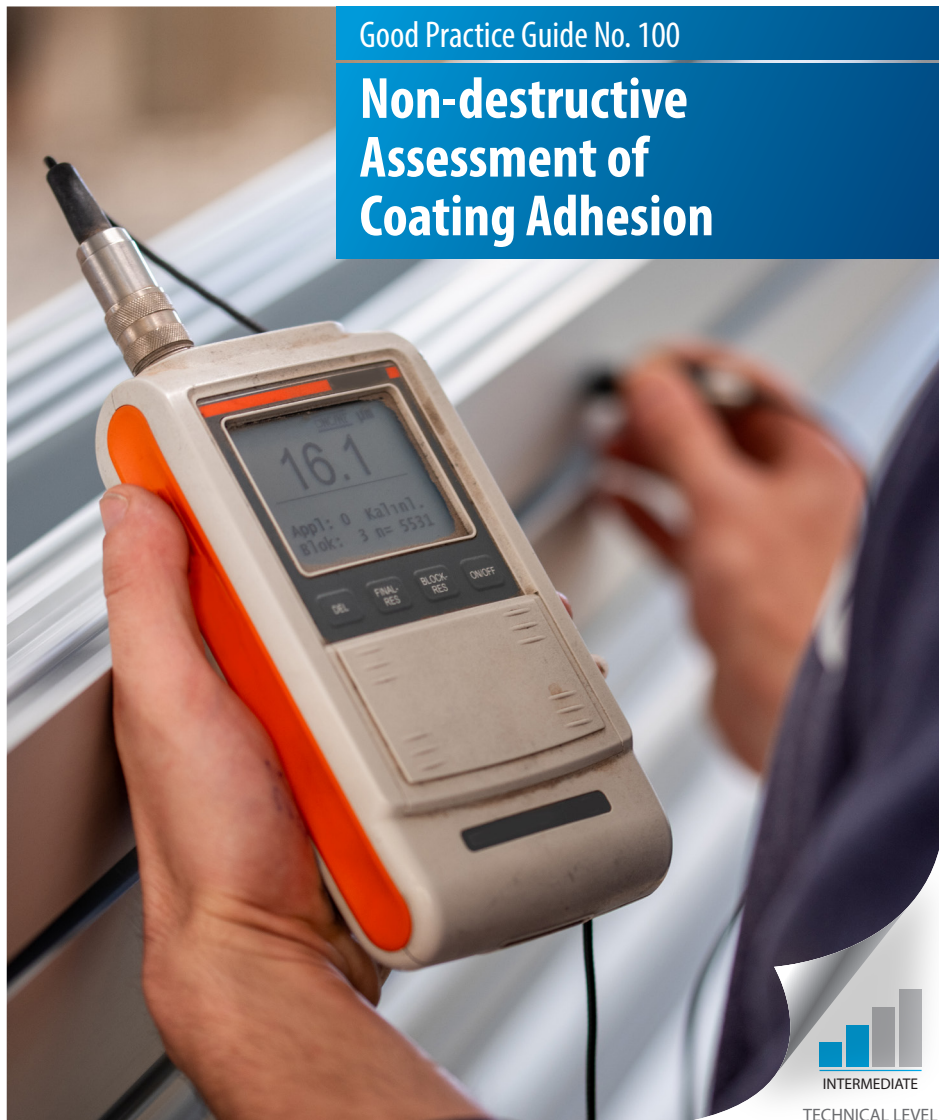


Good Practice Guide No. 100

Non-destructive Assessment of Coating Adhesion



INTERMEDIATE

TECHNICAL LEVEL

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Abstract

This document is a guide to the non-destructive assessment of coating adhesion both for the purposes of manufacturing quality assurance and onsite inspection. The different non-destructive techniques that are available are described highlighting their advantages and limitation. The key factors that should be taken into account when selecting a technique, for a particular application, are covered and short protocols on the use of the most promising techniques are provided.

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Chapter 1: Introduction

1. Introduction

Surfaces, surface engineering and coatings form the key interfaces between components and their service environments. Surface performance is critical to almost every engineering material and the ability to apply a durable coating (functional and/or decorative) is a key requirement. It is critical that regions of poor adhesion between a substrate and a coating or adhesive can be determined reliably for quality assurance and for onsite inspection.

The development of non-destructive test methods for coating adhesion are most important for functional engineering coatings. These are coatings that protect components from mechanical damage such as abrasion, erosion, adhesive wear and fatigue; and from chemical attack such as galvanic corrosion in aqueous media and high temperature corrosion and oxidation resulting from combustion and chemical processes. The greatest need for NDE techniques are for quality assurance purposes during manufacture; and for assessment of components in service, where fretting (a form of contact fatigue), general component fatigue and corrosion undermine coating adhesion. Coating adhesion weaknesses can occur at the time of application and during service. At the time of application they could be due to inadequate surface preparation, or poorly controlled application parameters, which in turn could lead to flaws such as micro-cracking and porosity, or inadequate thickness. In service, corrosion and fatigue may cause failure of a coating, as may general degradation of the coating over time (for example by migration of molecules within the coating). These may not be strictly a loss of adhesion, but are nevertheless highly important to users of the coating.

Currently a wide range of different adhesion measurement techniques are used in industry [1] however these are all destructive and a non-destructive technique is of extreme interest to industry. Many non-destructive techniques have been proposed for the measurement of coating adhesion and there is a need to evaluate the suitability of these different techniques.

Over the last two years a wide range of techniques have been examined at NPL and TWI and the results of this study are summarised here. A couple of techniques that were initially considered promising were excluded after evaluation these included:

- Electronic Speckle Pattern Interferometry (ESPI)
- Digital Image Correlation Strain Mapping

These techniques measure the strain in coatings that is produced by either mechanical or thermally stresses into the coating. By comparing the strain that is produced in a well-adhered coating to that in a poorly adhered or delaminated coating it was originally thought that it would be possible to identify poorly adhered coatings. Unfortunately, the differences in strain were not sufficiently large to detect poor adhesion or even delamination. These techniques have therefore not been included in this guide.

The techniques that are included in the guide include:

- Static thermography
- Flash thermography
- Ultrasonics
- Scanning acoustic microscopy
- Acoustic emissions
- Laser surface acoustic wave
- Terahertz spectroscopy

These techniques are described in Chapter 2, with examples of how they have been used to examine delamination or poor adhesion in a range of different coatings. Key factors that should be taken into account when selecting one of these techniques are discussed in Chapter 3 and short protocols on the use of these techniques are provided in Chapter 4.

Chapter 2: Techniques

- Static thermography
- Flash thermography
- Ultrasonic testing
- Scanning acoustic microscopy
- Acoustic emission
- Laser surface acoustic wave
- Terahertz

2. Techniques

2.1 Static Thermography

Static thermography techniques involve heating and thermally imaging coatings to detect regions of differential heating or cooling caused by defects or poor adhesion. It can be used for rapid inspection of large structures and is capable of detecting gross defects such as delamination of coatings. The technique requires the inspected component to be heated to produce a surface temperature distribution that can be correlated with a structurally intact specimen. In all cases the spatial and temporal temperature distribution is measured using infrared imaging CCTV cameras, and analysed either in real time or post processed to find hidden features. The cameras with best thermal resolution are cooled either with Peltier coolers or with liquid Nitrogen. Conductive heating through the substrate and radiant heating of the coating's surface are both possible and are described below.

