Chapter 6

PDS FOR INDUSTRIAL APPLICATION

6.1 Introduction

Finished materials, products and equipments, which fail to achieve their design requirements or projected life, due to undetected defects, may require expensive repair or early replacement. Such defects may also cause unsafe conditions or could lead to catastrophic failure, as well as loss of revenue due to unprecedented plant shutdown. Thus material analysis through Non-Destructive Testing (NDT) becomes very important. NDT can be applied in each stage of an item's construction to understand materials homogeneities or to determine its quality [1].

NDT, Non-Destructive Evaluation (NDE), Non-Destructive Inspection (NDI) are the terms commonly used in connection with the techniques that are based on the application of physical principles employed for the purpose of determining characteristics of material/components/systems, for detecting and assessing the inhomogeneities and harmful defects without impairing the usefulness of the material/components/systems. In other words, it is used for the examination of materials and components in such a way that, it allows materials to be examined without changing or destroying their usefulness. NDT of engineering materials, components and structures has steadily increased in recent years because of the all-round thrust for improving material quality and performance reliability.

The concept of component design strives towards greater efficiency and improved performance. This can be achieved by employing materials with
acceptable but minimum level of defects analysed through quality testing. NDT can be used to find out the size and to locate superficial as well as subsurface flaws and defects. Welded joint, cracks, porosity, voids, inclusions, etc., can be examined using NDT.

Performance of surface hardened parts is a major issue in automotive and aerospace industries. There is, therefore, a strong need to improve the quality control by introducing new measurement systems which allow for non-destructive, non-contact hardness profile measurement as an alternative to the presently used destructive inspection method [2]. Table 6.1 is the list of the merits and demerits of NDT with respect to destructive techniques.

<table>
<thead>
<tr>
<th>Non-Destructive tests</th>
<th>Destructive tests</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Advantages</strong></td>
<td><strong>Limitations</strong></td>
</tr>
<tr>
<td>1. Tests are made directly on the object. 100% testing on actual components is possible.</td>
<td>Tests are not made on the objects directly. Hence correlation between the sample specimen used and object needs to be proved</td>
</tr>
<tr>
<td>2. Many NDT methods can be applied on the same part and hence many or all properties of interest can be measured</td>
<td>A single test may measure only one or a few of the properties</td>
</tr>
<tr>
<td>3. In-service testing is possible</td>
<td>In-service testing is not possible</td>
</tr>
<tr>
<td>4. Repeated checks over a period of</td>
<td>Measurement of properties over a</td>
</tr>
</tbody>
</table>
time are possible | cumulative period of time cannot be possible

5. Very little preparation is sufficient | Preparation of the test specimen is costly

6. Most test methods are rapid | Time requirements are generally high

**Limitations**

7. Measurements are indirect. Reliability is to be verified | Measurements are direct and reliable

8. Usually qualitative measurements | Usually quantitative measurements

9. Skilled judgments and experience are required to interpret indications | Correlation between test measurements and material properties are direct

| **Advantages** |

Table 6.1: List of merits and demerits of NDT with respect to destructive techniques.
6.2 The commonly used NDT methods

(i) **Liquid penetration inspection:**
This technique is based on the ability of a liquid to be drawn into a "clean" surface-breaking flaw by capillary action. After a period of time (called the "dwell"), excess surface penetrant will be removed and a developer will be applied. This acts as a "blotter" and it draws the penetrant from the flaw to reveal its presence. Major demerit of this technique is that this gives information about cavities that are open to the surface only. Very viscous liquids become unsuitable as penetrants, because they do not flow. Moreover, coloured penetrants require 'good white light while fluorescent penetrants need to be used in darkened conditions with an ultraviolet "black light".

(ii) **Eddy current:**
This technique uses magnetic lines of force, when an energised coil is brought near to the surface of a metal component. Due to magnetic induction, the lines of force get distorted by the presence of a flaw. Major disadvantages of these technique is that, as Eddy current testing is an electromagnetic technique, it can only be used on conductive materials. Surface irregularities and scratches can also give misleading indications. Therefore it is necessary to ensure very careful preparation of the surface before magnetic particle testing is to be undertaken.

(iii) **X-Ray and Gamma rays detection:**
The x-ray or gamma rays, is allowed to pass through the material to be inspected, and is then captured on a film. This film, when processed, gives an
image with a series of grey shades between black and white. The choice of type of radiation to be used, as to whether X-Ray or gamma ray, depends on thickness of the material to be tested. Major disadvantage with this method is that, X-rays and gamma rays are very hazardous. Special precautions must be taken when performing radiography. Hence the operator should use appropriate barriers and warning signals to ensure that there are no hazards to personnel.

