Non-contact techniques for measuring moisture related properties of Ethylene Vinyl Acetate as a photovoltaic module encapsulant

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Non-contact techniques for measuring moisture related properties of Ethylene Vinyl Acetate as a photovoltaic module encapsulant

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This dissertation is submitted for the degree of
Doctor of Philosophy

2015
This thesis is dedicated to the memory of my loving father, Khalil Rashtchi.
I miss him every day.
I am who I am because of him.
He thought me never back down.
To stand tall even when standing is impossible.
Even though he’s gone I’ll stand with all my passion for life.
Now, I could gladly say:
I am his daughter.
Acknowledgements

-I will always remember these faces...
Abstract

Polymer based encapsulant materials such as Ethylene Vinyl Acetate (EVA) are commonly used in silicon-based PV modules and play a central role in the structural integrity and hence long term performance of the device. In spite of many advantages, however, water can diffuse through them, leading to swelling and chemical degradation that can make the device susceptible to accelerated corrosion and failure. This thesis reports on efforts to provide novel methodologies to investigate the opto-mechanical response of EVA due to water diffusion and on the quantification of water concentration in deployed PV modules. A novel non-contact time-resolved measurement technique based on Wavelength Scanning Interferometry (WSI) is proposed to determine thickness, refractive index, thickness change and refractive index change in EVA during water uptake. This is preceded by numerical simulations to inform the design of the experimental methodology and setup, such as thickness of EVA layer, maximum surface velocity during measurements, type of data filters to use for data analysis and also for validating the proposed approach. The measured time-resolved thickness profiles provide a unique opportunity to study the swelling behaviour of EVA. Using a novel inversion technique, the hygroscopic expansion coefficient (HEC) as a function of water concentration is established for the first time. A hygro-mechanical Finite Element model is also developed to predict time-resolved hygroscopic expansion of EVA and compare it with experimental measurements. The hygro-mechanical stress is further studied through a set of deflection tests on EVA-glass bi-material strip specimens immersed in water and intermittently measured with an optical profilometer.
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Nomenclature

Alphabetical Symbols

$A_1$ Amplitude of the object waves from front surface of EVA
$A_2$ Amplitude of the object waves from back surface of EVA
$A_{ref}$ Amplitude of the reference wavefront
$A_s$ Amplitude of light source
$C$ Water concentration
$C_{ave}$ Average water concentration
$C_{sat}$ Saturation water concentration
$D$ Diffusion coefficient
$D^*$ Normalized detectivity of detector
$E$ Young modulus
$E_i$ Indenter modulus
$E_r$ Reduced elastic modulus
$E_s$ Indentation modulus of the specimen
$f$ Frequency (cycles per frame)
$h(C)$ Hygroscopic expansion coeffient as a function of concentration
$I$ Moment of inertia
$\tilde{I}(f)$ Fourier transform of intensity $I(t)$
$I(t)$  Intensity of interference signal as a function of time

$J$  Mass flux

$K$  Conductivity

$k$  Wavenumber

$\delta k$  Interframe wavenumber increment

$\Delta k$  Wavenumber range

$k_c$  Central wavenumber

$L_{eq}$  Equivalent length

$M(t)$  Weight of the sample at time $t$

$M_q$  Saturated mass

$M_t$  Instantaneous mass of the sample

$n_{air}$  Refractive index of air

$n_{EVA}$  Refractive index of EVA

$N_t$  Number of frames

$n_{water}$  Refractive index of water

$\mathbb{P}$  Momentum

$q$  Heat flux

$R$  Radius of curvature

$r$  Reflectivity

$t$  Time

$\bar{t}$  Transmissivity

$TH$  Total thickness in bi-strip

$th(t)$  Sample thickness as function of time

$V$  Volume
\( \hat{W} (\hat{f}) \) Fourier transform of window function \( W(t) \)

\( x \) Through thickness direction

\( \Delta x \) EVA thickness change

\( y \) Through length direction

\( z \) Through width direction

\( z_0 \) Geometrical path length between ZDL and glass substrate in HEC laminated sample

\( z_1 \) Geometrical path length between ZDL and front surface of EVA in HEC laminated sample

**Abbreviations**

**CTE** Coefficient of thermal expansion

**HEC** Hygroscopic Expansion Coefficient

**ZDL** Zero delay line

**Greek Symbols**

\( \delta \) Deflection

\( \delta \Lambda \) Optical path resolution

\( \Gamma \) \( E_1/E_2 \) in bi-strip

\( \gamma \) Constant that depends on the shape of the sampling window function \( W(t) \). \( \gamma = 2 \), \( 4 \) for rectangular or hanning windows, respectively.

\( \Lambda \) Optical path difference

\( \lambda_c \) Central wavelength

\( \mu \) Absorption

\( \nu \) Poisson’s ratio

\( \phi \) Phase

\( \sigma_{max} \) Maximum Shear stress
\( \Upsilon \quad th_1/th_2 \) in bi-strip

\( \varepsilon_{\text{ave}} \quad \text{Average strain} \)
Chapter 1

Introduction

1.1 Motivation for the present work

Cost effectiveness of energy systems cannot be achieved by focusing research on improving the efficiency alone, the focus also need to cover high life-time energy yield as well. Stability and Performance of Photovoltaics (STAPP) is a research project focusing on understanding the building blocks of stability and performance in PV systems as well as studying uncertainties in energy yield and the possible causes of degradation. To investigate the missing elements in long-term performance, STAPP aimed for developing a multiphysics numerical model to scrutinize the durability of PV module under combination of in-service loadings. This model takes into account a number of interconnected factors graphically shown in Fig. 1.1. This graph represents a comprehensive approach to the durability analysis of photovoltaic module by addressing the six main areas including mechanical, electrical, thermal, optical, chemical aspects and moisture which directly and indirectly influence the functionality and life of PV modules.

The present thesis, as part of the above general picture, is focused on two areas: effects of moisture on mechanical as well as optical properties of encapsulant. Encapsulation takes the central stage in providing structural integrity of the module, however, encapsulation related degradation is a key failure mode for PV systems Quintana et al. (2002). Numerous reliability and lifetime concerns in PV modules are associated with encapsulation which are believed to originate from moisture diffusion during service life Ardebili et al. (2003). Consequences are loss of adhesion, degradation of packaging materials, degradation of cell/module interconnects, drive performance loss and possibly failure for the PV modules Quintana et al. (2002).

In order to achieve the intended predictive ability, the comprehension of underpinning
Figure 1.1: STAPP comprehensive approach aims to provide multiphysics insight to the durability analysis of PV modules. The diagram is addressing six main interconnected areas including mechanical, electrical, thermal, optical, chemical aspects and moisture which directly and indirectly influence the functionality and life of PV module. The present thesis is focused on moisture related properties, mechanical and optical properties of PV.
mechanisms and responses in the materials and interfaces to environmental conditions is crucial. One of these mechanisms associated with moisture ingress into encapsulant is swelling. Several failure modes in electronic packaging devices are believed to have links with this material attribute of polymeric encapsulants Ardebili et al. (2003). When a polymer encapsulant swells upon moisture absorption and adjacent materials do not, hygroscopic stresses arise due to the mismatch. The build-up of stress from a number of contributing parameters including thermal, hygroscopic and mechanical loading, could eventually affect the structural integrity of the system Wong et al. (2002b). Accurate HEC measurements are essential for evaluating the effect of hygroscopic stress on package reliability. Hygroscopic swelling of EVA, the dominant PV encapsulant, is not adequately studied in the existing literature.

Several techniques have been introduced in the past to characterize hygroscopic swelling Ardebili et al. (2003). Amongst those, a widely used approach in electronic packaging materials is employing a Thermo-Mechanical Analyser (TMA) in conjunction with a Thermo-Gravimetrical Analyser (TGA) by finding the dimensional changes of the sample through a contact probe in the TMA while the moisture content is obtained from TGA in a desorption procedure, to finally extract the strain vs. moisture concentration curve Ardebili et al. (2003); Tee et al. (2003); Wong et al. (2002b); Zhou (2006a); Zhou et al. (2005a,b). Alternatively, non-contact approaches include optical methods such as Moiré interferometry, used to measure ‘in-plane’ hygroscopic strain in mold compounds Stellrecht et al. (2003).

The high compliance of EVA renders contact approaches such as TMA-TGA unsuitable. TGA is a destructive test that leads to chemical degradation, besides it accounts for desorption behaviours rather than absorption. Moreover, EVA free standing films tend to warp due to water uptake when placed in a humid environment. For this reason, monitoring the ‘in-plane’ dimensions of an initially flat sample is not possible and an alternative approach to Moiré interferometry is required.

In this research, a non-contact method based on Wavelength Scanning Interferometry (WSI) is proposed to determine thickness, refractive index, thickness change and refractive index change in EVA due to water uptake. It is highly desirable to have a single measurement technique to obtain both refractive index and thickness, and efforts have been made to find valid approaches to measuring refractive index and thickness of material simultaneously or separately or at least by using the same apparatus. Gahm and Witte (1986); Martinez-Anton and Bernabeu (1996); Maruyama et al. (2002); Tearney et al. (1995) presented methods for determining the thickness, as well as the refractive index employed low coherence interferometry in combination with microscope to
simultaneously measure both. Fukano and Yamaguchi (1999) presented independent measurement of refractive index and thickness by combining WSI and confocal microscopy. Simultaneous measurements has also been demonstrated by Martinez-Anton and Bernabeu (1996) using interferential spectrogoniometry. Most of above approaches however, require complex instrumentations such as high resolution translation stage due to use of single wavelength in addition that they only deal with a single layer of material and not laminated sample. Coppola et al. (2003) demonstrated a simple technique using tunable light source and developed a lateral-shear, wavelength scanning interferometey to obtain thickness and refractive index simultaneously.

The presented system uses a tunable near-infrared laser to illuminate a layer of EVA laminated onto a glass substrate. The interference signal between light reflected at different interfaces of laminated EVA on glass and a reference beam is modulated with temporal frequencies proportional to the optical path difference between them. Fourier transformation of the interference signal leads to peaks that locate the position of the interfaces. The refractive index and thickness of the EVA layer can be determined and tracked as a function of time by comparing the distances between pairs of peaks in the Fourier spectrum. In this technique detected optical path changes are separated into refractive index and thickness changes.

Taking advantage of a time-resolved thickness profile measurement using WSI on EVA film laminated on glass, a numerical technique was developed to find an approximation of the HEC vs. concentration relationship. HEC is widely accepted as a material constant, however our assumption of dependency of HEC to moisture concentration is expected to improve the predictive power of forward FE models that evaluate mechanical stress in response to water ingress. In addition the proposed technique uses a single measurement of swelling from dry to saturated conditions which save experimental time and effort. The study on hygroscopic swelling induces stress is also performed through measurement of deflections in bi-material strips exposed to water. This experiment represents another independent observation on the effects of hygroscopic swelling which is also compared with corresponding FEA predictions to verify the HEC calculations.

1.2 Novelty of research

In this research the following novel aspects could be highlighted:

In using WSI technique explored in this work:

- Development of time resolved wavelength scanning interferometry
• Simultaneous measurement of thickness and refractive index change was carried out

• Comprehensive numerical simulation, prior to above, to help for designing the experimental set up details such as thickness of EVA layer, maximum surface velocity in measurement, type of filter to use for data analysis of results and also validating the approach.

In measurement of HEC the proposed characterization methodology features the following unique attributes:

• Use of WSI high sensitivity

• Assumption of HEC as a non-constant material property

• Linking the material strain to non-uniform moisture distribution in the material-to avoid averaging through the thickness

• Use of moisture absorption rather than desorption in the specimen

• Use of water immersion condition to speed up swelling response

• Use of FEA to observe material response to the moisture. The results obtained from this methodology offers challenges to widely accepted approaches and assumptions in calculation of hygroscopic expansion.

1.3 Organisation of the thesis

This thesis is organized as follows:

Chapter 1 presents a description of the motivations and highlighting the scope of the project. Chapter 2 reviews previous works in relevant areas including general introduction to PV and durability problems and a brief description of the existing inspection technologies and the achievements of other researchers.

Chapter 3 cover the principles of WSI, development of theoretical calculations, the concepts of processing as well as preliminary simulation. The simulation allows an inspection on different aspects and details of the experimental set up, as well as system performance and limitations.
Chapter 4 describes the experimental WSI set up and main instrument subsystems as well as sample preparation. This chapter explains validation tests to establish the analysis technique to reconstruct the moving surface, followed by actual experiment and results.

Chapter 5 presents a numerical approach to identify the HEC as a function of water concentration. The time-resolved thickness profile measurement from previous chapter is the input to a developed numerical technique along side the experimental water diffusion tests. FE simulation is then used to provide the comparison between the results from the presented technique and available methodologies.

Further tests for understanding the hygroscopic swelling induced stress is presented in Chapter 6.

Finally Chapter 7 consists of a conclusion and recommendations for future works.
Chapter 2

Photovoltaic modules: moisture associated mechanical reliability concerns

2.1 Introduction to photovoltaic modules

The sun plays as the immediate vital element for life on Earth, supplying light and keeping the essential energy cycles. Sunlight can be utilized in the production of electricity. Photovoltaic (PV) comprise the technology to generate electricity directly from sunlight. The basic unit sensitive to sunlight is the photovoltaic cell which is a semiconductor device with an area between 1 and 100 \( cm^2 \) that generates electricity when exposed to sunlight. Commonly, arrays of individual cells are connected together to form modules with an area between 0.5 and 2 \( m^2 \) and modules are combined in order to create a PV system that produces the intended output voltage and current. Today’s available photovoltaic cells are predominantly made from silicon. Thanks to the extensive progress on solar cells research, a broad range of alternative cell solutions have been introduced; however, the focus of this research is on the silicon based photovoltaic technology. In 1839 the first observation of the photovoltaic effect was reported by Edmund Becquerel, a French scientist. However it was not totally understood before the advances of quantum theory of light and solid state physics in early to middle 1900s. Orbital satellites in space programs in the 1950s constituted the first commercial use of PV technology. Since then, PVs have undergone remarkable progress and development Mah (1998). A common PV module consists of solar cells embedded in an encapsulation material between front glazing and a back-sheet which provides rigidity to the module.
Transparency is required only on one side of the module therefore the back sheet is usually made of material other than glass. The purpose of the encapsulant in a module is to bond the multiple layers of module together. Other critical characteristics of the encapsulant include high optical transmittance as well as very good adhesion to other components. Moreover, it should provide adequate mechanical strength to withstand induced stresses due to thermal expansion difference between components. An exploded view of a PV module is depicted in Fig. 2.1.

![Exploded diagram of a PV module](image)

Figure 2.1: Exploded diagram of a PV module

Ethyl Vinyl Acetate (EVA) is a polymer widely use for the encapsulation in PV modules McIntosh et al. (2009b), which also acts as bonding to the front and back sheet. It contributes to the structural integrity of the modules, provides optical coupling as well as electrical insulation and also acts as a physical insulation protection and environmental barrier. Photovoltaic modules are expected to have a life time of above 20-years and therefore it is necessary to understand the processes which contribute to module failure as long term PV reliability is crucial to the cost-effectiveness as well as commercial success of PV.

The cost involved in producing energy by a PV module comprises the initial cost of manufacturing as well as installation and commissioning, then over time amortization costs, and at the end decommissioning costs. Amortization costs are somehow the stealth part of the equation. The panels are expected to hold the primary efficiency; however the amount of produced energy through the operational life of a solar panel is highly
2.1 Introduction to photovoltaic modules

dependent on the durability of the panel. Durability itself is a function of ageing, water ingress and associated damages, change in the material properties in terms of mechanical, electrical, optical, chemical attributes and structural integrity of the panel. Gaining the capability to anticipate the durability of PV modules would lead to better estimates of amortization cost, warranty cost, and government subsidies.

2.1.1 Different types of encapsulants

There are different encapsulant materials used in PV systems. Some of them are well established in the industry and have been used for over 3 decades and some others are novel materials. The most prevalent and most studied materials are ethylene vinyl acetate (EVA), thermoplastic polyurethane (TPU), polyvinyl butyral (PVB), ionomers (ION) and thermoplastic silicone (TSI).

Presently, EVA is the predominant encapsulation material utilized in the PV industry. It is inexpensive and one of the first encapsulant materials investigated; its formulation has been well developed for exposure to sunlight Cuddihy et al. (1986); Holley Jr et al. (1996); Kempe (2006); Pern and Glick (1997). EVA is a copolymer of ethylene and vinyl acetate denoted by the molecular formula \((C_2H_4)_x(C_4H_6O_2)_y\) as shown in Fig. 2.2, where \(x\) and \(y\) indicate the ratio by weight of ethylene unit \((C_2H_4)\) which is non-polar, and the Acetoxy group or vinyl acetate unit (VA) \((C_4H_6O_2)\), which is polar. For different types of EVA the proportion of vinyl acetate usually varies between 10% and 40% (by weight), with the remaining being ethylene. The water permeability of EVA increases steadily with VA content Marais et al. (2002). The VA units are randomly distributed along the EVA chain and their percentage can enhance the elasticity and thermo-plasticity of EVA.
2.1.2 Role of PV in the energy sector

Solar energy is the most abundant type of energy resource on the planet. Amazingly, the amount of energy that reaches the Earth’s surface from the sun in an hour almost equals the energy consumed by all human activities in a whole year. Direct conversion of sunlight into electricity through PV modules is one of the three main solar operating technologies; the two others are concentrating solar power (CSP) and solar thermal collectors used for heating and cooling (SHC). PV is expanding very quickly thanks to effective supporting policies and cost reductions and is expected to contribute 16% of global electricity supply by 2050 [IEA (2014)]. Photovoltaic technology is known for providing clean energy, without any kind of harmful emissions or released polluting gases during operation Battisti and Corrado (2005). Photovoltaic systems play a particular role in supplying electricity in remote regions where no electric grid is available or it would be too costly to extend the grid. The power generation in PV modules is in part, function of the module technology and more importantly at any given moment it is a function of the perpendicular sunlight intensity on its surface. Reduction of power generation takes place if it is cloudy or if the sunlight incident angle becomes large. Generally, the average power generation of a photovoltaic system is possible to be well anticipated on a monthly basis using previously measured meteorological data Lund (2006). PV modules can be considered as a reliable alternative in thousands of villages which are running on diesel generators, which are difficult to maintain as well as expensive to fuel, and after all harmful to the environment. Even in developed countries there are still some communities or enterprises which rely on diesel generators while PV could reduce operational costs and reduce environmental impact. In many areas worldwide, water pumping systems are energized by PV-powered water pumps, replacing internal combustion engines supplying water for different end users ranging from households to industry and livestock. In the developing world, expansion of small industries requires improving indoor lighting and power. There is a growing list of applications of PV that include vaccine refrigeration, electric-vehicle charging, pond aeration, just to name a few NREL (2000). PV technology also contributes to energy source diversification. It can complement other means of energy supply. As for the electricity in particular, PV can suitably provide additional power during peaks of demand, which in many regions take place in the sunniest hours and days especially as a result of air conditioning Kurokawa (2003); PV-TRA (2005).
2.2 Durability problems

For many years, a primary concern in research and development related to photovoltaic systems has been development of cost-effective PV modules with useful lifetimes of 20-years. There are different critical issues in the life cycle of PV modules which are efficiency, durability and cost. The main problem regarding solar energy technologies is not discovering how to collect the radiant flux, but how to collect it in a cost competitive way against conventional power generation. Service lifetime prediction estimation of the photovoltaic device determines life cycle costs. The cost-effective deployment of any PV device is limited by the durability and life cycle cost of the material used Scott (2010). Uncertainties associated with PV module technologies are commonly major obstacles in solar energy deployment.

Significant progress has been made in quality tests and investigation of ageing processes so as to predict failure mechanisms. However, additional research is still needed to achieve necessary product quality towards the long-term goal. The long term reliance on photovoltaic modules is a function of durability in the actual operational conditions in the field and not just the primary performance under standard test conditions, because the initial capital intends to harness nominal energy over 20 years. Research on durability aims to anticipate the long term performance PVs through applying intense conditions on them to reveal possible failure mechanisms, degradation and loss of performance. It is also concerned with the study of inter-related stresses from the weather or climate that may act in complex patterns which are very difficult to replicate through tests.

Within this context one of the major challenges for module durability is corrosion as it affects module output power by raising electrical resistance of the interconnects. Ingress and presence of moisture into encapsulation materials are known for accelerating the impact of corrosion. Degradation of the polymeric encapsulant materials is dependent on the weathering conditions caused mainly by temperature, ultraviolet irradiation and humidity. One way a module can fail in an accelerated fashion is when the optical transmission of its encapsulant drops dramatically. It has been found that damp heat (85°C, 85%RH) as well as UV reduce the optical transmission of EVA and silicones, mostly at wavelengths below 500 [nm] Ohl and Hahn (2008). However, the changes in transmission have not been quantified in terms of a rise in absorption coefficient and a drop in scattering by water molecules McIntosh et al. (2009a).
Moisture related effects on encapsulants

Polymer materials are vulnerable to water diffusion in the presence of either liquid water or water vapour in the surrounding medium. The diffusion and sorption of water (or other vapour molecules) can result in different kinds of damage in such materials. Since the encapsulant plays a very important role in the structure of PV modules, any damage can dramatically influence their performance Sargent and Ashbee (1984). Water vapour is one of the main causes of corrosion and aging within photovoltaic modules MacDonald et al. (2009).

Water ingress into composites can cause loss of strength. When water enters into the polymer matrix, it can decrease the glass transition temperature $T_g$ and consequently can allow molecular motion to happen easily and may raise impact strength. Roughly, absorption of 1% (by weight) water into an epoxy resin matrix can reduce $T_g$ by $10^\circ C$. A decrease of $T_g$ at an interface can relax interfacial stresses and can allow creep of polymer matrix to cause de-wetting or disbanding; or can raise impact strength in resin materials Banks et al. (2000); Smith (1988). Down at the molecular level, water interacts with organic or polymeric molecules through hydrogen bonding Iwamoto et al. (2003).

Generally speaking, moisture adversely influences materials in terms of dimensional stability, mechanical properties, corrosion, chemical stability, thermal properties, handling characteristics, and more Pilkington Microwhite (2012). Increased level of moisture content in polymers leads to loss of molecular weight, loss of adhesion, modifications or degradation of the polymer, polymer fragments leaching, effects on underlying substrates, loss of electrical insulation properties, and higher vulnerability for mechanical failure due to environmental stress cracking. Tensile stress in polymers increase moisture absorption as cracks and defects are opened up letting even more moisture to be absorbed. Increased moisture content can lower the viscosity of polymers by a factor of 100 as its cuts polymer chains, making the material flow more easily Carroll P. A. (2008). Reliable moisture measurement can be a key to innovation in a wide variety of areas. Some photovoltaic devices are sensitive to even low amounts of moisture and the presence of water into a module can lower its performance considerably during the life of module. Quantification of water diffusivity and solubility in encapsulants is crucial to anticipate how long it would take moisture to enter a module. This can be achieved by measuring the transient water vapour transmission rate (WVTR) through the encapsulant layer. In outdoor conditions, the moisture penetration rate and direction is a function of environmental conditions including relative humidity, temperature, air mass movement, as well as of climate cycles. In order to explore the effect of moisture related damage on PV module, a comprehensive study based on existing literature is presented.
in next sections. It is meant to find the best possible approach to measure the amount of moisture in PV module encapsulants. Fig. 2.3 shows some of the moisture related aspects to be looked into which eventually leads to understanding the failure of the panel due to presence of water.

![Figure 2.3: Monitoring the moisture map](image.png)

### 2.2.1 Hygroscopic swelling behaviour

Hygroscopic swelling is the expansion of a material upon moisture absorption. Swelling mismatch between adjacent materials induce additional stresses, so called a hygroscopic stress, which is known to be a major cause of failure in electronic packaging Zhou et al. (2005b). Fig. 2.4 shows resultant warping appearing in the cured EVA after exposure to humidity and temperature (85%RH, 85 °C). Understanding encapsulant behaviour in the presence of water is therefore crucial for any durability and failure predictive models Ardebili et al. (2003); Wong et al. (2002b). Hygroscopic swelling of EVA, the dominant PV encapsulant, is not adequately studied in the existing literature.
Several techniques have been introduced in the past to characterize hygroscopic swelling Ardebili et al. (2003). Amongst those, a widely used approach in electronic packaging materials is employing a Thermo-Mechanical Analyser (TMA) in conjunction with a Thermo-Gravimetrical Analyser (TGA) by finding the dimensional changes of the sample through a contact probe in the TMA while the moisture content is obtained from TGA in a desorption procedure, to finally extract the strain vs. moisture concentration curve Ardebili et al. (2003); Tee et al. (2003); Wong et al. (2002b); Zhou (2006a); Zhou et al. (2005a,b). Alternatively, non-contact approaches include optical methods such as Moiré interferometry, used to measure ‘in-plane’ hygroscopic strain in mold compounds Stellrecht et al. (2003).

The high compliance of EVA renders contact approaches such as TMA-TGA unsuitable. TGA is a destructive test that leads to chemical degradation. Moreover, EVA free standing films (0.3 mm to 1 mm thickness) tend to warp due to water uptake when placed in a humid environment. For this reason, monitoring the ‘in-plane’ dimensions of an initially flat sample is not possible and an alternative approach to Moiré interferometry is required.

### 2.2.2 Optical and dimensional changes

As stated in previous chapter, one of the areas of focus in this thesis is studying dimensional and optical changes as a result of water absorption. It is intended to carry out
this study through high precision optical non-contact system. Optical changes in EVA could, in part, appear as changes in refractive index. The existing literature shows the achievements and challenges in obtaining refractive index and dimensional measurement. Gahn and Witte (1986) presented a method for determining the mechanical and optical thickness, as well as the refractive index of transparent tissue layers in living specimens. They used a microscope to focus on the surface of a thin transparent layer. The physical thickness of the specimen was measured with a high resolution inductive pathfinder, and the optical thickness was calculated with the known stage movement between the two foci and marginal ray analysis. Tearney et al. 1995 Tearney et al. (1995), presented two methods to determine the refractive index of transparent tissue. The first method uses the ability of OCT to measure the optical path length of excised tissue specimens. The second method employs the coherence gating properties of OCT to track the focal position as the sample is moved along the optical axis. A new method later in 2003, for measuring simultaneously the thickness and the refractive index of a transparent plate was proposed by Coppola et al. (2003). The method is based on a simple, variable lateral-shear, wavelength-scanning interferometer, however it is applicable to single transparent layer and does not measure thickness and refractive index change.

