed down the diffusion of water. In Figure 35 the Fickian behaviour does not fit with the experimental results. This kind of behaviour has also been reported in other studies on epoxy resins [Leg09]. This can be explained by non-Fickian sorption of water into a polymer [Bon06][Fan09]. In this model the absorption of water into polymer depends on the binding probability of the water molecule. The diffusion can be divided into two phases. First the probability that water molecules interact with the polymer is quite small and the diffusion follows Fickian sorption [Bon06][Pop06]. As greater amounts of moisture are absorbed the probability that water molecules interact with the polymer increases and the transport process is slowed down [Bon06].

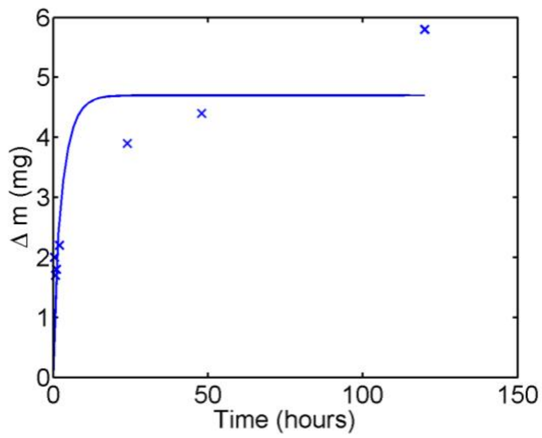


Figure 35 Moisture mass gained in immersion test vs. Fickian approximation for FR-4 samples without parylene C coating.

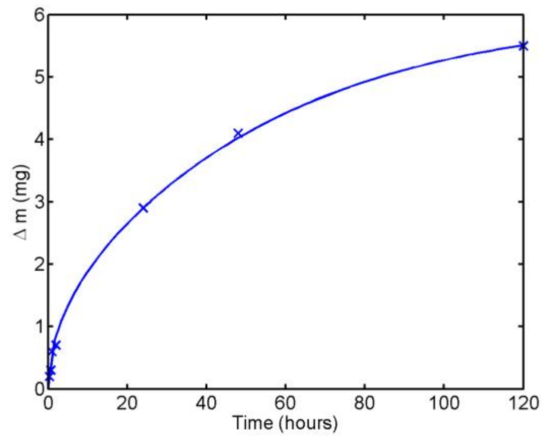


Figure 36 Moisture mass gained in immersion test vs. Fickian approximation for FR-4 samples with parylene C coating.

Parylene C coated samples and immersion in boiling water followed the Fickian diffusion model. For all these test lots the testing time was apparently too short to show the saturation of moisture mass. If the test had lasted longer the same kind of behaviour might have been seen as in Figure 35. With parylene C the diffusion process is slower and here only the Fickian behaviour can be seen. The immersion tests in boiling water only lasted for 60 min and again only the Fickian behaviour can be seen. The moisture masses gained in test lots both without and with parylene C coating in boiling water are presented in Figures 37 and 38 respectively. In boiling water the effect of parylene C is smaller, as can be seen in both Table 4 and the figures.

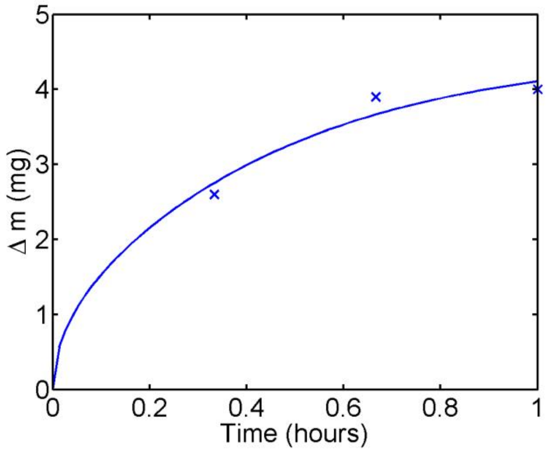


Figure 37 Moisture mass gained in test lot without parylene C coating in boiling water.