2.1.1 Conductive heating method

Conductive thermography involves heating the substrate of the component and monitoring the heat that passes through the coating. This involves heating the specimens on an electric hot plate and allowing them to cool down under the infrared camera (Figure 1). If the coating is well adhered to the substrate the material should cool uniformly and the thermal image appear uniform. If, however, there is delamination or other defects within the coating the heat conducted through the coating will be reduced and that part of the specimen will appear slightly cooler than the surrounding area.

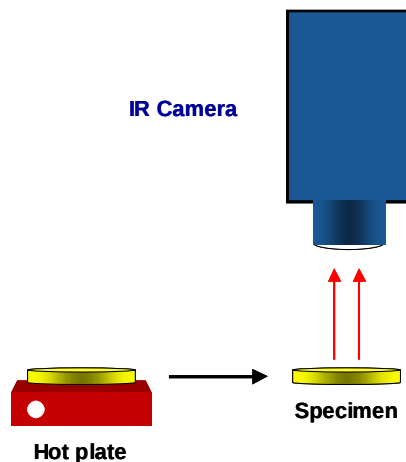


Figure 1 Schematic diagram of the conductive heated method.

2.1.2 Radiated method

When a coated specimen is irradiated with infrared radiation (Figure 2), the surface of a defect free coating should warm up uniformly. However, if there are defects in the coating the thermal image will appear non-uniform. In the case of delamination the surface will heat up faster than the surrounding area, as heat in the delaminated region will not be able to diffuse into the substrate beneath, as it will no longer be in thermal contact.

The difference in the results obtained from the two techniques is clearly shown in Figure 3. A hydroxyapatite coated stainless steel specimen with a defect caused by a fingerprint has been imaged using both techniques. The results obtained clearly show how the defect causes the image to appear cooler using the conductive method but hotter using the radiated method.

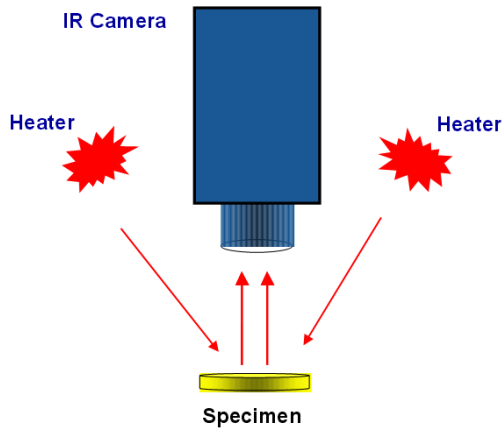


Figure 2 Schematic diagram of a simple radiation method.

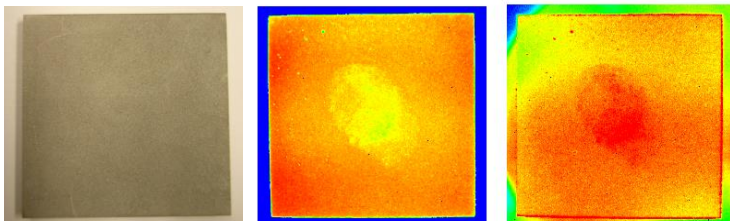
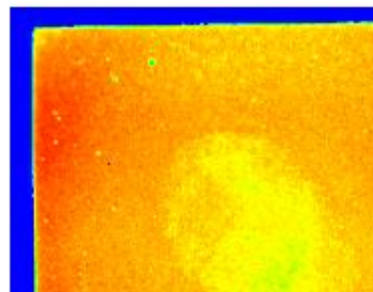


Figure 3 Results obtained using conductive and radiated h



2.1.3 Case study: Static thermography of turbine blades

Static thermography has been used to study a PVD coated turbine blade that has been oxidised prior to coating to reduce the coating adhesion. The titanium blade was initially grit blasted and then oxidised by heating it in a furnace at 500 °C for 15 hours. The middle section of the blade was then again grit blasted to return it to its original state (Figure 4a). The blade was then coated with a chromium nitride using physical vapour deposition (PVD), to produce a visibly uniform coating (Figure 4b). An image of the blade obtained using conductive heating thermography is shown in Figure 4c. In order to visualise differences in temperature the image has been artificially coloured using a range of colours from blue to red. Blue representing the coldest regions in the image and red the hottest. Regions where the coating is poorly adhered to the substrate can clearly be seen to be colder than where the oxide has been removed to provide a good bond for the coating (i.e. blue or green rather than yellow or red).

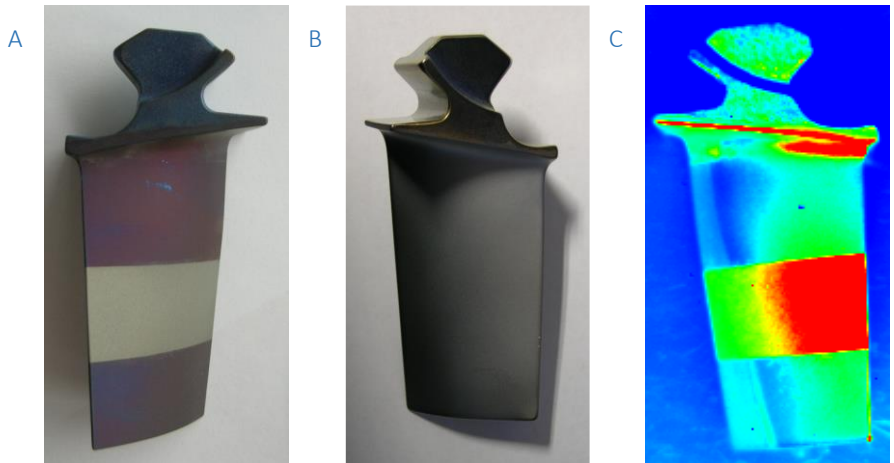


Figure 4 Images of a chromium nitride coated turbine blade, A) oxidised turbine blade with central region grit blasted, B) PVD coated blade, C) thermogram of coated blade

2.2 Flash Thermography

In flash thermography, a brief pulse of light is used to heat the surface of a sample rather than using continuous heating. As the sample cools, thermal radiation from the surface of the specimen is monitored as a function of time using the ThermoScope PT system (Figure 5). Defects that can cause poor adhesion at the coating/substrate interlayer such as delamination, voids or inclusions, obstruct the flow of heat through the coating into the substrate. As a consequence the surface temperature above such defects is increased enabling the defects to be identified (Figure 6).



ThermoScope™ PT system – (supplied by LOT -Oriol)

Figure 5 Experimental set up used in flash thermography

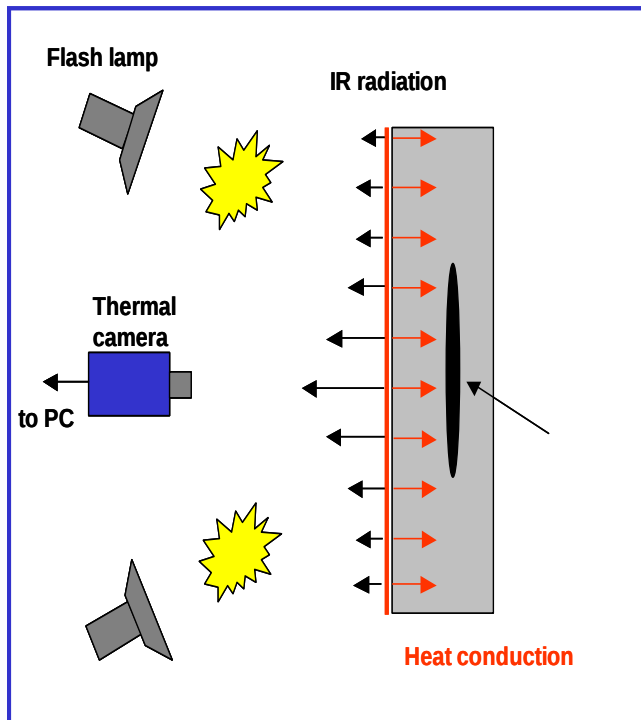


Figure 6 Schematic illustrating how defects are detected in flash thermography

2.2.1 Case study: Flash thermography of chromium nitride coatings

Flash thermography has been examined using five stainless steel specimens coated in chromium nitride (Figure 7). The adhesion of these coatings has been controlled by treating the surface of the substrates prior to applying the coating, to disrupt the chemical bonding between the coating and the substrate. Results obtain from the flash thermography on the PVD coated specimens are shown in Figure 7. The graphs chart the heat that is radiated by the specimens as a function of time following the initial heat pulse. As can be seen the poorly adhered coatings are significantly hotter than the well-adhered coatings. Clearly demonstrating how flash thermography can be used to assess the adhesion strength of coatings.