Ohlidal et al. (2011) presented an original method for the complete optical characterization of thin films exhibiting area thickness where non-uniformity was presented. This method was based on interpreting experimental data obtained using an original imaging spectroscopic photometer operating in the reflection mode at normal incidence of light on carbon-nitride films Ohlidal et al. (2011). Takashi Fukano and Ichirou Yamaguchi in 1999 Fukano and Yamaguchi (1999), measured the refractive index and the geometrical thickness by using hybrid configuration of a confocal microscope and a wavelength-scanning heterodyne interferometer with a laser diode, the material they used was glass slide, optical parallel (BK7) and (Fused Silica).

### 2.3 Non-destructive measurement of moisture concentration in laminated EVA: a feasibility study

#### 2.3.1 Existing inspection techniques:

##### 2.3.1.1 Raman spectroscopy

Raman spectroscopy takes advantage of inelastic scattering of monochromatic light, commonly from a laser source. In these technique photons are absorbed and then re-
emitted by the sample. The frequency of re-emitted photons is shifted up or down relative to the excitation photon’s frequency, a phenomenon which is called Raman shift which is shown in Fig. 2.5. This shift gives information on vibrational, rotational and other low frequency transitions inside the molecules. The Raman effect is inherently due to molecular deformation in the presence of an electric field \( E \) determined by molecular polarizability.

\[
E_R = h\nu_0 - \Delta E_{vib} \tag{2.1}
\]

where \( E = E_0 \cos(2\pi\nu_0 t) \) represent electromagnetic field which is associated with radiation of frequency \( \nu_0 \) with \( t \) being the time. The intensity of Raman scattering is proportional in the polarization during the vibration as it shown bellow:
2.3 Non-destructive measurement of moisture concentration in laminated EVA: a feasibility study

\[ I_{\text{Raman}} \sim \left( \frac{\alpha}{\pi} \right)^2 \]
\[ I_{\text{Raman}} \sim \left( \frac{1}{3\pi} \right) \] (2.2)

In the above equations \( \alpha \) is molecular polarizability and \( E_R \) denotes energy of inelastically scattered photon (Stokes Raman scattering). Confocal Raman spectroscopy allows point-wise, non-destructive spectral measurements inside the encapsulant in PV modules. Raman spectroscopy was used for analysing the degradation behaviour of encapsulants of cSi based PV modules Peike et al. (2011). Material changes were explored during the ageing process for two types of cSi PV modules with EVA aged indoor under two set of conditions:

1) damp-heat condition with 85%RH and 85°C and 2) combined UV/moisture condition. A lateral non-uniformity of the fluorescence intensity of \( CH \) stretching vibration intensity ratios was noticed, depending on the position above the cell as well as from the ageing condition, which might be an indicator for the diffusion of water in the encapsulant. However, not direct measurement of water concentration in the EVA has been reported with Raman spectroscopy.

**Pilot study**

A Raman spectroscopy system (Horiba LS5 -see Fig. 2.6) was used to analyse free standing EVA films 0.5 mm thick that have been subjected to the following environmental conditions: 1) 85%RH and 85°C inside a climate chamber for 40 days; 2) 5% RH and 23°C inside a desiccator for 40 days. Fig. 2.7 shows a Raman spectrum for the mid-thickness depth which is a plot of the intensity of Raman scattered radiation as a function of its frequency difference from the incident radiation with the units of wavenumbers \([cm^{-1}]\).
Photovoltaic modules: moisture associated mechanical reliability concerns

Figure 2.6: Confocal RAMAN spectroscopy system.

Figure 2.7: Raman shift for hydrated sample (red) and a dehydrated sample (green) 40 days in 85%RH and 85°C. Typical Raman spectra of EVA peaks at 629 (ν(CO)), 1067 (ν(CC)), 1126 (ν(CC)), 1295 (t(CH), δ(CH)), 1438 (δ(CH)), 1724 (ν(CO)), and 2832, 2863, 2880, 2895, and 2913 [cm⁻¹] (ν(CH₂) and ν(CH₃)) Peike et al. (2011, 2012).

The increased fluorescence background in the Raman spectra is interpreted as encapsulant degradation. Since it is correlated to the amount of chromophores formed, the
2.3 Non-destructive measurement of moisture concentration in laminated EVA: a feasibility study

fluorescence intensity could be used to identify ageing process Peike et al. (2011).

A reason for unsuitability of this technique for our intended measurement is that as shown in above pilot study, even though Raman spectroscopy has been used to map EVA degradation in PV modules, the spectra seem to indicate that the 3400 and 3700 [cm$^{-1}$] region is not Raman active (i.e. no peaks are detected) Peike et al. (2011).

2.3.1.2 Nuclear Magnetic Resonance (NMR)

Nuclear Magnetic Resonance (NMR) is a non-destructive technique which can be applied to determine the moisture content of many materials including foodstuffs, agricultural products, powders and polymers. This method determines a through thickness profile of hydrogen content and movement in the material. As the moisture content is proportional to the hydrogen content, the molecular mobility of the hydrogen leads to determination of the binding energy of the water, therefore bounded water and free water can be determined separately. This measurement requires applying a high magnetic field Malm (2008b). Application of techniques such as gravimetric, optical microscopy, X ray or radioactive tracer studies in order to measure the system’s reaction to water ingress in terms of swelling, diffusion, dissolution and segmental mobility inherently involves disadvantages since stopping of the diffusion process or dropping and destruction of the sample are inevitable. NMR imaging is an ideally suitable method to examine dynamic process such as diffusion in a non-invasive fashion through detecting the mobile molecules of solvent matrix systems. Therefore the main advantage of the method is the possibility of extracting the diffusion profile, as well as the concentration and location of the permeating liquid in a solid specimen, to finally determine diffusion coefficients in poly (tetrahydrofurfuryl methacrylate-co-hydroxyethyl methacrylate) Ghi et al. (1997). Using NMR imaging to observe the ingress of solvent into solid systems has been around for almost two decades Webb and Hall (1991). In 1998 NMR was utilized to study the ageing process of rubber filler materials Knorgena et al. (1999). Knorgena et al. (1999) studied the fraction of voids in 0.4 [mm] thick plates of a cresol-novalac molding compound from Plaskon by solid state proton nuclear magnetic resonance imaging (NMR) using the strategy of void filling with water. Information about the distribution of water between the voids and the polymer matrix is also obtained since spectral line width and chemical shift differences distinguish water in these two regions Qian and Liu (1999).

This technique is suitable for quantitative spatially resolved measurement of diffusion in polymers Knorgen et al. (1999), however there are some limitations in using this technique especially with regards to the sample shape and size. The samples should be
cylindrical shape in small size (1 – 2 [mm] diameter by 10 – 15 [mm] long) and there is no chance of laminated sample.

### 2.3.1.3 Permeation tests

The PERMATRAN-W@3/33 (Fig. 2.8) system uses a modulated infrared sensor capable of detecting water vapour transmission with parts-per-million sensitivity, through both flat films and flexible and rigid packages. Testing is conducted over a range of RH conditions as well as temperatures. One side of a film is exposed to water vapor ingress, as water permeates through the sample material, nitrogen sweeps the other side of the film which transports the transmitted water vapour molecules to the infrared sensor to eventually be reported as transmission rate which can be transformed to permeability rate by multiplying by the average thickness of the material.

![Figure 2.8: PERMATRAN-W Model 3/33 Plus Macon (2014)](image)

In a recent work, WVTR and adhesion properties of EVA and a replacement for EVA were measured in a range of temperatures using a Mocon Permatran-W 3/31 device. WVTR at test conditions up to 85°C and 100% RH as well as adhesion values were measured before and after UV exposure and damp heat at 85%RH, 85°C Jorgensen et al. (2003).
2.3.1.4 Fourier Transform Infrared spectroscopy (FTIR)

Infrared spectroscopy is undoubtedly one of the most valuable analytical techniques available to today’s scientists. A molecule needs to possess a specific feature to show infrared absorption, i.e. the molecule electric dipole moment must change due to molecular vibration. This is the selection rule behind infrared spectroscopy Stuart (2005), which is known as a powerful technique to study interactions. In the case of water detection, the frequency and shape of the OH stretching absorption of H₂O are very sensitive to interaction (with the polymer) Iwamoto et al. (2003). The study of water interactions in polymers has long been subject of scientist’s attention using various methods. Recently, many scientists have found IR as a valuable approach to investigate intermolecular interaction of water in polymers Bashir et al. (1994). In 1997 FTIR was used to identify the internal reflection mode that enables gaining near infrared data in solid or liquid Linossier et al. (1997). In that study for Polystyrene and Poly (methyl methacrylate) films, one of the non-destructive spectroscopic test methods was applied to measure small water molecules diffusivity for a polymeric coating. This study addressed the comparability of FTIR versus gravimetric method for measuring water or diffusion of solvent in those polymeric films. It was observed that one of the advantages of FTIR is to avoid the difficulty of gravimetric technique in handling and measuring precise mass gain for thin films of less than 10 µm. Secondly, it allows measuring the uptake of liquid in polymeric films with high diffusion coefficients, whereas in gravimetric tests a significant amount of penetrant may desorb when the sample is removed from the solution. In a recent work in 2009, FTIR was used to measure moisture ingress through encapsulant materials (EVA, PVB) from the glass edge and towards the module centre after damp heat exposure. The moisture level determination was performed in the encapsulants by integrating the IR spectrum from 1880 – 1990 [nm] (in case of PVB (Poly Vinyl Butyral)). Weathering parameters consisted in 85°C and 85% RH during 5000 hours on 20 × 20 [cm] glass laminates Kapur et al. (2009). In another research Pelisset (2009) FTIR was applied to identify the moisture content of encapsulants which was PVB. The measurements were fitted to a Fickian diffusion model with good compliance. The polymer characteristic peaks lie between 6000 and 5300 [cm⁻¹] and water peaks appeared within 5300 and 5000 cm⁻¹. Swonke and Auer (2009) used gravimetry and FTIR for several encapsulant polymers so as to determine the WVTR. The wavelength range for FTIR test was between 1.7 µm to 2.9 µm. The test was carried out on thermoplastic polyurethane (TPU) as well as other encapsulant polymers including PVB, fast-cure ethylene vinyl acetate (EVA fc), ION and TSI Swonke and Auer (2009). In 2005 FTIR was reported to be applied in Poly (ethylene-Co-Vinyl Acetate) in order to find the
interaction of water on various Poly (vinyl Acetate) of 3, 5, 10, 11, 14 and 18% (VA). Film samples were hydrated by immersion in water for 30 to 60 minutes Iwamoto and Matsuda (2005). Fig. 2.9 (a) shows the result of FTIR for one type of encapsulant polymer, TPU in the near infrared range. The absorption bands of water are indicated in the figure. Glass-encapsulant-glass plates were used as shown in Fig. 2.9 (b), which had been exposed to different temperatures and high air humidity in a climatic chamber for accelerated ageing.

![Figure 2.9: (a) Normalized infrared absorption characteristics of TPU for different exposure times at 85°C, 85%RH. (b) Glass/TPU/Glass laminates Swonke and Auer (2009).](image)

**Summary**

Table 2.1 summarizes moisture related properties ($C/\text{m}^3$, $D/\text{m}^2$) in the literature reported for EVA to the best of the author’s knowledge. It emphasizes on the significant features of those research such as measurement techniques, testing conditions and sample layout.

There are limitations for each measurement method in identifying water concentration in EVA inside PV modules. The above mentioned techniques cannot be applied to measure water content in PV modules, or more generally, there is currently no instrumental method available commercially for measurement of low moisture permeation through polymer substrate Hulsmann et al. (2009). Since the encapsulant is usually between a glass front sheet and a back sheet made of glass or multi-layered films and we need to find the method which is most suitable for encapsulant material like EVA; NMR is not suitable for laminated samples, and gravimetry involves difficulties imposed due
2.3 Non-destructive measurement of moisture concentration in laminated EVA: a feasibility study

to handling the sample and more importantly incapable of distinguishing the moisture diffusion in layers. Nonetheless Mocon devices were used for identifying moisture diffusion, it is not suitable for high barrier films with water vapour and gas transmission rate below $10^{-6}[kg/(m^2d)]$ Kumar et al. (2002). It was finally concluded that FTIR seems to offer the best chances to develop a system that could measure water concentration through the front glass, if combined with depth-encoding of the spectral signal.
Table 2.1: Summary of moisture parameters in EVA related literature

<table>
<thead>
<tr>
<th>Material</th>
<th>Technique</th>
<th>Time [h]</th>
<th>%RH</th>
<th>T °C</th>
<th>C_sat [kg/m²]</th>
<th>D [m²/s]</th>
<th>Other Remarks</th>
<th>Ref</th>
</tr>
</thead>
<tbody>
<tr>
<td>EVA</td>
<td>Gravimetric</td>
<td>21</td>
<td>2.5</td>
<td>1.2 e - 10</td>
<td>8 e - 11</td>
<td>cured type: fast cure</td>
<td>Kohl (2010)</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>40</td>
<td>@ 40°C</td>
<td>3.2 e - 10</td>
<td>8 e - 10</td>
<td>Laminate EVA with 3TPT layer-back sheet (Tedlar/ PET/ Tedlar)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>60</td>
<td>630 min</td>
<td>8 e - 10</td>
<td>8 e - 10</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>EVA</td>
<td>Innovative setup with mass spectrometer and climate chamber</td>
<td>21</td>
<td>4.2 e - 11</td>
<td>4.2 e - 11</td>
<td>4.2 e - 11</td>
<td>Hulsmann et al. (2009)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>EVA</td>
<td>Mocon Permatron</td>
<td>23</td>
<td>1.6</td>
<td>8.5 e - 11</td>
<td>8.5 e - 11</td>
<td>film diameter:12 cm</td>
<td>Kempe (2006)</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>55</td>
<td>@ 27°C</td>
<td>6.14 e - 11</td>
<td>6.14 e - 11</td>
<td>EVA1h: 0.46 to 2.84 mm</td>
<td></td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>97</td>
<td>71%RH</td>
<td>8.07 e - 10</td>
<td>8.07 e - 10</td>
<td>67%wt Etylen, 33%wt VA</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>6</td>
<td>@ 85°C, 85%RH</td>
<td>85%RH</td>
<td>85%RH</td>
<td>Fickian diffusion model</td>
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<td></td>
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<td>EVA</td>
<td>Moisture sensors</td>
<td>2000</td>
<td>85</td>
<td>65</td>
<td>2.4 e - 10</td>
<td>2.4 e - 10</td>
<td>glass/EVA/glass</td>
<td>Mon et al. (1988)</td>
</tr>
<tr>
<td></td>
<td>Permeation test</td>
<td>22</td>
<td>9.2 e - 12</td>
<td>9.2 e - 12</td>
<td>9.2 e - 12</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>40</td>
<td>2 e - 12</td>
<td>2 e - 12</td>
<td>2 e - 12</td>
<td></td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>50</td>
<td>3.2 e - 12</td>
<td>3.2 e - 12</td>
<td>3.2 e - 12</td>
<td>Hulsmann et al. (2009)</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>60</td>
<td>4 e - 12</td>
<td>4 e - 12</td>
<td>4 e - 12</td>
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<td>80</td>
<td>1.4 e - 11</td>
<td>1.4 e - 11</td>
<td>1.4 e - 11</td>
<td></td>
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<tr>
<td>EVA</td>
<td></td>
<td>25</td>
<td>6 e - 12</td>
<td>6 e - 12</td>
<td>6 e - 12</td>
<td>EVA with different VA% (19wt%-70wt%), Kempe (2005)</td>
<td></td>
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</tbody>
</table>
Near infrared absorption of water

Near infrared absorption of water has been the main subject of several previous investigations since the early 1900s. Notably in the work reported by Hulburt and Hutchinson (1917) the water absorption data covered the wavelength from 0.704 to 1.344 micrometers. Later on, Collins (1925) published data for water absorption in the wavelength range between 0.70 to 2.10 micrometers. He focused on the shift in position of absorption bands, resulting from water temperature change, rather than the absorption coefficient. In Collins’ measurements in the range between 0.70 to 2.50 [µm], there are 5 prominent water absorption bands in near infrared which occur at 0.76, 0.97, 1.19, 1.45 and 1.94 [µm] and one weaker band at 0.845 [µm]. It was observed when water temperature rises to 95 °C the intensity of bands enhances. Collins also found that increasing the temperature of water shifts the position of maximum absorption to slightly shorter wavelength. Fig. 2.10 is a re-plot of extracted points out of published data from Collins’ measurements, represented as single curve, excluding the overlapping range. The intention behind replicating this graph is to get a clearer overlook of the water absorption peaks in near infrared range.

Properties of water in EVA

Polymers in general are vulnerable to water diffusion in the presence of either liquid water or water vapour in the surrounding medium. Within the EVA polymer matrix, water can exist either as free or bonded water. Bonded water can be of two types: one-bonded
and two-bonded water. In the case of one-bonded water, one \(OH\) is hydrogen-bonded to the a \(C = O\) ester group and the other \(OH\) is free Iwamoto and Matsuda (2005). In two-bonded water, each \(OH\) is hydrogen-bonded to one \(C = O\), -see Fig. 2.11.

In the case of two-bonded water, each \(OH\) is hydrogen bonded to \(C = O\). While free water penetrates into micro cavities within the polymer structure and moves through it Maffezzoli et al. (1993), bonded water makes up strong molecular interactions with the matrix; it is somehow ‘locked’ and thus constrained to move. The diffusion and sorption of water could result in different kinds of damage in such materials. In the case of EVA, the presence of water changes the level of vinyl acetate as it reacts with the acetate group to produce acetic acid, which in turn diffuses out of the matrix. This results in a gradual change in the chemical formulation and molecular weight of EVA.

2.3.1.5 Possibility of using FTIR to detect water inside encapsulant materials

Numerous attempts have been made to estimate water concentration in EVA using gravimetric methods Kohl (2010), water vapour transmission rate measurements Bashir et al. (1994); Carlsson et al. (2006a,b); Carroll P. A. (2008); Kempe (2005, 2006); Kohl (2010); Maffezzoli et al. (1993); Malm (2008a); Swonke and Auer (2009) and embedded moisture sensors Carlsson et al. (2006a), to name a few. Iwamoto and Matsuda Iwamoto and Matsuda (2005) studied the presence of free and bonded water in EVA.
2.3 Non-destructive measurement of moisture concentration in laminated EVA: a feasibility study

Figure 2.12: OH stretching absorption of water in EVA Iwamoto and Matsuda (2005).

copolymer co-polymers with different proportions of vinyl acetate, using FTIR. By analysing the spectral change in very specific spectral bands between 3400 and 3700 [cm\(^{-1}\)], they found that one- and two-bonded water coexists in the co-polymer and that two-bonded water increases in relative proportion with increasing vinyl acetate composition. TPU showed clear absorption bands at 1.9[\(\mu m\)] and 2.7[\(\mu m\)] (5263 [cm\(^{-1}\)] and 3703 [cm\(^{-1}\)], respectively), corresponding to water vapour absorption, indicating that TPU mainly hosts free water that can move through its matrix. Water concentration was obtained from the absorbance data using the wavelength dependent extinction. Furthermore, they found that ION and TSI store practically no free water as no changes were observed in the absorbance at the water vapour absorption bands. No results were reported for EVA nor was the presence of bonded water addressed in their study. The frequency and shape of the \(OH\) stretching absorption of water are very sensitive to interaction with the polymer Iwamoto and Matsuda (2005). Iwamoto and Matsuda (2005) identified four bands that are linked to absorption of different vibrational modes of \(OH\) groups, as shown in Fig. 2.12.

The A band, with a high frequency around 3688 [cm\(^{-1}\)], is assigned to the stretching mode of a free \(OH\) group of one-bonded water \(v_f(OH)\) - see Fig. 2.13. The B band, at around 3640 [cm\(^{-1}\)] and significantly broader than the A band, is assigned to the anti-symmetric \(OH\) stretching mode \(v_a(OH)\) of two-bonded water. The C band seems to be a combination of two vibrations that appear at the same frequency: a) the \(OH\)-stretching band of the bonded \(OH\), \(v_b(OH)\), of one-bonded water and b) the symmetric \(OH\)-stretching band, \(v_s(OH)\), of two-bonded water. The frequency and the relative intensity of the C band do not apparently depend on the VA composition. The B and C
bands that correspond to a bonded OH group of one-bonded or two-bonded water are significantly larger in bandwidth than the A band of a free OH, probably a result of the statistical spread of the distances between water molecules and the VA groups. Within the VA ester group, the hydrogen bonding site is either –O– or C = O. The spectrum shows a band at about 3440 [cm$^{-1}$], the so called D band, which is lower in frequency to the C = O stretching frequency (3457 [cm$^{-1}$]) of an ester group of VA. Bonded water has the effect of a downshift of the C = O frequency by about 16 to 20 [cm$^{-1}$]. They concluded that the D band is linked to hydrogen bonding of the dissolved water to a C = O group instead of the –O– site and was thus assigned to the hydrated C = O group, which suggests that the intensity of the D band should be proportional to the amount of the dissolved water, which is roughly proportional to the intensity of the C band. They also concluded that the ratio of the peak-height of the D band to that of the C band should be a constant independent of VA composition. Interestingly, they also found that the hydrated fraction of C = O was around 2% for 18% VA composition by weight. This is linked to the relative distances between C = O groups in the polymer matrix. In summary, their study suggests that water dissolved in EVA copolymers is not interstitially situated in the matrix but instead it is hydrogen-bonded to one or two C = O groups depending on their distribution density in the matrix.
2.3 Non-destructive measurement of moisture concentration in laminated EVA: a feasibility study

Figure 2.14: Shimadzu FTIR 8400s, EVA free standing film on the sample holder of the instrument.

Pilot study

Following the work by Iwamoto and Matsuda (2005), absorbance on EVA free standing films was measured subjected to different conditions. The films were manufactured in a PV module laminator with 33% VA composition, 80% gel content, and were 0.5 mm thick. The absorption was measured by using a Shimadzu FTIR 8400s on free stand film of EVA. Fig. 2.14 shows the FTIR machine and how the samples are placed inside for the measurement.

Fig. 2.15 shows that the A, B, C and D bands for different conditions. The ratio between the measured absorbance spectra and the absorbance value at 2679 [cm$^{-1}$] (which corresponds to an Ethylene group in the polymer matrix and is thus independent of water content) is shown in Fig. 2.15. EVA samples were kept at 60% RH at 20°C during 10 days. After that, one sample was kept in an oven at 85°C for 72 hours and another was immersed in liquid water at room temperature for 96 hours. The spectra show a D band whose absorbance changes very little with hydration and desorption. The sample kept in the oven shows a reduction in the absorbance in all bands, indicating loss of water. It is interesting to note that the absorbance of the C band is reduced more than that of the B band. This is consistent with an initial loss of one-bonded water followed by a loss of two-bonded water. The B band only refers to two-bonded water. The absorbance of the sharp A band, also associated to one-bonded water, was also reduced. Iwamoto et al. (2003). The results for the hydrated sample show water absorption in all bands, but more in the B band, indicating a dominant presence of two-bonded water.
Figure 2.15: OH stretching absorptions of the water contained in EVA with 33% VA composition and 80% gel content using transmission FTIR. The A band is associated to free OH stretching mode; the B band to the antisymmetric OH stretching in two-bonded water; the C band to the symmetric OH stretching of two-bonded water and the stretching of the bonded OH in one-bonded water; and the D band to the hydrated carbonyl group in the vinyl acetate Rashtchi et al. (2012).

With regards to challenges associated with EVA in detection of water in PV modules: 1) as there is virtually no interstitial free water, the strong absorption bands at 1.9 and 2.7 [$\mu m$] cannot be relied upon. Instead, the A, B, C and D bands between 3400 and 3700 [cm$^{-1}$], with differential absorbance levels below ~1.0 between the dry and hydrated states seem to be the best indicators of the presence and concentration of water;

2) a transmission configuration is not always possible, as only a small fraction of PV modules have glass front and back-sheets. The great majority uses Tedlar® back-sheets, a multilayer polyvinyl fluoride (PVF) film. The c-Si cells and the electrodes constitute extra obstacles for light transmission through the panel; 3) reflection FTIR could provide a viable mean to detect the required spectral absorbance but it would suffer from lower signal levels; 4) ageing of EVA will change VA composition and will add extra products to the matrix, further complicating matters.

2.4 Concluding remarks

In this Chapter, a summary of durability concerns of photovoltaic module’s were discussed. As a main problematic one, which is ingress of water in PV modules, was chosen
for more investigation. A summary of available literature for measurement of water in encapsulant materials was presented in a table that includes the different methods to measure water used for different encapsulant material in PV modules, the diffusion coefficient, concentration after ageing and time required for measurements.

In this thesis, a novel non contact (optical) method of measuring the Hygro-mechanical effect of water ingress in EVA is proposed, where thickness and refractive index change of EVA are determined in dry and hydrated conditions. This is achieved by simultaneously measuring the interference intensity signal produced for different environmental conditions during a wavelength scan. In the next chapter the working principle of WSI is described and some design considerations are discussed to build the simulated WSI system for a designed sample and measurement conditions, which can be used to determine the surface displacement and refractive index change of the material after absorbing water.
Chapter 3

Non-contact measurement of the swelling behaviour of EVA using Wavelength Scanning Interferometry: feasibility study and numerical simulation

3.1 Introduction

In order to design a reliable electronic package like a PV module, it is important to understand the mechanical response of associated materials and interfaces to environmental conditions. One of the reliability concerns associated with moisture in encapsulating packaging is swelling, and there are failure modes in these devices which are believed to have roots in diffusion of moisture during operation Ardebili et al. (2003).

Generally speaking the measurement of transparent plates and films thickness is very important for quality control purposes in industry. Several optical techniques, such as low-coherence interferometry (LCI) Danielson and Boisrobert (1991); Tsuruta and Ichihara (1975), and confocal microscopy Sheppard et al. (1994) are used. These techniques, however, deliver not the geometrical thickness but the optical thickness, which is equal to the product of the geometrical thickness and the refractive index. It is highly desirable to have a single measurement technique to obtain both refractive index and thickness, and efforts have been made to find valid approaches to measuring
Non-contact measurement of the swelling behaviour of EVA using Wavelength Scanning Interferometry: feasibility study and numerical simulation

refractive index $n$ and thickness of material simultaneously or separately or at least by using the same apparatus Coppola et al. (2003).

