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Measurement of the local displacement field generated by a microindentation using digital speckle pattern interferometry and its application to investigate coating adhesion

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ABSTRACT

This paper presents a technique to investigate the adhesion of thin coatings which combines digital speckle pattern interferometry and an indentation test. The proposed approach is based on the measurement of the local displacement field produced by a microindentation introduced on the coated surface of a specimen. It is experimentally demonstrated that the buckling of the coating generated by the microindentation depends on its adhesion to the substrate. Experiments carried out in specimens with different conditions in the coating–substrate interface show that digital speckle pattern interferometry can be used to determine the size of the buckled region and to give a measurement of the coating adhesion strength.

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1. Introduction

The application of a thin coating to a material increases the lifetime of many mechanical components made in conventional form by modifying their surface properties. Coatings are mainly used when the mechanical protection of a component against corrosion or exposure to high temperature is necessary. Moreover, they also find application in components that must be reinforced against high localised loads. Even though coating failure in service can be due to several causes, adhesion is usually the most important one. For this reason, it is essential to have suitable test methods to guarantee the adhesion of a given coating–substrate system.

As the definition of adhesion is not a simple issue, the characterization of this property has also generated considerable debate in the technical literature [1]. For the practitioner, adhesion can be measured as the maximum applied tension to separate the coating from the substrate. However, there are several other test methods that can be used to evaluate the adhesion of a given coating–substrate combination. Many of these tests are qualitative or semi-quantitative and the adhesion is assessed from the appearance of the fracture surface [2]. According to the properties of fragility or ductility of the coating and the substrate, different failure modes can be produced which contribute to propagate a crack or a delamination through the interface [3]. Therefore, various methods to measure adhesion are based on two steps. In the first step, a perturbation is introduced in the coated specimen to produce a delamination. The second step consists in the evaluation of the extension of the generated delamination, which can be directly related to the adhesion strength.

The local displacement field that is produced by a delamination in a coated specimen can be measured accurately using digital speckle pattern interferometry (DSPI) [4]. This is a very attractive technique in optical metrology, not only for its non contacting nature but also for its relative speed of inspection procedure, mainly due to the use of video detection and digital image processing. The application of digital techniques in DSPI allows the automation of the data analysis process, which is usually based on the extraction of the optical phase distribution encoded by the generated correlation fringes. Quite recently, the authors of this paper presented a method which combines DSPI with a bending test to evaluate coating adhesion [5]. This method is based on the measurement of the deflections produced by a pre-notched coated specimen subjected to a four-point bending test. Using specimens containing different simulated delaminations, the authors demonstrated that the deflections measured with the DSPI technique can be used to estimate the lengths of the artificially introduced defects. However, even though this method can be used to estimate the

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adhesion of a real coating–substrate interface, it is not a very practical approach as the application of the bending test needs specially prepared coated specimens.

A more practical way of introducing a perturbation in a coating–substrate system is by using an indentation test. Although the indentation techniques have become the most widely used approaches to characterise thin coating adhesion, the quantitative analysis and the interpretation of the obtained results is quite a complex problem. It is known that the introduction of a conical microindentation on the coated surface of a specimen will generate a buckling that will depend on the combination of the mechanical properties of the coating and the substrate, and also on the indentation depth [6]. At low indentation loads, radial cracking can appear in the coating. As the load is increased, a circular delamination in the coating–substrate interface is usually generated [7,8]. It can also be demonstrated that the extension of the delaminated region is directly related to the strain energy release rate, which can be used to evaluate the adhesion of the coating to the substrate [7,9,10].

In this paper, we present a combined technique based on DSPI and a microindentation test to investigate coating adhesion. It is worth to note that this optical technique had been already applied to measure the local displacement field generated by an indentation test. As a typical example, Viotti et al. [11] recently presented similar approach to measure elastic moduli of materials. Using specimens formed by a very thin bronze sheet glued to steel substrates and subjected to different adherence conditions, it is demonstrated here that DSPI can be used to measure the small local deflections generated by the buckling due to the microindentation. It is also shown that these displacement data make it possible to estimate the size of the buckled region generated by the microindentation, which can be used to analyse the adhesion of the coating–substrate system.