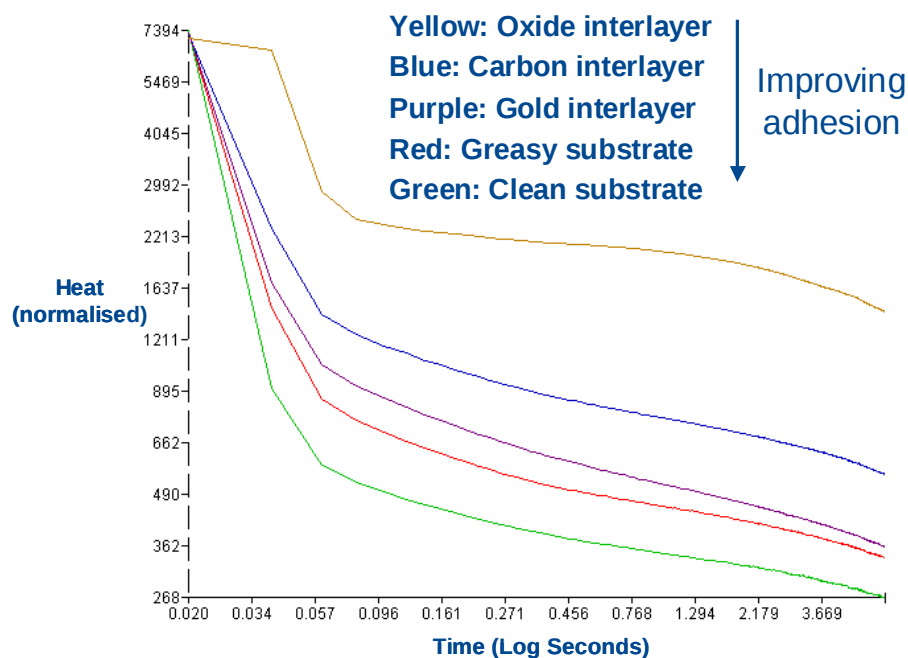


Figure 7 Heat dissipation as a function of time in flash thermography for five chromium nitride coated specimens

2.3 Ultrasonic Testing

Ultrasonic inspection techniques are widely used for the non-destructive evaluation of materials. The techniques can be used to detect, measure and characterise a wide range of manufacturing and in-service defects, including the type of delamination that often results from poor adhesion of coatings. The main method of ultrasonic inspection uses a conventional ultrasonic thickness tester, which gives an A-scan presentation of signal versus time. The reflected signals can be analysed in a number of ways. Figure 8 shows a frequency analysis. Ultrasonic C-scan inspection can be used to produce a planar image map corresponding to the surface of the object by measuring the amplitude of the signal at some point after the surface reflection. A scanning system is required to produce C-scan images and is often used for manufactured components. Ultrasonic C-scan inspections are often carried out under water using a scanning frame either in transmission or by reflection; this ensures good coupling.

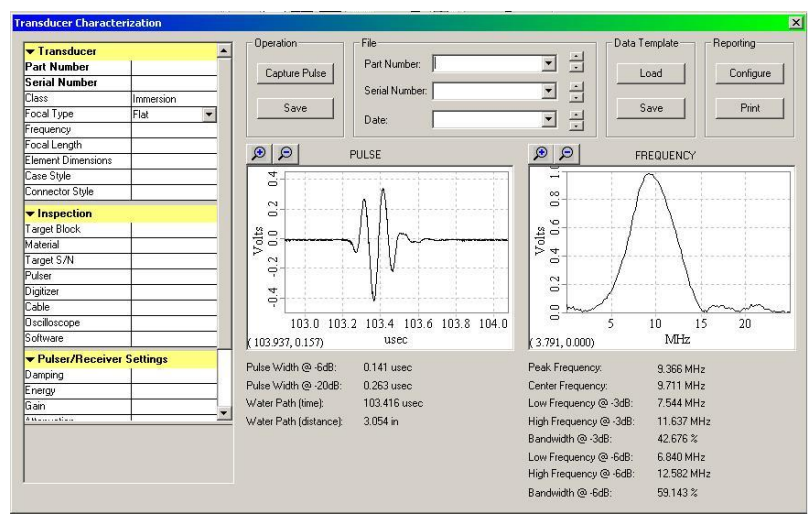


Figure 8 Analysis of reflected ultrasonic pulse (frequency spectrum)

Significant recent trends in ultrasonics include the use of ultrasonic linear scanning methods including Time of Flight Diffraction (TOFD), the use of ultrasonic lamb-wave or guided wave methods, air coupled ultrasound and ultrasonic phased arrays. A phased array comprises an array of miniaturised probes, often produced by etching a flexible PVDF film. If 0° probes are used this can be used to produce real time C-scan images. Alternatively, altering the firing sequence of individual cells can produce a range of beam angles.

For inspection of coatings one method of avoiding the surface reflection is to use an angled scan. Figure 9 shows an example of the detection of an area of paint-contaminated surface under a thermally sprayed coating.

It is known that spectral intensity at a certain frequency band increases with adhesive strength correspondingly. The wavelet transform or frequency analysis can be used to analyse the ultrasonic waveform. By using this method, the echo from the boundary between a coating and base metal could be readily determined. It is thought that this analysis method could be applied to the coated specimens to be examined in this project.

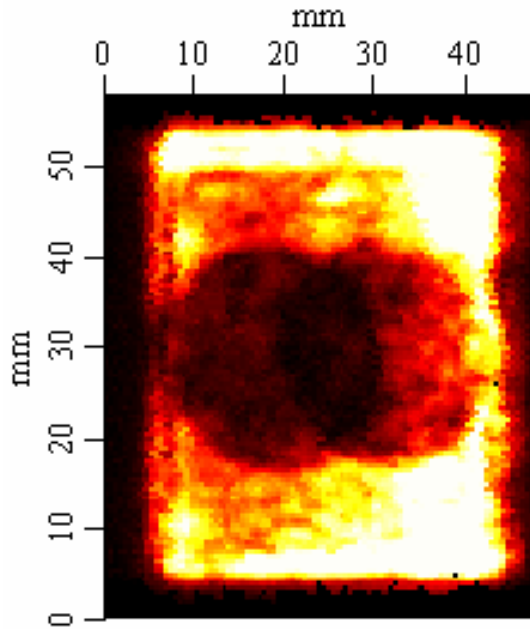
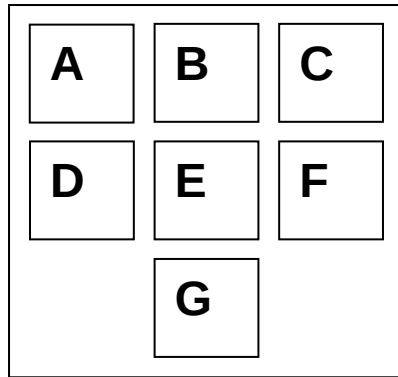


Figure 9 Frontwall Inspection (C-Scan) of aluminium thermally sprayed sample with paint contaminant (circular) with oblique-incidence technique.