In this chapter, a non-contact method based on Wavelength Scanning Interferometry (WSI) is proposed to determine thickness, refractive index, thickness change and refractive index change in EVA due to water uptake. The approach proposed in the thesis separates optical path changes into refractive index and thickness changes.

The novel aspects of using WSI explored in this work are:

1. Time resolved WSI.
2. Measurement of thickness and refractive index and their changes.
3. Numerical simulation to help for designing the experimental set up details, such as thickness of EVA layer, maximum surface velocity in measurement, type of filter to use for data analysis of results and also validating the approach.

The system uses a tunable near-infrared laser to illuminate a layer of EVA laminated onto a glass substrate. The interference signal between light reflected at different interfaces of laminated EVA on glass and a reference beam is modulated with temporal frequencies proportional to the optical path difference between them. Fourier transformation of the interference signal leads to peaks that locate the position of the interfaces. The refractive index and thickness of the EVA layer can be determined and tracked as a function of time by comparing the distances between pairs of peaks in the Fourier spectrum.

This chapter provides the mathematical framework of the technique and a simulation case study to probe its feasibility for measuring hygroscopic expansion coefficient of EVA.

3.2 WSI principle and theory

Wavelength scanning interferometry, also known as wavelength-tuning interferometry (WTI), frequency scanning interferometry (FSI) or swept-source OCT (SS-OCT), is now an established optical technique used in profilometry and tomography. WSI roots back to the emergence of tunable laser sources and has found a wide range of applications in the area of phase shifting interferometry. WSI is an effective technique for measuring engineered and structured surfaces. Previous research highlighted the application of this technique for measuring transparent surfaces like film layers of Parylene on glass slide surface, crystalline silicon Si and a uniaxial crystal of lithium niobate LiNbO$_3$ Coppola et al. (2003); de Groot (2000); Fukano and Yamaguchi (1999); Gao et al. (2011).
Improvements in tunable lasers helped WSI to gain popularity as an imaging tool over the last couple of decades.

In mid-1990s, parallel versions of WSI were developed for profilometry applications Kuwamura and Yamaguchi (1997); Takeda and Yamamoto (1994), in which each pixel in a 2-D array is tuned into an independent range sensor. de Groot (2000) described a wavelength scanning interferometer able to separate the interferograms from two different optical surfaces on a common optical path. Low-coherence interferometry (LCI) has been used to simultaneously measure the refractive index and thickness of transparent plates but not their changes as a function of time Fukano and Yamaguchi (1999); Maruyama et al. (2002).

Requiring a long measurement time for scanning the sample as well as the reference mirror is one of the drawbacks of this technique when integrated into a confocal microscope Fukano and Yamaguchi (1999). By using WSI, the optical path difference (OPD) could be directly evaluated by measuring the resultant interferometric phase shift, which is proportional to the OPD and is caused by a change in the wavelength of a continuously tuned laser. WSI can provide absolute distance measurements that exceeds the optical wavelength without any mechanical moving components.

WSI seems a good candidate to measure EVA response to water uptake. In this section, the principal concepts behind WSI are described, and a feasibility study based on numerical simulations is performed.

Consider a Michelson interferometer setup as shown in Fig. 3.1, comprised of the following components: a tunable laser source, a beam splitter, reference and sample arms and finally the detector. The zero delay line (ZDL) represents the apparent position of the reference mirror along the sample arm.

The mathematical description of WSI presented here has been derived from previous works by Ruiz et al. Chakraborty and Ruiz (2012); Huntley and Ruiz (2011); Ruiz et al. (2004, 2005). Consider the layout shown schematically in Fig. 3.1. At $z = 0$, due to the same path lengths of reference and sample arms from BS there is a zero phase difference. Assuming that the sample is modelled as a set of $N_s$ discrete thin scattering layers parallel to its surface, and illuminated by a beam of wavelength $\lambda$ which varies with time and is partially reflected from different layers within the sample. Considering that multiple reflections are neglected within the layers and that light is imaged onto a pixel of a photo-detector array with indices $(m, n)$, the intensity as seen at the detector due to the interference of the sample and the reference wavefronts can be written as:
Non-contact measurement of the swelling behaviour of EVA using Wavelength Scanning Interferometry: feasibility study and numerical simulation

Figure 3.1: Michelson interferometer - A schematic diagram of WSI, showing tunable laser (TL); sample (S); reference mirror (RM); beam splitter (BS) and photo detector (PD);

\[ I(m, n, t) = \left| A_{ref}(m, n, t) + \sum_{j=1}^{N_S} A_j(m, n, t) \exp [i\phi_j(m, n, t)] \right|^2 \]  

(3.1)

Where \( A_{ref} \) represents the amplitude of the reference wavefront and \( A_j (j=1,2,\ldots,N_S) \) is the amplitude of the wavefront reflected at the \( j^{th} \) interface in the object and \( i \) is the complex number \( (i = \sqrt{-1}) \). \( \phi_j \) represent the phase difference between light reflected at the \( j^{th} \) interface and the reference beam. The spatial indices \( m \) and \( n \) on the detector are \( m = 0, 1, 2, \ldots, M - 1 \); \( n = 0, 1, 2, \ldots, N - 1 \) and \( t \) is a non-dimensional time defined as the true time divided by the camera interframe time. Expanding Eq. 3.1 and dropping the \((m, n, t)\) dependence in all the amplitude and phase variables in right hand side of the equation for clarity gives

\[ I(m, n, t) = A_{ref}^2 + \sum_{j=1}^{N_S} A_j^2 + 2 \sum_{j=1}^{N_S-1} \sum_{l=j+1}^{N_S} A_j A_l \cos(\phi_j - \phi_l) + 2A_{ref} \sum_{j=1}^{N_S} A_j \cos \phi_j \]  

(3.2)

The right hand side of Eq. 3.2 consists of four terms. The first two correspond to dc terms and term 3 represents interference between the light reflected back from the \( j^{th} \) and the \( l^{th} \) layers. Together, terms 2 and 3 are referred as the autocorrelation terms (AC). Term 4 represents the interference between the reflected light from each of the
different $j^{th}$ interfaces and the reference beam, and is referred to as the cross correlation term (CC). This last term contains the information about the structure of the sample. The phase $\phi_j$ can be represented as

$$\phi_j(m, n, t) = \phi_{xj}(m, n, t) + k(t)\Lambda_j(m, n) \tag{3.3}$$

Where $\phi_{xj}$ is a phase change induced upon reflections of the sample at the $j^{th}$ interface contributing to the amplitude at pixel $(m, n)$. $k(t)$ is the wavenumber $2\pi/\lambda(t)$ and $\Lambda_j$ is the optical path difference between the wavefront reflected at the $j^{th}$ interface and the reference beam. Wavelength scanning interferometry involves changing $k$ with time over a total range $\Delta k$, while image sequences are recorded using the 2-D photo-detector array.

$$k(t) = k_c + \delta kt \tag{3.4}$$

Where $k_c$ is the central wavenumber, $\delta k$ is the increment in wavenumber between successive frames and $t$ changes from $-\Delta k/2\delta k$ to $+\Delta k/2\delta k$. Since the angular frequency is defined as the rate of change of phase, it can be defined as:

$$\omega = \frac{d\phi}{dt} = 2\pi f. \tag{3.5}$$

Substitution of Eq. 3.4 into Eq. 3.3 leads to the result:

$$\phi(m, n, t) = k_c\Lambda(m, n) + \phi_{s}(m, n) + \delta k\Lambda(m, n)t \tag{3.6}$$

Two important consequences follow from Eq. 3.6. First, the linear variation of $\phi_j(m, n, t)$ with $t$ leads to an intensity modulation frequency $f_{\Lambda j}(m, n)$ in units of “cycles per frame” which is directly proportional to the optical path difference between the wavefront reflected from the $j^{th}$ interface and the reference wavefront:

$$f_{\Lambda j}(m, n) = \delta k\Lambda_j(m, n)/2\pi \tag{3.7}$$

An alternative formula for frequency can be defined in units of cycles per scan duration:
Non-contact measurement of the swelling behaviour of EVA using Wavelength
Scanning Interferometry: feasibility study and numerical simulation

\[ \hat{f}_{\Lambda_j}(m, n) = f_{\Lambda_j} N_t = \Delta k \Lambda_j(m, n)/2\pi \]  

(3.8)

Where \( N_t \) is number of frames. Measuring \( f_{\Lambda_j} \) or \( \hat{f}_{\Lambda_j} \) thus provides a measure of \( \Lambda_j(m, n) \).

Second, if two scans are performed before and after a deformation of the sample, then provided \( \phi_{sj} \) remains the same between scans, the phase change \( \Delta \phi_j(m, n, 0) \) provides a direct measure of the change in optical path lengths \( \Delta \lambda_j(m, n) \) due to the deformation.

The Fourier transform of a set of \( N_t \) sampled intensity values \( I(t) \) can be written:

\[ \hat{I}(\hat{f}) = \int_{-\infty}^{\infty} I(t) W(t) \left[ \sum_{i=\infty}^{\infty} \delta(t - \tilde{t}) \right] \exp(-2\pi i \hat{f} \tilde{t}/N_t) d\tilde{t} \]  

(3.9)

where \( \delta \) is the Dirac delta function, \( \tilde{t} \) is a continuous version of the discrete non-dimensional time variable \( t \), \( W(\hat{f}) \) is a continuous window function assumed to be even and nonzero only for \( \|\tilde{t}\| \leq N_t/2 \), and \( \hat{f} \) is a (continuous) non-dimensional temporal frequency.

The Fourier transform of the interference intensity signal \( I(m, n, t) \) in Eq. 3.2 can be written as an infinite sum over an integer index that locates identical spectra \( N_t \) samples apart in the frequency domain Huntley and Ruiz (2011). The goal of the measurement is to reconstruct the amplitude \( A_j(m, n) \) and the distributions of phase \( \phi_j(m, n) \) of the scattered light field from each of the \( N_s \) scattering layers. The Fourier transformed data of the intensity in Eq. 3.2 may be written as:

\[ \tilde{I}(\hat{f}) = \bar{W}(\hat{f}) * \left\{ \begin{array}{c} (I_0 + \sum_{j=1}^{N_s} I_j) \delta(\hat{f}) + \sum_{j=1}^{N_s} \sqrt{I_0 I_j} \exp(\pm i \phi_{0j}) \delta(\hat{f} \mp \hat{f}_{\Lambda_j}) \\ N_s - 1 \quad N_s \\ + \sum_{j=1}^{N_s} \sum_{l=j+1}^{N_s} \sqrt{I_j I_l} \exp(\pm i (\phi_{0j} - \phi_{0l})) \delta(\hat{f} \mp (\hat{f}_{\Lambda_j} - \hat{f}_{\Lambda_l})) \end{array} \right\} \]  

(3.10)

in this case * represents convolution, and \( \bar{W}(\hat{f}) \) is the Fourier transform of \( W(t) \), a continuous window function that represents the finite sampling duration and the envelope of the laser power spectrum. The first term between curly brackets in Eq. 3.10 is part of the autocorrelation term and represents the DC term due to the reference
and scattered wave-fronts. The second, known as the cross-correlation term, represents the interference between the reference beam and the illumination beam scattered at all layers within the sample and is given by the superposition of Dirac deltas $\delta$ that fall within bands in the frequency domain between frequencies $\hat{f}_{Ap1}$ and $\hat{f}_{ApN}$, which are, according to Eq. 3.7 proportional to the optical paths $\Lambda_{p1}$ and $\Lambda_{pN}$ corresponding to the first and last layers. The phase at the origin of each of these frequency components is given by $\phi_{0j} = \phi_{j}(m, n, 0)$. The third term, again known as autocorrelation, represents the interference between pairs of scattering layers due to the same illumination beam. It is represented by Dirac deltas at frequencies $\pm(\hat{f}_{ Aj} - \hat{f}_{Al})$. Before the application of the Fourier transform, the mean value of the intensity signal is subtracted and the signal is multiplied by a Hanning window. The peaks are due to the interference between the light beams reflected from different $j^{th}$ slices and the reference beam.

### 3.3 Numerical simulation

In this section, a numerical simulation is performed to establish the right data processing protocol and to identify optimum experimental parameters required. A 2-D cross-section of a simulated sample is presented in Fig. 3.2. Fig. 3.2(a) show the overall sample outlook: a layer of EVA is laminated on the surface of a glass substrate coated as a partial mirror. This mirror is meant to enhance the reflection of light at the interface between EVA and substrate. The air-EVA and EVA-glass interfaces are labelled '1' and '2' respectively, and the air-glass interface is labelled '0'. Fig. 3.2(b) shows the optical path distance (OPD) including the ZDL, $OPD_0$, $OPD_1$ and $OPD_2$. In this arrangement the sample is situated in an environmental chamber with a window glass through which the sample is illuminated.

The purpose of this simulation is to incorporate all influential parameters including geometry of the sample, amplitudes, phase, intensity of the WSI test within a comprehensive model that describes the interference signal. This is followed by imposing a hypothetical thickness change in the EVA layer as a function of time, as it would be the case due to moisture ingress. This key input to the numerical simulation along with a known tuning of the laser would result in an intensity signal variation. Following the calculation of the expected intensity signal, which takes place in a forward model, an inverse model is then used to analyse the intensity signal to evaluate the EVA thickness.
Non-contact measurement of the swelling behaviour of EVA using Wavelength Scanning Interferometry: feasibility study and numerical simulation

Figure 3.2: (a) Sample geometry with amplitude reflected from each interfaces, (b) Optical path length for different layers of sample

change. This approach enables us to check the consistency of the forward and inverse models. From known sample parameters (dimensions and refractive index) and temporal changes, the forward model estimates the signal that would be measured by a WSI system. This in turn is used to recover the known parameters with the inverse model.

The forward model that generates the intensity is schematically shown in Fig. 3.3. In this model the main preliminary data are as below:

- geometrical parameters including $z_0$, $z_1$, $th_{\text{EVA}}$ and the imposed changes in $th_{\text{EVA}}$
- optical properties, $n_{\text{air}}$, $n_{\text{EVA}}$ and $n_{\text{water}}$
- wavenumber changes linearly with time.

The aforementioned parameters are used to calculate the amplitude and phase of wavefronts reflected at each interface, which lead to the interference intensity signal. The intensity is indeed associated with changes in the refractive index of the medium and the refractive index of EVA due to swelling. The sample is assumed to be immersed in either dry air or liquid water. Choosing water as the medium removes the need to
3.3 Numerical simulation

add a controlled humidity system in the measurement chamber and at the same time permeates relatively fast through the material to cause swelling.

![Forward model diagram]

Figure 3.3: Forward model.

In the inverse part of the simulation, the temporal variation in the EVA thickness is sought through evaluating phase change at different spectral peaks by using specific carrier frequencies (using Kreis FFT method of phase recovery Kreis (1986)). The analysis employs two sets of intensity signals, intensity in dry air and intensity in liquid water. The intensity signal for dry air, acts as reference, enabling us to characterize the phase change when the thickness changes. This dry condition is generated simply by tuning the laser while $th_{EVA}$ remains constant. Fig. 3.4 shows a block diagram of the inverse model.

The phase vs. wavenumber detected in the dry condition would be linear, while in
Figure 3.4: Inverse model.
the hydrated condition, the thickness change due to swelling will show up as a deviation of the phase from that linear behaviour. The difference between the retrieved phase for the hydrated condition and the phase for the dry condition, leads to the isolation of the effect of hydration in the phase change.

The inverse model consists of the following steps:

• Apply a fast Fourier Transform (FFT) to interference signal $I(t)$ to compute the amplitude $\tilde{I}(f_k)$ and phase (approach 1);
• Apply filter in the spectral domain for peaks 0, 1 and 2;
• Apply inverse FFT to evaluate wrapped phase $\phi_{wrapped}$;
• Phase unwrapping to calculate unwrapped phase and OPDs (approach 2);
• Calculate $th_{EVA}(t)$ and $n_{EVA}(t)$

In addition to the above, the initial dry condition is used to estimate $z_0$ and $z_1$ (illustrated in Fig. 3.2) both required in the hydrated condition to obtain $th_{EVA}(t)$.

3.3.1 Forward model

From Fig. 3.2:

\[ z_1 = z_0 - th_{EVA} \quad (3.11) \]

The optical path difference in any layer is equal to the product of the geometrical path difference times the refractive index of the layer. If the incident ray is perpendicular to the surface, the OPDs are:

\[ \Lambda_0 = 2n_{air} z_0 \quad (3.12) \]

\[ \Lambda_1 = 2n_{air} z_1 \quad (3.13) \]

\[ \Lambda_2 = \Lambda_1 + 2n_{EVA} th_{EVA} \quad (3.14) \]
Considering the wavefront reflected at the front face of the window glass as a reference, the phase terms as a function of OPD can be formulated as:

\[ \phi_0 = k\Lambda_0 \]  
\[ \phi_1 = k\Lambda_1 \]  
\[ \phi_2 = k\Lambda_2 \]

where \( k \) represents wavenumber in \((rad.m^{-1})\).

The amplitude of the reference and object waves (front and back surface of EVA 1, 2) are denoted by \( A_{\text{ref}} \), \( A_1 \) and \( A_2 \) respectively. In Fig. 3.2 (a) all amplitude reflection from each interface are shown with different arrows. Transmission \((\bar{t} = 1 - r)\) in these equations refers to transmission coefficient at each interface, \( \mu \) represents the absorption coefficient, and \( r \) the reflection coefficient which is function of refractive indices -see Subsection 7.4.1. Then the amplitude coefficients are:

\[ A_1 = A_{\text{ref}} r_1 \]
3.3 Numerical simulation

\[ A_2 = A_{ref} t_1^2 \quad (3.19) \]

\[ A_0 = A_{ref} \quad (3.20) \]

The amplitude vectors associated to the reflected light from each interface can be written as follows:

\[ \vec{A}_{ref} = A_{ref} \exp(-i\phi_{ref}) \quad (3.21) \]

\[ \vec{A}_0 = A_0 \exp(-i\phi_0) \quad (3.22) \]

\[ \vec{A}_1 = A_1 \exp(-i\phi_1) \quad (3.23) \]

\[ \vec{A}_2 = A_2 \exp(-i\phi_2) \quad (3.24) \]

The intensity in the EVA region, \( I_{EVA} \) (referred to as point 1 in Fig.3.5), and the intensity outside the EVA region, \( I_{substrate} \) (point 0 in Fig.3.5), can be written as:

\[ I_{Substrate} = \| \vec{A}_{ref} + \vec{A}_0 + \vec{A}_{noise} \|^2 \quad (3.25) \]

\[ I_{EVA} = \| \vec{A}_{ref} + \vec{A}_1 + \vec{A}_2 + \vec{A}_{noise} \|^2 \quad (3.26) \]

where \( \vec{A}_{noise} \) is a random variable with a Gaussian distribution to account for noise in the signal as generated and presented in Figs.3.6 and 3.7. The spectral amplitude of the resultant signal after FFT is shown in Figs.3.8 and 3.9 for both dry and hydrated conditions. A list of all parameters and inputs for the aforementioned simulation are
Table 3.1: Input parameters for the WSI simulation

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>$n_{\text{air}}$</td>
<td>$1$</td>
</tr>
<tr>
<td>$n_{\text{water}}$</td>
<td>$1.33 + 0.0001(1 - \exp(-N_i/N))$*</td>
</tr>
<tr>
<td>$n_{\text{EVA}}$</td>
<td>$1.47 + 0.03(1 - \exp(-N_i/N))$*</td>
</tr>
<tr>
<td>$k$</td>
<td>$4.62e + 6 &lt; k &lt; 4.9867e + 6$</td>
</tr>
<tr>
<td>$N$</td>
<td>8192</td>
</tr>
<tr>
<td>Initial EVA thickness $t_{\text{EVA,initial}}$ [$m$]</td>
<td>0.005</td>
</tr>
<tr>
<td>$A_{\text{ref}}$</td>
<td>0.5</td>
</tr>
<tr>
<td>$A_0$</td>
<td>0.5</td>
</tr>
<tr>
<td>$A_1$</td>
<td>0.05</td>
</tr>
<tr>
<td>$A_2$</td>
<td>0.45</td>
</tr>
<tr>
<td>Exponential thickness change for EVA $t_{\text{EVA}}$ [$m$]</td>
<td>$5 \times 10^{-6}(1 - \exp(-N_i/N))$</td>
</tr>
</tbody>
</table>
| Triangular thickness change of EVA $t_{\text{EVA}}$ [$m$] | \[
\begin{cases}
\frac{5 \times 10^{-6}}{N/2} N_i & 1 < N_i < N/2 \\
-\frac{5 \times 10^{-6}}{N/2} N_i + 10^{-5} & N/2 < N_i < N
\end{cases}
\]

* Dummy functions used to test the prediction of $n$ out of $I(k)$ by inverse model

Tabulated in Table 3.1.

Fig. 3.7 shows the intensity signal with respect to wavenumber simulated in dry condition. Fig. 3.7(a) shows the intensity at the substrate, Fig. 3.7(b) shows the intensity in the EVA region and Fig. 3.7(c), (d) shows zoomed-in versions of Fig. 3.7(a) and (b).

3.3.2 Inverse model (Phase evaluation)

Signal processing involves the Fourier transform as a tool to connect the intensity signal in the time and frequency domains.

Simulated intensity of the same arrangement but in hydrated condition is depicted in Fig. 3.6. For the case of an exponential EVA thickness change input function (see Table 3.1), Fig. 3.8 shows the Fourier transformed intensity signal vs. $f_A$ for an exponential thickness change input. The intensity for both dry and hydrated conditions
3.3 Numerical simulation

Figure 3.6: Intensity signal simulated in hydrated condition: (a) the intensity corresponds to the interference between light reflected at the coated substrate glass, (c) same signal in a smaller range. (b) The intensity corresponds to the interference between light reflected at the EVA front surface, (d) same signal in smaller range.

Figure 3.7: Intensity signal simulated in dry condition: (a) the intensity corresponds to the interference between light reflected at the air-glass interface, (c) same signal in a smaller range. (b) The intensity corresponds to the interference between light reflected at the air-EVA interface, (d) same signal in a smaller range.
are shown in the graph, where three significant peaks of interest are readily seen which belong to interfaces 1, 0 and 2 (front surface of EVA, air-glass interface and back surface of EVA respectively). In Fig. 3.8 (a), the amplitude of peak 1 is relatively lower than peak 2. This comes from the difference in refractive index of the EVA front surface and the back of EVA which is the mirror interface. The separation gap between the peaks 1 and 2 is related to the EVA thickness, therefore in hydrated condition shown in Fig. 3.8 (b), swelling causes the separation to be more pronounced. There is also an overall shift between dry and hydrated conditions (Figs. 3.8 (a) and (b). This is clearly visible in the location of peak 0, which comes from the glass substrate, that repositioned to a higher $\hat{f}_A$ due to higher refractive index of surrounding medium ($n_{\text{water}}$).
Figure 3.8: Fourier transform of simulated signal shown in Fig. 3.7 and Fig. 3.6. (a) dry condition (b) hydrated condition with exponential thickness change input.
Similarly, the Fourier transform of a simulated interference signal due to triangular EVA thickness change input for hydrated condition is demonstrated in Fig. 3.9.

The intensity signal was multiplied by $w(n) = 0.5(1 - \cos(2\pi \frac{n}{N}))$ $0 \leq n \leq N$ a Hanning window to reduce spectral leakage between the otherwise prominent secondary lobes of a rectangular window. Fig. 3.10 demonstrates the Hanning window envelope overlaid on the peaks 0, 1 and 2.

Figure 3.10: Hanning window envelope overlaid on the peaks 0, 1 and 2.
3.3 Numerical simulation

Figure 3.9: Fourier transform of interference signals shown in Fig. 3.7 and 3.6. (a) dry condition (b) hydrated condition with triangular thickness change input.
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Figure 3.11: Schematic phase change obtained by inverse FFT applied on three filtered peaks 0, 1 and 2 demonstrated in Figs. 3.8 and 3.9.

Pursuant to filtering the signal using Hanning window down to our intended peaks, the inverse FFT is applied on filtered signal and then by performing unwrapping, the phase change \( \phi(k) \) for each peak is looked out for (see the block diagram in Fig. 3.4). It is worth mentioning that phase is computed from \( \text{arctan}(\text{Im}/\text{Re}) \) which is the phase shift at a frequency. Phase unwrapping is the process of recovering unambiguous phase values from phase data that are measured in modulo \( 2\pi \) [rad] (wrapped data) Huntley (2001); Huntley et al. (2006); Salfity et al. (2006a,b). The result is similar to the schematic graph shown in Fig. 3.11. As earlier described in Eq. 3.3, the first part \( \phi_{sj} \) is zero in this case so the equation is simplified as follows:

\[
\phi = k\Lambda
\]

where \( \Lambda \) is optical path (\( \Lambda = 2nz \)), hence the slope of the phase would be:

\[
\frac{d\phi}{dk} = \Lambda.
\]

This slope is related to the frequency of the signal, i.e. to the peak positions in Fig. 3.8 and 3.9. \( \Lambda \) can be established through two possible ways as elaborated in the following subsections.

### 3.3.2.1 Approach 1

The first approach involves finding the positions of peaks in the Fourier transform spectrum with sub-pixel resolution. This is shown in the inverse model flowchart (Fig. 3.4).
Let’s denote $N \in \mathbb{R}$ as the number of fringes through a full scan for the case of a single interface, $N_1$ as the position of peak 1 (Fig. 3.10) and $\Lambda_1$ as a constant through the scan. The phase change during a linear wavelength scan will be:

$$\Delta \phi = \Lambda_1 \Delta k \quad (3.29)$$

A linear phase slope is established to represent phase at the first interface, that can be used as a reference -see Fig. 3.13.

Figure 3.13: A static interface leads to a linear $\phi(k)$ behaviour. Peak position in the spectral domain is directly related with the slope of $\phi(k)$.