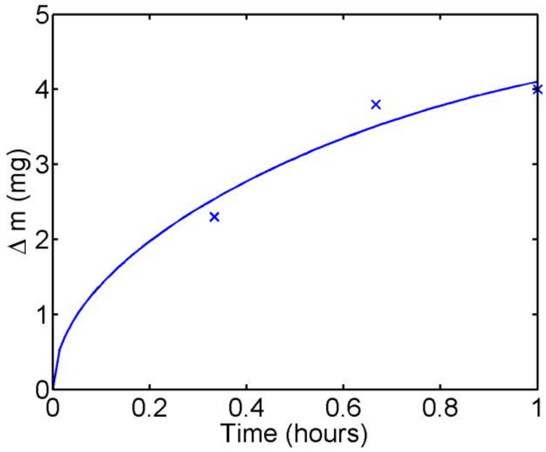


Figure 38 Moisture mass gained in test lot with parylene C coating in boiling water.

5 CONCLUSIONS AND FINAL REMARKS

A work is perfectly finished only when nothing can be added
and nothing taken away.
–Joseph Joubert

Electronics used in harsh environments impose extra demands on reliability and protection. In many cases electronics that operate in demanding environments are also the electronics that need to stay functional i.e. the reliability needs to be good. To achieve this, conformal coatings are used to protect the electronics from the environment. One such environment is inside the human body, where metal cations, salts, acids, dissolved oxygen and cells are present. If electronics are placed inside the human body, it is vital that the device is protected against this environment. Conformal coating protection works in two ways. It needs to protect the electronics from the environment but on the other hand it needs to protect the ambient environment from the electronics. Since electronic devices consist of many different materials that may be toxic and not biocompatible, the coating material needs to isolate these materials from the human body.

In this work the effects of conformal coatings upon the reliability of anisotropically conductive adhesive joined flip chips were studied. In these studies the main focus has been for use in medical electronics applications, where the effects of humidity, body fluids, sterilization, and salty atmospheres were studied. The conformal coating materials selected and used in this work were applicable in medical electronics applications. The conformal coatings used are epoxy and parylene C, which have both been approved for their biocompatibility by USP. The work has been extended to also cover other use environments by testing the reliability also in such environments which are not likely for medical implants but for other applications, i.e. temperature cycling test.

In the constant humidity tests, parylene C proved to be the most reliable choice to protect the electronics. Epoxy or epoxy parylene C composite coating structure enhanced the reliability from non-coated samples but obviously caused stresses inside the package causing different failure modes than without conformal coating. Parylene C slowed down the moisture absorption through the package to the adhesive and thus changed the failure mode to a slow increase of daisy chain resistance. This enhanced the long-term reliability and especially when the failure criterion was set higher, parylene C coated test samples endured humid environments well. Non-coated test samples showed peaks in daisy chain resistance prior to failure and thus the effect of failure criterion selection was clear. Epoxy had such an effect on the adhesive joints that the failures appeared as abrupt changes in daisy chain resistance and thus the selection of a failure criterion did not have much effect on the results.

Failure analysis of all the test lots with different or no-coating, showed delamination as the failure mechanism. However, without coating the delamination occurred only between bump and pad while with coating the delamination was found either between bump and pad or between chip and bump. Gamma sterilization of the devices affected the structure so that the delamination occurred only between bump and pad, also with conformal coating.

Thermal cycling testing indicated that the coating material did not impair or improve reliability. The failures were caused by differences in CTEs in thermal cycling and a thin layer of polymer coating did not markedly change the behaviour of the structure. Parylene C, however, delaminated from the silicon chip during testing due to weaker adhesion. This did not cause reliability issues to the structure, since the PI substrates performed well in the test even though they suffered from this delamination.

Salt spray testing caused severe corrosion to the ACF interconnections when no coating was protecting the flip chips. Parylene C conformal coating protected the interconnections well and prevented the formation of corrosion. In hydrolysis testing the atmosphere differed somewhat from salt spray and even in hydrolysis testing parylene C withstood the influences of hydrolysis solution.

