

# Detection of the Corrosion of Aluminium Alloy by Optical Method

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## Abstract

The Speckle effect is a discipline with whole share. It is exploited in various scientific fields. On the application plan, it became a very powerful tool for the characterization and the non destructive testing. This technique which makes it possible to control and quantify in real time several parameters such as: the roughness of surface, the vibrations, the constraints, microphone-displacements, the microstructure of surfaces, can be applied, at the same time, with the objects transparent and diffusing, solid or liquids. The development of flexible tools of capture of images to high resolution (camera CCD) coupled to information processing systems, nowadays makes the techniques speckle particularly important and interesting for industrial control. Our work consists in controlling qualitatively and quantitatively the variation thickness extracted the surface of the Duralumin sample (Aluminium alloy) immersed in a Salt water (to 3.5%), by using a camera CCD connected to a computer equipped with a software of image processing PRO-PLUS

**Key words:** DSPI Digital Speckle Pattern Interferometry, Phase Evaluation Method; Phase unwrapping; Non destructive testing, Image processing.

## 1. Introduction

The speckle effect is the manifestation of the strong space coherence of a source of light. It is observed when one lights an object diffusing by a laser beam. This luminous granularity is not other than the result of the interference of the waves coherent but by chance out of phase (shift of phase) resulting from the object. Consequently, if this shift had been suddenly modified, it means that if surface is deformed, the structure of the speckle would be also modified. By studying the modification of the structure of the speckle (displacement, disappearance or appearance of luminous points), one can go up with one of the parameters (deformation, roughness, microphone-displacement, vibration etc...) of the surface of the object in a qualitative but especially quantitative way. The technique which makes it possible to go up with the parameters quoted above, is digital interferometry speckle. It consists in making the difference or the addition in two figures of fields of speckle before and after having undergone with the object a modification. The result can generate fringes of correlation which correspond exactly to the variation of the state of the object. The present study, consists of controlling qualitatively and quantitatively the variation thickness extracted the surface of a Duralumin sample by interferometry speckle, since the immersion of material in salt solution until the beginning of the dissolution of metal, while passing by the various stages included in the process of the rupture of the passive film which precedes the pitting. This control is done by collecting the images by a camera CCD connected to a computer

equipped with a software of image processing PRO-PLUS [1].

## 2. Theory

Interferometry speckle uses the information of the phase contained in each grain of speckle. To obtain this information of phase, it is necessary to make interfere the wave of speckle with a wave of reference (the device can be assimilated to

The interferometer of Michelson). This requires rather strict conditions on the slope of the wave of reference and the curve of its face of wave. That is to say the image of speckle coded [2, 3, 4]:

$$R + Se^{i\varphi}$$

Either  $R(x, y)$  the average amplitude of the wave of reference on the grain to the point  $(x, y)$ , or  $S(x, y)$  that of the wave of speckle and or the difference in average phase between these two waves.

Note: vary by chance from one grain to another because unspecified surface is supposed.

The intensity observed is given by the module to the square of the amplitude [5,6]:

$$I(x, y) = (R + Se^{i\varphi})(R + Se^{-i\varphi}) \quad (1)$$
$$= R^2 + S^2 + 2RS \cos\varphi$$

By posing :  $I_0(x, y) = R^2 + S^2$

$$\text{and: } V(x, y) = \frac{2RS}{R^2 + S^2}$$

One obtains:

$$I(x, y) = I_0(x, y)(1 + V(x, y)\cos\varphi) \quad (2)$$

Will the disturbance of the object create a local dephasing (it means that depend on the point (x,y)) between the wave of speckle and the wave of reference. The intensity is then given by:

$$I_2(x, y) = I_0(x, y)(1 + V(x, y)\cos(\varphi + \Delta\varphi)) \quad (3)$$

Consequently, the technique applies as follows: one takes an image of the object before disturbance, which gives us  $I_1(x, y)$ . One takes then an image of the object after disturbance, which gives us  $I_2(x, y)$ .

