Problems in Quantification of Pitting Corrosion

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Abstract

Pitting corrosion is a localized type of corrosion in the form of holes that can penetrate in materials extremely rapidly. It is one of the most frequent forms of corrosion in stainless steels, mainly caused by chlorides. Quantification of pitting attack has been the focus of intense inquiry in recent years. Pitting corrosion is quantified in different ways e.g. average pit depth measurement, maximum pit depth measurement, remaining wall thickness and affected area due to pitting.

Measurement of average pit depth is complicated by the fact that there is a statistical variation in depth of pits. There is always a large variation in pit size on the same specimen so interpreting results in terms of average pit depth is critical. Measurement of the maximum pit depth is also not a reliable way to express the pitting damage. In this paper, advantages and disadvantages of some used methods of quantitative characterization of pitting corrosion are explicated. A detailed comparison of three methods, i.e. optical microscopy, 3D optical microscopy and X-ray radiography have revealed that high resolution X-ray radiography is a powerful and easy method to quantify pitting corrosion both in terms of pit depths and percentage of affected area. This method overcomes common problems like high scatter of results, high personal and cost efforts with low reproducibility for other methods. Principle, accuracy, merits and limitations of the proposed methodology are discussed.

Keywords: Pitting corrosion, pit depth, optical microscopy, 3D optical microscopy, x-ray-radiography.

Introduction

Pitting corrosion is one of the most prevalent forms of localised corrosion that is a dangerous phenomenon because it is difficult to detect and predict. The attack is highly localised and pits can penetrate inwards extremely rapidly and the deepest of them can damage the structure by perforating the material. The pitting corrosion of stainless steels is the most important corrosion processes that affect the service behaviour of these materials [1]. Weight loss and wall thickness reduction is not an appropriate and trustworthy way to interpret the pitting corrosion because pitting corrosion is a localised form of attack. Quantification of pitting corrosion is very important for understanding the behaviour of material and help to measure the corrosion distribution leading to a better understanding of how the material is affected during its service. This also helps to develop a ranking for a choice of the right material for a specific application.

There are various ways to quantify the pitting corrosion attack e.g. average pit depth measurement, maximum pit depth measurement, pitting density (pits/cm^2) and remaining wall thickness due to pitting. A detailed comparison of three techniques, i.e. classical optical microscopy, 3D optical microscopy and X-ray radiography was done during this research. The application of a recently modified radiographic technique for corrosion quantification is described in this paper. This system has some possible advantages compared to other conventional quantification methods.

X-ray radiography:

Digital radiography is one of the oldest NDT techniques, which is in use since long time ago as a monitoring method to detect major flaws and severe corrosion attacks in many industrial systems. X-rays are generated by means of specially designed high vacuum tubes (X-ray tubes). When electrically operated this tube emits penetrating radiation beams known as X-rays. The beam from equipment penetrates a piece of metal, and the amount the beam is attenuated depends on the thickness of the material, and hence the intensity of the transmitted beam varies with position. Step wedges are used for the calibration and standardization of X-ray machines. Also, when an object with various thicknesses is radiographed, a step wedge of the same material is used. The resulting radiograph shows the dimensional features of the part. In a photograph, the thinnest portion of a sample will cause dark image while the thickest portion where the intensity was low will cause a bright image. The principle of digital radiography is shown in figure 1.