2. Experimental procedure

A diagram of the DSPI system used to measure the deflections of the coating due to the introduction of the microindentation is shown in Fig. 1. Basically, it was a conventional speckle interferometer designed to measure the out of plane displacement component w at the coated surface of the specimen. The light beam of a He–Ne laser with a wavelength $\lambda = 632.8\,\text{nm}$ was first divided into the object and reference beams by a beam splitter (BS). The reference beam was directed to a mirror (M) linked to a piezoelectric transducer (PZT), which was controlled by an electronic unit (PCU) that was used to introduce the phase shifts needed to evaluate the phase distribution. The reference beam was then expanded by the microscope objective (L) and was directed to the CCD camera (Pulnix TM-620) through an another BS, where it was recombined with the speckle pattern scattered by the specimen. The video camera had a zoom lens (CL) which allows to image a small region of the specimen of approximately $14 \times 14\,\text{mm}^2$ in size. The camera output was fed to a frame grabber (Matrox Pulsar) located inside a personal computer which digitised the images in grey levels with a resolution of 512×512 pixels $\times$ 8 bits.

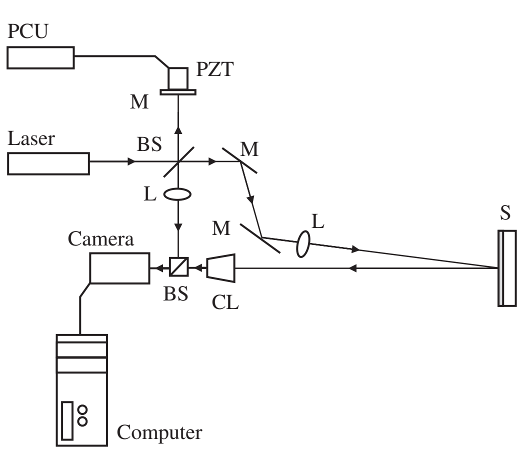

Fig. 1. Diagram of the experimental setup: beam splitters (BS), piezoelectric transducer (PZT), piezoelectric control unit (PCU), mirrors (M), camera lens (CL), microscope objectives (L), and specimen (S).

The microindentation was introduced using a scratch tester device (Teer coatings ST30) without displacing the specimen. This device has a Rockwell C spherical diamond tip of 0.2 mm of radius and a cone angle of 120°, and was located outside of the optical bench. Therefore, the specimen had to be repositioned into the same position that it had when the reference speckle interferogram was recorded. It must be noted that this procedure must be carried out with a high degree of accuracy to prevent the introduction of speckle decorrelation between both interferograms recorded before and after the introduction of the microindentation. The repositioning was performed using a specimen holder with high stability and no moving parts [12]. In this holder, the specimen rested by gravity by leaning it slightly backwards with its back surface placed against three hard metallic balls, which determined the specimen plane. Another three support pins fully determined its position. The specimen was placed with its bottom side lying on the two support pins until one of the vertical sides came to rest against the third pin.

The substrate of the specimens to be tested was a steel plate with a rectangular cross-section and a size of $50 \times 35 \times 5\,\text{mm}^3$. As one of the main problems to study adhesion problems is the difficulty of preparing specimens in which this property can be varied over a wide range; as coating, we have decided to use a bronze sheet with a thickness of 0.25 mm. The bronze sheet was glued to one of the surfaces of each steel plate by means of an epoxy resin. This methodology allows us to prepare specimens in which the adhesion can be changed through the smoothness variation of the coated surface of the substrate and also by increasing the adhesive curing time.