Also:
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\[ N_1 = \frac{\Delta \phi_1}{2\pi} = \Lambda_1 \frac{\Delta k}{2\pi} \]  
(3.30)

Therefore the optical path length that leads to peak 1 is

\[ \Lambda_1 = \frac{2\pi N_1}{\Delta k} \]  
(3.31)

\[ \Lambda = 2\pi f_k = \frac{d\phi}{dk} = \frac{\Delta \phi}{\Delta k} \]  
(3.32)

in which \( f_k \) is in units of (\( m/\text{rad} \)). Therefore the change in \( \Lambda_1 \) necessary to shift the peak by 1 cycle in the spectrum (i.e. one sample interval in the spectrum obtained through an FFT) is therefore:

\[ \Delta \Lambda_{\text{cycle}} = \frac{2\pi}{\Delta k} \]  
(3.33)

in this case \( \Delta \Lambda_{\text{cycle}} \approx 20 [\mu m] \) with \( \Delta k \sim 3.66 \times 10^{-5} [\text{rad.m}^{-1}] \). If the sub-pixel peak detection improves in a sense that the peak position estimation becomes accurate to 1/100 of a cycle, it is possible to establish \( \Lambda_1 \) to around 0.2 [\mu m], and hence \( z_1 \) to around 0.1 [\mu m].

Another important step in data analysis is to refine peak position with subpixel resolution to find the OPD to the glass substrate. This important step is carried out using the optimization method presented by Huntley (1986). It produces an improved estimate of the position of a peak in the Fourier transform of the complex row vector. Employing this method was a crucial part in tracking the thickness changes.

### 3.3.2.2 Approach 2

The second approach estimates the optical path length \( \Lambda \), by measuring the slope of \( \phi_1(k) \) in dry condition. As illustrated in Fig. 3.4, the phase is established from an inverse FFT of filtered signal. A linear fit is used to produce a linear representation of the phase at the first interface, \( \phi_{1,\text{dry}}^{\text{lin}} \), that can be used as a reference as shown in Fig. 3.14.

From the linear fit data, \( \Delta \phi_1 \) is calculated as follows:
Figure 3.14: Schematic phase change for peak 1 and its linear representation to measure its slope.

\[
\Delta \phi_{1,\text{dry}}^{\text{lin}} = \Lambda_{1}^{\text{dry}} \Delta k
\]

(3.34)

after rearranging:

\[
\Lambda_{1}^{\text{dry}} = \frac{\Delta \phi_{1,\text{dry}}^{\text{lin}}}{\Delta k}.
\]

(3.35)

Thus \( z_1 \), the distance between front surface of EVA and reference glass window \( ZDL \) can be calculated as follows:

\[
 z_1^{\text{dry}} = \frac{\Lambda_{1}^{\text{dry}}}{2n_{\text{air}}}
\]

(3.36)

This estimate of the geometrical distance from \( ZDL \) to EVA front surface corresponds to the very initial geometrical distance upon immersion in water, thus \( z_1^{\text{dry}} = z_1^{\text{Hyd}} \). Therefore:

\[
\Lambda_{1}^{\text{Hyd}} = 2 z_1^{\text{Hyd}} n_{\text{water}}
\]

(3.37)

where \( n_{\text{water}} \) is refractive index of water which was assumed to be a time varying parameter. It is introduced in Table 3.1 as a function of frame number. By substituting Eq. 3.36 in Eq. 3.37 leads to:

\[
\Lambda_{1}^{\text{Hyd}} = \frac{n_{\text{water}}}{n_{\text{air}}} \Lambda_{1}^{\text{dry}}
\]

(3.38)
or in terms of phase that we should measure if there was no swelling during immersion:

\[ \phi^{\text{lin}}_{1,\text{Hyd}}(k) = \Delta k \Lambda_1^{\text{Hyd}} = 2 z_1^{\text{dry}} n_{\text{water}} \Delta k. \]  

(3.39)

This linear phase change can be used as a reference to establish EVA thickness variations. By subtracting the linear phase component that corresponds to peak 1 (i.e. EVA front) from the measured phase \( \phi_1^{\text{Hyd}}(k) \), \( \phi_{\text{def}} \) is obtained as:

\[ \phi_{\text{def}}(k) = \phi_1^{\text{Hyd}}(k) - \phi^{\text{lin}}_{1,\text{Hyd}}(k) \]  

(3.40)

\( \phi_{\text{def}} \), which is defined as the \( \phi \) for deformation due to swelling, is then proportional to the thickness change. This is calculated as follows; accumulating the changes in \( \Lambda \) from the beginning of the scan as \( \phi = k\Lambda \). Fig. 3.15 shows how phase change and wavenumber are related in an incremental discretized fashion.

From Fig. 3.15, the first phase value, \( \phi_1 \), corresponds to wavenumber \( k_1 \):

\[ \phi_1 = \phi(k_1) = k_1 \Lambda(k_1) = k_1 \Lambda_1 \]  

(3.41)

\[ \phi_2 = \phi(k_1 + dk) = (k_1 + dk) \Lambda(k_1 + dk) \]
\[
= (k_1 + dk) (\Lambda(k_1) + d\Lambda_1)
\]

\[
= k_1 \Lambda_1 + k_1 d\Lambda_1 + \Lambda_1 dk + dk d\Lambda
\]

\[
\cong k_1 \Lambda_1 + dk \Lambda_1 + k_1 d\Lambda_1
\quad (3.42)
\]

From Fig. 3.15, the incremental phase change is given by:

\[
d\phi_1 = \phi_2 - \phi_1
\]

\[
= \phi(k_1 + dk) - \phi(k_1) = \Lambda_1 dk + k_1 d\Lambda_1
\quad (3.43)
\]

then by rearranging:

\[
d\Lambda_1 = \frac{d\phi_1 - \Lambda_1 dk}{k_1}
\quad (3.44)
\]

and eventually in generic format:

\[
d\Lambda_j = \frac{[\phi_{j+1} - \phi_j] - \Lambda_j dk}{k_j}.
\quad (3.45)
\]

For the OPD:

\[
\Lambda_2 = \Lambda_1 + d\Lambda_1
\quad (3.46)
\]

and in generic form with \( j = 1, ..., N \):

\[
\Lambda_j = \Lambda_{j-1} + d\Lambda_{j-1}.
\quad (3.47)
3.4 Refractive index change

Variation of refractive index of medium and EVA during the measurement is likely to occur. Hence the model was enabled to identify changes in refractive index and take these variations into account while estimating the thickness change profile. In this chapter to simulate the measurement conditions in water, varying refractive indices are introduced in a forward approach to the model, and they were inversely identified through detailed formulations provided in the following subsections. Later in this chapter, the input and estimated refractive indices are presented to verify the technique.

3.4.1 Refractive index of water

The following approach was developed to obtain $n_{\text{water}}$ during a laser scan, which is based on phase changes.

The phase change for peak 0 vs wavenumber is shown schematically in Fig. 3.16 (a), and it can be written based on Eq. 3.15 as:

$$\phi_0 = 2z_0 n_{\text{water}} k.$$  

(3.48)

This relates the phase for peak 0 with the geometrical distance between surface of EVA and the ZDL ($z_0$ as shown in Fig. 3.2 is considered here as a constant determined by a glass spacers used in the experimental set up to position the samples in measurement chamber). By differentiating $\phi_0$ with respect to $k$ due to tuning the wavenumber and taking $dn_{\text{water}}$ as our unknown parameter -see Fig. 3.16 (b):

$$\frac{d\phi_0}{dk} = 2z_0 (n_{\text{water}} + k \frac{dn_{\text{water}}}{dk})$$  

(3.49)

$$d\phi_0 = 2z_0 (n_{\text{water}} dk + kdn_{\text{water}})$$  

(3.50)

where $k$ ranges between $k_1$ and $k_2$, the initial and final wavenumbers. $dn_{\text{water}}$ could be extracted from Eq. 3.50 as the height of each step in Fig. 3.16 (b). $n_{\text{water}}(k)$ is calculated by accumulating the changes $dn_{\text{water}}$ from the beginning of the scan. At $k = k_1 + dk$; from Eq. 3.50:
3.4 Refractive index change

Figure 3.16: (a) Estimated phase change for peak 0 as a function of wavenumber, (b) Schematic diagram for estimated refractive index of water change with wavenumber.
which represents the first incremental change in the refractive index of water between $k = k_1$ and $k = k_1 + dk$. From Fig. 3.16 (b) and Eq. 3.51:

$$n_{\text{water}}(k_1 + dk) = n_{\text{water}}(k_1) + dn_{\text{water},1}$$

(3.52)

$$dn_{\text{water},j} = \left[ \frac{d\phi_0(k_j)}{2z_0} - n_{\text{water}}(k_j + dk) \right] \frac{1}{k_j + dk}.$$ 

(3.53)

Therefore:

$$n_{\text{water}}(k_{j+1}) = n_{\text{water}}(k_j) + dn_{\text{water},j}$$

(3.54)

In order to obtain $n_{\text{water}}$ from Eq. 3.54, $n_{\text{water}}(k_1)$ at the beginning of the scan is required. Within simulation this value is simply calculated based on the formula introduced to the forward model presented in Table 3.1. However in actual test (Chapter 4), it could be calculate as:

$$n_{\text{water}}(k_1) = \frac{\phi_{0,\text{Hyd}}^\text{im}(k_{\text{end}}) - \phi_{0,\text{Hyd}}^\text{im}(k_1)}{\phi_{0,\text{Dry}}^\text{im}(k_{\text{end}}) - \phi_{0,\text{Dry}}^\text{im}(k_1)}.$$ 

(3.55)

Eq. 3.55 involves a margin of uncertainty, which comes from approximation involved in linear fit on phase changes in dry and hydrated conditions.

### 3.4.2 Refractive index of EVA

In order to calculate the refractive index change of EVA, $n_{EVA}$, due to water uptake, the unwrapped phase changes of both sides of EVA are taken into account. $\phi_1 + \phi_2$ include information of refractive index and thickness of EVA, plus the influence of $n_{\text{water}}$. Although change in $\phi_2$ alone does carry information about the $n_{EVA}$, it is under influence of $n_{\text{water}}$ as well. To account for both parts $\phi_1$ is added to the equation. Following calculations are considered:
3.4 Refractive index change

\[ \phi_1 + \phi_2 = k(\Lambda_1 + \Lambda_2) = k(2n_{\text{water}}z_1 + 2n_{\text{EVA}}th_{\text{EVA}}) \] (3.56)

By differentiating Eq. 3.56, and taking \( n_{\text{water}}, n_{\text{EVA}}, z_1 \) and \( th_{\text{EVA}} \) all to be functions of \( k \) or time:

\[
\frac{d\phi_1 + d\phi_2}{dk} = (2n_{\text{water}} z_1 + 2n_{\text{EVA}}th_{\text{EVA}})
+k(2dn_{\text{EVA}}th_{\text{EVA}} + 2n_{\text{EVA}}\frac{dth_{\text{EVA}}}{dk} + 2dn_{\text{water}}\frac{dz_1}{dk})
\] (3.57)

The change in the refractive index of EVA is thus:

\[
dn_{\text{EVA}} = \frac{(d\phi_1 + \delta\phi_2)}{2k th_{\text{EVA}}} - \frac{dk (\Lambda_1 + \Lambda_2)}{2k th_{\text{EVA}}}
-n_{\text{EVA}}dth_{\text{EVA}} + dn_{\text{water}}z_1 + n_{\text{water}}dz_1
\] (3.58)

\( n_{\text{water}} \) is evaluated first, as illustrated in previous section (Eq. 3.53). \( dz_1 \) is replaced by \( d z_0 - dth_{\text{EVA}} \) and \( z_1 \) replaced by \( z_0 - th_{\text{EVA}} \) where \( z_0 \) is obtained from Eq. 3.48 as shown in following equation:

\[
z_0 = \frac{\Delta \phi_{\text{lin}}}{2n_{\text{air}}\Delta k}. \] (3.59)

The optical path between glass substrate and the ZDL is recalculated based on the obtained refractive index of water \( n_{\text{water}}(k) \).

As a result, changes in \( n_{\text{EVA}} \) can be calculated as follows:

\[
dn_{\text{EVA}} = \frac{(d\phi_1 + \delta\phi_2)}{2k th_{\text{EVA}}} - \frac{dk (\Lambda_1 + \Lambda_2)}{2k th_{\text{EVA}}}
-n_{\text{EVA}}dth_{\text{EVA}} + dn_{\text{water}}(z_0 - th_{\text{EVA}}) + n_{\text{water}}(dz_0 - dth_{\text{EVA}})
\] (3.60)
Non-contact measurement of the swelling behaviour of EVA using Wavelength Scanning Interferometry: feasibility study and numerical simulation

3.5 Effect of film thickness on optimum filtering and maximum measurable surface velocity

The EVA layer is seen as two peaks 1 and 2 (front and back surfaces) which separation determines the maximum spectral bandwidth of the filters that could be used to separate the signal corresponding to each peak. In other words, to avoid crosstalk between the signals corresponding to these peaks, the filters have to be band limited and their width cannot exceed 2 times the peaks separation. This imposes a limit on the surface velocity during a wavelength scan.

In general, we can write the width of the spectral peak, i.e. the optical path resolution, as:

$$\delta\Lambda = \gamma \frac{2\pi}{\Delta k}$$  \hspace{1cm} (3.61)

where the constant $\gamma$ takes the value 4 for a Hanning window Huntley and Ruiz (2011). Therefore for our laser tuning parameters of $k_1 = 4.62 \times 10^6 [rad/m]$ and $k_2 = 4.9867 \times 10^6$, the depth resolution $\delta\Lambda$ is 68 [$\mu m$].
3.5 Effect of film thickness on optimum filtering and maximum measurable surface velocity

As the simulation intended to assess the feasibility of the method in identifying the thickness change through the phase signal, here the output of the simulation is compared with the input. The thickness is given as an input to the forward model in two forms, triangular and exponential thickness profile with initial thickness of 0.5 [mm] and amplitude of 5 [µm]. The choice of initial thickness and amplitude is inline with the specimen and expected thickness change for EVA in our measurements.

The triangular thickness profile happens with \( \frac{dth}{dN} = 0.0011 \text{ [µm/frame]} \), in a matter of \( N = 8192 \text{ frames} \) (see Table 3.1). As shown in Fig. 3.17, triangular thickness profile causes two sub-peaks in envelope for peak 1. The ramp up in blue signal in Fig. 3.18 (a) with positive slope results in the right peak, and the ramp down with negative slope results in the left peak of envelope 1 in Fig. 3.17. The higher the slope of thickness change, the wider the gap between these two sub-peaks will be, and the central point would remain unchanged.

In Fig. 3.18 (a) the red curve shows estimated thickness change from triangular input signal. A generally good agreement between the two input and estimated curves are observed, however there are initial and final errors as well as offset between red and blue curves. The initial and final error comes from the filtering process. Since the curve is formed in a build up process of frame by frame calculations, the initial error cause the overall offset in the estimated curve.

Similarly the input and estimated profile is presented for an exponential thickness change in Fig. 3.18 (b). Generally good agreement is observed for this case as well, however there is a poor match at the start of the curve. This comes from filtering and the inverse FFT. In order for the high slope of the thickness change to be reconstructed, high frequency Fourier components are required which are excluded by the reduced bandwidth of the filters.

As explained earlier the refractive index of both water and EVA were assumed to be varying values. They were defined in the form of exponential function as demonstrated in Table 3.1. The developed formulation in section 3.4, estimates the refractive indices of water and EVA. Figs. 3.19 (a) and (b) show the estimated refractive indices against input functions for water and EVA respectively. The reasonable agreement between the two curves in both cases verifies the method for extracting refractive index from phase change, except that there are initial errors that lead to an offset throughout.
Figure 3.18: (a) Input and estimated triangular thickness, (b) Input and estimated exponential thickness.
3.5 Effect of film thickness on optimum filtering and maximum measurable surface velocity

Figure 3.19: (a) Refractive index of water input and output signal, (b) refractive index of EVA input and output signal.
Non-contact measurement of the swelling behaviour of EVA using Wavelength Scanning Interferometry: feasibility study and numerical simulation

Maximum surface velocity

As mentioned earlier the rate of thickness change is limited by the allowable bandwidth of the filters to avoid cross talk between the peaks. This sub-section is intended to calculate the maximum velocity of the measured moving interface given that the filters has a finite width.

Differentiating the relation between phase change and OPD, $\phi = k \Lambda$, and considering $k = k_c + dk t$ leads to:

$$2\pi f = \frac{d\phi}{dt} = k' \frac{d\Lambda}{dt} + \Lambda \frac{dk}{dt}$$

and then by rearranging:

$$f = \frac{1}{2\pi} \left( k' \frac{d\Lambda}{dt} + \Lambda dk \right) \text{ [cycle/frame].}$$

Rewriting in the form of non-dimensional temporal frequency gives:

$$\hat{f} = N f = \frac{k N}{2\pi} \frac{d\Lambda}{dt} + \frac{\Lambda \Delta k}{2\pi},$$

in which the first term of right hand side of equation is due to swelling and the second term is due to laser tuning. In presence of swelling the first term is a negative values, therefore following inequality is obtained:

$$\hat{f} < \frac{\Lambda \Delta k}{2\pi}.$$  \hspace{1cm} (3.65)

$N_{21}$ is the bandwidth between the two peaks (front and back surface of EVA '1' and '2' -see Fig. 3.20). Substituting $N_{21} = \frac{\Delta k}{2\pi} \Lambda_{21}$ leads to:

$$\Delta \hat{f} < N_{21}.$$  \hspace{1cm} (3.66)

Considering $\Delta \hat{f} = \frac{\Delta \Lambda}{2\pi}$, and differentiating gives:

$$\frac{d\Lambda}{dt} < \frac{N_{21} 2\pi}{k} = \lambda_c N_{21}$$

which could rewritten in terms of geometrical distance ($z$):

$$\dot{z} = \frac{dz}{dt} < \frac{N_{21} \lambda_c}{N 2n_{\text{water}}}.$$  \hspace{1cm} (3.68)

Eq. 3.68 describes the maximum surface velocity. For the present system and sample geometry, surface velocity is limited to $6.011e-9 \text{ [m/s]}$. 


3.6 System performance and limitations

The EVA layer is seen in the spectral domain as two peaks (front and back surfaces) which separation determines the spectral bandwidth of the filters that can be used to separate the signal corresponding to each peak to avoid crosstalk. This imposes a limit on the surface velocity during a wavelength scan. In other words, the frequency of the interference signal is proportional to the OPD. The tuning rate creates a carrier frequency that can be changed by changes in the OPD. When the OPD increases, the frequency increases and vice versa. Moreover, if the OPD increases quickly, i.e. due to an accelerated movement of the target interface under observation, there will be a sudden increase in the frequency of the interference signal. If the change in OPD is such that the instantaneous frequency is higher than the filter bandpass frequencies (the filters used to isolate individual peaks from the spectrum evaluated with the FFT), then those frequencies will not be used in the inverse FFT to reconstruct the original phase with the original slopes. The slopes of the phase are therefore truncated which leads to a loss of information on the changes of the OPD. Sharp variations in OPD, i.e. due to a step, can even cause unwrapping errors if the absolute value of the phase change is larger than $\pi$. As the slopes increase, the spectral peaks broaden too much and start to interfere and to spread cross-talk, which leads to a poor phase reconstruction. What can make phase reconstruction even more challenging is a short OPD between interfaces in

Figure 3.20: Peaks 1 and 2 in frequency and number of sample axes where $f_k^{21} = \frac{N_{21}}{\Delta k}$.
the sample, e.g. a thin EVA layer, and also if the substrate is too close to the $ZDL$. This is so because peak’s proximity limits the bandwidth of the filters required to correctly recover phase information. The maximum thickness change rate detectable in the set up is analytically presented and is dependent on geometrical and system parameters. The main limitations can be summarized as follows:

1) Phase slope reconstruction is limited by filter bandwidth.

2) Filter bandwidth is ultimately limited by the spectral proximity of peaks that correspond to different interfaces in the sample.

3) Maximum detectable thickness change rate is related to $\frac{N_1}{N_2}$ as well as $\Delta k$.

3.7 Conclusion

In this chapter a method based on Wavelength Scanning Interferometry is proposed to determine thickness change of EVA film over a substrate. The mathematical framework of the technique and a simulation case study to probe its feasibility for thickness change monitoring of our intended material was presented. This simulation takes advantage of a forward model that takes a hypothetical surface deformation as an input and replicates the intensity signal reflected from the illuminated sample surface. This is followed by an inverse model that evaluates the surface deformation from the interference intensity signal. The system relies on a tunable laser to illuminate a layer of EVA laminated onto a reflective substrate. The interference signal between light reflected at different interfaces and a reference wavefront is modulated with temporal frequencies proportional to the optical path difference between them. Fourier transformation of the interference signal leads to peaks that locate the position of the interfaces. The refractive index and thickness of the EVA layer are determined by evaluating changes in OPD by measuring phase changes.

The limitations of the method in terms of the sample thicknesses and also the maximum detectable swelling rate, which served us to design the experimental set up, sample configuration, data acquisition parameters and data analysis which are presented in the next chapter.
Chapter 4

Non-contact measurement of the swelling behaviour of EVA using Wavelength Scanning Interferometry: optical system and results

4.1 Optical setup

Fig. 4.1 shows a schematic diagram of the optical set up of WSI. In this set up the reflection from the window glass is taken as reference beam which recombines with reflections from sample interfaces (see Fig. 3.2). The interference pattern was detected by a 2-D photo-detector array (SU640SDV – 1.7RT/RS170, Goodrich corporation, 640 × 512 pixels, 14 bit).

As previously mentioned in Chapter 3, the idea is to measure the EVA swelling through two sets of measurements. The first run serves as a reference in a sense that there is no thickness change by performing the test in dry condition. This is followed by the second measurement that takes place in presence of water (immersion of sample in water), hence with thickness change. Processing the acquired data from both measurements leads to identifying EVA thickness change over the course of test.

Regarding test conditions, both measurements were carried out at room temperature, and the choice of water immersion for the second test comes from the fact that the saturation with any level of RH would otherwise happen over a very long period, which
was not practically feasible for the WSI measurement set up.

Therefore the measurement chamber was designed to ensure the following issues:

1. dry and immersed conditions for EVA on glass substrate
2. an optical window that acts as interferometric reference
3. thermo-mechanically stable construction
4. chemically stable materials under water immersion.

The schematic design is shown in Fig. 4.2 (a) in which the sample is placed horizontally and illuminated using a 45° mirror as depicted in Fig. 4.1. The glass spacers provide
a thermo-mechanically stable distance between reference window and EVA sample. Fig. 4.2 (b) demonstrates a detailed view of the sample components and glass window.

![Figure 4.2: (a) Side view of the designed environmental chamber with sample in testing position, (b) detail of chamber window and sample.](image)

The environmental chamber was made of food grade polycarbonate plastic. The bottom was replaced with a 4 mm thick glass sealed using a high temp resistant sealant, as shown in Fig. 4.3. The whole chamber was mounted on three equal length rods that are attached to glass bottom plate.

![Figure 4.3: Fabricated new environmental chamber.](image)

The experimental setup is shown in Fig. 4.4.
4.2 Sample features and fabrication

In this section the several stages of sample design and fabrication is discussed. The design of this sample intended to address a number of concerns:

- Execution the lamination and curing process in accordance with the standard and predominant practice PV industry.
- Provision of wide exposure area to environmental condition (water) in order to fast track water absorption process.
- Provision of enough prospective of water uptake for the scale to pick up.
- Featuring a substrate layer insensitive to water exposure (mechanical, chemical and optical) and refraction contrast with EVA and surrounding medium.

The above features link the sample and actual PV system in terms of materials, layout, lamination and curing processes, while fulfilling the requirements of the WSI test setup. The following subsection explains different stages of design and fabrication of the sample to meet different aforementioned aspects.

4.2.1 Sample fabrication

The test samples consist of a cured $\sim 0.5\{mm\}$ EVA layer on top of a 2 $\{mm\}$ thick glass substrate. The composition of the EVA used in this study included 33% vinyl acetate
and 80% gel content. Before the lamination process a circular Aluminium mirror was sputtered onto the glass to increase the reflectivity of the substrate at the EVA/substrate interface for optical measurement, as described above. Fig. 5.2 shows a typical sample produced in the way just described. The area of the sample and the EVA film was established according to the balance resolution and the expected changes in weight due to water uptake.

The fabrication of samples was first out-sourced to Solar Capture Technologies Ltd. (Narec solar Ltd. at the time) which is specialist in manufacturing of photovoltaic products. The results was not close to intended samples as the industrial laminating machines are not suitable to do tailored EVA layout. Therefore a number of approaches was attempted to fabricate the samples in-house.

Initially, a number of samples were prepared by using an Autoclave machine. This technique required thin spacers, as thick as the final EVA thickness, and a block to apply pressure on the whole sample while curing. Despite of applying a vacuum, all the attempts ended up with unacceptable and broken samples. A samples prepared with this approach is shown in Fig. 4.7. A very poor and faulty adhesion is visible in this figure.

A second approach was tried using hot press machine and a vacuum pump shown in Fig. 4.8 (a). A different lay-up of spacers and top and bottom blocks was found which resulted in a better integrity between glass and EVA but yet remarkable level of bubbles

![Figure 4.5: (a) Initial sample 3-D view, (b) 2-D drawing.](image-url)
Non-contact measurement of the swelling behaviour of EVA using Wavelength Scanning Interferometry: optical system and results

Figure 4.6: (a) Coating machine used to increase reflectance at the EVA-substrate interface. (b) Sample after coating a 20 [mm] circular region at its center.

Figure 4.7: EVA laminated in Autoclave, with unsatisfactory results full of wrinkles and bubbles.
4.2 Sample features and fabrication

Figure 4.8: (a) Vacuum bag used in a hot press for laminating EVA on glass, (b) samples prepared with this method contain bubbles.

as presented in Fig. 4.8 (b). The improvements observed through this approach, helped us to come up a with an alternative technique and different EVA layout.