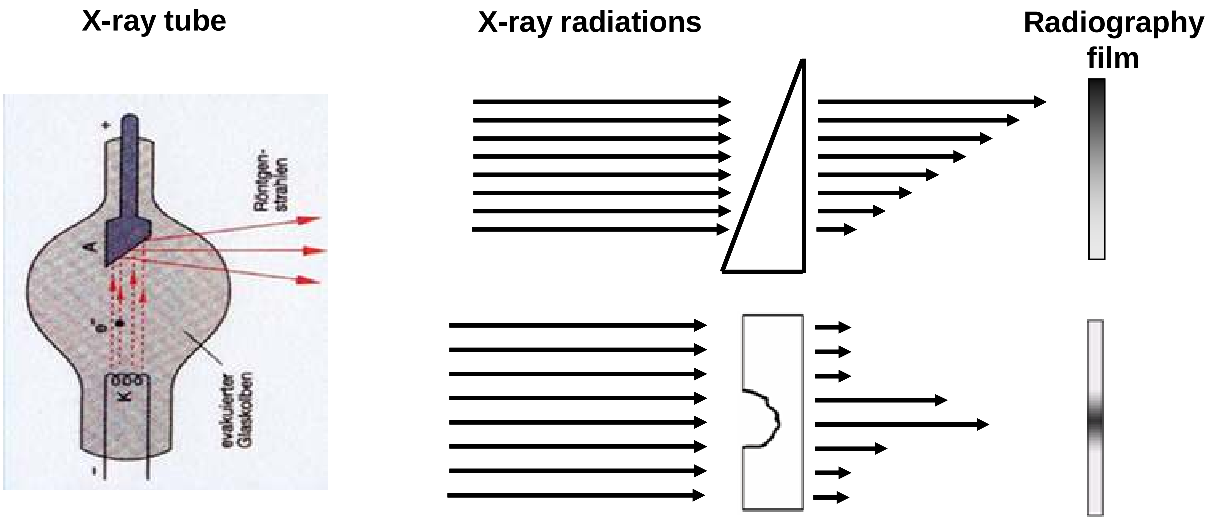

Figure 1: Radiographic image of calibration sample and corroded sample [3]

In this work specimens from cyclic corrosion tests were investigated with the help of this method. Figure 2 shows a coupon and its radiographic images with two different filters.

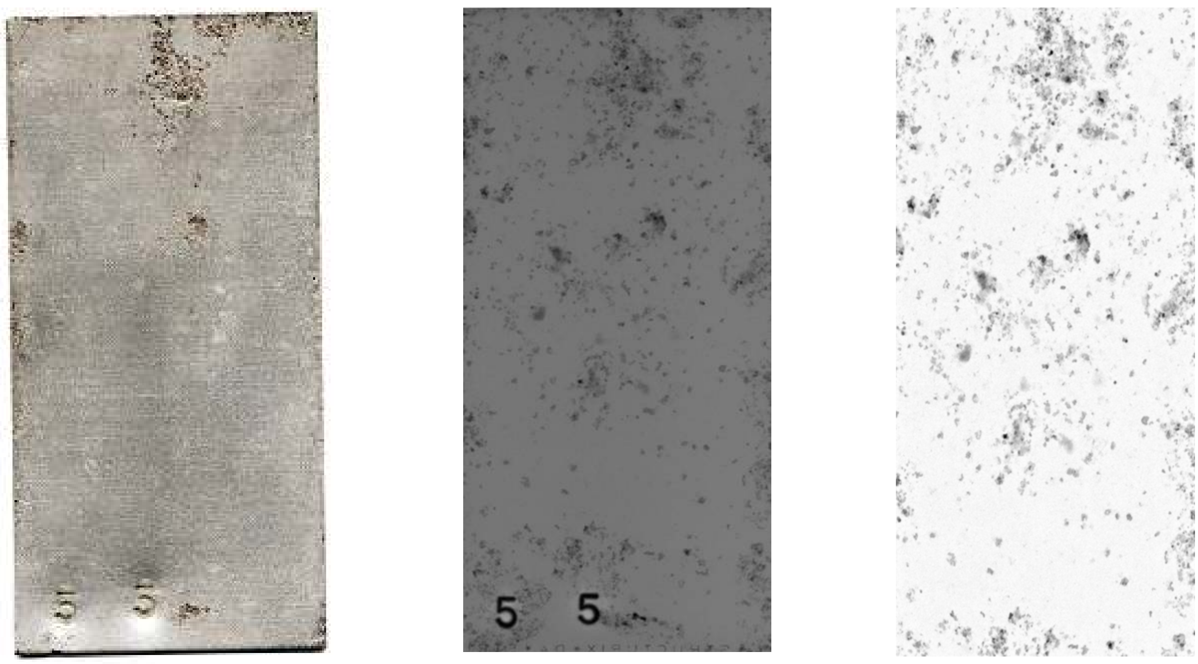

Figure 2: Corrosion coupon with its radiographic images

The figure 3 shows an example of quantification of corrosion attack in terms of percentage affected area and reduction in wall thickness of a specimen due to corrosion attack.