The study and research on medical electronics is nowadays intense, and the potential in that area is vast. Research needs to be performed carefully since human lives depend on the basic research work. The reliability of electronics is vital, likewise the miniaturization of such devices is essential for the use of the applications. The study of different miniaturization methods and their reliability, and different shielding options are areas for further study.

In the following paragraphs the main results of the publications are summarized.

Publication I, entitled “Thermal cycling of ACF joined flip chips on FR-4 and PI substrates with parylene C coating”, described the effects of conformal coating on thermal cycling reliability. Two different substrate materials were used since the substrate material has a significant effect on long-term reliability. The results showed that PI substrate endures better in thermal cycling loading than FR-4. Test samples with PI did not fail during the test but the samples on FR-4 failed and failure analysis revealed the failure mechanisms to be the same both with and without a parylene C conformal coating. The root cause of failures was delamination between chip and bump and bump and pad.

Publication II, entitled “Corrosion protection of anisotropically conductive adhesive joined flip chips”, studied the effects of salt spray testing on ACA interconnections both with and without a protective coating. The aim was to detect the failure mechanisms occurring in ACA interconnections in salt atmosphere and to ascertain the effect of a parylene C conformal coating to protect the interconnections. The functioning of the coating was further studied with moisture absorption testing in this paper. As a result, severe corrosion was found in the Ni layers that protect the Cu pads on substrates. Corrosion was only detected with test lots that did not have a parylene C coating. It was found that even though parylene C absorbs moisture, it prevents corrosion by inhibiting the salts and other corrosive substances from penetrating the coating.

Publication III, entitled “Effects of conformal coating on anisotropically conductive adhesive joints; a medical perspective”, focused on ACA interconnection reliability using constant humidity testing. The study considered test samples without conformal coating, with epoxy coating and with parylene C coating. The failure mechanisms were studied and the effects of both coatings on reliability were evaluated. Moreover, the adhesion of coating materials was studied after exposure to humidity. The results revealed delamination in all test lots. The difference in these three test lots was that without a coating delamination was only found on the interface between bump and pad while with either an epoxy or a parylene C coating delamination was found between bump and pad or between chip and bump.

Publication IV, entitled “Influence of medical sterilization on ACA flip chip joints using conformal coating”, concentrated on the effects of medical sterilization on the reliability of ACA joints. The sterilization was done in this study using a gamma sterilization method. When electronics are used in medical applications it needs to withstand all the procedures needed prior to use. Sterilization is needed for all devices in contact with blood or other body fluids. In this paper reliability was studied using constant humidity testing and the results of sterilized test lots were compared with those of the non-sterilized test lots from Publication III. The results showed that gamma sterilization changed the ACF material, however, the changes were minor and the reliability was not markedly changed due to sterilization.

Publication V, entitled “Composite coating structure in an implantable electronic device”, introduced a bi-layer structure which consisted of both an epoxy and a parylene C coating layer to protect medical devices from humid environments. The aim of the bi-layer structure was to combine the good properties of both coating materials and to smooth the surface of the implant device. The bi-layer structures were compared to the test lots with only one coating material from Publications III and IV. The bi-layer structures were tested both non-sterilized and sterilized in gamma radiation. As a result, the bi-layer structure was found to be as reliable as a pure parylene C coating, but the benefit of bi-layer structure is that the geometry of the implant can be altered using an epoxy layer underneath the parylene C coating.

Publication VI, entitled “Hydrolysis testing of ACF joined flip chip components with conformal coating”, concentrated on testing the dummy implant devices *in vitro*. The testing was conducted for epoxy, parylene C and epoxy-parylene C bi-layer coated samples in Na-PBS solution. The testing was conducted for ten months and the results showed that each of the coatings gave sufficient protection to the electronics inside. Delamination of coating materials from silicon chip and NiAu coated Cu wires were found, but apparently this did not impair the reliability results.

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