As EVA is a strong adhesive after curing on glass surface a special temperature resistant tape with moderate adhesion was employed to mask parts of the glass surface that needed to be clear in finished sample, then this glass was fully laminated by EVA. The lamination process involved using a number of EVA film layers which initially exceeded the spacers’ height, so as to prevent the bubbles by applying a subtle pressure on the EVA films first. As EVA starts to melt the pressure was increased to cause a slow flow of EVA running all over the glass surface. This technique was complemented by adding a flow hump to slow down the EVA flow off the edge of the glass again by using the tape. This flow hump ensured a uniform thickness of EVA free from bubbles. Fig. 4.9 shows the details of actual lay-up with a schematic side view. After curing, the lay-up is placed in a cold press to cool the sample down while maintaining the pressure. Finally the areas with the tapes, including the edges, were cut out and detached from the glass surface and a sample as per intended layout is obtained.

In the above explained approach the temperature settings before placing the lay-up and instantaneous temperature of the hot press machine after that was recorded. In Fig. 4.10 time starts after placing the block in the machine which cause a drop in the temperature. A 20°C higher temperature setting for the bottom block (in contact with glass) was obtained through trial and error to ensure a good adhesion in lamination.

Because of the CTE (Coefficient of thermal expansion) mismatch of different layers, residual stress remains in the laminated sample after sample fabrication and cooling down. The scale of the residual stress under different lamination process parameters
Figure 4.9: Schematic (a) and actual lay-up, (b) of successful sample fabrication technique, before placing into hot press machine.
4.2 Sample features and fabrication

Figure 4.10: Temperature of the hot press machine plates during curing of EVA on top of glass substrate.

for different assembly specifications Dechthummarong et al. (2010); Oreski and Wallner (2005); Wang et al. (2013); Zhang et al. (2012). This effect is addressed in Section 6.4.

The final outcome of this stage is depicted in Fig. 5.2.

Figure 4.11: Laminated EVA (Sample size: 105 mm × 105 mm × (2 mm glass thickness + 0.5 mm EVA thickness)).

After preparation the samples were acclimatized in a desiccator at 10% RH during 30 days.
4.3 Instrument validation

A validation test was required to verify the WSI setup and the analysis techniques followed to measure thickness changes. For this purpose a known displacement is presented to the system so as to evaluate the accuracy of the measured displacements. The WSI setup is meant to capture small thickness changes of the EVA layer in the range of a few microns, therefore the validation test included an element with controlled motion within that range, based on a piezoelectric actuator. This section explains the steps followed to conduct the validation test, including the calibration of the PZT actuator.

Using a known displacement introduced with a piezoelectric actuator

In order to introduce a controlled displacement a piezoelectric translator (PZT) was used. Supplying voltage variations to the PZT changes the optical configuration of the interferometer, resulting in changes to the OPD between the arms of the interferometer Ochoa and Huntley (1998). Based on requirements of the WSI analysis technique, the arrangement shown in Fig. 4.12(a) was employed. Two mirrors were assembled on top of a solid block. One was fixed, representing the glass substrate in the actual sample, and the other was attached to the PZT actuator to represent the moving surface of EVA during hygroscopic swelling. A Thorlabs piezoelectric actuator AE0203D04F was used, with a displacement span of $4.6 \pm 1.5 \mu m$ with $150 \left[V\right]$ maximum drive voltage. The mirrors where then mounted on a post and held stationary in the sample position in the chamber.

The technique presented by Ochoa and Huntley (1998) for calibrating non-linear phase modulators was used for the PZT. This method is based on the minimization of a function obtained from a sequence of interferometric images. Two images $I_0$ and $I$ are digitized, where $I$ is a phase shifted version of $I_0$. The first image is used as a reference and the second has an unknown phase step as a result of an applied voltage to the PZT.

By considering the squared difference between $I_0$ and $I$:

$$(I_0 - I)^2 = B^2[\cos(\phi + \alpha_0) - \cos(\phi + \alpha)]^2 \quad (4.1)$$

in which $\alpha$ is function of applied voltage, $\phi$ is the phase difference between the two arms of the interferometer, and $\alpha_0$ is an initial phase. Therefore, by averaging Eq. 4.1,
Figure 4.12: a) Mirrors used to represent the fixed surface of glass and moving surface of EVA, b) Mirror-PZT assembly mounted on a pole and facing down to be positioned in the chamber
assuming independence between $B$ and $\varphi$, the following equation is obtained:

$$S = \langle B^2 \rangle \langle [1 - \cos(2\varphi + \alpha + \alpha_0)] [1 - \cos(\alpha - \alpha_0)] \rangle,$$

(4.2)

where $S = \langle (I_0 - I)^2 \rangle$.

The error between experimental data and an estimator of the mean square intensity function when an in-plane speckle interferometer is phase shifted using a the PZT driven by a linear voltage ramp is evaluated and minimized Ochoa and Huntley (1998). It is assumed a second order dependence of the PZT displacement (and therefore phase) with the applied voltage.

The required data for calibration was acquired using the WSI configuration by recording images of interference at the moving mirror fringes. The first image acts as reference fringe pattern which is shown in Fig. 4.13 (a). As mentioned earlier a sequence of such images were recorded while the PZT was powered by a controlled changing voltage. A small region in the images are used to evaluate average of the squared differences value in the sequence. Fig. 4.13 (b) represents the mean square intensity signal vs. voltage. Once the displacement vs. voltage relationship of the PZT is established, then PZT can now be used in the validation experiment with different displacement vs. time relationships. The slight drop in the start of the WSI output ramp as compared to PZT input, in Fig. 4.16, comes from the inevitable mismatch between experimental and fitted curves in Fig. 4.13.
4.3 Instrument validation

After the calibration, a validation test was performed using two temporary varying displacements, triangular and exponential. Fig. 4.15 shows the image from the NIR camera. Both mirrors are visible, the fix mirror on the left hand side and the moving mirror on the right hand side of the image. In this test the PZT control and the WSI system are run in tandem.

![Image of fringes during the test](image)

Figure 4.15: Images of the fringes during the test, the moving mirror is on the right hand side, and the fixed mirror on the left hand side.

Fig. 4.16 (b) shows the linear ramp input displacement to the PZT and the one detected through WSI analysis. An agreement is observed between the two curves.

Further to the linear ramp test, an exponential input for the PZT was also tested. Fig. 4.16 (c) shows the detected displacement. It can be seen that the plot includes 3 sequential segments 15 min each. The reason for breaking the input curve in 3 segments was to replicate the actual sequential measurement condition on the WSI set up when laminated EVA sample undergoes a sequence of multiple scans while immersed in water to the point of saturation. Fig. 4.16 (c) shows the predicted three segment curve. The
Figure 4.16: Validation experiments (20°C, 10%RH), (a) PZT intensity for linear ramp signal, (b) PZT output for linear ramp vs. input signal, (C) PZT output displacement for exponential initial signal.
reason for a growing discrepancy between the measurement and the actual exponential displacement in Fig. 4.16 (c) is that the second segment is connected to the end point of the first segment, therefore with the inaccuracy for the final point in fist segment a slight deviation appears. A similar situation has happened in the third segment leading to an accumulated discrepancy.

4.4 Time resolved measurements of swelling of EVA immersed in water

Experimental setup

In this study an in house developed optical set up based on Wavelength Scanning Interferometry (WSI) is used to determine thickness change in EVA immersed in water. The laminated samples described in Section 4.2 were subjected to a moisture loading in the measurement chamber shown in Fig. 4.17 (b) immersed in water, while they were illuminated with a tunable laser (TSL-510) in the WSI set up.

The system uses a tunable laser near-infrared laser to illuminate a layer of EVA laminated onto a mirror as shown in Fig. 4.1. The interference signal between light reflected at different interfaces and a reference wavefront is modulated with temporal frequencies proportional to the optical path difference between them. Fourier transformation of the interference signal leads to peaks that locate the position of the interfaces. The refractive index and thickness of the EVA layer can be determined by comparing the distances between pairs of peaks in the Fourier spectrum. As a matter of fact, since the direct impact of the hygroscopic swelling in encapsulant material is volumetric and refractive index changes over the course of exposure to moisture, employing WSI would be an effective mean to investigate these two factors.
Figure 4.18: EVA surface image recorded by detector. Region of interest includes both mirror and EVA surface.

Figure 4.17: (a) WSI measurement chamber filled with silica gel for dry condition, (b) and filled with water.

Fig. 5.2 shows the test sample. It consists of an EVA layer laminated on top of mirror coated glass. The measurement of thickness is carried out on the central strip with the coated mirror. As explained earlier two sets of measurements are performed. The first run serves as a reference in a sense that there is no thickness change by performing the test in dry condition. Then the second measurement that takes place in presence of water (immersion of sample in water), which involves thickness change. Two identical samples have been in tandem immersed in water through the test, one of which inside the WSI measurement chamber (Fig. 4.1), and the other outside, used to track the water uptake.

Upon filling the bottom of the chamber with distilled water to immerse the samples, the WSI measurements started. A thermometer probe was immersed into the water
4.4 Time resolved measurements of swelling of EVA immersed in water

Figure 4.19: Recorded water temperature during WSI measurement.

to record the temperature during the test (see Fig. 4.19). NIR detector continuously recorded images of EVA surface through the window glass as shown in Fig. 4.18.

14 consecutive scans were performed back-to-back every 6 hours. This total of 3.5 day measurements was carried out to capture the swelling response of the material up to the saturation point. The required total saturation time span was determined from an independent test running for 21 days in which an optical profilometer was intermittently used to track the thickness change of sample in similar condition and no remarkable thickness change was observed after the first two days -see Section 4.6.

The choice of 6 hr scans was to record at a reasonable temporal resolution. Since manual intervention was required to operate the set up upon start and stop, performing shorter tests to achieve higher temporal resolution were not practically feasible. This was inline with the theoretical study performed in the previous chapter on minimum required frame rate to detect surface speed.

A sequence of 8748 frames of $426 \times 16$ pixels images was recorded whilst scanning the laser frequency over the full range from $v_1 = 237.9305 \ [THz]$ to $v_2 = 220.4356 \ [THz]$ ($v = c/\lambda = ck/2\pi$, with $c = 2.9979243 \times 10^8 \ [m/s]$ the speed of light in vacuum) which corresponds to a wavelength range $1260 \ [nm] \leq \lambda \leq 1360 \ [nm]$ and a wave-number range $4.9867 \times 10^6[m^{-1}] \leq k \leq 4.6200 \times 10^6[m^{-1}]$ and the intensity $I(m, n, t)$ was encoded in 14 bits. The frequency sweep was done in ‘step mode’ and the step in between two consecutive frequencies was fixed at 0.002 $[THz]$, corresponding to the wavelength step of $0.011 \ [nm]$ and wave-number step of $41.9 \ [m^{-1}]$. The field of view was ultimately limited by the random access memory (RAM) available for data processing, but $512 \times 640$ pixels (rows, columns) images could be acquired with the NIR camera. After a complete scan, the stack of images of a 3-D intensity volume was stored -see Fig. 4.20.
4.5 Data analysis and results

The experimental data analysis procedure, takes its building blocks from the inverse part of the simulation, elaborated in Section 3.3. The temporal variation in the EVA thickness is sought through evaluating phase change at different spectral peaks. The inputs of the analysis are two sets of intensity signals, one recorded in dry air and the other in distilled water. The intensity signal for dry air, acts as reference to identify the phase change due to EVA thickness change. Fig. 4.21 shows the flow diagram of data analysis. The only difference is an additional peak refinement prior to filtering. This step is to track down the spectral peaks at sub-pixel resolution by producing an improved estimate of the position of a peak in the Fourier transform of the intensity vector Huntley (1986).

Data analysis starts with introducing the the intensity signals to the code. Fig. 4.22 (a) and (b) shows the intensity signals for one pixel respectively in dry and hydrated conditions. The pixel is inside the recorded field of view, depicted by the rectangle in Fig. 4.18.

Fourier transformation of the recorded 3-D (for a pixel \((m, n)\)) intensity distribution was performed along the \(t\) axis, leading to a spectrum that is directly related to the position of the surface along the observation direction. The tunable laser delivered a nearly constant power during the whole scan, which translated in a good level of signal modulation.

The mean value of the intensity signal was subtracted to eliminate the dominant DC peak. Also the intensity signal was multiplied by a Hanning window \(w(n) = 0.5(1 - \cos(2\pi \frac{n}{N}))\) \(0 \leq n \leq N\) and the reason is to reduce spectral leakage between the otherwise prominent secondary lobes of a rectangular window. The Fourier transform of the signal from the EVA surface in Fig. 4.22 (a) and (b) are shown in Fig. 4.23 (a) and (b) respectively in the frame number axis. Peak 1 indicates interference between the
Figure 4.21: Data analysis flow chart.
Figure 4.22: (a) Intensity minus mean intensity vs. wavenumber at EVA / dry condition, (b) hydrated condition.
reference window and front surface of EVA, and peak 2 indicates interference between reference window and back surface of EVA. The radius of filter envelope is \( r_{\text{filter}} = 10 \) for peak 1 and \( r_{\text{filter}} = 40 \) for peak 2. Fig. 4.23 (c) shows a closer look at peak 1 from Fig. 4.23 (a) against OPD axis (\( \Lambda \)). It has a width of 70 \( \mu m \), which compares well with the expected value of 68 \( \mu m \) from Eq. 3.56.

Alternatively, rather than a single pixel, a rectangular region was selected as region of interest (ROI). For the pixels of this region, in the recorded intensity signal, the first image is used as a reference and the squared difference of the signal with regards to the reference is calculated. Then the average value of the squared difference is calculated over the full images and analysed. The resultant signal after FFT is shown in Fig. 4.24 in which the peaks are shown. The position of peak 1 was identified by the optical path distance of the EVA front surface, also by the optical path difference of the peaks 1 and 2. The adjacent peaks could be easily mistaken for the actual peak 1 without scrutiny.

A sensitivity analysis was carried out on the filter bandwidth. Three different filter radius, \( r_{\text{filter}} = 7 \), 20 and 45 were applied to peak 1 which are shown as “Peak 1 envelope” in Figs. 4.24 (a), (b) and (c) respectively. The obtained thickness change profiles as a result of these three filter sizing, plus the one obtained from the aforementioned single pixel analysis, are depicted in Fig. 4.25. A noticeable variation between the thickness change profiles is visible. Compared to the results obtained from independent profilometer measurement, the best answer comes from \( r_{\text{filter}} = 20 \) with the closest final thickness prediction. The under-estimation of the \( r_{\text{filter}} = 7 \) is probably due to lack of data under the envelope for the sub-pixel peak finder. The over-estimation of \( r_{\text{filter}} = 45 \) however is due to contribution of the peak to the left side of peak 1 which drifted the optimized peak position towards left, implying a shorter OPD for peak 1, and hence more thickness change. This also might come from cross talk between peak 1 and 2 with a wider filter envelope. This compassion also highlights that the obtained result from a single pixel might not be a true representation of thickness change across the surface as the final thickness change in the red curve is apparently an under-estimation.
Figure 4.23: Fourier transform of intensity signal recorded at one pixel within EVA surface during a WSI scan (Fig. 4.22), (a) in dry condition, (b) in hydrated condition, (c) peak 1 in more detail vs. \( \Lambda \) axis (dry condition).
4.5 Data analysis and results

Figure 4.24: Fourier transform of intensity signal recorded at region of interest within EVA surface during a WSI scan with different filter radius for peak 1 (a) $r_{\text{filter}} = 7$, (b) $r_{\text{filter}} = 20$, (c) $r_{\text{filter}} = 45$. Peak 2 has filter envelope of $r_{\text{filter}} = 55$.

Fig. 4.26 shows the final estimate thickness change profile due to swelling in EVA with filter radius of $r_{\text{filter}} = 20$ for peak 1. This measurement was performed over
Non-contact measurement of the swelling behaviour of EVA using Wavelength Scanning Interferometry: optical system and results

Figure 4.25: Obtained EVA thickness change profiles analysed for region of interest with different filter sizing for peak 1. The red curve shows the result for a single pixel with filter envelope of $r_{filter} = 10$ for peak 1.

course of 3 days and the experimental results show a thickness increase of $7 \times 10^{-6}[m]$ for the EVA. The stable values at the end are in agreement with water uptake as within the first 48 hours the material is fully saturated, as shown later in Chapter 5.

Figure 4.26: Thickness change in EVA after absorption of water along with the RMS error against exponential fitted curve.
4.5 Data analysis and results

Refractive indices

figure 4.27: (a) Refractive index of water for each test interval during 3 days measurement with WSI, (b) changes of the refractive index of water for all test intervals in the same time period.

As part of the analysis, an approach was developed to obtain refractive index of the EVA and surrounding medium. The formulation and the algorithm to obtain refractive indices is detailed in Section 3.4. Fig. 4.27 (a) presents refractive index change of water. 14 segments corresponding to 14 scans are plotted vs. time. It is visible that within each scan the variations of \( n_{\text{water}} \) is relatively small, however there is an increase in the starting point of each segment. Fig. 4.27 (b) shows overlaid graphs of refractive index change, for all individual segments. It is visible that individual variations of \( n_{\text{water}} \) are in line with \( 1 \times 10^{-4} \). It is worth noting that the temperature change as reported in Fig. 4.19, could impact \( n_{\text{water}} \) within range of \( 1 \times 10^{-6} \) as analytically formulated by Thormählen et al. (1985).

The exponential-like trend in ascending starting points was attributed to sample deflection. Development of inter-laminar stress associated with water diffusion and swell-
Non-contact measurement of the swelling behaviour of EVA using Wavelength Scanning Interferometry: optical system and results

Figure 4.28: Schematic representation of deflected sample after absorbing water over the course of WSI measurement.

Figure 4.29: Refractive index change of EVA during swelling vs. measurement time.

...leads to marginal reduction of $z_0$, which is the gap between window glass and glass substrate (see Fig. 4.28). Referring to Eq. 3.48, this would result in an increase in $n_{\text{water}}$. Since evolution of inter-laminar stress is in accordance with the hygroscopic swelling behaviour, the resulted deflection has an exponential-like trend. This deflection behaviour is empirically observed and linked to hygroscopic swelling induced stress in Chapter 6.

Similarly the EVA refractive index for all 14 measurements are calculated based on developed formulations in Sub-section 3.4.2. These values are plotted in Fig. 4.29 for all segments. The changes in refractive index of EVA are inline with its thickness change and speaks to the fact that diffusion of water into the EVA gradually influence the refractive index. This exponential trend in terms of reaching its maximum level complies with the thickness change (see Fig. 4.26).

The invoking associativity of $n_{\text{EVA}}$ and water concentration, suggested a potential link between the two parameters. This idea is studied after water concentration cal-
4.5 Data analysis and results

Figure 4.30: Associativity between refractive index of EVA and water concentration with the RMS error against linear fitted curve. The triangular points represents data from Abbe refractometer.

Calculation through gravimetric measurements presented in Chapter 5 (see Fig. 5.7). The result however is presented here in the form of a graph demonstrating $n_{EVA}$ vs. water concentration. The underlying link between the exponential trends of the two parameters, leads to a relatively linear relationship. Fig. 4.30 shows an increase in refractive index of EVA vs. water concentration. RMS error band of the WSI curve shows that the accuracy of this measurement still requires refinements, however the trend is clear and it was validated using a refractometer. This phenomenon could potentially be a mean to in situ evaluate presence of water inside EVA layer of PV module.

The Abbe refractometer is also suited for measuring the refractive indices of EVA in dry and saturated condition, (see Fig. 4.30 the result of refractive index measurement for several films of EVA before and after saturation in water. The accurate readings to 4 decimal places RI was possible with this machine. The average difference of 0.0192 in refractive index of EVA was observed with refractometer from dry to saturated conditions (see Table 4.1), this value was around 0.01905 from WSI data analysis results. The refractive index of EVA is relatively insensitive to wavelength (for $\lambda > 500 \text{ nm}$), therefore there is almost no chancing due to type of light source McIntosh et al. (2009b). Wavelength of light source in Abbe 5 Refractometer is 589 nm Abbe5-userguide (2015).
Table 4.1: Refractive index data for some EVA samples measured with Abbe refractometer, dry condition (20°C, 10%RH) and hydrated condition (20°C, in water, after 48 [hrs])

<table>
<thead>
<tr>
<th>n_{EVA}</th>
<th>Δn</th>
<th>t_{EVA} [mm]</th>
<th>dimension [mm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dry</td>
<td>Saturated</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.4748</td>
<td>1.4971</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.4765</td>
<td>1.4953</td>
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<tr>
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<tr>
<td>1.4791</td>
<td>1.4900</td>
<td></td>
<td></td>
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<tr>
<td>Ave.</td>
<td>1.4764</td>
<td>1.4956</td>
<td>0.0192</td>
</tr>
</tbody>
</table>

4.6 Comparing WSI with an independent method

An optical profilometer (Taylor–Hobson Talystep as shown in Fig. 4.31 (a)) was employed to measure the EVA thickness across the centre of the middle stripe in the sample, -see Fig. 5.2. This was done at different times to capture the swelling response of the material during 13 days until no changes were detected (indicating saturation). The measured profile included the EVA central strip and a portion of glass substrate at either side. The EVA thickness was established in the following way: first, outliers and spurious values measured at the edges of the EVA stripe were excluded from the measured profile; then linear fits were performed to the profile corresponding to the glass substrate to establish a reference, and also to the middle region of the EVA profile. Finally, the difference between the constant terms is evaluated to obtain a thickness estimate. Three measurements were averaged to account for potential repositioning errors. In Fig. 4.31 (b), the solid line shows the actual measured profile, while the dotted and dashed lines correspond to the glass substrate and EVA fitted lines, respectively. Fig. 4.31 (c) shows the EVA thickness measured in this way over the course of the test. This is in agreement with water uptake (-see Chapter 5) and indicates that the diffusion transient occurs during the first two days, (around 98000 [s]).

The comparison between the results obtained from WSI (Fig. 4.26) and current method (Fig. 4.31) shows a comparable thickness change detected with both techniques. The idea was to identify the prospective thickness change, so a different sample was used with slight difference in initial EVA thickness.
Figure 4.31: (a) Optical Profilometer Taylor–Hobson Talystep, (b) Surface profile analysis, (c) Total displacement of laminated EVA due to swelling measured with Optical profilometer.
4.7 Conclusion

In this chapter, a displacement and refractive index change measurement method with wavelength scanning interferometry (WSI) was introduced. This experimental measurement was carried out in accordance to the developed and simulated approach, presented in Chapter 3. The expediency of the actual system was demonstrated using piezoelectric translator to verify the algorithm of data analysis as well as the accuracy of measurement. The measurements performed on EVA laminate were also verified with an independent measurement using an optical profilometer. The present chapter also highlighted the importance of sub pixel peak refinement as well as the sensitivity of analysis method to a number of parameters including the filtering. Filtering of identified peaks influences the level of accuracy in thickness measurement regardless of whether a region of interest is studied or a single pixel. The variation in refractive index of water as a medium over the course of measurement is obtained through the phase change, similarly $n_{EVA}$ is also extracted and addressed. The refractive index of EVA as a characteristic parameter could potentially act as a mean to identify the presence of water and differentiate the levels of concentration. The relatively linear $n_{EVA}$ vs. water concentration curve is eventually presented to demonstrate the observed variations from dry EVA to fully saturated one.
Chapter 5

Numerical approach for the determination of the hygroscopic expansion coefficient of EVA as a function of water concentration

5.1 Introduction

One of the underpinning cause of failure modes in PV module is the state of stress across the module. The build-up of stress from number of contributing factors including thermal, hygroscopic and mechanical loading, could eventually affect structural integrity of the system. Mechanical loading occurs when the PV module is exposed to wind or a layer of snow Reil et al. (2012) as well as transport shocks Kontges et al. (2013) and installation loads Olschok et al. (2013). Mechanical stress may cause cracks in the solar cells, which have been shown to reduced long time stability of PV module power Potthoff et al. (2008). Direct exposure to the incoming irradiation of sun as well as the operation of the PV cells impose stresses due to mismatching thermal expansions and redistribute the residual thermal stresses of the manufacturing process Chatterjee et al. (2011); Dietrich et al. (2010); Eitner et al.; Yixian and Tay (2011).

Amongst various stress sources that over time could cause interconnect failures in polymeric encapsulated packages, one of the concerns is associated with moisture and caused by hygroscopic swelling. Polymeric encapsulants are widely used in the electronic packaging industry due to low costs, and improved part quality. These materials however, are hydrophilic, i.e. they tend to absorb moisture in a humid environment.
Numerical approach for the determination of the hygroscopic expansion coefficient of EVA as a function of water concentration

Hygroscopic swelling is the expansion of a material upon moisture absorption. Swelling mismatch between adjacent materials induce additional stresses, so called a hygroscopic stress, which is known to be a major cause of failure in electronic packaging Zhou et al. (2005b). Understanding encapsulant behaviours in the presence of water is therefore crucial for any durability and failure predictive models Ardebili et al. (2003); Wong et al. (2002b). Hygroscopic swelling of EVA, the dominant PV encapsulant, is not adequately studied in the existing literature.

Several techniques have been introduced in the literature to investigate hygroscopic swelling behaviour see-Chapter 2.

In this chapter, the hygroscopic swelling behaviour of EVA is studied through 4 different approaches including an inversion method to express the HEC as a function of water concentration. This inversion method takes advantage of unique opportunity provided by the time-resolved thickness changes of the EVA film due to water uptake, measured experimentally as presented in Chapter 4.

The proposed characterization methodology features the following attributes:

a) Assumption of HEC as a non-constant material property.

b) Linking the material strain to non-uniform moisture distribution in the material-to avoid averaging through the thickness.

c) Use of moisture absorption in the specimen.

d) Use of water immersion condition.

e) Use of high accuracy instruments.

f) Use of FEA to observe material response to the moisture. The results obtained from this methodology offers challenges to widely accepted approaches and assumptions in calculation of hygroscopic expansion confident.

5.2 Hygroscopic swelling

Ethylene vinyl acetate (EVA) is a common encapsulant material used in silicon-based PV modules. It is relatively cheap and has been one of the first investigated encapsulant materials Cuddihy et al. (1986); hence, it has been well formulated for exposure to direct sunlight Holley Jr et al. (1996) and is known to be adequate for most crystalline silicon PV devices. Compared to silicon wafer-based PV modules the new thin film PV technologies could be potentially much less expensive, however they require greater protection from environmental exposure Kempe (2006).