(iv) Ultrasonic detection:
It is based on the principle that, ultrasonic waves produced inside any material, could propagate through the material, since solid materials are good conductors of sound waves. The waves are not only reflected at the interfaces but also by internal flaws such as material separations, inclusions, etc. This is one of the most popular NDT and is the "present day quality testing tool".

(v) PDS technique
PDS technique is one among the NDT tool. It works on the principle of understanding thermal wave generation and propagation within the material. The thermal wave is generated inside the material through optical absorption. Since this technique uses laser beam, localized information to an area of μm range can be derived. Moreover this makes it non-destructive. Companies such as Jenoptik, PHOTOTHERM in Europe and Thermawave Inc. in US, have started to manufacture apparatus based on PDS technique, for quality control (mainly of silicon wafers) which are based on optically and thermally modulated optical reflection from semiconductors [3]. This is for measuring thickness of surface coating like paint etc,. Lot of effort has gone in for the
transfer of technology from laboratory scale to industrial plant scale. This was because, it requires additional optimization comprising of cost considerations, security and reliability aspects. Most importantly the device has to work under the special conditions dictated by the industrial demands, with considerable reduction of the available measuring time [3].

In the present study, we attempt to show the use of PDS tool for industrial application. We tried to study the quality of welded joint, to determine the thickness of paint coated over a substrate and to understand the homogeneity of polymer doped with various filler components.

6.3 Review

Gibkes et al [4] had developed a trace gas detector using photothermal technique for industrial application. It uses either a tunable laser adjusted to an absorption line of the trace molecule or spectrometer to scan the whole absorption spectrum. The advantage of this detector is that it can be adapted to different gases by changing simply a narrow band IR-filter in the front of the excitation beam. The use of limited number of electronic, mechanical and optical components makes the system reliable and less sensitive to environmental changes.

Busse et al [5] measured thickness of paint, using modulated photothermal radiometry, as this was an appropriate method for remote and nondestructive evaluation of thickness and the thermal properties of thin films / coatings deposited on thick substrates. A 48-μm-thick layer of grey paint on brass was investigated. The measurements were performed with the Photothermal Microscope (model PTM1) made by PHOTOTHERM -
Germany [6]. Wu et al [7] investigated turbine blades for hidden defects using this technique. They used combination of Lock-in technique and commercial IR-thermography. This allowed a fast, remote and nondestructive inspection of components. Even at low modulation frequencies (0.03 Hz) the measurement time was within 3 minutes.

Walther [8] inspected rough steel surface using photothermal technique. He used an effective layer model and compared his results with experimental data. He also observed that the signal corresponds to the spatially averaged surface temperature, if the diameter of the heating spot was larger than the correlation length (which is the mean lateral distance between characteristic hills or valleys of the rough surface) and fulfills the conditions of one dimensionality. He had also published another work, which was based on the equivalent layer model. The effect of roughness induced thermal wave dispersion was studied on hardened steel. He could successfully demonstrate the validity of the theory by comparing calculations with photothermal radiometric measurements [9].

Pandey et al [10] studied polymeric materials using photothermal techniques. Thermal diffusivity values for polymer materials such as, Biaxially oriented polypropylene film (BOPP), Tubular Quinched polypropylene film (TQPP), Carbon fibers, fullerene and composite ceramics were determined using this technique.
6.4 Experimental Details

6.4.1 Welded joint

Two individual Steel plates (MS EN8) of dimension 2 x 1 inch and of thickness 5 mm were welded along a “V” Groove using a 3 mm welding rod, maintained at 120 Amp current, with an input voltage of 440 V. Protruding surface at this joint, after welding, was grounded and polished, so that the joint becomes visually invisible. Fig. 6.1 shows the schematic of the welded joint. This joint was analyzed using PDS technique, and was also tested using mechanical testing methods.

Debris at the surface was thoroughly washed and finally rinsed with acetone. For PDS studies, sample was loaded in the sample cell immersed in CCl₄ solution. Chopped laser beam from a semiconductor diode laser (NdYAG - CASIX) of wavelength 532 nm and of power 25 mW was used as the pump beam. Pump beam scanning was done over an area of 600 x 1000 μm² within the welded portion for two different chopping frequencies of 250 Hz and 3000 Hz.