To generate the speckle patterns, the smooth bronze surface of each specimen was covered with a very thin layer of white paint. The thicknesses of the paint layer and the epoxy resin were evaluated by analysing a cross-section of one specimen, which was obtained by slicing it with a diamond saw. Both thicknesses were measured using an optical microscope. The average thickness of the paint layer resulted to be approximately 6 μm, which can be neglected in comparison to the thickness of the bronze sheet. This measurement also confirmed that the adhesive layer had an average thickness of 60 μm, which means that it was more than four times thinner than the bronze sheet.

As previously mentioned, different interface conditions were obtained by polishing the surface of the steel substrates that were glued to the bronze sheets with sandpapers of different grain sizes. In order to obtain reproducible interface conditions, the polishing was performed by displacing the sandpaper along the same direction in all specimens. It was supposed that the different depths of the parallel microgrooves that were generated in the polished substrate surfaces will change the effective adherence.

The experimental procedure used to record the pair of speckle interferograms to be correlated was as follows. First, each specimen was located at the specimen holder and the reference speckle interferogram was recorded. Then, the specimen was positioned in the scratch tester device, which was located outside the optical bench. The microindentation was introduced over the painted bronze surface by applying the selected load to the diamond tip during approximately 30 s. Finally, the specimen was repositioned at the specimen holder and the second speckle interferogram corresponding to the deformed state was recorded.

3. Data analysis

The out of plane displacement component w produced by the introduction of the microindentation in each specimen was determined from the phase difference $\Delta\phi$ coded by the generated DSPI fringes. As it is well known, the relationship between the measured displacements and the phase difference in a speckle interferometer with out of plane sensitivity and normal collimated illumination is given by [4]

$$w = \frac{\lambda}{4\pi}\Delta\phi \quad (1)$$

The wrapped phase distribution was evaluated using the Carré phase-shifting technique, which is based on the processing of two sets of four phase-shifted speckle interferograms recorded before and after introducing the microindentation [4]. Additionally, a local smoothing with a kernel of 3×3 pixels was introduced in order to increase the signal-to-noise ratio of pixels having low intensity modulation.

The unwrapped phase was determined using an iterative robust unwrapping algorithm based on the minimum L^0-norm solution [13]. This algorithm allows the evaluation of the continuous phase distribution and is not influenced by inconsistent pixels generated by noise or erroneous discontinuities which are generally present in DSPI phase maps. Additionally, all phase maps obtained with this procedure were processed with a method applied to remove the rigid body displacements that can be introduced when each specimen was repositioned after the introduction of the microindentation [14]. This method is based on a least square calculation of three correction parameters determined from the displacements measured at pixels located along a circular path centred at the microindentation. As the radius of this path was large in comparison with the size of the microindentation, it can be assumed that the displacement field produced at the previously mentioned pixels was negligible.

4. Experimental results

4.1. Size of the buckled region for specimens with different interface conditions

The local out of plane displacement component generated by the microindentation was first evaluated in specimens having