In order to design a reliable photovoltaic modules, understanding the behaviour of materials and interfaces within the actual conditions to which they will be exposed is
crucial. In this part of section an investigation of absorption of moisture in EVA encapsulant was conducted. Test samples had a glass/EVA configuration which comprised laminated EVA on top of the glass. The samples are identical with the ones used for WSI measurement. In order to accelerate the moisture ingress, immersion in water was used in the set up to find the exact values for each parameters for tested samples ($C_{sat}, D$).

### 5.2.1 Fickian model of moisture diffusion

Generally speaking, diffusion describes the movement of matter from one part of a medium to another and it is basically because of random motion of molecules Crank (1970). In case of vapour, diffusivity commonly depends on the concentration of local permeance. There are several mathematical expressions explaining the dependency of the diffusion coefficient on concentration. However, exponential dependence is the most preferred Crank and Park (1951).

The most common approach for modeling moisture absorption or desorption is Fickian’s diffusion law Crank (1970). Fick’s first law describes that the diffusion flux along direction $x$ is proportional to the concentration gradient (Eq. 5.1).

$$ J = -D \frac{dC}{dx} $$  \hspace{1cm} (5.1)

Fick’s Second Law of diffusion describes water transport in a material, linking temporal behaviour and the spatial distribution of moisture concentration. The majority of gas species including water vapour (moisture) follow Fick’s laws when they permeate through polymer chain structures Fan and Suhir (2010). Mass flux in polymers is governed by Fick’s first law, which is expressed as:

$$ J = -D \nabla C $$  \hspace{1cm} (5.2)

where $J$ is the moisture mass flux vector [$kg/m^2s$], $D$ is the diffusivity [$m^2/s$], $\nabla$ is the gradient operator, and $C$ is the moisture concentration [$kg/m^3$]. The conservation of mass within an infinitesimal volume yields Fick’s second law as follows:

$$ \frac{\partial C}{\partial t} = \frac{\partial}{\partial x} \left( D_s \frac{\partial C}{\partial x} \right). $$  \hspace{1cm} (5.3)

Analytical solutions of Fick’s second law are available for some single (homogeneous) material problems with simple geometry. The diffusion Eq. 5.3 for an isothermal 3-D
Numerical approach for the determination of the hygroscopic expansion coefficient of EVA as a function of water concentration

Figure 5.1: The geometry and dimension of specimens.

The problem is reduced to:

$$\frac{\partial C}{\partial t} = \frac{\partial}{\partial x} (D_x \frac{\partial C}{\partial x}) + \frac{\partial}{\partial y} (D_y \frac{\partial C}{\partial y}) + \frac{\partial}{\partial z} (D_z \frac{\partial C}{\partial z})$$

(5.4)

where $D [m^2/s]$ is the diffusion coefficient, $C [kg/m^3]$ is the moisture concentration of the diffusing substrate, $t [s]$ is time and $x, y, z$ refer to Cartesian coordinates along the concentration gradient. For isotropic materials $D_x = D_y = D_z$, then:

$$\frac{\partial C}{\partial t} = D (\frac{\partial^2 C}{\partial x^2} + \frac{\partial^2 C}{\partial y^2} + \frac{\partial^2 C}{\partial z^2})$$

(5.5)

For the case of a rectangular bar as shown in Fig. 5.1, Eq. 5.5 can be solved through separation of variables, yielding an expression for the local moisture concentration as a function of time, position, and diffusivity.

Assuming a two dimensional case when the third dimension is considerably larger than the other two, and considering $x$ as the depth dimension, the $y$-axis along the width, and $l_x$ and $l_y$ the thickness and width respectively, the analytical solution is given by Crank (1970); Kempe (2006):

$$C = C_{Sat} \times \left(1 - \frac{16}{\pi^2} \sum_{m=0}^{\infty} \frac{1}{2m+1} e^{\frac{-D(2m+1)^2\pi^2t}{l_x^2}} \sin \left(\frac{(2m+1)\pi x}{l_x}\right)\right)$$

$$\times \sum_{n=0}^{\infty} \frac{1}{2n+1} e^{D(2n+1)^2\pi^2t} \sin \left(\frac{(2n+1)\pi y}{l_y}\right)$$

(5.6)

where $C_{Sat}$ is the concentration at saturation and $m$ and $n$ are integer indexes. Local concentration data are not measurable but the total weight gain. Hence the above equation is integrated over the thickness to give the fractional mass uptake of the
Figure 5.2: EVA laminated on to glass.

specimen as a function of time Crank (1970).

\[
\frac{M_t}{M_q} = 1 - \sum \frac{8}{(2n+1)^2} \exp\left(-\frac{D(2n+1)^2}{L_{eqv}^2} t\right)
\]

\[
L_{eqv}^2 = \left\{ \left( \frac{(2n+1)\pi}{x_0} \right)^2 + \left( \frac{(2n+1)\pi}{y_0} \right)^2 \right\}
\]

where \(M_t\) is the instantaneous mass of the sample, \(M_q\) is the saturated mass, \(L_{eqv}\) is an equivalent length, \(x_0\) and \(y_0\) are the thickness and length dimensions, respectively.

5.2.2 Gravimetric measurement plan and procedure

Samples were subjected to two different environmental conditions (one which was dry and one in water) and their weights were periodically monitored until a virtual saturation state was reached. The virtual saturation state is defined as the occurrence of no additional weight gain within the resolution of the balance for 48 to 72 hours.

The test samples consist of a laminated/cured EVA layer on top of a glass substrate. The fabrication process is detailed in Section 4.2. Fig. 5.2, shows a WSI measurement sample.

In order to measure the dry weight of the samples, all laminated EVA samples were first kept in a desiccator at 20°C, 10%RH for 3 months, then their weight was measured using an electronic balance (HA180 A&D Instruments Ltd) with a precision of ±0.1 [mg], and finally they were placed in the chamber where they were immersed in water at 20°C. The samples were removed periodically from the chamber for weight measurement after blow drying the sample so as to remove all surface water droplets. This step took around 5 min and remained consistent for all measurements. Then the samples were placed back in the chamber for further absorption. Assuming an initial dry sample at the start of the absorption tests, the weight gain corresponds to the weight of the moisture ingressed into
Numerical approach for the determination of the hygroscopic expansion coefficient of EVA as a function of water concentration

the sample. It is reasonable assumption that only the EVA is responsible for the moisture absorption as the glass is impermeable to moisture with no significant contribution to the mass gain.

Plotting mass uptake as a function of $t^{1/2}$, diffusion coefficient can be determined from the linear portion of the curve, as shown in Fig. 5.3. By using Eq. 5.8, the error is in the range of 0.1% when the ratio of $M_t/M_q$ is lower than 0.5 (Kennedy and Taylor (1991)). This makes an estimation of the diffusion coefficient and a comparison of diffusion behavior.

$$\frac{M_t}{M_q} = 2 \left( \frac{D}{\pi} \right)^{1/2} t^{1/2}$$  \hspace{1cm} (5.8)

The mass uptake of the polymer film at time $t$ has been determined from and presented for all measured samples in Fig. 5.4:

$$Mass \ uptake (\%) = \frac{M(t) - M_{Dry}}{M_{Dry}} \times 100$$  \hspace{1cm} (5.9)

where $M(t)$ is the weight of the sample at time $t$ and $M_{Dry}$ is the dry weight before moisture preconditioning.

Saturated moisture concentration ($C_{Sat}$) determines material’s moisture absorption capacity and diffusivity ($D$) determines the rate of moisture diffusion. $C_{Sat}$ and $D$ can be determined by exposing the testing samples to a specified humidity for a specified time. In this study $C_{Sat}$ was defined by Eq. 5.10 where $V$ is the volume of EVA.

$$C_{Sat} = \frac{\Delta m}{V} \left[ kg/m^3 \right]$$  \hspace{1cm} (5.10)
5.2 Hygroscopic swelling

In order to evaluate $D$, the Fickian diffusion solution in Eq. 5.7 was fitted to the experimental data by using a least squares method. The results are tabulated in Table 5.1 for all measured samples. Fig. 5.5 shows the experimental measurements and the fitted curve for one sample. The diffusion was found to be Fickian, because considering the experimental uncertainty the fit of the Fickian diffusion model to the experimental data was in a good agreement. This is also in agreement with literature Kempe (2006).

Based on the diffusion coefficient obtained from the experiment, Fig. 5.6 shows water concentration versus time at different depths in the sample. The water concentration is solved using Eq. 5.6, for middle point of width ($y$ direction), and through the depth of sample ($x$ direction). $X_1, X_2, \ldots, X_{10}$ are equally spaced layers through the EVA film thickness, starting from the top surface of the EVA. Layers close to the top surface reach saturation much faster than the ones close to the bottom surface. The average

Figure 5.4: Water uptake of EVA at $20^\circ C$.

---

Figure 5.5: Fractional mass uptake of specimen (sample 17).
Numerical approach for the determination of the hygroscopic expansion coefficient of EVA as a function of water concentration

Figure 5.6: Water concentration at different depths through the EVA thickness.

Table 5.1: Diffusion Model parameters

<table>
<thead>
<tr>
<th>Sample</th>
<th>Thickness [m]</th>
<th>V [m^3]</th>
<th>D [M^2/s]</th>
<th>C_{sat} [kg/m^3]</th>
</tr>
</thead>
<tbody>
<tr>
<td>14</td>
<td>430.8202e − 6</td>
<td>3.1019e − 06</td>
<td>1.2460e − 11</td>
<td>0.2579</td>
</tr>
<tr>
<td>16</td>
<td>429.8449e − 6</td>
<td>3.0949e − 06</td>
<td>5.6899e − 12</td>
<td>0.3231</td>
</tr>
<tr>
<td>17</td>
<td>333.6483e − 6</td>
<td>2.3906e − 06</td>
<td>7.660e − 12</td>
<td>0.2928</td>
</tr>
<tr>
<td>15</td>
<td>430.8202e − 6</td>
<td>2.34e − 06</td>
<td>2.96e − 12</td>
<td>0.2901</td>
</tr>
</tbody>
</table>

concentration through the thickness is highlighted with a solid line.

Gravimetric tests reveal a fast initial moisture uptake that gradually slows down to the point of saturation when the material stops gaining weight Rashtchi et al. (2014). The values of corresponding diffusion model parameters are tabulated for this measurement in Table 5.1:

5.2.3 Models of hygroscopically swelling characterization

The main parameters commonly used in the literature to characterize the HEC are the overall experimental strain $\varepsilon_{ave}$, (of the material full thickness) and material ‘mean water concentration $C_{ave}$. It is also widely assumed that a linear relationship between these two holds Shirangi et al. (2008); Shirangi (2010); Stellrecht et al. (2004); Tee et al. (2003); Wong et al. (2002b); Yazdi (2011); Zhou (2008). The strain and water concentration are
5.2 Hygroscopic swelling

Figure 5.7: (a) Thickness change of laminated EVA due to swelling as measured with WSI (described in Chapter 4), (b) Strain.

defined with respect to dry condition as a reference state Zhou (2008).

\[
\varepsilon_{ave}(t) = \frac{th(t) - th_0}{th_0} = \frac{\Delta th(t)}{th_0} \quad (5.11)
\]

\[
C_{ave}(t) = \frac{M(t) - M_0}{V_0} = \frac{M_{Moisture}(t)}{V_0} \quad (5.12)
\]

In which \( th(t) \) is sample thickness as function of time and \( th \) is the thickness in dry condition. \( M_0 \) and \( V_0 \) are specimen weight and volume in dry condition, respectively, \( M(t) \) is the weight of specimen due to water uptake and \( M_{Moisture}(t) \) is the moisture weight at time \( t \). Fig. 5.7 (a) shows the EVA thickness measurement results from WSI measurement (elaborated in Chapter 4) over the course of the experiment. As thickness change needs to be defined as a function of time, an exponential curve is fitted to experimental data to be utilized in subsequent calculations. This fitted curve in this chapter represents the thickness change throughout the current chapter. Corresponding strain, which is evidently engineering strain \( (\varepsilon_{ave}) \) is shown Fig. 5.7 (b).

The thickness change result shows an agreement with water uptake (shown in Fig. 5.5) in a sense that within the first 48 hours the material is fully saturated.

By using the measured strain from the experiment along with the corresponding mean water concentration within the material, a conventional illustration of swelling strain vs. concentration is shown in Fig. 5.8 (solid line).

Basically three different approaches are observed in the literature to characterize HEC from experimental data Fan et al. (2008); Hsu et al. (2008); Stellrecht et al. (2004); Wong et al. (2002b); Zhou (2006b); Zhou et al. (2005a):
Numerical approach for the determination of the hygroscopic expansion coefficient of EVA as a function of water concentration

1. Evaluating HEC from a linear fit to the strain vs. average water concentration:
   As shown in Fig. 5.8 a linear fit with equation of \( \varepsilon = \beta c + b \) represent the swelling behaviour, where \( \beta \) is the slope and hence the HEC.

2. Evaluating HEC considering the average strain vs. average water concentration:
   this approach simply calculates the HEC based on \( \beta = \varepsilon_{ave}/C_{ave} \). Average strain in this context is the strain obtained from top surface displacement, as opposed to localised strain. This average strain is represented as solid line in Fig. 5.8.

3. Evaluating HEC at the point of \( C_{sat} \) which means that the strain is also in saturated condition (\( \beta = \varepsilon_{sat}/C_{sat} \)).

Amongst the above methods, the linear fit seems to be a fairly suitable estimation of this behaviour and has been used in majority of researches (dotted line in Fig. 5.8). However taking into account the local slope of the experimental result shown in Fig. 5.8, the widely accepted linear relationship between strain and moisture concentration seems to be idealized. In this context in an attempt to achieve a more precise approach a forth method is proposed in this research by introducing a dependency between HEC and concentration. Similar observation on dependency of HEC to water concentration were reported by Loh et al. (2005); Park et al. (2009). Therefore the following methodology is proposed which takes advantage of time resolved displacement data obtained form WSI measurement Chapter 4 and Fick’s moisture diffusion assumption.

5.2.4 Proposed methodology

In this research an inversion approach is presented in order to establish EVA’s HEC as a function of water concentration. Fig. 5.9 shows the flow diagram of the technique.
Figure 5.9: Inversion approach.
Numerical approach for the determination of the hygroscopic expansion coefficient of EVA as a function of water concentration

The thickness of the EVA layer is first split into 9 sub-layers. As water diffuses through the thickness of the EVA, these sub-layers will expand depending on the local water concentration. The total thickness change is then equal to the sum of the individual thickness changes of all sub-layers. On this basis, nine ‘thickness change’ equations can be written at 9 time instants, and simultaneously solved to obtain HEC values corresponding to the localized water concentration at each sub-layer. This way of breaking down the overall material thickness change into layer-wise strain accumulation allow us to partially avoid the errors arising from average strain and the average moisture concentration in the specimen Zhou (2008). Fig. 5.10 shows the sub-layers which are spaced in a logarithmic sequence and the top edge exposed to water. As shown in Fig. 5.10, times \( t_1 \) to \( t_9 \) indicate the elapsed times at which water concentration of the corresponding bottom boundary of a given sub-layer reaches 99.9% of \( C_{sat} \). Since the Eq. 5.6 is in the form of series, reaching absolute \( C_{sat} \) is rather unlikely. This divides the time span of moisture ingress into nine intervals, by picking the sequential moments when the next layer boundary is fully saturated.

The water concentration shown in Fig. 5.11 is solved using Eq. 5.6, when the middle point of width (\( y \) direction), and through the depth of sample (\( x \) direction). \( X_1, X_2, \ldots, X_{10} \) are logarithmically spaced layers through the EVA film thickness, starting from the top surface EVA. Layers close to the top surface reach saturated much faster than the ones close to bottom surface. The average concentration through the thickness is depicted with a solid line. \( C(t) \) for the 10 layer boundaries (9 layers) are demonstrated in Fig. 5.11. The logarithmic spacing allows a wider range of local concentrations across the layers for instance at time \( t_1 \), the concentration at the bottom layer (the layer between boundary lines 9-10) is virtually zero, whereas in the top layers it is as high as \( C_{sat} \).

Figure 5.11: Water concentration through the EVA thickness with logarithmic spaced layers.
5.2 Hygroscopic swelling

Figure 5.10: Ingress of moisture through the material at lapse times when the next layer sequentially reaches 99.9% of saturation.
Numerical approach for the determination of the hygroscopic expansion coefficient of EVA as a function of water concentration

The hygroscopic strain is normally assumed to be proportional to the moisture concentration Ardebili et al. (2003); Tee et al. (2003); Zhou et al. (2005b). Taking into account the variation of HEC as a function of concentration, this relation could be:

$$\varepsilon_h = h(C)C$$

where $\varepsilon_h$ is the hygroscopic strain, $h(C)$ [$m^3/kg$] is the HEC and $C$ [$kg/m^3$] is the water concentration. The experimentally observed ‘overall’ EVA thickness change $\Delta x$, at any instant of time $t_m$ can be written as summation of changes in layers 1 to $n$:

$$\Delta x(t_m) = \sum_{n=1}^{9} C_n(t_m)h_n(C)l_n$$

where $n$ represents the layer number, $l_n$ is the thickness of $n^{th}$ layer, $C_n$ is the mean concentration within that layer and $h_n(C)$ is the HEC value corresponding to $C_n$. Considering a schematic thickness change as opposed to elapsed times, shown in Fig. 5.12; $t_m = [t_1, \ldots, t_9]$. 

Figure 5.12: Schematic representation of overall displacement, and elapsed time at which corresponding layers reach saturation.
5.2 Hygroscopic swelling

Figure 5.13: Through thickness logarithmic spaced layers.

Given that $\Delta x(t_m)$ is the experimentally observed displacement at time $t_m$, at $t_1$, the following holds:

$$h_{0,1}C_{0,1}l_{0,1} + h_{1,2}C_{1,2}l_{1,2} + h_{2,3}C_{2,3}l_{2,3} + h_{3,4}C_{3,4}l_{3,4} + h_{4,5}C_{4,5}l_{4,5} + h_{5,6}C_{5,6}l_{5,6} + h_{6,7}C_{6,7}l_{6,7} + h_{7,8}C_{7,8}l_{7,8} + h_{8,9}C_{8,9}l_{8,9} = \Delta x(t_1) \quad (5.15)$$

In above equation $h_{i,j}, C_{i,j}$ and $l_{i,j}$ represent the parameter between layer interface $i$ and $j$ shown in Fig. 5.13. The reason why such notation is used is that values of concentration, for instance; is dependent on the depth, so the representative values for a layer is shown by the top and bottom interfaces. However $C_i$ refers to the concentration of $i^{th}$ interface. Eq. 5.15, refers to instant of time ($t_1$) when $C_1 \equiv C_{sat}$ as the layer is 99.9% saturated at $t_1$.

Moving on to $t_2$, when the first two layers are saturated and hence their corresponding $h$ are nearly the same ($h_1 \approx h_2$); it is obvious that $h_{1,2}C_{1,2}l_{1,2} \approx h_{0,1}C_{0,1}l_{1,2}$, therefore $\Delta x(t_2)$ can be rewritten as follows:

$$h_{0,1}C_{0,1}l_{0,1} + h_{0,1}C_{0,1}l_{1,2} + h_{2,3}C_{2,3}l_{2,3} + h_{3,4}C_{3,4}l_{3,4} + h_{4,5}C_{4,5}l_{4,5} + h_{5,6}C_{5,6}l_{5,6} + h_{6,7}C_{6,7}l_{6,7} + h_{7,8}C_{7,8}l_{7,8} + h_{8,9}C_{8,9}l_{8,9} = \Delta x(t_2) \quad (5.16)$$
A similar approximation can be written for every time lapse, substituting the products $h_i C$ by $h_{0,1} C_0$ in all layers in which saturation is met.

The aim here is to get nine simultaneous equations to find nine unknowns, however; what is presented in Eq. 5.18 is underdetermined system since number of unknowns exceeds the number of equations. This comes from the fact that, despite the similar notations; for instance $h_{7,8}$ in first seven rows; the value behind them varies. This is because it is assumed that $h$ is dependent on concentration, and as the equations are written at different instants of time, the instantaneous concentrations vary, so do $h$. This is illustrated in Eq. 5.17 which represents the average concentration within layers at elapse times in array format.

\[
C = \begin{bmatrix}
0.2931 & 0.2931 & 0.2929 & 0.2925 & 0.2910 & 0.2861 & 0.2697 & 0.2051 & 0.0969 \\
0.2932 & 0.2931 & 0.2931 & 0.2929 & 0.2925 & 0.2910 & 0.2862 & 0.2706 & 0.2113 \\
0.2932 & 0.2932 & 0.2931 & 0.2931 & 0.2929 & 0.2925 & 0.2911 & 0.2863 & 0.2718 \\
0.2932 & 0.2932 & 0.2932 & 0.2932 & 0.2931 & 0.2931 & 0.2930 & 0.2925 & 0.2911 & 0.2864 \\
0.2932 & 0.2932 & 0.2932 & 0.2932 & 0.2932 & 0.2932 & 0.2931 & 0.2930 & 0.2926 & 0.2912 \\
0.2932 & 0.2932 & 0.2932 & 0.2932 & 0.2932 & 0.2932 & 0.2931 & 0.2931 & 0.2930 & 0.2926 \\
0.2932 & 0.2932 & 0.2932 & 0.2932 & 0.2932 & 0.2932 & 0.2931 & 0.2931 & 0.2930 & 0.2926 \\
0.2932 & 0.2932 & 0.2932 & 0.2932 & 0.2932 & 0.2932 & 0.2932 & 0.2932 & 0.2931 & 0.2931 \\
0.2932 & 0.2932 & 0.2932 & 0.2932 & 0.2932 & 0.2932 & 0.2932 & 0.2932 & 0.2932 & 0.2931 \\
\end{bmatrix}
\]

(5.17)

Therefore to solve the problem an approximation is required. In Eq. 5.17 it is observed that at the instant of saturation in first and second layers ($t_2$); the value of $C_{2,3}(0.2929)$ is very close to the value of $C_{1,2}(0.2931)$ at $t_1$. Similar situations are observed among values shown in bold font. so they all could potentially be replaced by $C_{1,2}$ at $t_1$. Looking at the diagonals parallel to the bold values, this situation could spread towards the top-right corner of the array. This means that the distribution of concentrations at $t_1$ across the depth was in such a way that the concentrations of sub-layers at times $t = t_2, \ldots, t_9$ can be approximated with a concentration at $t_1$. By doing so the corresponding $h$ would also replace with one from first row. This approximation reduces the number of unknowns ($h$) in this system of equations to 9, presented in Eq. 5.19.