The subtraction of these two images is made pixel by pixel. The result is given by:

$$I_{tot}(x, y) = I_1 - I_2 = I_0(x, y)V(x, y)[\cos\varphi - \cos(\varphi + \Delta\varphi)] \quad (4)$$

By making a trigonometric transformation, one finds:

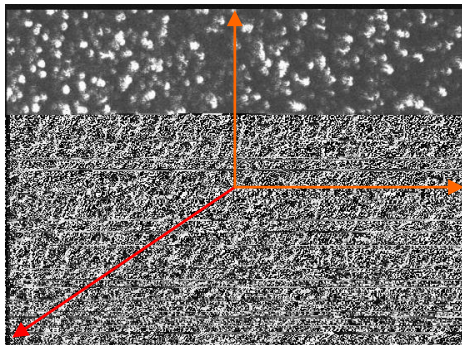
$$I_{tot} = 2I_0V\sin\left(\frac{\Delta\varphi}{2}\right)\sin\left(\varphi + \frac{\Delta\varphi}{2}\right) \quad (5)$$

They are fringes of intensity.

Dephasing  $\Delta\varphi$  on the difference of way optical  $\Delta L$  depends between the state of reference and the disturbed state, and is expressed as follows [7, 8]:

$$\Delta\varphi = (2\pi/\lambda)\Delta L = (2\pi/\lambda).2\Delta(n.e).\cos\varepsilon \quad (6)$$

Where « n » is the index of refraction of the medium, « e » the thickness of the extracted layer and « ε » is the angle of refraction inside the layer.



### 3. Principle of the technique

#### 3.1. Experimental device

The diagram of the breadboard construction employed to acquire the figures of fields of speckle is represented by figure 1.

Is the source of light used a laser source He-Ne (emitting in the red with  $\lambda = 632.8\text{nm}$ ), in order to have a length of sufficient coherence and a high brightness. The beam widened by an objective of microscope (OB) of enlargement 10x is filtered spatially (filter of diameter  $25\ \mu\text{m}$ ) then collimated by a photographic objective (O.P.H.)4.5/ 210. The outgoing beam of this last, crosses a first separating cube, which separates it in two parts. A part of the light goes directly on a second separating cube, arrives at the  $M_1$  mirror, which it considered on a third separating cube and falls then on the matrix from the CCD. This beam is called beam of reference. The second beam falls on the mirror  $M_2$  which it considered on the sample (this one is fixed vertically

by its higher end on a metal support using a glue) of Duralumin plunged in a rectangular basin out of glass containing a salt water solution with 3,5%. This beam covers a great part of the sample of dimension 2cm X 1cm. The beam diffused by the sample (called beam object), positioned the plan of capture parallel to, crosses the photographic objective (O.P.H.) 4.5/75, then the third cube and is interfered then with the wave of reference on the same matrix of the CCD, connected to a computer making it possible to acquire the images and to digitize them on 256 levels of gray. The images are then sent towards the software to treat them.

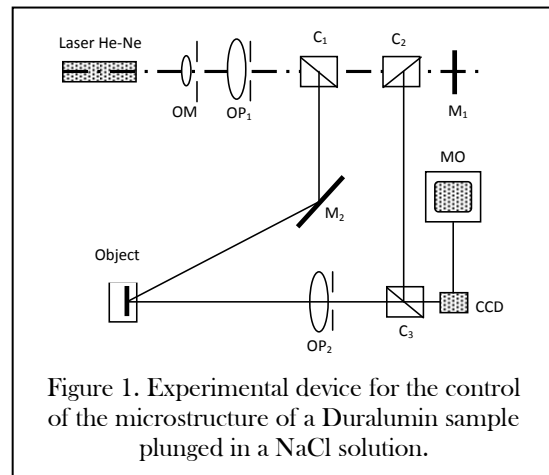


Figure 1. Experimental device for the control of the microstructure of a Duralumin sample plunged in a NaCl solution.

#### 3.2. Analyze

Before carrying out the qualitative and quantitative control of the variation thickness extracted from our samples, one takes an initial image with the state of the interference of the two waves of an object plunged in the solution, represented by the equation (2).

Then one takes other images (interferograms) after having immersed the sample during a certain time, and which is represented by the equation (3).

With one  $\Delta\phi$  who varies according to the removed thickness of the sample.

#### 4. Results and interpretations

To show the method described previously, one takes image of reference and several images of the same sample immersed in the solution chlorinated to different times: from 2 minutes up to 120 minutes. Figure (2) represents 12 specklogramms, which show the images of each state of variation of the influence of the solution on material to be controlled. These specklogramms is not other than parallel interference rings with a spacing (distance between interference rings) no constant for each image.