![Fig. 6.1 Schematic representation of the welded joint.](image-url)
6.4.2 Thickness of paint

A layer of black paint was coated over a glass substrate using an artist brush. Fig. 6.2 is the schematic of paint coated over glass substrate. Thickness was calculated using gravimetric method, to be ~0.8 mm.

The painted substrate of dimension 1 x 5 cm was mounted in the sample cell. Chopped laser beam from a semiconductor diode laser (NdYAG - CASIX) of wavelength 532 nm and of power 25 mW was used as the pump beam. CCl₄ was not used as the coupling medium this time, as we observed that the painted layer got eroded when dipped in this liquid. For surface imaging, scanning was done over an area of 800 x 1000 μm² at a fixed chopping frequency of 3200 Hz, so as to reconstruct a two-dimensional image of the paint coated area over the substrate. To determine the thickness, PDS data was collected over a range of chopping frequency of 5 Hz to 700 Hz. We used one layer model for curve fitting, as the painted layer was absorbing layer \( (l_1) \) and the substrate was non-absorbing \( (l_2) \). Detailed description of thickness calculation is given in chapter 1 (section 1.4).

![Fig. 6.2 Schematic of painted substrate](image)

6.4.3 Effects of filler in Polymer material

The sample used for this measurement was developed for encapsulating or potting heat generating devices. Polyurethane (PU) prepared
from Empeyol sp 101 and MDI Cosmonate LK obtained from Manali petrochemicals, Chennai, was used as the polymer matrix. Required amount of SP 101 and Cosmonate LK were taken in the ratio of 100:30. This proportion was thoroughly mixed and filler components were added immediately. The composition was kept in continuous stirring condition till the filler got dispersed in the polymer. Entrapped air was removed by applying vacuum. This mixture was then poured into the mould for casting and left for 24 hours.

Unlike any conventional methods, where the sample is directly heated, PDS method is based on the principle of generation of thermal waves within the material by optical excitation. The thermal waves so generated traverse through the material, which in turn is governed by the material’s thermal property. Thus, a direct relation could be obtained and the thermal diffusivity of the material could be determined nondestructively for PU doped with various fillers such as Mica, Boron Nitrate (BN), Zinc Oxide (ZnO), Aluminium Oxide (Al₂O₃). Samples were identified with running numbers as “1 to 9” and the sample detail is tabulated in Table (6.2).
<table>
<thead>
<tr>
<th>Sample Id</th>
<th>Sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>PolyUrethane(PU)</td>
</tr>
<tr>
<td>2</td>
<td>PU+Mica</td>
</tr>
<tr>
<td>3</td>
<td>PU+BN(1g)</td>
</tr>
<tr>
<td>4</td>
<td>PU+BN(0.1g)</td>
</tr>
<tr>
<td>5</td>
<td>PU+ZnO(1g)</td>
</tr>
<tr>
<td>6</td>
<td>PU+BN(0.5g)+Mica(0.5g)</td>
</tr>
<tr>
<td>7</td>
<td>PU+BN(0.5g)+Silica(0.5g)</td>
</tr>
<tr>
<td>8</td>
<td>PU+Silica(1g-precipitate)</td>
</tr>
<tr>
<td>9</td>
<td>PU+BN(0.1g)+ZnO(0.9g)</td>
</tr>
<tr>
<td>10</td>
<td>PU+BN(0.1g)+Al2O3(0.9g)</td>
</tr>
<tr>
<td>11</td>
<td>PU+Al2O3(1g)</td>
</tr>
<tr>
<td>12</td>
<td>PU+Silica(1g-Ultrafine)</td>
</tr>
<tr>
<td>13</td>
<td>PU+BN+ZnO(2.5g)</td>
</tr>
</tbody>
</table>

Table 6.2 Details of undoped and doped PU polymer samples

The samples used for PDS studies were cut in a disc shape of 1cm radius. Chopped laser beam from a semiconductor diode laser (NdYAG - CASIX) of wavelength 532 nm and of power 25 mW was used as the pump beam. In this case too we did not use CCl₄, as CCl₄ was observed to degrade the polymer material. Details of thermal diffusivity measurement is described in chapter 1 (section 1.5.1).
6.5 Results and Discussion

6.5.1 Welded joint

Two-dimensional PDS amplitude plots obtained for two different chopping frequencies of 250 and 3000 Hz is as shown in Fig. (6.3). It was observed that for 3000 Hz the signal was almost uniform. When the chopping frequency was decreased to 250 Hz, non uniformity in the PDS amplitude signal was observed.