Factoring and rearranging turns the latter equation to Eq. 5.20. Eventually it could be transformed to the form of matrix multiplication as Eq. 5.21.
\[ h_{0,1} C_{0,1} l_{0,1} + h_{1,2} C_{1,2} l_{1,2} + h_{2,3} C_{2,3} l_{2,3} + h_{3,4} C_{3,4} l_{3,4} + h_{4,5} C_{4,5} l_{4,5} + h_{5,6} C_{5,6} l_{5,6} + h_{6,7} C_{6,7} l_{6,7} + h_{7,8} C_{7,8} l_{7,8} + h_{8,9} C_{8,9} l_{8,9} \]

\[ h_{0,1} C_{0,1} l_{0,1} + h_{0,1} C_{0,1} l_{1,2} + h_{2,3} C_{2,3} l_{2,3} + h_{3,4} C_{3,4} l_{3,4} + h_{4,5} C_{4,5} l_{4,5} + h_{5,6} C_{5,6} l_{5,6} + h_{6,7} C_{6,7} l_{6,7} + h_{7,8} C_{7,8} l_{7,8} + h_{8,9} C_{8,9} l_{8,9} \]

\[ h_{0,1} C_{0,1} l_{0,1} + h_{0,1} C_{0,1} l_{1,2} + h_{0,1} C_{0,1} l_{2,3} + h_{3,4} C_{3,4} l_{3,4} + h_{4,5} C_{4,5} l_{4,5} + h_{5,6} C_{5,6} l_{5,6} + h_{6,7} C_{6,7} l_{6,7} + h_{7,8} C_{7,8} l_{7,8} + h_{8,9} C_{8,9} l_{8,9} \]

\[ h_{0,1} C_{0,1} l_{0,1} + h_{0,1} C_{0,1} l_{1,2} + h_{0,1} C_{0,1} l_{2,3} + h_{0,1} C_{0,1} l_{3,4} + h_{0,1} C_{0,1} l_{4,5} + h_{5,6} C_{5,6} l_{5,6} + h_{6,7} C_{6,7} l_{6,7} + h_{7,8} C_{7,8} l_{7,8} + h_{8,9} C_{8,9} l_{8,9} \]

\[ h_{0,1} C_{0,1} l_{0,1} + h_{0,1} C_{0,1} l_{1,2} + h_{0,1} C_{0,1} l_{2,3} + h_{0,1} C_{0,1} l_{3,4} + h_{0,1} C_{0,1} l_{4,5} + h_{0,1} C_{0,1} l_{5,6} + h_{0,1} C_{0,1} l_{6,7} + h_{0,1} C_{0,1} l_{7,8} + h_{0,1} C_{0,1} l_{8,9} \]

\[ h_{0,1} C_{0,1} l_{0,1} + h_{0,1} C_{0,1} l_{1,2} + h_{0,1} C_{0,1} l_{2,3} + h_{0,1} C_{0,1} l_{3,4} + h_{0,1} C_{0,1} l_{4,5} + h_{0,1} C_{0,1} l_{5,6} + h_{0,1} C_{0,1} l_{6,7} + h_{0,1} C_{0,1} l_{7,8} + h_{0,1} C_{0,1} l_{8,9} \]

\[ \begin{bmatrix} \Delta x(t_1) \\ \Delta x(t_2) \\ \Delta x(t_3) \\ \Delta x(t_4) \\ \Delta x(t_5) \\ \Delta x(t_6) \\ \Delta x(t_7) \\ \Delta x(t_8) \\ \Delta x(t_9) \end{bmatrix} \in \mathbb{R}^9 \]

(5.18)
Numerical approach for the determination of the hygroscopic expansion coefficient of EVA as a function of water concentration.

\[
\begin{pmatrix}
\Delta x(t_1) \\
\Delta x(t_2) \\
\Delta x(t_3) \\
\Delta x(t_4) \\
\Delta x(t_5) \\
\Delta x(t_6) \\
\Delta x(t_7) \\
\Delta x(t_8) \\
\Delta x(t_9)
\end{pmatrix}
\]

(5.19)
\[
\begin{bmatrix}
h_{0,1}C_{0,1}l_{0,1} + h_{1,2}C_{1,2}l_{1,2} + h_{2,3}C_{2,3}l_{2,3} + h_{3,4}C_{3,4}l_{3,4} + h_{4,5}C_{4,5}l_{4,5} + h_{5,6}C_{5,6}l_{5,6} + h_{6,7}C_{6,7}l_{6,7} + h_{7,8}C_{7,8}l_{7,8} + h_{8,9}C_{8,9}l_{8,9}
\end{bmatrix}
\begin{bmatrix}
h_{0,1}C_{0,1}(l_{0,1} + l_{1,2}) + h_{1,2}C_{1,2}l_{2,3} + h_{2,3}C_{2,3}l_{3,4} + h_{3,4}C_{3,4}l_{4,5} + h_{4,5}C_{4,5}l_{5,6} + h_{5,6}C_{5,6}l_{6,7} + h_{6,7}C_{6,7}l_{7,8} + h_{7,8}C_{7,8}l_{8,9}
h_{0,1}C_{0,1}(l_{0,1} + l_{1,2} + l_{2,3}) + h_{1,2}C_{1,2}l_{3,4} + h_{2,3}C_{2,3}l_{4,5} + h_{3,4}C_{3,4}l_{5,6} + h_{4,5}C_{4,5}l_{6,7} + h_{5,6}C_{5,6}l_{7,8} + h_{6,7}C_{6,7}l_{8,9}
h_{0,1}C_{0,1}(l_{0,1} + l_{1,2} + l_{2,3} + l_{3,4}) + h_{1,2}C_{1,2}l_{4,5} + h_{2,3}C_{2,3}l_{5,6} + h_{3,4}C_{3,4}l_{6,7} + h_{4,5}C_{4,5}l_{7,8} + h_{5,6}C_{5,6}l_{8,9}
\end{bmatrix}
\]

\[
\Delta x(t_1) \\
\Delta x(t_2) \\
\Delta x(t_3) \\
\Delta x(t_4) \\
\Delta x(t_5) \\
\Delta x(t_6) \\
\Delta x(t_7) \\
\Delta x(t_8) \\
\Delta x(t_9)
\]

\[
\mathbb{R}
\]

(5.20)
Numerical approach for the determination of the hygroscopic expansion coefficient of EVA as a function of water concentration

For the sake of simplifying the format, a new notation is introduced as Eq. 5.22 that replaces the concentration in the latter equation with \( C_n \) representative of the concentration of the \( n^{th} \) sub-layer. Using the latter notation and replacing \( l_{n-1,n} \) by \( l_n \) and \( h_{n-1,n} \) by \( h_n \) gives the final format shown in Eq. 5.23.

\[
\begin{bmatrix}
C_{0,1}l_{0,1} & C_{1,2}l_{1,2} & C_{2,3}l_{2,3} & C_{3,4}l_{3,4} & C_{4,5}l_{4,5} & C_{5,6}l_{5,6} & C_{6,7}l_{6,7} & C_{7,8}l_{7,8} & C_{8,9}l_{8,9} \\
C_{0,1}l_{0,2} & C_{1,2}l_{1,2} & C_{2,3}l_{2,3} & C_{3,4}l_{3,4} & C_{4,5}l_{4,5} & C_{5,6}l_{5,6} & C_{6,7}l_{6,7} & C_{7,8}l_{7,8} & 0 \\
C_{0,1}l_{0,3} & C_{1,2}l_{1,3} & C_{2,3}l_{2,3} & C_{3,4}l_{3,4} & C_{4,5}l_{4,5} & C_{5,6}l_{5,6} & C_{6,7}l_{6,7} & 0 & 0 \\
C_{0,1}l_{0,4} & C_{1,2}l_{1,4} & C_{2,3}l_{2,4} & C_{3,4}l_{3,4} & C_{4,5}l_{4,5} & 0 & 0 & 0 & 0 \\
C_{0,1}l_{0,5} & C_{1,2}l_{1,5} & C_{2,3}l_{2,5} & C_{3,4}l_{3,5} & 0 & 0 & 0 & 0 & 0 \\
C_{0,1}l_{0,6} & C_{1,2}l_{1,6} & C_{2,3}l_{2,6} & C_{3,4}l_{3,6} & 0 & 0 & 0 & 0 & 0 \\
C_{0,1}l_{0,7} & C_{1,2}l_{1,7} & C_{2,3}l_{2,7} & C_{3,4}l_{3,7} & 0 & 0 & 0 & 0 & 0 \\
C_{0,1}l_{0,8} & C_{1,2}l_{1,8} & C_{2,3}l_{2,8} & C_{3,4}l_{3,8} & 0 & 0 & 0 & 0 & 0 \\
C_{0,1}l_{0,9} & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\
\end{bmatrix}
\times
\begin{bmatrix}
h_{0,1} \\
h_{1,2} \\
h_{2,3} \\
h_{3,4} \\
h_{4,5} \\
h_{5,6} \\
h_{6,7} \\
h_{7,8} \\
h_{8,9} \\
\end{bmatrix}
= \begin{bmatrix}
\Delta x(t_1) \\
\Delta x(t_2) \\
\Delta x(t_3) \\
\Delta x(t_4) \\
\Delta x(t_5) \\
\Delta x(t_6) \\
\Delta x(t_7) \\
\Delta x(t_8) \\
\Delta x(t_9) \\
\end{bmatrix}
\]

\( C_n = \left( \frac{C_{n-1} + C_n}{2} \right) \)  

(5.22)
\[ \begin{bmatrix} C_1 l_1 & C_2 l_2 & C_3 l_3 & C_4 l_4 & C_5 l_5 & C_6 l_6 & C_7 l_7 & C_8 l_8 & C_9 l_9 \\ C_1 (l_1 + l_2) & C_2 l_3 & C_3 l_4 & C_4 l_5 & C_5 l_6 & C_6 l_7 & C_7 l_8 & C_8 l_9 & 0 \\ C_1 (l_1 + l_2 + l_3) & C_2 l_4 & C_3 l_5 & C_4 l_6 & C_5 l_7 & C_6 l_8 & C_7 l_9 & 0 & 0 \\ C_1 (l_1 + l_2 + l_3 + l_4) & C_2 l_5 & C_3 l_6 & C_4 l_7 & C_5 l_8 & C_6 l_9 & 0 & 0 & 0 \\ C_1 (l_1 + l_2 + l_3 + l_4 + l_5) & C_2 l_6 & C_3 l_7 & C_4 l_8 & C_5 l_9 & 0 & 0 & 0 & 0 \\ C_1 (l_1 + l_2 + l_3 + l_4 + l_5 + l_6) & C_2 l_7 & C_3 l_8 & C_4 l_9 & 0 & 0 & 0 & 0 & 0 \\ C_1 (l_1 + l_2 + l_3 + l_4 + l_5 + l_6 + l_7) & C_2 l_8 & C_3 l_9 & 0 & 0 & 0 & 0 & 0 & 0 \\ C_1 (l_1 + l_2 + l_3 + l_4 + l_5 + l_6 + l_7 + l_8) & C_2 l_9 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ C_1 (l_1 + l_2 + l_3 + l_4 + l_5 + l_6 + l_7 + l_8 + l_9) & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \end{bmatrix} \begin{bmatrix} h_1 \\ h_2 \\ h_3 \\ h_4 \\ h_5 \\ h_6 \\ h_7 \\ h_8 \\ h_9 \end{bmatrix} = \begin{bmatrix} \triangle x(t_1) \\ \triangle x(t_2) \\ \triangle x(t_3) \\ \triangle x(t_4) \\ \triangle x(t_5) \\ \triangle x(t_6) \\ \triangle x(t_7) \\ \triangle x(t_8) \\ \triangle x(t_9) \end{bmatrix} \]
Numerical approach for the determination of the hygroscopic expansion coefficient of EVA as a function of water concentration

Based on this formulation presented in Eq. 5.23, solutions for 9 values of HEC \((h_1 \text{ to } h_9)\) are sought through general form of \(A.x = b\). Each of \(h_1 \text{ to } h_9\) corresponds to a different concentration level. Solving this system of equations is fairly straight forward as the \(A\) is a full rank square matrix.

5.2.5 Summary of presented methods

The HEC obtained from the approach presented in previous section is plotted in Fig. 5.14. In this figure nine points representing the hygroscopic swelling against water concentration. A solid line is representing a curve fitted on the data and its equation is provided in Table 5.2. In Fig. 5.14 a secondary axis shows the corresponding water concentration from 0 to 100% of \(C_{\text{sat}}\).

![Figure 5.14: HEC from present numerical solution vs. average water concentration.](image)

The summary of HEC obtained from four methods are tabulated in Table 5.2.

<table>
<thead>
<tr>
<th>Method</th>
<th>Formula</th>
<th>(HEC \ [m^3/kg])</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Linear fit slope</td>
<td>(\varepsilon = \beta c + b)</td>
<td>0.06431</td>
</tr>
<tr>
<td>2 Average method</td>
<td>(\beta = \varepsilon_{\text{ave}}/C_{\text{ave}})</td>
<td>0.041199</td>
</tr>
<tr>
<td>3 Saturation</td>
<td>(\beta = \varepsilon_{\text{Sat}}/C_{\text{Sat}})</td>
<td>0.061213</td>
</tr>
<tr>
<td>4 Present numerical</td>
<td>(\alpha) data presented in Fig. 5.14</td>
<td>(0.844 C^2 + 0.027 C)</td>
</tr>
</tbody>
</table>
5.3 Finite Element modeling of hygroscopic swelling

In order to verify the effectiveness of presented inverse method, a forward model was simulated taking into account parameters including $D$, $C(t)$, $HEC(c)$. Fig. 5.15 illustrate the flow diagram of the forward method. The FEA model of EVA layer on glass substrate, could predict EVA thickness change over the course of the water absorption which will be compared against the exponential fit on experimental results used to extract the hygroscopic expansion coefficient. This allows the validation of HEC obtained from different techniques. The idea is to apply the same concentration profile across the material cross-section as used throughout the calculations for diffusion and HEC, and then comparing the resultant displacement-time graph with the initial displacement profile from the experiment. The hygroscopic swelling coefficient that replicates the experimental result the best, would be the right HEC value. In the following sub sections the steps toward this goal is elaborated.

5.3.1 Modelling strategy for hygro-mechanical analysis

The total deformation in a material subjected to both temperature and relative humidity change, could be represented by:

$$
\varepsilon = \varepsilon^o + \varepsilon^\alpha + \varepsilon^\beta
$$

(5.24)

where $\varepsilon^o$, $\varepsilon^\alpha$, $\varepsilon^\beta$ are the total, stress-induced (mechanical), free thermal, and free hygroscopic swelling strains, respectively. In order to obtain the stress/deformation response resulted from temperature and moisture diffusion, a force equilibrium equation along with the governing equations of heat conduction and moisture diffusion need to be solved. This could potentially be too complicated for practical engineering applications to solve three simultaneous field equations. Temperature and moisture concentration fields are almost independent of the stress state. Hence heat transfer analysis and moisture diffusion analysis could describe temperature and moisture concentration prior to a stress analysis, and then they can be incorporated into a subsequent stress analysis.

The modelling flow diagram of the thermo-hygroscopic stress analysis is illustrated in Fig. 5.16. The heat transfer analysis describes temperature distribution, the temperature could also strongly influence the diffusion of moisture within the material. Then the moisture concentration field as well as temperature field are incorporated into the thermal–hygroscopic stress analysis to predict the resultant stress due to combined effect of temperature and moisture.
Numerical approach for the determination of the hygroscopic expansion coefficient of EVA as a function of water concentration.

Figure 5.15: Forward model.
5.3 Finite Element modeling of hygroscopic swelling

The present study predominantly focuses on the effect of moisture concentration in material swelling. The temperature is assumed to be constant and within normal range of room temperature. This allows to have a hygro-mechanical modelling and analysis which compares to the experimental results.

![Diagram](image)

Figure 5.16: Modeling hierarchy for thermo-hygroscopic stress analysis.

Implementing this procedure into a commercial FEA software would require a capability of mass (moisture) diffusion analysis, whereas most FEA software packages do not handle the moisture (or mass) diffusion function Samson Yoon and Wang (2007). An effective way to address this issue in modelling is to take advantage of a similarity. Heat conduction is a diffusive process and is governed by Fourier’s law of heat conduction as:

\[
q = -K \nabla T \quad (5.25)
\]

where \( q \) is the heat flux \([W/m^2]\) and \( K \) is the conductivity \([W/mK]\). By assuming the energy balance in the absence of internal heat generation, the governing equation of heat conduction yields as follows:

\[
\rho c_p \dot{T} = \nabla \cdot (K \nabla T). \quad (5.26)
\]

Due to the similarity in above heat conduction governing equations, and mass diffusion equation Eq. 5.25 and Eq. 5.26, these two situations are often treated in a “unified” way M. D. Mikhailov (1984). This “thermal–moisture” analogy is quite attractive because moisture diffusion could be solved using the heat transfer function which is available in any FEA software package. As a consequence, the thermal–moisture analogy has been widely employed to address water diffusion inside electronic packages Ardebili et al. (2003); Tee et al. (2003); Vaddadi et al. (2003); Wong et al. (2002a,b). Hence, by substituting the moisture concentration and coefficient of swelling with temperature
Numerical approach for the determination of the hygroscopic expansion coefficient of EVA as a function of water concentration

and thermal expansion coefficients respectively, hygroscopic stress, and strain could be modelled with commercial finite element software.

5.3.2 Implementation using MSC.Marc

MSC.MARC provides general mass diffusion analysis capability MSC.Marc (2013). Nonetheless, the results would be only a temporal diffusion across the model and this can not describe the subsequent hygroscopic swelling. Therefore it does not lead to a hygroscopic stress analysis and this approach is ruled out.

The alternative approach is to obtain transient field moisture concentration, using the diffusion solution, and apply them as temperatures at nodal points. In order to implement this scheme a specific code should be developed in Python. Taking advantage of py_mentat capabilities of MARC which provide a library of command to access and manipulate the modelling environment, allows to run a python script within MARC (Marc has an embedded Python compiler) which creates the model along with calculating the transient moisture diffusion for each and every node depending on position in width and thickness direction of the EVA layer. A 2-D Fickian diffusion model was adopted to calculate moisture concentration in this code. The Nodal quantities are produced in the form of time dependent tables and applied as thermal boundary condition to the corresponding node.

The script follows the typical steps of creating a thermal-structural analysis in MARC. The model of the sample consists of an EVA strip on top of a glass as shown in Fig. 5.17. Plain-strain analysis is employed to represent the sample as the third dimension is considerably larger than the other two. The HEC calculated in the previous section is an input to the model and it substitutes the thermal expansion coefficient.

The straight line in Fig. 5.17, represents the surface of the glass substrate in the model and all nodes along the line are constrained to it using glue-type contact interaction. The material parameters used in this model are listed in Table 5.3. It is worth noting that
5.3 Finite Element modeling of hygroscopic swelling

the dependency of young modulus of EVA to moisture ingress which is experimentally obtained and presented in next chapter, is addressed in modeling

Table 5.3: Material parameters

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Poisson’s ratio</td>
<td>0.39 (-see Table 6.1)</td>
</tr>
<tr>
<td>Width [m]</td>
<td>0.003</td>
</tr>
<tr>
<td>Thickness [m]</td>
<td>$3.30 \times 10^{-4}$</td>
</tr>
<tr>
<td>CTE</td>
<td>$H E C = 0.844 C^2 + 0.027 C$</td>
</tr>
<tr>
<td>$E [M P a]$</td>
<td>6.98 (-see Table 6.1)</td>
</tr>
</tbody>
</table>

Fig. 5.18 depicts the cross-sectional 2-D concentration distribution obtained with the FE model and a concentration dependent HEC. Water concentration is higher at the top and side edges as they are exposed to water.

Figure 5.18: 2-D concentration map [kg/m$^3$] on the cross-section of EVA strip.

The displacement (perpendicular to the EVA surface $y$ axis in Fig. 5.19) distribution is shown in Fig. 5.19 for the fully saturated EVA strip. The colour distribution shows maximum displacement at the centre top of the EVA surface. To carry out a comparison, this simulation was repeated for different HEC values obtained from different approaches, as discussed in next section.
Numerical approach for the determination of the hygroscopic expansion coefficient of EVA as a function of water concentration

5.4 Data analysis and results

In Fig. 5.20 the swelling response predicted by the FEA model described in Sub-section 5.3.2 is compared with EVA surface displacement as measured with WSI, shown in Fig. 5.7, which provides a time-resolved surface profile with very high temporal resolution as the sample is immersed in water. It is readily seen that the HEC from the present numerical approach that uses a concentration dependent HEC leads to the closest prediction of the surface profile. The widely adopted method which takes the slope of the fitted line on strain-concentration curve (method 1 as mentioned above) does not seem to provide a close agreement with the experiment. The same is true for the case in which the HEC is evaluated considering the strain vs. average water concentration (method 2 solid line in Fig. 5.8) and when the simple ratio of final strain to saturated concentration is used. Taking into account the total surface displacement of 7 [$\mu m$] observed in the test, represented by the solid line Fig. 5.8; the latter three methods, (with $13 - 15$ [$\mu m$] displacement) might entail 80-100% error in displacement calculation. However the current results clearly show that the proposed technique has the potential to provide highly accurate results. The study on variation of HEC as a function of moisture concentration in the current study suggests that it is a more precise approach to assume a link between these two parameters. This correlation is observed to be in the form of a second order polynomial and provided in Table 5.2.
5.4 Data analysis and results

Figure 5.20: Thickness change profile of the material surface for various HEC values.

The level of resultant final displacements shown in Fig. 5.20 are corresponding to the level of HEC values summarized in Table 5.2. However as seen in Fig. 5.14, scattered points shows HEC value up to 0.08 at $C_{sat}$ which is above all other values, but results in less displacement. To clarify this by differentiating Eq. 5.13 we have:

$$\frac{d\varepsilon_h}{dC} = dh(C).C + h(C)$$

(5.27)

Considering HEC as a function of concentration and by integrating:

$$\varepsilon = \int_{0}^{C_{Sat}} [C.dh(C) + h(C)]dC$$

(5.28)

Therefore the strain would be the result of above integral for the provided $h(C)$ in Table 5.3, which is as follows:

$$\varepsilon = \int_{0}^{0.2932} [2.53C^2 + 0.054C]dC = 0.02359$$

(5.29)

The above strain multiplied by initial EVA thickness corresponds to 7.78 [$\mu m$] of final displacement which is shown in Fig. 5.20.

In order to compare the effect of different methods to estimate HEC and swelling behaviour on the mechanical integrity of the laminated EVA glass sample, the maximum predicted shear stress at the EVA-glass interface was evaluated with the FE model.
Numerical approach for the determination of the hygroscopic expansion coefficient of EVA as a function of water concentration

Fig. 5.21. Shear stress, especially close to the edge can result in delamination which would further increase moisture ingress by extending the free surface. The maximum shear stress values obtained with methods 1-3 above are between 1.5 and 2.5 times larger than the value obtained with the proposed approach which uses a concentration dependent HEC. A more accurate stress calculation contribute to more accurate durability assessment.

Figure 5.21: Predicted shear stress at the interface of EVA and glass.

5.5 Conclusion

In this study the hygro-mechanical swelling behaviour of Ethylene Vinyl Acetate (EVA) was investigated. Experimentally measured hygroscopic expansion coefficient is needed in order to feed durability predictive models for PV modules vs. water ingress. Taking advantage of a time-resolved thickness profile measurement on EVA film laminated on glass, a numerical technique was presented to find an approximation of the HEC vs. concentration relationship. It is expected that this approach would improve the predictive power of forward FE models that evaluate mechanical stress in response to water ingress. In addition this technique uses a single measurement of swelling from dry to saturated conditions to save the experimental effort of having to measure samples saturated at different %RH and eliminating repositioning errors. This would take far too long exposure of samples to humidity especially at low %RH when the saturation is difficult to detect with marginal water uptake in the material.

The alternative approach to perform a similar study would be FEMU through which an unknown material behaviour is estimated by an initial function. The parameters of this initial function are to be optimized so as to match the intended output with the relevant experimental data. However this process is not a trivial process as similar
research is presented by Kou et al. (2011) in which elastic parameters of a 3-D printed polymeric material is sought to match experimental result. The optimization module is implemented with COMSOL Multiphysics with Matlab script in an iterative update of convergence plot of numerical curve to the experimental elongation-force curve. The presented technique in this chapter is a non-iterative approach, similar to inverse method reported by Lifshitz et al. (1999) to obtain CTE of solids using the expansion obtained from interferometry.
Chapter 6

Hygroscopic swelling induced stress in bi-material EVA/glass strip

6.1 Introduction

Most polymer based materials used in plastic encapsulated packages are hydrophilic and tend to absorb moisture upon exposure to a humid environment. Absorption of moisture in the encapsulant induces swelling and deformation, leading to package failure in some cases. When a polymer encapsulant swells upon moisture absorption and nonpolymeric adjacent materials such as glass or electronics do not, hygroscopic stresses arise due to the mismatch \cite{Wong2002}. It has been reported that swelling induced stresses are comparable to thermally induced stresses \cite{Stellrecht2003} and similar to the thermal stress caused by the mismatch in thermal expansion coefficient of adjacent materials. There is a direct relationship between induced hygroscopic stress and the hygroscopic swelling coefficient of the material. Accurate HEC measurements are essential for evaluating the effect of hygroscopic stress on package reliability.

The study presented in this chapter was an independent observation on the effects of hygroscopic swelling in the encapsulant, through measurement of deflections in bi-material strips exposed to water. EVA laminated to glass strip bends due to development of swelling stress upon immersion of the strips in water for three weeks. Changes of curvature were recorded periodically by scanning the surface of the sample using an optical profilometer. The maximum tangential bending stress at the EVA/glass interface is calculated from the measured deflection which is then used to estimate the maximum shear stress values occurring at both ends. The presented measurement also leads to evaluate the mechanical stress that arises due to hygroscopic swelling of EVA. The theoretical treatment of stress present in such a bent bi-material was first given by
Timoshenko (1925) Timoshenko et al. (1925) which could adequately estimate the values of peak stresses. In the experimental section, the basic principles of the approach are presented. Results are subsequently analysed for various bi-material thickness ratios.

6.2 Theory of bi-material strip deflection

Timoshenko’s classic analysis of thermal stress in a bimetal thermostate Timoshenko et al. (1925) based on strength of materials theory has been widely used for designing laminated structures. However this theory is unable to predict the distribution of interlaminar stresses. The axial normal stress (normal to the cross section) which is the result of shearing forces could be obtained for the bi-material along the strip which is inversely proportional to the radius of curvature of bent strip. According to Timoshenko the interfacial shear stress is restricted to a particular area near the ends of the strip (no shear in the centre of the strip occurs) and could not be calculated in an elementary way. Hence more sophisticated theories like extended strength of materials theory Suhir (1986, 1988, 1989) and theory of elasticity Hess (1969a,b) have been used to provide analytical prediction of normal and shear stress distribution specially near the edges. Alternatively FEA is found to be just as an effective tool for evaluating the stress distribution Eischen et al. (1990), which is used in this chapter. A closer look at the description and approach is presented below.

Description of the problem and approach:

In analysing the deflection of a bi-material strip subject to uniform heating, the overall geometry is illustrated in Fig. 6.1. In this analysis coefficients of thermal expansion of layers 1 and 2 are denoted by \( \alpha_1 \) and \( \alpha_2 \), the elasticity moduli \( E_1 \) and \( E_2 \), the Poisson’s ratios \( \nu_1 \) and \( \nu_2 \) and the temperature change from the stress free state is denoted by \( \Delta T \).
The fundamental assumption in the following analysis is that a cross section of the strip, originally plane and perpendicular to the axis; remains plane during bending and become perpendicular to the curved axis of the strip. Considering that:

\[ \frac{E_1}{E_2} = \Gamma \]  

\[ \frac{t h_1}{t h_2} = \Upsilon \]  

Also that \( R \) is the radius of curvature and \( EI \) the flexural rigidity of the layer, the moment can be calculated as follows:

\[ P_1 = \frac{E_1 I_1}{R} \]  

\[ P_2 = \frac{E_2 I_2}{R} \]  

From the equal elongation of both materials at the interface, the following general equation for the curvature is obtained Timoshenko et al. (1925):

\[ \frac{1}{R} = \frac{6 ( \alpha_2 - \alpha_1 ) \Delta T (1 + \Upsilon)^2}{\Theta II (3(1 + \Upsilon)^2 + (1 + \Upsilon \Gamma) (\Upsilon^2 + \frac{1}{\Upsilon \Gamma})))} \]  

\[ \frac{1}{R} = \frac{6 ( \alpha_2 - \alpha_1 ) \Delta T (1 + \Upsilon)^2}{\Theta II (3(1 + \Upsilon)^2 + (1 + \Upsilon \Gamma) (\Upsilon^2 + \frac{1}{\Upsilon \Gamma})))} \]
The curvature is proportional to the difference in elongation of the materials and inversely proportional to the thickness of the strip Timoshenko et al. (1925). As mentioned earlier shear stress in concentrated near the ends of the strip, however Timoshenko states that the magnitude of peak shear stress can have the same order as the peak of tangential bending stress $\sigma_{\text{max}}$ occurring in the layer directly adjacent to the interface, which is given by:

$$\sigma_{\text{max}} = \frac{1}{R} \left( \frac{2}{TTH \, b \, th_1} (E_1 \Pi_1 + E_2 \Pi_2) + \frac{th_1 E_1}{2} \right).$$  \hspace{1cm} (6.6)$$

As elaborated in Chapter 5 in the development of stress in our study is due to hygroscopic swelling, hence temperature change $\Delta T$ is substituted by moisture concentration change $\Delta C$. Accordingly, $\alpha$ is substituted by the hygroscopic expansion coefficient, HEC.