On the figure (2), One notices that there is increase in the number of fringes (decorrelation between the fields of speckle), therefore the distance between interference rings « i » decreases. This is explained by the fact that there was a variation of the difference in walk  $\Delta L$ , therefore formation of a passive film on the surface of the sample. The variation thickness of this film is deduced starting from the equation (6) and the following relation:

$$i = \lambda/\Delta L.$$

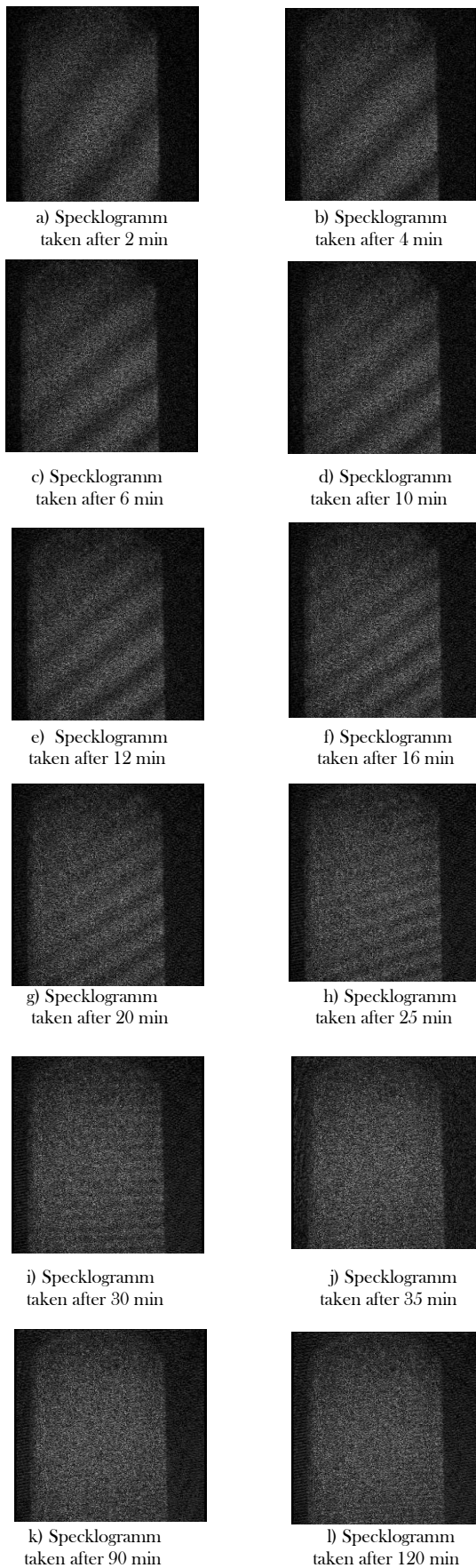
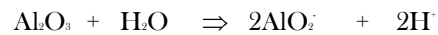


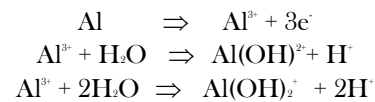
Figure 2. Representation of specklogramms relating to the states of change of the microstructure of the Duralumin sample plunged in the NaCl solution.

The specklogramms (b), (c), (d), (e), (f) and (g) correspond at the first stage which is the formation of the passive film  $Al_2O_3$  with reinforcement starting from the molecules of  $H_2O$  (from the point of view chemical or electrochemical, the formation of this film requires at least three stages which correspond to the quoted specklogramms: initial dissolution of the matrix, hydrated oxide formation or  $Al(OH)_3$  hydroxide then dehydration of this last and obtaining Alumina. (G.Okamoto, T.Shibata, 1978 et C.Vargel,1999).

After the adsorption of Cl on the surface, the passive film will be destroyed according to the following reaction:



This stage corresponds to the specklogramms (h) and (i), which show a decorrelation of the fringes by the introduction of new fringes. One thus attends a beginning of corrosion. While specklogramms (j), (k), and (l) (preserve the same figure of the object) corresponds at the stage of dissolution of the base metal. Once the dissolved passive film, the base metal is found naked and one can have the possibility of the following reactions:



Cependant the two harmful reactions which can take place is:

- Formation of chlorides such as:  
 $Al^{3+} + 3Cl^- \Rightarrow AlCl_3$  (which are soluble)
- And that of  $AlOHCl$  in solution :  
 $Al^{3+} + H_2O + Cl^- \Rightarrow AlOHCl$  (soluble also).

the process of formation and dissolution of these two products, will activate the process of the oxidation of the base metal and thus one attends the starting of a piquuration. From these results, one could estimate the variation thickness of passive film formed on metal.