(Note: there are two circles because the defect is passed by both the incident and reflected waves)

2.3.1 Case study: Ultra-sonic testing of thick coatings

Samples were prepared with various surface conditions as shown in the upper picture (Figure 10), corresponding to different levels of adhesion. The lower picture shows the amplitude image when a 10MHz ultrasonic beam is reflected from the interface with the beam sent through the substrate. The different conditions can be differentiated reasonably well, in particular the poor adhesion that results from the contamination on specimens E and F. Inspection is less easy when carried out from the coating side.



- A - Grit blast with NK20 grit
- B - Partially grit blasted to NK20
- C - Ground surface
- D - Hi-temp oxide contaminant
- E - Copper grease contaminant
- F - Zinc phosphate contaminant (paint)
- G - Rusty surface

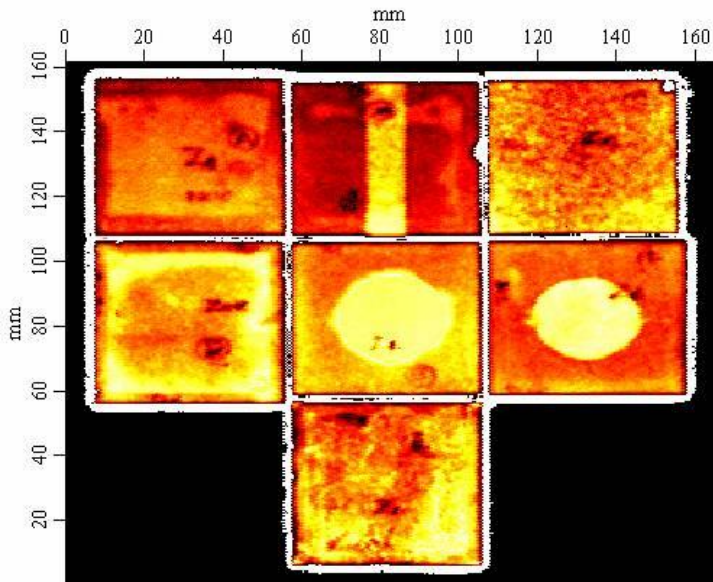


Figure 10 Ultra-sonic images obtained from coatings with different adhesion levels.

2.4 Scanning Acoustic Microscopy

Scanning Acoustic Microscope (SAM) uses very high frequency ultrasound (75 MHz) to image the internal integrity of coatings. The system is operated in pulse-echo mode with a single ultrasonic transducer emitting and receiving the ultrasonic beam (Figure 10). The ultrasound waves are generated using a piezoelectric crystal transducer. Specimens are submerged during the experiments in deionised water that acts as a couplant to transmit the ultrasound into the specimen. The ultrasonic beam is focused using lenses to a specific range of depths enabling the depth of defects to be determined. The ultrasonic pulses travel through the specimens until they strike an interface within the material where they reflected back to the transducer producing a signal. The ultrasound data is digitised and amplitude and the time-of-flight data is processed and imaged.

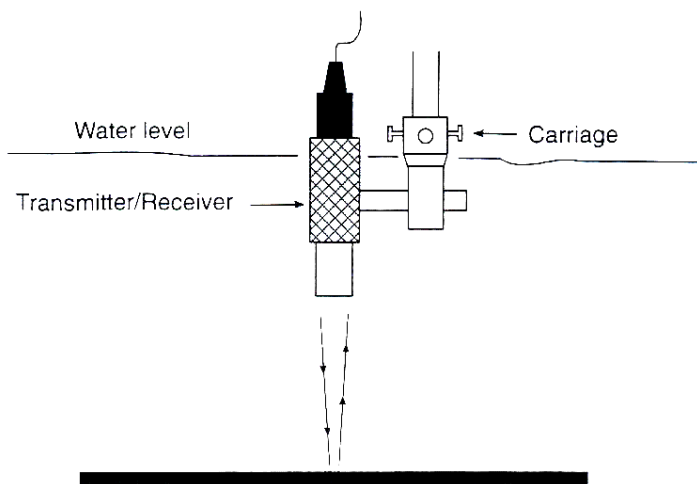


Figure 11 Schematic of Scanning Acoustic Microscope in pulse-echo mode

2.4.1 Case study: Scanning acoustic microscopy of a delaminate coating

The potential for using scanning acoustic microscope to detect poorly adhered coatings has been demonstrated using a partially delaminated chromium nitride coated stainless steel. Measurements were made as a function of time-of-flight across the surface of the specimen (Figure 12). The first large peak in the scan represents the reflections obtained from the surface of the specimen; the smaller peaks that occur to the right represent reflections from discontinuities and defects at the interface of the coating. A composite image was then obtained of the whole coating close to its interface with the substrate, which clearly shows the presence of delamination (Figure 13). This clearly demonstrates the ability of the technique to detect gross coatings defects. The technique is, however, unable to distinguish between well-

adhered and poorly adhered coatings where the coating is still in contact with the substrate. Moreover, extreme care must be taken when interpreting SAM data, as the images do not represent an image of the specimen at constant depth but represents data obtained with the same time-of-flight. It is therefore essential to verify the presence of a defect using other techniques such as optical microscopy or SEM.

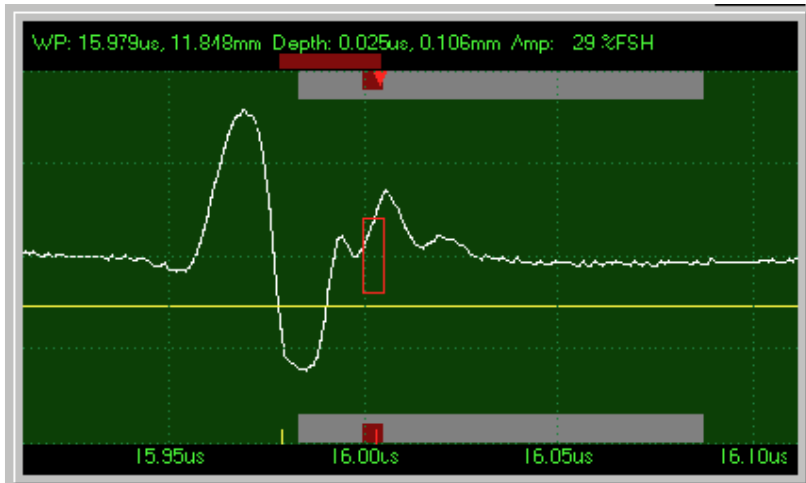


Figure 12 Signal obtained from SAM as a function of time-of-flight

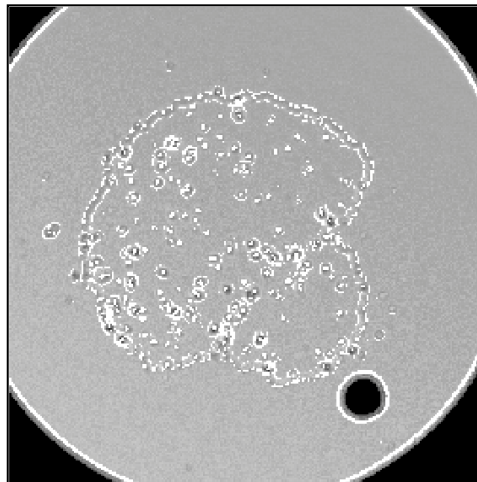


Figure 13 SAM image of delamination on a chromium nitride coated specimen.