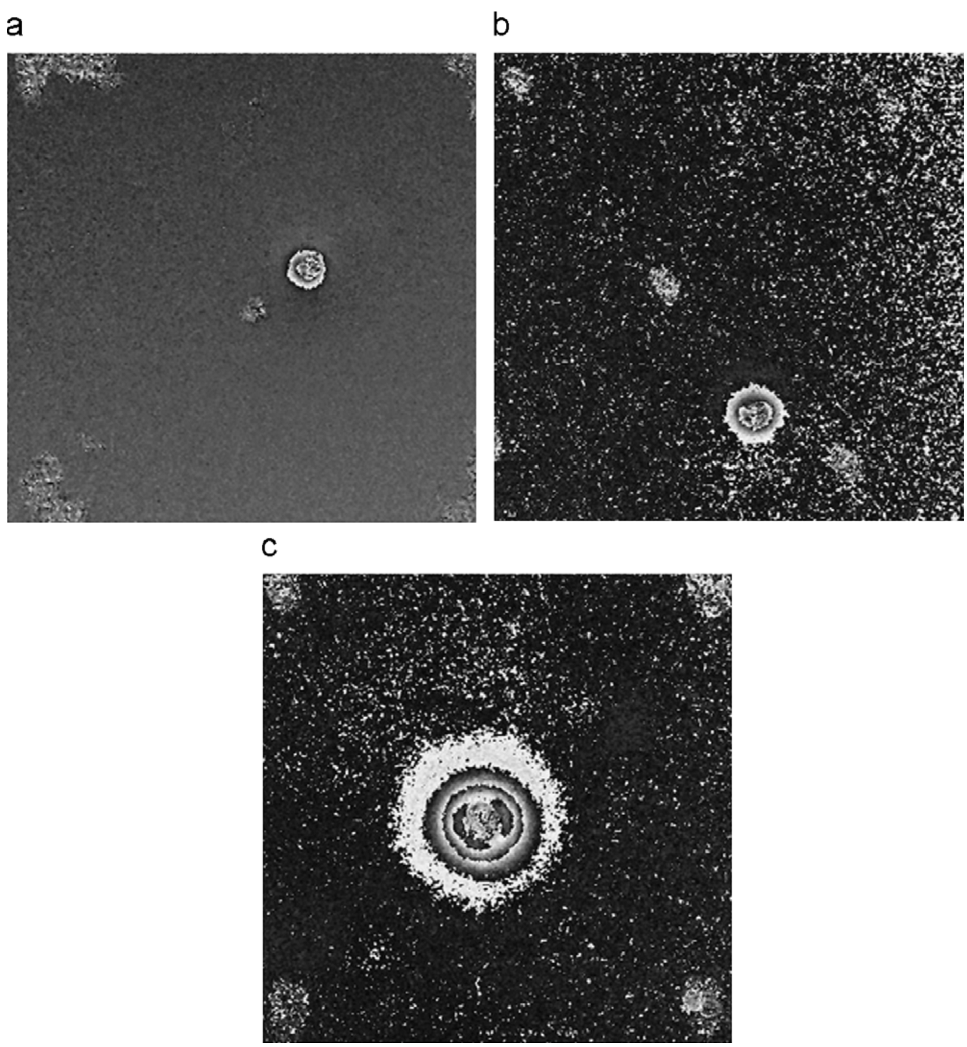

Fig. 2. Wrapped phase maps produced by specimens with different interface conditions: (a) substrate polished with sandpaper number 80; (b) substrate polished with sandpaper number 240; and (c) substrate polished with fine grain alumina.

different coating–substrate interface conditions. These specimens were obtained by polishing each steel substrate with sandpapers having different grain sizes. In all tests, the speckle interferograms were acquired 24 h after the coating was glued. Also, all tests were performed using an indentation load of 30 N.

Fig. 2(a) shows the wrapped phase map obtained by testing a specimen with its substrate polished with sandpaper number 80. Fig. 2(b) and (c) display the wrapped phase distributions produced by specimens with their substrates polished with sandpaper number 240 and fine grain alumina, respectively. In these figures, it is observed that the local wrapped phase maps have a circular shape. Therefore, these local wrapped phase distributions confirm that the buckling introduced by the microindentation was axisymmetrical, as it was expected.

In our coating–substrate system, we could not confirm if the buckling produced by the introduction of the microindentation was caused by a delamination related to a loss of adhesion or cohesion in the adhesive layer. However, by plotting the out of plane displacement component measured along a radial direction, with the origin of the coordinate system located at the indentation centre, it was possible to estimate the radius R of the circular buckled region. Fig. 3 shows the out of plane displacement component w plotted as a function of the radial coordinate r, that was obtained from a typical phase map similar to those shown in Fig. 2. The out of plane displacement curve shown in Fig. 3 clearly displays two minima located at both sides of the origin of the radial coordinate r. The distance l between both minima was taken as the diameter of the buckled region generated by the microindentation over the coated surface of the specimen. Therefore, the radius R of the buckled region that can be used to characterise the adhesion of the coating system is given by $R = l/2$. Fig. 3 also displays the diameter d_i of the indentation mark and the diameter d_s of the region presenting speckle decorrelation. This last region, which was generated by the severe change of the microstructure generated in the neighbourhood of the microindentation, does not produce reliable displacement data. The top right position of Fig. 3 shows a photograph of the microindentation mark.