### 5. Detection of the beginning of corrosion by method ESPI

#### 5.1. Capture figures of speckle

An initial figure of speckle ( $S_0$ ) of the object after its immersion in the solution of 3.5% of NaCl, is recorded. After 12 minutes of immersion, one moves the piezoelectric one by quantities  $0, \pi/2, \pi, 3\pi/2$  successively. Four figures of speckle of ( $S_1$  to  $S_4$ ) corresponding to each shift of phase are recorded.

#### 5.2. Treatment of the interferograms

Four interferograms of ( $I_1$  to  $I_4$ ) are generated by the calculation of the square of the difference between the figures of speckle of the beginning of the immersion of the object up to 12 min of immersion ( $I_i = (S_i - S_0)^2, (i = 1-4)$ ). These operations are carried out using the software Image-Pro Plus.

The analysis of these interferograms will be carried out by the software Fringe Processor 3, from where procedure of calculation of the phase which proceeds according to the following stages.

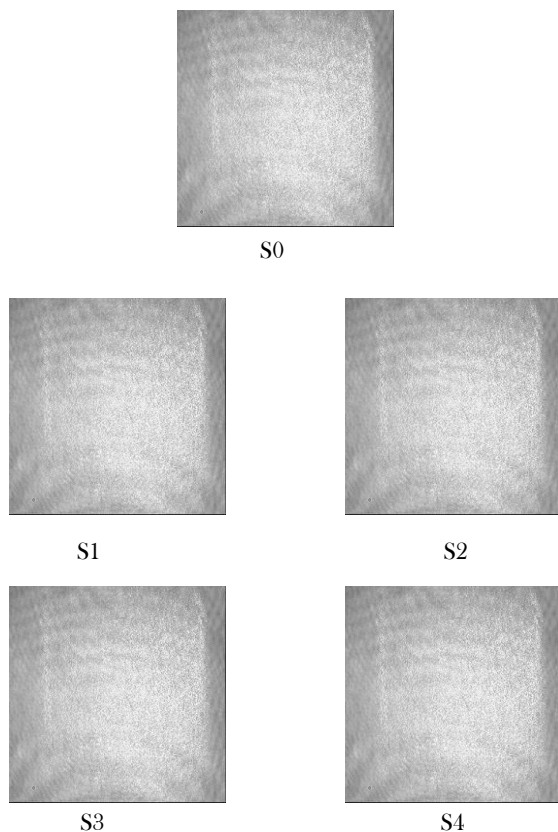


Figure 3. Images of speckle, (S0) image initial, (S1 to S4) images collected after 12 min of immersion.

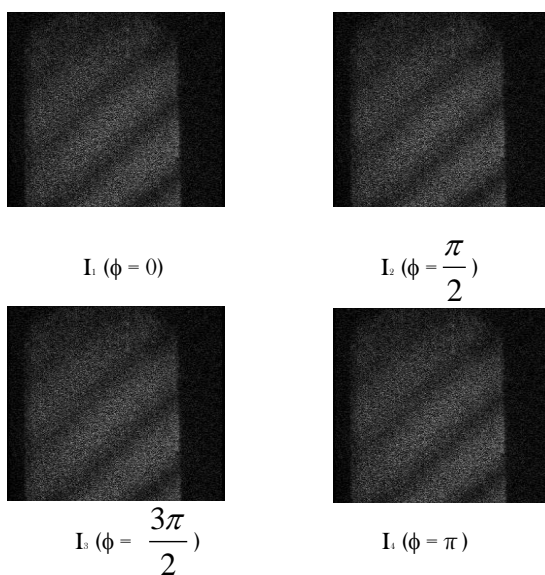


Figure 4. Interferograms obtained by the technique of shift of phase after 12 min of immersion.