2.5 Acoustic Emission

The Acoustic Emission Technique involves passive 'listening' to bursts of acoustic waves emitted within a component. The technique usually refers to emissions in the range 30kHz to 30MHz. The prime source of Acoustic Emission is the release of energy as stress is relieved during crack growth. The amount of energy released depends on the details of the material and the nature of the damage. The success of this for adhesion measurement therefore depends on the partial damage to the interface caused by an applied stress.

The principles of AE testing are relatively straightforward. AE is defined as 'transient elastic waves generated by the rapid release of energy from localised sources within a material'. These AE sources can be detected by standard piezoelectric sensors with a frequency response greater than 100kHz.

Application of the technique involves the placement on the component of at least two, and often many, transducers. The signal bursts from these are monitored and recorded continuously, or over periods at regular interval. The equipment therefore entails a number of transducers, with signal amplifiers, filters and a recording device such as a PC. A video display of the signal vs. time is usually also displayed. Recognition of clear signal is often difficult against background noise. The signals can be analysed in a number of ways:

- Amplitude versus time.
- Number of signals exceeding a threshold versus time.
- Cumulative energy of signal received versus time.
- Frequency spectrum of signals.

Acoustic emission can be coupled with destructive tests (e.g. portable adhesion testers) in order to provide information on coatings failure in destructive tests. Cumulative acoustic counts are plotted against the applied loading (or temperature in the case of thermal fracture tests) in order to assess the onset of failure, as shown in the Figure 14 below.

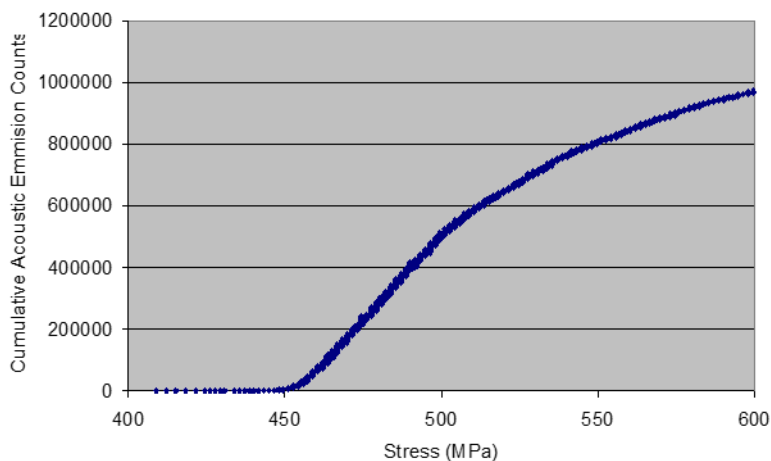


Figure 14 Cumulative acoustic emissions from a 1 μm thick CrN coating.

In order to ensure global coverage of a component, pencil leads should be broken at various distances from one sensor to establish the distance at which signal amplitude falls below the nominal threshold (40dB AE). Sensors are then spaced at a maximum of one and a half times this distance. Pencil lead breaks are a highly reproducible AE source similar in nature to AE signals produced by crack growth. Pencil lead breaks can also be performed in close proximity to each sensor in order to test how well the sensors are acoustically coupled to the test vessel.

Environmental noise, both electronic and mechanical, can make AE testing difficult. Prior to testing, one or two minutes of data should be acquired, as a background noise check and this should also be repeated post-test. Noise sources should also be identified within the test data based on signal waveform shape. Application of data filters within the acquisition software can remove these sources.

2.6 Laser Surface Acoustic Wave

Laser surface acoustic wave is a technique that measures the fundamental properties of a coating by measuring the velocity of acoustic waves through the surface of the coating. Specimens are mounted in the LaWave SAW spectrometer as shown in Figure 15. Short ($\sim$ nano second) pulses from a nitrogen laser are focused on to the sample surface by a cylindrical lens causing rapid localised heating of the material. The expansion and contraction of the sample induces a large bandwidth of white noise surface acoustic waves, which propagate along the surface of the sample. These waves are detected using a piezo-foil held in position by a knife-edge contact. A high frequency data-logging oscilloscope is used to record the signal. The accuracy of the velocity determination is improved by moving the point of excitation for the SAWs with respect to the sample and detector using a high-resolution translation stage. Sixteen pulses are averaged for each position and a time base window filter used to improve the signal to noise ratio. A Fast Fourier Transform of the collected signal generates a dispersion curve of wave velocity vs. frequency, the slope of which can be related to the properties of the coating.

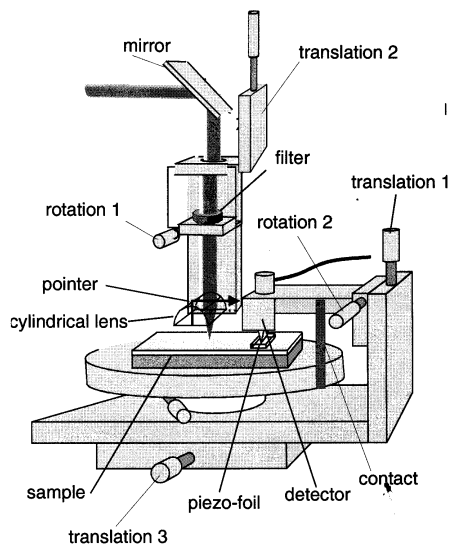


Figure 15 Schematic of the LaWave measurement stage

2.6.1 Case study: Laser surface acoustic wave of PVD coatings

The laser surface acoustic wave technique has been used to examine five chromium nitride coated specimens that have been treated prior to coating to vary their adhesion properties. Dispersion curves of wave velocity vs. frequency obtained from the PVD coated specimens are shown in Figure 16. These graphs demonstrate how the gradient of the dispersion curves

increases with the coating adhesion enabling an operator to distinguish between specimens with good and bad adhesion. Furthermore, it is also possible from the dispersion curves to detect regions where the coating has actual delaminated. This can be clearly seen in the specimen with a carbon layer as the dispersion curve appears to be almost a horizontal line where the coating has delaminated from the substrate. The technique is, however, limited in that at present it can only be used on relatively smooth, flat specimens.

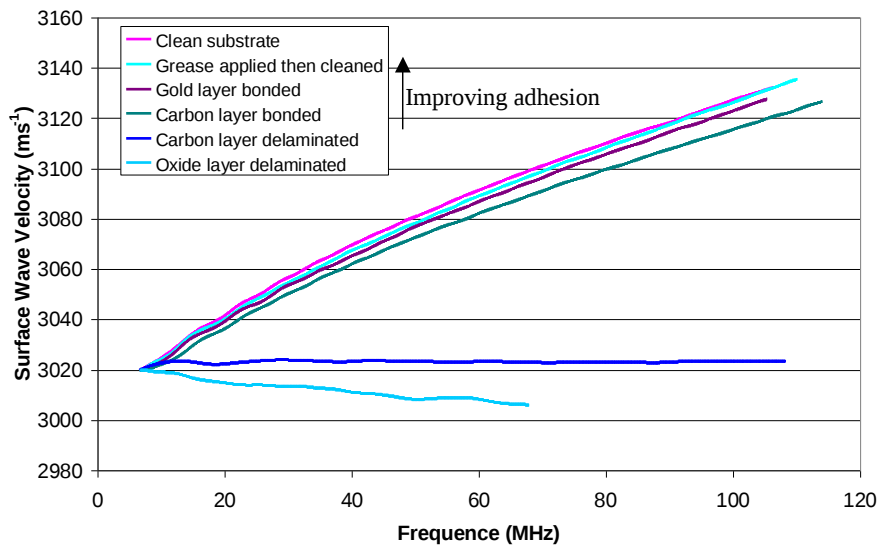


Figure 16 Dispersion curves for PVD coated specimens obtained using laser surface acoustic wave.