The radius R of the buckled region that was produced by each of the three specimens, whose wrapped phase maps were shown in Fig. 2(a–c) and was determined using the previously described approach, was 0.9, 1.3 and 3.1 mm, respectively. As expected, these results show that the radius of the delaminated region increases as the grain size of the sandpaper was reduced. This behaviour can be due to the fact that a substrate polished with a polishing paper having a small grain size reduces the anchoring of the epoxy adhesive, so that the effective adhesion strength between the substrate and the coating is decreased.

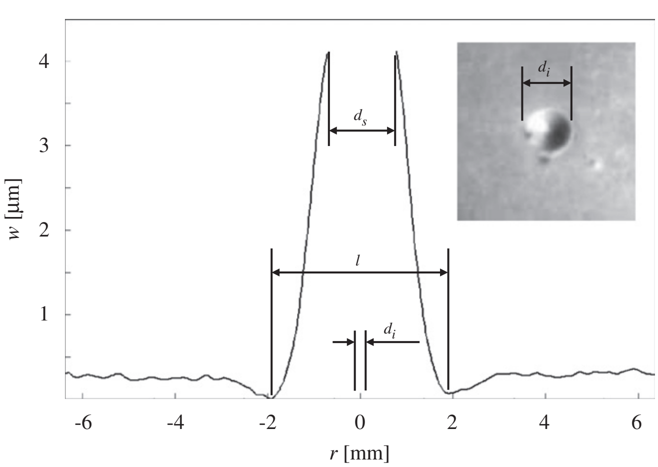

Fig. 3. Out of plane displacement component w produced by a microindentation along a radial direction. The figure also displays the diameter of the circular buckled region (l), of the region presenting speckle decorrelation (d_s) and of the indentation mark (d_i).

4.2. *Size of the buckled region as a function of the adhesive curing time*

The effect of the curing time of the epoxy resin was analysed by measuring the local displacement component w generated by various microindentation tests performed at different times after the coating was glued to the substrate. To make sure that the same interface conditions were used in all tests, all microindentations were introduced at different places over the coated surface of a single specimen. This procedure can be used because the local displacement field introduced by a microindentation is negligible at a distance of 1 cm from its centre.

Fig. 4 shows the out of plane displacement component w that were measured along the radial direction r when the microindentation was introduced at 30, 90 and 150 min after the coating was glued. In these tests, the substrate was polished with sandpaper number 80 and the indenter load was 30 N. The radius values R of the buckled regions that were determined at 30, 90 and 150 min after the coating was glued were 2.1, 1.2 and 1.0 mm, respectively. As the radius of the buckled region is inversely proportional to the adhesion strength, as expected these results confirm that the adhesion of the epoxy resin increases with the curing time. Additional tests carried out at times longer than 3 h after the coating was glued, did not show any change in the measured displacements. Therefore, these last results suggest that the technique proposed in this paper allows the confirmation that the epoxy resin used to glue the coating cures completely at times longer than 3 h.