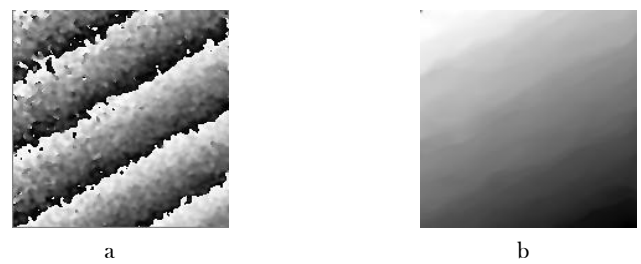


Figure 5. a- Distribution of wrapping phase. b- Distribution of unwrapping phase

5.3. Representation of the wrapping and unwrapping phase

To represent the distributions of the wrapping and unwrapping phase (see figure 5 a and b), we chose the algorithm with four images (with a constant increment  $\pi/2$ ) (D.W.Robinson, 1993)

5.4. Exploitation of the phase in three dimensions

The exploitation of the phase unwrapping in three dimensions enables us to know the value and the direction of the evolution thickness of film formed on the base metal. Once the phase is determined, the product «  $e.n = a$  » can be calculated starting from the distribution of the unwrapping phase. The following equation enables us to calculate «  $e.n$  » out of the plan, which is expressed by (J-I Kato, I. Yamaguchi, and Q. Ping, 1993):

$$\Delta\phi = (2\pi/\lambda)\Delta L = (2\pi/\lambda).2\Delta(n.e).\cos\epsilon''$$

with  $\epsilon''$  the angle of incidence under which the object is illuminated, «  $e$  » is the thickness of film and «  $n$  » is the index of refraction of alumina.

From the results recorded for various immersion times, we can represent the variation of the thickness of the passive film with time of immersion of the metal.

one notices that the more the duration of the samples in the chlorinated medium increases, the more the thickness increases.

One as sees on this figure as :

- Firstly this thickness does not evolve/move in the same way on all surface, that can be due :
- A nonuniformity of surface (difference of roughness and the slope of the sample);
- Or with the difference in concentration of the dissolved oxygen on both sides of the surface of the sample;
- Or many two at the same time.
- Secondly the values found thicknesses more important are compared with the values quoted in the bibliography and which are of the order of (nm). This difference is probably due to the hydration of the sample.

The values obtained in experiments can agree with the thicknesses of the layer of Alumina formed in various mediums. In fact this thickness depends on the nature of material, of the conditions of its preparation, the nature of the electrolyte (medium) and the duration.

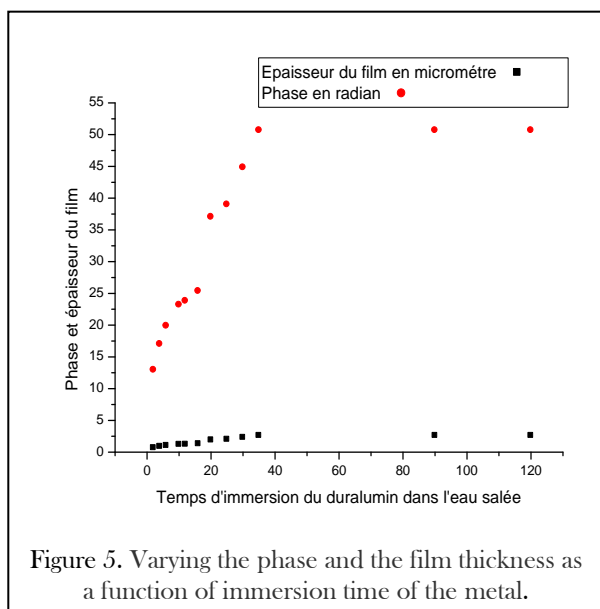


Figure 5. Varying the phase and the film thickness as a function of immersion time of the metal.

## 6. Conclusion

The technique speckle is a technique no destructive rapid and precise. It makes it possible to take qualitative and quantitative measurements static or dynamic deformations at weak frequencies, provided that the deformation does not exceed the diameter of the grain of speckle. It is also shown that a mathematical operation such as the difference in two images in two different states, makes it possible to generate fringes of correlation. In this work one showed that the

chemical reactions under consideration for the formation and the destruction of passive film, agree well with our practical results. With the technique of interferometry speckle and by combining to him the technique of shift of phase (phase shifting), one also could evaluate the variation thickness of passive film formed according to the time of immersion of our samples in a salt solution.

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