2.7 Terahertz

Ultra-fast pulsed lasers with a suitably engineered semiconductor crystal have recently led to the development of new Terahertz pulse imaging systems (THz = 10^{12} Hz) which when used to visualise internal structures work much like a radar system transmitting and receive reflected Terahertz pulses (Figure 17). A Terahertz pulse illuminates the target, and portions of the pulse are reflected from the internal layers. The delay of the pulse as it passes through the object gives a precise measure of the distance to the various surfaces inside the object. So by scanning the beam across the target a complete three-dimensional picture can be built up of the internal structure. The coherent detection of these Terahertz pulses is then achieved by illuminating a second crystal with the visible beam. The electric field of the Terahertz field is measured as a function of time with femto-second resolution yielding depth information, and this same waveform can then also be mathematically transformed to produce an absorption spectrum for the sample.

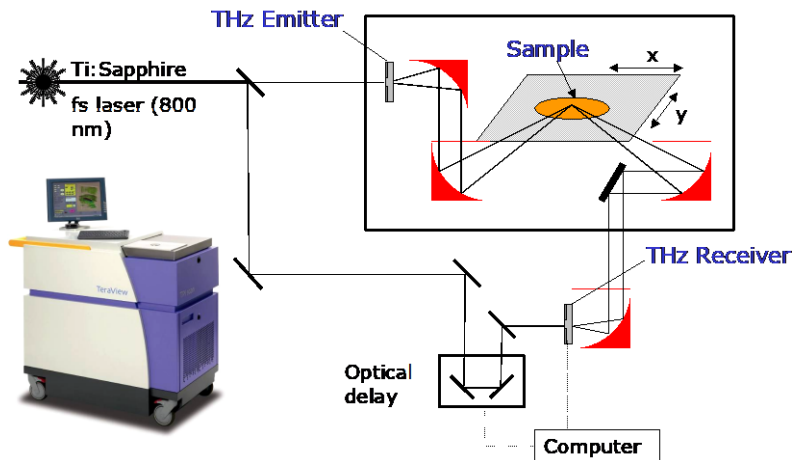


Figure 17 Schematic diagram of the Terahertz pulse imaging system

2.7.1 Case study: Terahertz imaging of paint systems

The terahertz pulsed imaging (TPI) technique has been used to characterise the thickness and uniformity of coating layers in paint systems used for ballast tanks in marine applications. Figure 18 demonstrates how this technique can be used to image interfaces between different paint systems and identify defects.

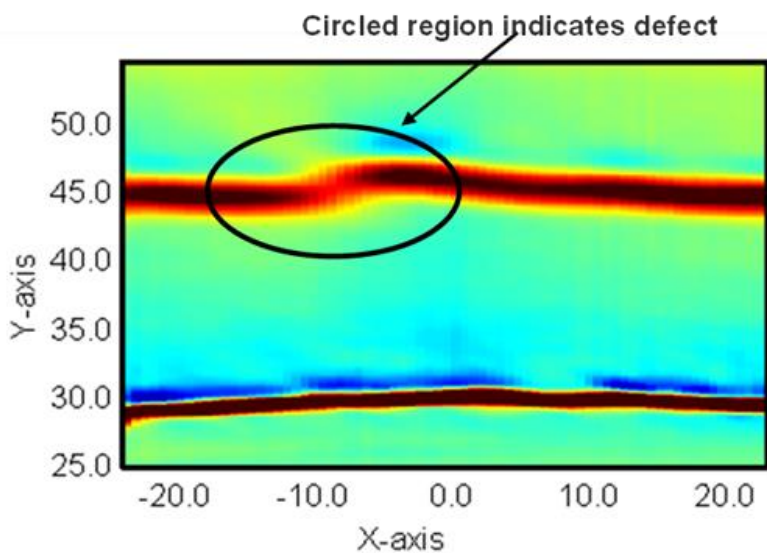


Figure 18 Image of defect in paint system identified using terahertz pulse image

Chapter 3: Test selection

- Introduction
- Selection tables

3. Test selection

3.1 Introduction

The selection of a suitable test method for the detection of coating defects is a complex task, which depends on a wide range of different factors. The five key factors that need to be considered when selecting a test method are:

- Sensitivity
- Coverage
- Speed
- Reliability
- Cost

Sensitivity describes the type and size of the defect that can be detected. In certain, application this may simply be the ability to detect delamination or cracks within the coating, whereas in other cases it may be necessary to detect the fact that although the coating is still intact the adhesion strength is insufficient for the intended application.

Coverage is the area of the component that can actually be inspected by the technique; this is particularly important where the technique is to be used for on-site inspections.

Speed affects the cost of the inspection and the coverage that it is possible to achieve within the required time, this is particularly important where the technique is to be used for quality control and essential when this is to be conducted on-line.

Reliability of a technique is the probability that a real defect will actually be detected and the number of spurious results or false-calls that are also detected. False-calls are where defects are indicated by the technique but no defect is present. False-calls are a key issue in many automated inspection processes and often restrict the reliability that can be achieved.

Cost is probably the biggest fact in the selection of any test method, not just the capital cost of the equipment but also the cost of conducting the inspections and maintaining the equipment in good working order. The cost equation should, however, always be balanced against the cost of failures, lost custom and possible litigation.

3.2 Selection tables

Although the task of selecting a suitable technique is complex the different factors that govern the selection can be broken down into two main areas; those that depend on the properties of the specimens (e.g. geometry and material properties), and those that depend on the application (e.g. onsite inspection or quality control). To assist in this selection process the key requirements from each of these main sources have been listed in Tables 1 and 2. In addition, the key features of each technique including; resolution, coverage, type of specimen, and cost are given in Table 3.

		Thermography		Ultrasonics	SAM	Acoustic Emission	LSAW	Terahertz
		Static	Flash					
Is the coating thinner than 50 μm ?	Yes	✓	✓	✗	✗	✓	✓	✓
	No	✓	✓	✓	✓	✓	✓	✓
Is the coating electrically conductive?	Yes	✓	✓	✓	✓	✓	✓	✗
	No	✓	✓	✓	✓	✓	✓	✓
Is the coating porous?	Yes	✓	✓	✗	✗	✓	✓	✓
	No	✓	✓	✓	✓	✓	✓	✓
Is the coating rough?	Yes	✓	✓	✓	✗	✓	✗	✓
	No	✓	✓	✓	✓	✓	✓	✓
Does the coating have high acoustical damping?	Yes	✓	✓	✗	✗	✗	✗	✓
	No	✓	✓	✓	✓	✓	✓	✓
Is the surface of the specimen flat?	Yes	✓	✓	✓	✗	✓	✗	✓
	No	✓	✓	✓	✓	✓	✓	✓
Is the specimen area less than 1 cm^2	Yes	✓	✓	✓	✓	✓	✗	✓
	No	✓	✓	✓	✓	✓	✓	✓
Is the specimens adversely affected by water?	Yes	✓	✓	✓	✗	✓	✓	✓
	No	✓	✓	✓	✓	✓	✓	✓