Moreover, analyzing the experimental results in terms of deflection seems to be more practical than curvature $1/R$. In Fig. 6.2 a bi-material strip, simply supported at the ends, is shown in uniform bending condition. The curve ACB is an arc of a circle with radius of $R$, therefore it leads to $\delta(2R - \delta) = (l/2)^2$. It is assumed that the deflection is small in comparison to the radius of curvature. From which:
6.3 Experiment

Considering the fact that mechanical properties of plastics can change dramatically when certain additives are incorporated into the composition, it was decided to experimentally determine the Young’s modulus of EVA and glass rather than relying on available data in the literature. Tensile test and micro-indentation were carried out to obtain the Young’s modulus for EVA and glass respectively.

EVA tensile testing was performed in compliance with the relevant section of ASTM D638 standard Standard (2010). This part of ASTM D638 specifies the test conditions for determining the tensile properties of unreinforced and reinforced plastics. Dumbbell type specimens were prepared from the EVA film cured in identical conditions as the EVA in bi-material strips. The test provides mechanical properties such as ultimate strength, strain at failure, and modulus. The test procedure is enhanced by the use of a camera to track the changes of cross-sectional area.

The Young’s modulus of glass was determined using a micro-indentation machine. Obtaining hardness and elastic modulus by indentation techniques has widely been adopted and used in the characterization of mechanical behavior of materials. The approach works based on indentation load–displacement data obtained during a cycle of loading and unloading.

Eventually the centerpiece of this part of experimentation is the bi-material strip swelling-induced deflection measurement and analysis. Pertinent explanation regarding the preparation of specimens, test setup and data analysis is provided in the following sub-sections.

6.3.1 Young’s modulus measurement

Experimental apparatus

An Instron 4466 was used to perform the modulus test. Tensile test specimens were prepared in accordance to ASTM D638 TYPE IV from cured EVA film with a width of the narrow section of 6 mm. Curing conditions similar to the standard lamination
Hygroscopic swelling induced stress in bi-material EVA/glass strip

process (holding 150°C laminator temperature for 11 min) was used on a hot press machine to prepare 0.6 mm thick film between two release films. Since EVA is a kind of adhesive it was not possible to cure the specimen directly in the dog bone shape mould. Alternatively, specimens were cut out of the film. The specimen is shown in Fig. 6.3(a). A high-definition (HD) video camera was used to record the test for strain analysis, providing resolution of 1920 × 1080 (2.1 megapixel). The testing setup consists of a tensile testing machine and video camera is shown in Fig. 6.3(b).

![Figure 6.3: (a) Tensile sample in accordance with ASTM D638 TYPE IV, (b) Tensile test set up with camera.](image)

Tensile test sample was carried out on specimens kept in two environmental conditions (20°C -10%RH for 30 days, 20°C immersed in water for 20 days). The crosshead speed provides extension rates of approximately 10 mm/min. The recordings from the camera where timed with logged data on the tensile machine and used to determine longitudinal and transverse strain. Google-Sketchup, a simple freeware to extract distances in image, was used to perform strain analysis. The Young’s modulus is calculated from of the elastic region of the complete tensile test shown in Fig. 6.4 for one of the specimens. Based on Fig. 6.4 our elastic region is \( \varepsilon < 0.2 \), as it was observed through the test that the material quickly retracts to its original shape if exposed to strain within this domain. It is in compliance with the results presented by Eitner (2011).
The poisson’s ratio was also determined using the strain in the longitudinal and transverse directions measured with camera. The Stress-Strain curves of the elastic region are plotted in Fig. 6.5 (a) and (b) which demonstrate the behaviour of material in dry and wet condition respectively. In addition, the transverse strain vs. tensile strain graphs are shown in Figs. 6.5 (c) and (d) again for dry and wet conditions. The reason for discontinuity of the curve is that the extension was obtained from recorded video manually. Considering the limitations in reading the position of markings on the surface of the specimen due to image resolution, some discontinuity in extension reading occurred. This is more pronounced in Fig. 6.5 (c) and (d) as data in both axes are obtained from camera images. It means that the edge displacement shows intermittent jumps from one pixel to another. Based on the camera resolution and meter-stick the accuracy of measurement was 0.17 [mm] which translate to systematic uncertainty or error of 0.017 in $\varepsilon_t$ and in 0.0028 in $\varepsilon_l$. Because of the nature of the intended material parameters, obtaining a cloud of points could lead to reasonable accuracy through curve fitting, hence implementing a sub-pixel image-processing was not undertaken. The values of the elastic parameters $E$ and $\nu$ for each condition are extracted using a linear fit and summarized in Table. 6.1. The variation of results between the tests is in part due to systematic uncertainty of measurement and also randomness of material behaviour.
Figure 6.5: Stress-Strain curves of EVA in the elastic region: (a) 20°, 10%RH, (b) 20°, immersed in water. Transverse strain vs. tensile strain curves of EVA, (c) 20°C, 10%RH, (d) 20°C, immersed in water for 20 days.
Table 6.1: The values of the elastic parameters $E$ and $\nu$ for three specimens in two environmental conditions

<table>
<thead>
<tr>
<th></th>
<th>$20^\circ C, 10% RH$</th>
<th>$20^\circ C, in water$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E \ [MPa]$</td>
<td>9.0458</td>
<td>6.5501</td>
</tr>
<tr>
<td></td>
<td>8.3236 Ave: 8.56</td>
<td>7.4722 Ave: 6.98</td>
</tr>
<tr>
<td></td>
<td>8.3236</td>
<td>6.9472</td>
</tr>
<tr>
<td>$\nu$</td>
<td>0.3868</td>
<td>0.4068</td>
</tr>
<tr>
<td></td>
<td>0.3566 Ave: 0.38</td>
<td>0.3831 Ave: 0.39</td>
</tr>
<tr>
<td></td>
<td>0.4024</td>
<td>0.4029</td>
</tr>
</tbody>
</table>

The Young’s modulus of glass was obtained from a micro-indentation test. The glass was tested using a Micro Materials Ltd NanoTest equipment (MML NanoTest). The equipment used is a static system that functions with a horizontal loading mechanism. The micro-indentation tests were performed using a Vicker’s indenter with the applied load of 100 $[mN]$.

Slope of the load-depth curve during unloading stage of indentation is an indication of the contact stiffness. Generally this stiffness includes a contribution from both the tested material and the response of indenter itself. The stiffness of the contact can be used to calculate the reduced Young’s modulus $E_r$ Oliver and Pharr (2004). The Young’s modulus of the specimen $E_s$ is then calculated from the reduced elastic modulus $E_r$ and indenter modulus $E_i$ using the following relation ISO14577 (2007):

$$E_s = \frac{1 - \nu_s^2}{\frac{1}{E_r} - \frac{1-\nu_s^2}{E_i}}$$  \hspace{1cm} (6.8)

where $\nu_s$ and $\nu_i$ are the Poisson’s ratios for specimen and indenter respectively. Based on Eq. 6.8 and considering glass Poisson’s ratio $\nu_s = 0.23$ Chorfa et al. (2010), a Young’s modulus of 95 $[GPa]$ was obtained for the glass measured in our experiments.

### 6.3.2 Bi-material strip deflection test

For the purpose of this test, a number of bi-strip samples were prepared in a similar process to the WSI samples. For these samples a complete coverage of EVA on one side of the glass was crucial, therefore slightly different arrangement of spacers and holders were prepared. Layers of EVA virgin films were used on top of glass strip held between different set of spacers to achieve intended lamination thicknesses as shown in Fig. 6.6.
Standard curing process with 150°C temperature hot press machine was applied for 11 min. The thickness and dimensions of samples are listed in Table 6.2.

![EVA-glass bi-strip ready for curing in the hot press.](image)

**Figure 6.6:** EVA-glass bi-strip ready for curing in the hot press.

<table>
<thead>
<tr>
<th>Set</th>
<th>$t_{EVA}$ [mm]</th>
<th>$t_{glass}$ [mm]</th>
<th>Thickness ratio</th>
<th>Dimensions [mm]</th>
<th>N of samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Set 1</td>
<td>1.97</td>
<td>2.08</td>
<td>0.947 $\cong$ 1</td>
<td>10 × 90</td>
<td>3</td>
</tr>
<tr>
<td>Set 2</td>
<td>1.32</td>
<td>2.08</td>
<td>0.634 $\cong$ 0.6</td>
<td>10 × 90</td>
<td>3</td>
</tr>
<tr>
<td>Set 3</td>
<td>1.92</td>
<td>1.05</td>
<td>1.83 $\cong$ 2</td>
<td>26 × 75</td>
<td>3</td>
</tr>
</tbody>
</table>

Fig. 6.7 shows a bi-strip sample prepared following the described procedure. A set of three specimens was prepared for each intended thickness. The difference in elongations plays the main role in bi-strip curvature, however according to Eq. 6.5, thickness and
Young’s modulus ratios can both influence the results. Therefore three different thickness combinations for glass and EVA were picked to enable us to observe which combination would better work for us.

Figure 6.7: Bi-material sample.

The bi-strip samples remained in a desiccator for 1 month (at 20°C, 10%RH) before a dry reference measurement. The surface of each strip was then scanned to have a reference along the length direction using non-contact optical profilometer (TAYLOR HOBSON). This provided data to determine the curvature and subsequently deflection of each specimen after moisture absorption, similar to the schematic shown in Fig. 6.2. A sample positioned in optical profilometer is shown in Fig. 6.8. In order to perform a consistent set of measurements all along the experimentation, the same line was scanned every time by placing the sample against a fixed bracket on the stage and running a predefined routine on the machine.

Figure 6.8: Optical profilometer while sample scanning.
Samples were then immersed in distilled water as shown in Fig. 6.9. They were removed from the immersion chamber periodically to take measurements over the course of a 23-day experiment. Upon removal of the sample from the immersion chamber, it was air dried and the surface scanned from both glass and EVA side. The glass side scan provided poor and scattered results in terms of surface profile, this was due to strong reflections from the glass surface.

![Figure 6.9: Bi-strip samples in an immersion chamber.](image)

### 6.4 Data analysis and results

Following the experiments, a set of surface profiles are obtained. These raw data requires a few steps of processing including leveling, isolating the nuance in deflection and noise filtering. These steps are illustrated in Fig. 6.10 as an example. Levelling is an important part of processing because there could be a small misalignment compared to the profilometer horizontal reference plate due to for example dust under the sample or even non uniformity in EVA thickness. This stage is performed by identifying where sample surface starts and ends (Fig. 6.10 (a)) and removing the linear slope -see Fig. 6.10 (b). This levelled profile involves a contribution of initial curvature in the dry sample (nearly 90%) which comes from the residual stress of fabrication process, therefore the pertinent initial profile, which was also leveled, was taken off this profile to isolate the deflection merely coming from moisture ingress. The result which shows the deflection after slope and DC subtraction is presented in Fig. 6.10 (c-top). This step is further elaborated in subsection 6.4.2. A convolution filter is finally employed to remove noise and eventually obtain the curve shown in Fig. 6.10 (c-bottom).

It is important to note that since the above technique levels and zeros the base line of the profile based on the two top corner points of the surface profile (Fig. 6.10 (a)), the surface swelling is taken off and the presented final deflection profile shows merely the
As mentioned earlier three set of results were obtained for each thickness combination from the EVA side of bi-strip. Fig. 6.11 shows the accumulated deflection profiles obtained along the length direction for Set 1 over the course of the experiment. These results highlight the transition of deflection and the underpinning swelling phenomenon. Deflection profiles of Set 2 are provided in Fig. 6.12. Comparing both set of results it is visible that the final deflection is somehow proportional to the thickness ratio, which speaks to the fact that more material while swelling could impose higher stress and consequently more deflection on bi-strip.
Figure 6.10: Bi-strip experimental data processing steps: (a) raw data, (b) surface profile after leveling, (c-top) surface profile after slope and DC subtraction, (c-bottom) after noise filtering.
Figure 6.11: Accumulated deflection profiles obtained along the length direction for Set 1 (EVA Thickness = 1.97 [mm] Glass thickness = 2 [mm]).
Figure 6.12: Accumulated deflection profiles obtained along the length direction for Set 2 (EVA Thickness = 1.32 [mm] Glass thickness = 2 [mm]).
6.4 Data analysis and results

Figure 6.13: Deflection values picked up from a central band of surface profile curve with STD error for Set 1 (EVA Thickness = 1.97 [mm] Glass thickness = 2 [mm]).

The results obtained from Set 3 were unfortunately too poor in terms of quality of the signal to be analysed, therefore they were ignored. Another representation of the experimental results for Sets 1 and 2 are provided in Fig. 6.13 and 6.14. These two graphs show the deflection values picked up from a central band in Figs. 6.11 and 6.12 along with STD error bar and would be used for stress calculations.

Figure 6.14: Deflection values picked up from a central band of surface profile curve with STD error for Set 2 (EVA Thickness = 1.32 [mm] Glass thickness = 2 [mm]).

Eventually the experimental results bring us to the stress calculation part. Using Timoshenko’s formulation Timoshenko et al. (1925), since \( \delta \) (deflection) is small compared to the radius of curvature \( R \), it could be calculated from Eq. 6.7. Curvature
then leads to other parameters which are tabulated in Table 6.3 for Sets 1 and 2. The resultant shear stress between two layers cannot be calculated in an elementary way Timoshenko et al. (1925) as they are concentrated near the end of the strips, whereas no shear stresses occur at the interface in the center of the strip. However the magnitude of the maximum value of the shear stress can be estimated. According to Timoshenko Timoshenko et al. (1925), the shear stress may be of the same order as the maximum tangential bending stress $\sigma_{\text{max}}$ occurring in the layer directly adjacent to the interface, which is given in Eq. 6.6.

Table 6.3: Mechanical response to hygroscopic swelling of EVA/glass bi-strips.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Set 1</th>
<th>Set 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>EVA thickness: $h_1$ [m]</td>
<td>$1.97 \times 10^{-3}$</td>
<td>$1.32 \times 10^{-3}$</td>
</tr>
<tr>
<td>Glass thickness: $h_2$ [m]</td>
<td>$2 \times 10^{-3}$</td>
<td>$2 \times 10^{-3}$</td>
</tr>
<tr>
<td>$\delta$ [m]</td>
<td>$10.29 \times 10^{-6}$</td>
<td>$5.35 \times 10^{-6}$</td>
</tr>
<tr>
<td>$R$ [m]</td>
<td>98.39</td>
<td>189.25</td>
</tr>
<tr>
<td>$\sigma_{\text{max}}$ [Pa]</td>
<td>$1.56 \times 10^5$</td>
<td>$1.44 \times 10^5$</td>
</tr>
</tbody>
</table>

6.4.1 FEA simulation and results

In this subsection, FE simulation of EVA/glass bi-strip is presented. The idea is to compare the experimental results and estimated ones from FE so as to complement our observation and the analysis technique used to extract the deflection. Fig. 6.15 (a) shows the model developed using the same technique as elaborated in Chapter 5. Geometry is generated in MSC.Marc based on the dimensions provided in Table 6.2 in a plane-strain analysis. A glue contact is used to define the EVA/glass interface. Simply supported boundary condition is applied to the model and material properties as specified in Chapter 5 is assumed for EVA and glass. However as HEC is calculated using four different methods according to Table 5.2, the analysis for each geometry set is performed for all 4 values. Fig. 6.15 (b) shows the final deflections obtained from FE simulation. The graph shows the displacement profile of the EVA/glass interface along the sample, which could represent pure deflection in the bi-strip. The corresponding average experimental deflections (out of three samples) are also presented for each set.

Fig. 6.15 (b) generally shows a close agreement with the experimental results presented in Figs. 6.13 and 6.14. It is visible that the HEC values calculated through presented inversion method (Chapter 5) result in a very close estimation of the actual deflection
6.4 Data analysis and results

Figure 6.15: (a) 2-D FE bi-strip deflection mode, (b) Deflection results from FEA based on different HEC values presented in Chapter 5 for Set 1 (left) and Set 2 (right).

for set 2. As for set 1 experimental curve is closer to \( h_{sat} \), however it seems that the difference is coming from the approximations used in experimental data analysis process. The overall agreement between FEA and experimental measurements shows that the presented inversion approach, that calculates HEC as a function of water concentration, provides a reasonably reliable estimate of \( h(C) \).

The shear stress distribution is also obtained from FEA for both sets (Fig. 6.16). The alanythical Timoshenko’s estimate of the peak shear stress presented in Table 6.3 is close to the one predicted by FEM.
Hygroscopic swelling induced stress in bi-material EVA/glass strip

6.4.2 Distinguishing deflection

In order to calculate the experimental deflections it was needed to distinguish the swelling from deflection in the results. However there was lack of data on how much swelling is contributing to the surface profile readings. FEM was used to help us understand the correlation between swelling and deflection. Fig. 6.17 shows the approach. FE de-
6.5 Conclusion

The present chapter investigated the effect of hygroscopic swelling through observation of hygroscopic swelling induced deflection in bi-material strip samples immersed in water. The deflection of EVA-glass bi-strips due to development of swelling stress upon moisture uptake was measured over the course of three weeks. The changes of curvature were recorded periodically by scanning the surface of the sample with an optical profilometer. In order to do the calculations Young’s modulus of EVA and glass were determined using tensile and micro indentation tests respectively.

To obtain deflection, the data from profilometer was processed through several steps. As part of this analysis FEM was used to help distinguish deflection from swelling in the surface profile. This independent observation on the effects of hygroscopic swelling in bi-strips with different thickness ratios, were also simulated in FEM using HECs obtained in Chapter 5. A close agreement is found between these measurements and simulated deflections in FEM. This verifies that the FE model and parameters used to describe the material are correct.

As mentioned earlier in the thesis, the build-up of stress from number of contributing parameters including thermal, hygroscopic and mechanical loading, could eventually affect structural integrity of the system over time. Therefore in order to observe the effect of hygroscopic swelling induced stress in the PV module, development of a comprehensive FE modelling including all components, interactions, supports and loads is required. The presented shear stress study however, intended to observe the correlation between the analytical estimation and FEA.
Chapter 7

Conclusions and future work

7.1 Conclusions

The present study focused on the development of methods and techniques to study the mechanical behaviour of PV encapsulant materials due to water diffusion, as well as quantification of water concentration. As a common encapsulant, Ethylene Vinyl Acetate (EVA) was taken as a particular case.

A novel non-contact time resolved measurement technique is proposed based on Wavelength Scanning Interferometry (WSI) to determine thickness, refractive index, thickness change and refractive index change in EVA due to water uptake. This was preceded by a numerical simulation to inform the design of the experimental set up details, such as thickness of EVA layer, maximum surface velocity in measurement, type of filter to use for data analysis of results and also validating the approach. The time resolved thickness change profile provided unique opportunity to take the methodology in swelling behaviour study of EVA to the whole new level by introducing an inversion technique, proposed to establish the hygroscopic expansion coefficient as a function of water concentration. A Hygro-Mechanical FE model also developed to evaluate the HEC results. The Hygro-mechanical stress is further studied through a set of bi-strip EVA-glass exposed to moisture. This independent observation on the effects of hygroscopic swelling in bi-strips with different thickness ratios, also simulated in FEM using HECs obtained from inversion method.

The proposed WSI based technique is developed to determine time resolved thickness change of EVA film over a substrate. A mathematical framework of the technique and a simulation case study to probe its feasibility for thickness change monitoring of our intended material was presented. This simulation takes advantage of a forward model that takes a hypothetical surface deformation as an input and replicates the
Conclusions and future work

intensity signal reflected from illuminated sample surface. This is followed by an inverse model that evaluates the surface deformation from the interference intensity signal. The system assumes tunable laser to illuminate a layer of EVA laminated onto a reflective substrate. The interference signal between light reflected at different interfaces and a reference wavefront is modulated with temporal frequencies proportional to the optical path difference between them. Fourier transformation of the interference signal leads to peaks that locate the position of the interfaces. The refractive index and thickness of the EVA layer determined by evaluating changes in wavelength from measuring the phase change. The simulation shed light on the limitations of the method in terms of the sample geometry and also detectable rate and range of the surface deformation which served us to design the experimental setup, sample configuration, data acquisition parameters and data analysis.

The above was pursued by the actual test and analysis to demonstrate the applicability of the methodology. In verification, stage using piezoelectric translator to produce a controlled input in the form of displacing surface, a very good agreements between the input and estimated displacement were observed. In analysing the thickness change from experimental recorded data at the detector, a further level of improvement was incorporated in the analysis by using specific methods to identify the optimized position of peaks. Another key parameter was filter’s width which could be cause of cross talk between each identified peak in amplitude signal. The effect of phase change on changing refractive index of water as a medium, was addressed and the variation of refractive index of water in measurement time was extracted. Similar analysis for EVA also identified the refractive index change of the polymer during the progression of water diffusion.

The Hygro-mechanical swelling behaviour of Ethylene Vinyl Acetate (EVA) was also investigated. Taking advantage of a time-resolved thickness profile measurement on EVA film laminated on glass, a numerical formulation was to find an approximation of the HEC as a function of water concentration. It is expected that this approach would improve the predictive power of forward FE models that evaluate mechanical stress in response to water ingress. In addition the proposed technique uses of a single measurement of swelling behaviour from dry to saturated conditions to save the experimental effort of having to measure samples saturated at different %RH which would otherwise take far too long for samples exposed to humidity especially at low %RH when the saturation is difficult to detect with marginal water uptake in the material. The application of this technique to investigate the HEC as a function of concentration is simply expandable to other polymers and encapsulant.
Further investigation on the effect of hygro-mechanical material behaviour was carried out through observation of hygroscopic swelling induced deflection in bi-material strip sample exposed to humid condition. In the experiments EVA-glass bi-strip bent due to development of swelling stress upon moisture uptake over the course of three weeks. The changes of curvature were recorded periodically by scanning the surface of the sample using an optical profilometer. Maximum shear stress values occurring at both ends were estimated using the measured deflection. In order to do the calculations Young’s modulus of EVA and glass were determined using tensile test and micro indentation test respectively.

It is important to point out that as explained in pertinent chapters, all the water diffusion experiments are carried out in room temperature and through immersion. These testing conditions vary from the working temperatures, RH levels, or different ageing conditions which an actual PV system might be exposed to. This speaks to the fact that presented techniques are a step towards studying wider range of parameters and underpinning interactions, figures that could act as proxies of each other, e.g. water uptake level and nuances in NIR spectral response, or refractive index of EVA.

Another phenomenon which was not in the scope of our consideration is material reaction to desorption. Based on a number of researches, diffusion coefficient $D$ would not be the necessarily the same in sorption and desorption. Again the underlying reason is the intention to concentrate on characterization methodology rather than full EVA hygroscopic response.

Finally to summarize all the tests carried out in this research together with their pros and cons Table 7.1 is presented. Clearly the limitations highlighted in this table are the items relevant to this research.
<table>
<thead>
<tr>
<th>Method</th>
<th>Measured parameter</th>
<th>Advantages</th>
<th>Limitations</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>1. Time resolved output</td>
<td>1. Phase slope reconstruction is limited by filter bandwidth</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2. Measurement of thickness and refractive index and their changes simultaneously</td>
<td>2. Filter bandwidth is limited by the spectral proximity of peaks which correspond to different interfaces in the sample</td>
</tr>
<tr>
<td>WSI (Ch. 4)</td>
<td>EVA thickness, refractive index</td>
<td>3. Suitable for transparent and flexible materials</td>
<td>3. Maximum detectable thickness change rate is related to $\frac{N}{N_2}$ as well as $\Delta k$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4. Non contact and non destructive</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>5. Suitable in different environmental condition (temperature and humidity)</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>6. Suitable for laminated encapsulant material on glass</td>
<td></td>
</tr>
<tr>
<td>Optical profilometry</td>
<td>EVA thickness, deflection</td>
<td>1. Non contact and non destructive surface measurement</td>
<td>1. Sample size limitation (not larger than $10 \times 10 [cm]$) due to measurement table size</td>
</tr>
<tr>
<td>(Ch. 4 &amp; 6)</td>
<td></td>
<td>2. Accurate, and fast</td>
<td>2. Pre-set for operation in air as medium and not suitable for surface immersed in water</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>3. Drying and displacing the sample after removal from water makes the measurement susceptible to error</td>
</tr>
<tr>
<td>Method</td>
<td>Measured parameter</td>
<td>Advantages</td>
<td>Limitations</td>
</tr>
<tr>
<td>------------------------</td>
<td>--------------------</td>
<td>------------------------------------------------</td>
<td>----------------------------------------------------------------------------</td>
</tr>
<tr>
<td>Gravimetric (Ch.5)</td>
<td>Water concentration</td>
<td>1. Simple and fast technique</td>
<td>1. Drying and displacing the sample after removal from water makes the measurement susceptible to error</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2. Non destructive</td>
<td>2. Sample handling should be performed very delicately</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>3. Based on scale resolution, sample exposure area should be calculated</td>
</tr>
<tr>
<td>Abbe refractometer (Ch.4)</td>
<td>Refractive index</td>
<td>Simple and quick operation</td>
<td>1. Intermittent measurement</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>2. A single value of the refraction for whole surface is obtained</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>3. Using laminated sample is not possible</td>
</tr>
<tr>
<td>PZT / WSI (Ch.4)</td>
<td>Displacement</td>
<td>1. Applying a controlled displacement in range of µm, using a controlled input voltage</td>
<td>Requires stringent calibration in both directions (expansion and retractions)</td>
</tr>
</tbody>
</table>
7.2 Recommendations and future Work

It is important to note that for any PV module durability and failure predictive models, understanding material behaviour in the presence of water is crucial. The present study was an effort to introduce new techniques for material characterization. Future works and applications can be built from this study to expand the knowledge of polymeric material behaviour in humid environment and yield more robust products not only in PV industry and also other areas such as in microelectronic packaging applications.

This applications could include studying various PV encapsulant materials, e.g. polyvinyl butyral (PVB), silicone rubber, TPU, fluoropolymers (such as PVF, PTFE, ECTFE and ETFE), PMMA, thermoplastic elastomers, EPDM, polyamide and PET with these techniques. This method can be used for various environmental and ageing conditions like temperature, relative humidity and UV radiation. With the earlier works having shown the difference between absorption and desorption, studying properties of EVA and other encapsulants, would be a logical extension to this work.

Future contributions are needed to advance the understanding of the link between hygroscopic swelling coefficient and moisture concentration and plausible influence of temperature. The presented results in this research demonstrated that this fast technique is able to provide valuable information on the relation between the material swelling and diffusion behaviour.
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