4.3. *Size of the buckled region as a function of the indentation load*

Finally, the local displacement component w was also measured for different values of the indentation load. As before, the microindentations were introduced in a single specimen in order to make sure that the same interface conditions were used in all tests. Fig. 5 depicts the radius values of the buckled region plotted as a function of the indentation load, when a specimen with a substrate polished with sandpaper number 120 was used. The same figure also shows the linear fit obtained from a least square calculation of the measured data. It is observed that this line fits quite well with the measured data and proves the linear dependence between the radius of the buckled region and the

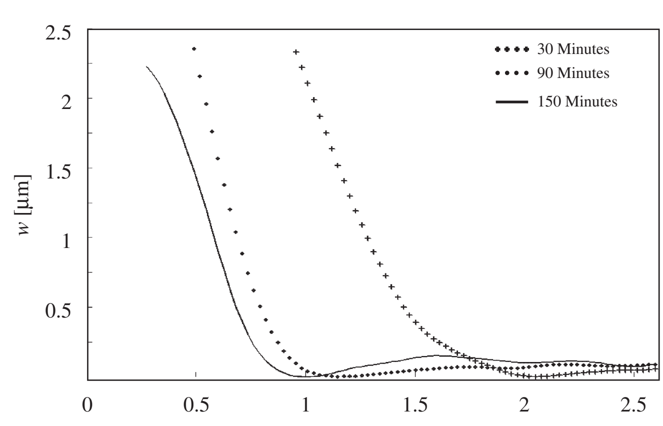

Fig. 4. Out of plane displacement component w measured along a radial direction at 30, 90 and 150 min after the coating was glued.

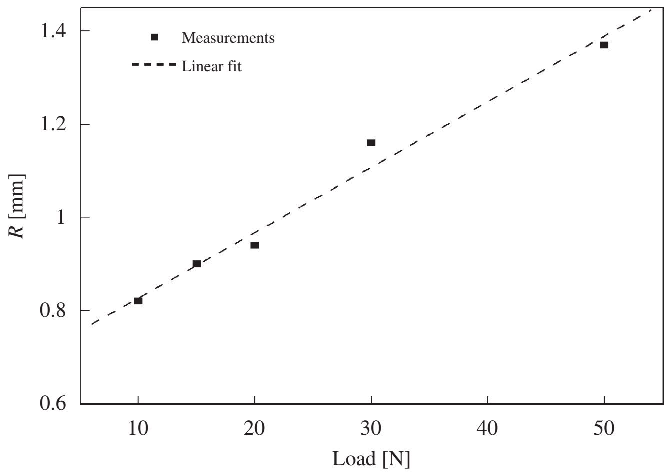

Fig. 5. Radius R of the circular buckled region as a function of the indentation load.

indentation load. It is also important to note that the linear behaviour obtained in the experiments agrees very well with the dependence of the radius of the delaminated region that is generated in a buckled coating as a function of the indentation load, which was proposed by the theoretical model presented in Ref. [10].

5. Conclusions

This paper presents the evaluation of a technique based on DSPI and a microindentation test, which is proposed to investigate the adhesive performance of coatings. Experiments performed on coated specimens with different interface conditions demonstrate that DSPI enables the measurement of the tiny buckling generated in the coating, after the introduction of a microindentation. Since these data allows the determination of the radius of the buckled region that is directly relevant for the estimation of the adhesion strength, the obtained results could be compared with the theoretical predictions given by existing models.

It is demonstrated that the proposed technique allows the determination of the radius of the circular buckled region produced by different substrate–coating interface conditions. The change in adhesion can also be successfully monitored as a function of the adhesive curing time. As it is expected, the experiments confirm that shorter delamination radii are obtained when the time between the coating adhesion and the introduction of the indentation is increased. Finally, the evaluation of the radius of the buckled region as a function of the indentation load is in good agreement with the predictions of linear dependence given by a theoretical model.

The obtained results demonstrate that the proposed technique could be used as a valuable tool to investigate the adhesion performance of coatings. Furthermore, as the size of the microindentation needed for the measurements is very small, the method can be considered as nearly non destructive. Therefore, this approach could result quite adequate to be used in the industry for the in-line testing of coated components. A portable speckle interferometer combined with a microindentation device will be the subject of future investigations.

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