Table 1 Specimen requirements for selection of coating adhesion tests

		Thermography		Ultrasonics	SAM	Acoustic Emission	LSAW	Terahertz
		Static	Flash					
Can the tests be used for quality control purposes?	Yes	✓	✓	✓	✓	✗	✓	✓
	No	✓	✓	✓	✓	✓	✓	✓
Can the tests be conducted on-line?	Yes	✓	✓	✓	✗	✗	✗	✓
	No	✓	✓	✓	✓	✓	✓	✓
Is the technique capable of rapid data acquisition?	Yes	✗	✓	✓	✗	✓	✗	✓
	No	✓	✓	✓	✓	✓	✓	✓
Can the tests be used for on site inspections?	Yes	✓	✓	✓	✗	✓	✗	✓
	No	✓	✓	✓	✓	✓	✓	✓
Can the tests be conducted in situ?	Yes	✓	✓	✓	✗	✓	✗	✗
	No	✓	✓	✓	✓	✓	✓	✓
Is the test capable of high sensitivity?	Yes	✗	✓	✓	✓	✗	✓	✓
	No	✓	✓	✓	✓	✓	✓	✓
Can non-specialist operators use the test method?	Yes	✓	✓	✗	✓	✗	✗	✗
	No	✓	✓	✓	✓	✓	✓	✓
Is the test non-contact?	Yes	✓	✓	✗	✓	✗	✗	✓
	No	✓	✓	✓	✓	✓	✓	✓
Can large test areas be inspected?	Yes	✓	✓	✓	✗	✓	✗	✓
	No	✓	✓	✓	✓	✓	✓	✓
Can the test be conducted at non-ambient temperatures?	Yes	✓	✓	✓	✗	✓	✗	✓
	No	✓	✓	✓	✓	✓	✓	✓

Table 2 Application requirements for selection of coating adhesion tests

	Thermography		Ultrasonics	SAM	Acoustic Emission	LSAW	Terahertz
	Static	Flash					
Analysis Method	Thermal radiation across surface of coating	Thermal radiation as function of time	Time of flight Attenuation / frequency	Time of flight Attenuation / frequency	Acoustic signal from fracture process	Time of flight as function of depth	Time of flight Attenuation / frequency
Depth Resolution	Not provided	Calculated from heating/cooling times	1 – 2 μm	1 – 2 μm	Not provided	1 μm	0.1 mm
Spatial Resolution	3-5 mm	1-2 mm	2-3 mm	> 1 μm	Only if two or more sensors are used	1 mm	0.1 mm
Inspection Area	300 mm ²	300 mm ²	25 mm ² or greater	100 – 150 mm ²	1000 mm ² or greater	10 mm ²	100 mm ²
Information Provided	Damage / defects	Damage and poor adhesion	Damage / defects Loss of adhesion	Damage / defects	Damage	Damage and poor adhesion	Damage / defects/ thickness
Type of Specimen	All shapes and sizes	All shapes and sizes	Non-damping coatings	Must be able to be placed in water bath	All non-damping coatings	Non-polymeric coatings	Non-conductive coatings
Relative cost of equipment	Low	Medium	Medium	Medium	Low (increase with area covered)	Medium	High
Relative cost of measurement	Low	Medium	Medium	Medium	High	High	Medium

Table 3 Physical characteristics of the different test methods

Chapter 4: Test protocol

- Specimen requirements and preparation
- Technique details
- Measurements and validation

4. Test protocol

4.1 Thermography

4.1.1 *Introduction*

In certain applications it is possible to detect sub-surface damage, or delamination using thermography. The different thermal diffusivities through damaged portions, inclusions or porosities give rise to small different surface temperatures which can be detected readily with a thermal camera.

4.1.2 *Specimen requirements preparation*

In order to minimise measurement artefacts and misleading images it is important to ensure that the surfaces are clean and dry. Thermography is very sensitive to surface emissivity which depends critically on colour. Wherever possible it should be ensured that the surfaces are uniformly clean. In some cases where it is not possible to achieve uniform appearance of the surface it may be advisable to coat the surface uniformly in mat black paint.

4.1.3 *Technique*

Heating can be applied through radiation or through conduction. A 500W lamp may be used to radiate the surface under study, or the object may be heated on a hot plate prior to measurement, and then transferred to the field of view of the camera. When using radiated heat it is important to make sure that the angle of the heat illumination is such that heat is not reflected from the surface into the camera. Objects close to room temperature radiate in the infrared part of the spectrum. Ensure that the thermal image obtained is not being influenced by the presence of overhead lamps or proximity of warm objects including human bodies. It is not generally necessary to heat objects above 60 °C, and frequently just a few degrees are necessary.

4.1.4 *Measurements*

While viewing live images it is frequently necessary to stretch the contrast in order to reveal the small thermal changes caused by delaminations and other surface defects. When a feature seems to become visible, the object should be moved around or rotated within the field of view in order to ensure that it is object related and not due to a stray reflection. Frequently it is possible to obtain thermal images through two different heating mechanisms. If the features are visible through both methods it adds confidence that what is being detected is a genuine feature of the object and not an artefact. While the object is still under the thermal camera it is often useful to compare the thermal image with a close visual inspection of the surface to reject any apparent thermal flaws that might have an obvious alternative explanation e.g. emissivity variation.

4.1.5 Validation

Wherever possible the sites indicated as damaged by the thermogram should be looked at closely with another technique such as optical microscopy or metallography in order to confirm the thermography diagnosis. For each application and type of specimen it may be necessary to build up a portfolio of detectable faults and limit of detectability before placing too much confidence in the thermography.

4.2 Ultrasonic Testing

4.2.1 Introduction

It is usually possible to detect sub-surface damage, or delamination using ultrasonics. Differences in mechanical impedance cause ultrasonic signals to be reflected (or partially transmitted) and this information can be used to detect flaws. It is however limited to some extent by the large change of impedance at the interface between the test object and the couplant (often water) or probe. Detection of adhesion change is more subtle, and for coatings is often complicated by the proximity and condition of the surface.

4.2.2 Specimen requirements preparation

The effect on the surface condition on the ultrasonic waves depends on the surface roughness and the wavelength of the ultrasound. Ideally smooth surfaces should be prepared, as to resort to lower frequencies to accommodate the surface condition reduces the sensitivity of the technique.

4.2.3 Technique

For inspection of a coated surface that is the backwall of an ultrasonic inspection, a 0° compression wave beam can often be used. This pulse can be analysed and displayed in various ways. However a simple C-Scan display showing signal amplitude can often show up the differences between various areas of the surface.

For analysis of a front surface echo, oblique incidence can be used to reduce the effect of a strong surface echo. Surface waves generated by different methods can also be used.

In general the area inspected by a single measurement can be determined by the choice of probe. Unfocussed beams will average over a larger area but will give a faster inspection and may be adequate to detect large flaws.

Water is usually the chosen couplant method, so inspection is carried out either in a tank or by means of squirters and water recovery systems. Conventional couplants such as gel or grease depend for the signal amplitude on hand pressure and are not really suitable for coating inspection.

4.2.4 Measurements

Measurements of signal amplitude, pulse waveshape, frequency spectrum, and various signal processing procedures such as wavelet transforms can be used to make the measurements. In general for these techniques to be effective a high quality pulse shape containing a wide frequency spectrum is necessary.

4.2.5 *Validation*

Validation of the technique experimentally is necessary, and much work needs to be done in this area. Preparation of suitable samples is a necessary prerequisite, and these have to be checked against destructive pull off tests. Formal validation of techniques in critical applications can be carried out by procedures of ENIQ (European Network for Inspection Qualification).

Once this preliminary work is done then the technique can be applied in the field.