For low chopping frequencies the signal is from the bottom layer of the sample and as the chopping frequency is increased the signal contribution is only for the surface. Thus, the non uniformity in PDS amplitude signal for 250 Hz could be due to hidden damage such as, entrapped air-pocket or unwelded portion inside the welded portion.

![Diagram of PDS analysis of welded joint](image.png)

Fig 6.3 PDS analysis of welded joint; Two dimensional Plot.
6.5.2 Thickness of Paint

Fig. 6.4 is a two dimensional image plot of PDS amplitude from paint coated over glass substrate. The data presented is for normalized PDS amplitude signal and the surface seems to be rough (Colour bar is given at the side is for indication). The irregularities in the plot represents roughness of painted portion. Thickness of this layer was also calculated using the curvefitting method, assuming a one layer model, the value was calculated to be \( \sim 0.72 \) mm. Fig. (6.5) is the PDS amplitude versus chopping frequency curve used for the determination of the paint thickness. Experimental data was curvefitted with the theoretically generated values. This study was for online monitoring of surface finishing of painted layer during mass.

![Two dimensional image plot of surface of paint coated layer over a glass substrate.](image)
6.5.3 Effect of filler in Polymer material

Fig. (6.6) is the typical log amplitude versus square root of chopping frequency of PU (which is the base material) and PU doped with Mica. Thermal diffusivity was determined using linear curvefitting of the experimental data. Thermal diffusivity of all other materials was determined the same way and the values are tabulated in Table (6.3). It was found that “sample 9” (PU doped with (0.1g) BN and (0.9g) ZnO) showed the maximum thermal diffusivity value of $0.4115 \times 10^{-6} \text{ m}^2/\text{s}$ and the least was observed for “sample 1” (pure PU) of value $0.00113 \times 10^{-6} \text{ m}^2/\text{s}$. Fig. (6.7) is the
normalised thermal diffusivity value for PU polymer samples. This gives a comparison study among the samples.

Thermal diffusivity is directly related to thermal conductivity \((a)\) by the equation,

\[
\alpha = \frac{k}{\rho c}, \quad \text{................................................................. (6.1)}
\]

Where,

\(k\) – Thermal conductivity,
\(\rho\) – Density of the material,
\(c\) – Specific heat.

The improvement in thermal diffusivity from the base material is largely related to the volume fraction of filler, and it’s thermal conductivity. The type of thermally conductive filler, including its size, shape, and dispersion, determine the thermal conductivity of the mixture. Spherical fillers have the least impact on viscosity increase, but also have the least impact on thermal conductivity improvement. When particles interact in such a way that a structured network is formed, thermal conductivity is enhanced. Some of the spheroid filler particles agglomerate forming a network and facilitate heat conducting through the material [11]. It also depends on the aspect ratio of the filler and it’s maximum packing fraction.
Fig. 6.6 Depicts the log amplitude versus square root of frequency for polymer samples
Fig. 6.7 Normalised thermal diffusivity values of polymer samples
<table>
<thead>
<tr>
<th>Sample Id</th>
<th>Thermal diffusivity (x10⁻⁶ m²/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.00113</td>
</tr>
<tr>
<td>2</td>
<td>0.02561</td>
</tr>
<tr>
<td>3</td>
<td>0.2951</td>
</tr>
<tr>
<td>4</td>
<td>0.1112</td>
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<tr>
<td>5</td>
<td>0.0313</td>
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<td>6</td>
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</tr>
<tr>
<td>7</td>
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</tr>
<tr>
<td>8</td>
<td>0.2879</td>
</tr>
<tr>
<td>9</td>
<td>0.4115</td>
</tr>
<tr>
<td>10</td>
<td>0.09034</td>
</tr>
<tr>
<td>11</td>
<td>0.1122</td>
</tr>
<tr>
<td>12</td>
<td>0.0615</td>
</tr>
<tr>
<td>13</td>
<td>0.0485</td>
</tr>
</tbody>
</table>

Table 6.3 Tabulated value of thermal diffusivity for PU and PU doped with various fillers

6.6 Conclusion

Welded joint was studied using PDS technique. Entrapped air-pocket or defect caused during the process of welding could be non-destructively detected. A layer of paint coated over a glass substrate was studied for its surface texture. Thickness of this layer of paint was also determined using PDS curve fitting method. Thermal diffusivity of polyurethane, before and after
adding filler components was also determined. Polyurethane doped with boron nitride and zinc oxide had the highest thermal diffusivity value and was found to be the best candidate amongst other filler combinations such as, Alumina, Mica, and Silica.
Reference