4.3 Scanning Acoustic Microscopy

4.3.1 *Introduction*

Scanning acoustic microscopy is an ultrasound technique that uses focused ultrasonic waves (5 to 150 MHz) to image defects in components. Ultrasonic sound waves through the specimen reflect at defects in the specimens and by interpreting the interaction of these waves with the defects within the specimen it is possible to determine whether there are defects within the specimen.

4.3.2 *Specimen requirements and preparation*

Specimens are normally immersed in deionised water during the experiments to act as a couplant so that the ultrasonic sound waves can enter the specimen. It is therefore essential to ensure that the water does not adversely affect the specimens. Scanning acoustic microscopy is also very sensitive to the surface topography of the specimen. It is therefore important wherever possible to ensure the surfaces are clean and that there are no air-bubbles on the surface of the specimen.

4.3.3 *Technique*

Scanning Acoustic Microscopy works by directing focused sound from a transducer at a small point on the specimen. A piezoelectric crystal or transducer produces a pulse of ultrasonic sound waves that pass into the specimen through a couplant, normally deionised water. The waves travel through the specimen with a portion of the waves being reflected back every time they hit a defect within the material. The reflected sound waves are then recorded by the transducer as a function of time. The transducer is then moved to a slightly different location and the process repeated in a systematic pattern until the entire region of interest has been investigated. The values obtained at each point on the specimen are then assembled to produce an image of the object. The resolution of the image is limited either by the physical

scanning resolution or the width of the sound beam (which in turn is determined by the frequency of the sound).

4.3.4 *Measurements*

The images that are built up using scanning acoustic microscopy represent the intensity of the reflected sound obtained at a particular time after the initial pulse has entered the specimen. This gives an indication as to the depth a particular defect is beneath the surface as the farther a defect is beneath the surface the longer it takes for the ultrasonic waves to return to the transducer. However, the time of flight cannot be directly related to depth as it also depends on the composition of the material. The contrast seen in the images is therefore based both on geometry and composition and may in fact represent features at different depths within the specimen.

4.3.5 *Validation*

Due to the composite nature of the images obtained from scanning acoustic microscopy it is important to confirm the presence of possible defects using other techniques such as optical or electron microscopy.

4.4 *Acoustic Emission*

4.4.1 *Introduction*

Acoustic Emission (AE) is the sound generated when a microscopic fracture of a material occurs, it is usually pulses (or hits) in the frequency band 30KHz – 30MHz. It is induced by a stress exceeding the breaking point of the particular area. It is usually detected with piezo-electric transducers specifically designed for receiving small ultrasonic signals.

When applied globally it can be used to detect problem areas for further investigation by other techniques.

4.4.2 *Specimen requirements and preparation*

Inspecting by acoustic emission requires no specific surface preparation, and large areas can be inspected with a small number of transducers. However some means of applying stress to the particular area of concern (i.e. the coating-substrate interface) is required. This technique must not in itself generate acoustic noise in the same frequencies as the acoustic emission.

4.4.3 *Technique*

The measurement technique for AE is relatively simple: the stress is applied (usually by an increasing ramp to failure) and the AE is detected.

Detection can be enhanced by arrays of transducers used to localise or separate the signal from the test area or by eliminating signals from other areas.

The sensitivity is set up using pencil lead breaks, such that the signal from each area under test is equal.

4.4.4 *Measurements*

Acoustic emission pulses are recorded and various parameters are extracted from the recorded signals. These are typically hits per unit time or stress unit, ring down count, energy, and frequency content.

Repeated application of stress or stress level holds can show up more severe defects, as normally once a given level of stress has been obtained, the acoustic emission ceases.

4.4.5 *Validation*

The AE tests must first be shown to detect the coating adhesion failure in laboratory tests. In practice, if an area showing a large acoustic emission count is detected, then it may be from a number of sources (e.g internal defects) other than coating adhesion failure, and these possibilities must be checked, usually by another non-destructive technique.

4.5 *Laser Surface Acoustic Wave*

4.5.1 *Introduction*

This technique measures the surface wave dispersion curves (wave velocity vs. frequency curves) of coated samples and can reveal the way different coating/surface treatments influence their quality and properties. The model used to fit the dispersion curve depends on elastic modulus, film thickness, poisson's ratio and density/porosity of the material. Depending on the knowledge of the coating/substrate system properties being studied, in some cases, it is possible to detect micro-defects in the film and at the interface, which cause lower stiffness and a reduction in the slope of the measured dispersion curve.

4.5.2 *Specimen requirements and preparation*

There are only a few requirements on the sample: a plane surface with a maximal roughness $R_a \sim 1 \mu\text{m}$, a minimum size of $15 \times 10 \text{ mm}^2$, and a thickness between 0.3 mm and 5 mm. Additionally it should be opaque for UV-light (337nm) or must be covered with a thin metal film (thickness $\sim 10\text{-}50\text{nm}$). If measuring curved (i.e. helicoidally) shaped samples, the measurement site has to be carefully selected so that the portion of sample surface tested is as parallel as possible with respect the sample stage.

4.5.3 *Technique*

The samples are mounted in a Lawave® SAW spectrometer. A Nitrogen pulsed laser beam (0.5ns at 337nm) is then focused on to the sample surface by a cylindrical lens in order to excite a line-shaped broadband SAW pulse via a thermoelastic mechanism. A piezoelectric polymer foil pressed onto the substrate by a steel wedge is used as a detector for the SAW pulse.

4.5.4 Measurements

SAW propagation measurement is performed for different propagation lengths. A high frequency data-logging oscilloscope was used to record the signal. The accuracy of the velocity determination was improved by moving the point of excitation for the SAW with respect to the sample and detector using a high-resolution translation stage. A fast Fourier transform of the collected signal generates a dispersion curve of wave velocity versus frequency. The slope of the experimental dispersion curve can be related to Young's modulus or thickness or density of the coating by fitting the related parameters of a SAW propagation model to the dispersion curve [2].

4.5.5 Validation

The dispersion curve increase with frequency, due to the higher Young's modulus of the coatings compared to the substrate. The results suggest that by comparing the slope of the curve for different specimens, it is possible to assess the degree of damage of the samples. However, as the slope also depends on coating/substrate Young's Modulus and film thickness, one has to ensure that comparison is done among similar coating/substrate systems in order to understand the obtained results.

4.6 Terahertz

4.6.1 Introduction

Terahertz waves have a wavelength of 0.1 -10mm and are in the range between microwaves and infra-red waves. They penetrate non-conductive materials and are affected by changes in the dielectric constant.

They are generated by special techniques from laser pulses and travel through air in a similar way. They are totally reflected by metals.

4.6.2 Specimen Requirements and Preparation

The samples do not need to be prepared specially except that dirt and dust should be removed. The surface should be placed such that the reflected waves reach the receivers.

4.6.3 Technique

The terahertz generator is placed over the specimen and terahertz receivers are placed nearby. The technique is essentially pulse-echo using analysis of reflections. Similar displays to those used in ultrasonics can also be used (e.g. with B-Scan, C-Scan etc).

4.6.4 Measurements

The measurements are initially of signal amplitude and time of flight. More sophisticated analysis of pulses can be carried out. The depth resolution is of the order of the lower wavelength, and the amplitude is proportional to the change in dielectric constant between materials.

4.6.5 *Validation*

To validate an indication obtained with Terahertz is difficult, as no other technique is sensitive to the same properties, therefore the best method is by sectioning, until confidence is such that the technique signals can be associated with specific defect conditions.

Chapter 5: Further reading & references

- Further reading
- References

5. Further reading and references

5.1 Further reading

For the reader who would like to enquire more into some of the techniques discussed in this guide the list of references below contains more detailed information.

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5.2 References

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