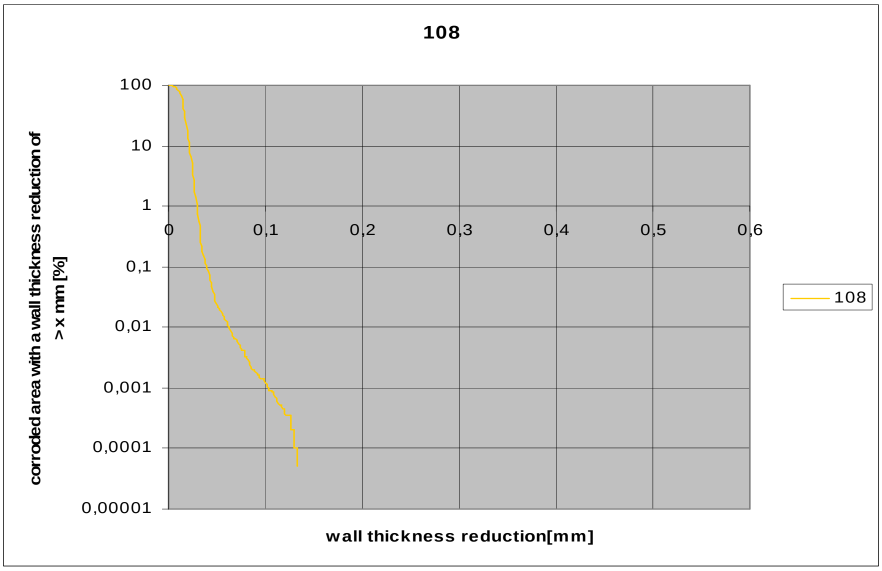

Figure 3: Wall thickness reduction VS percentage infected area

The probability of the pits that initiate on a specimen surface is calculated by using statistical extreme value distribution method. The development of extreme value theory for engineering applications has provided the mathematics to predict the deepest corrosive attack in a large structure from a limited number of measurements on a portion of the structure. An example of extreme value distribution plot of maximum pits is shown in figure 4.

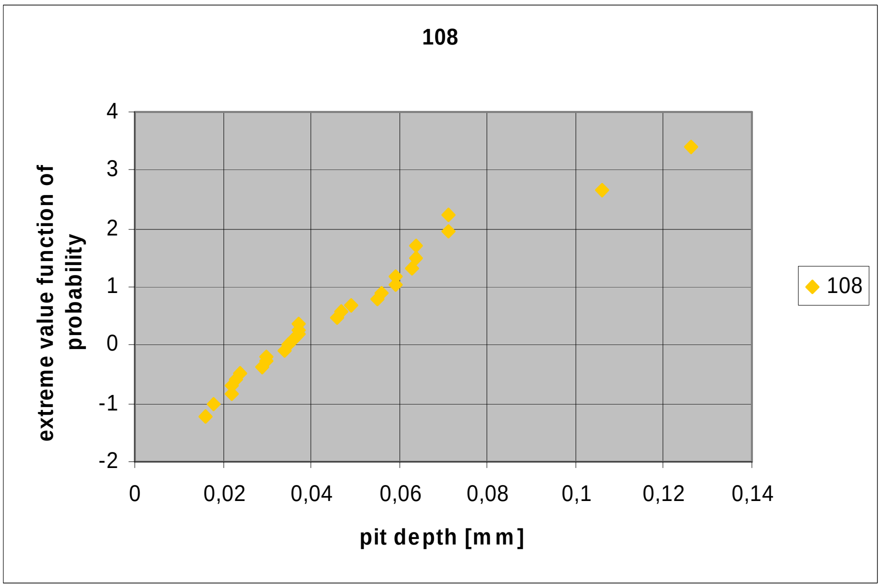

Figure 4: Extreme value distribution plot of the deepest pit data

3D-optical microscope:

The use of 3D optical microscope to quantify pitting corrosion is also common now a day. Using these measuring tools enables quantification of corrosion damage with high resolution visualisation. In this research an Infinite Focus Measurement Machine (IFM) was used to measure pit depths and to obtain 3D data set from a sample surface. Like other pitting quantification methods it's also a non-destructive method and in use in many corrosion laboratories around the world. Its operating principle is Focus Variation. This moves the focus plane of a known objective lens vertically over a surface which in conjunction with Smartflash illumination builds up a 3D full colour model of the surface to be examined. This provides the 3D topography of the analyzed surface, which enables to distinguish between corroded and un-corroded areas of the specimen. The IFM method allows capturing images with a lateral resolution down to 400 nm and a vertical resolution down to 10 nm. In this research entire surface of specimens were evaluated by an automated 3D optical microscope with statistical data analysis. Characterized parameters were the maximal pit depth and pit volume. An example of pit depth measurement with this method is shown in figure 5.

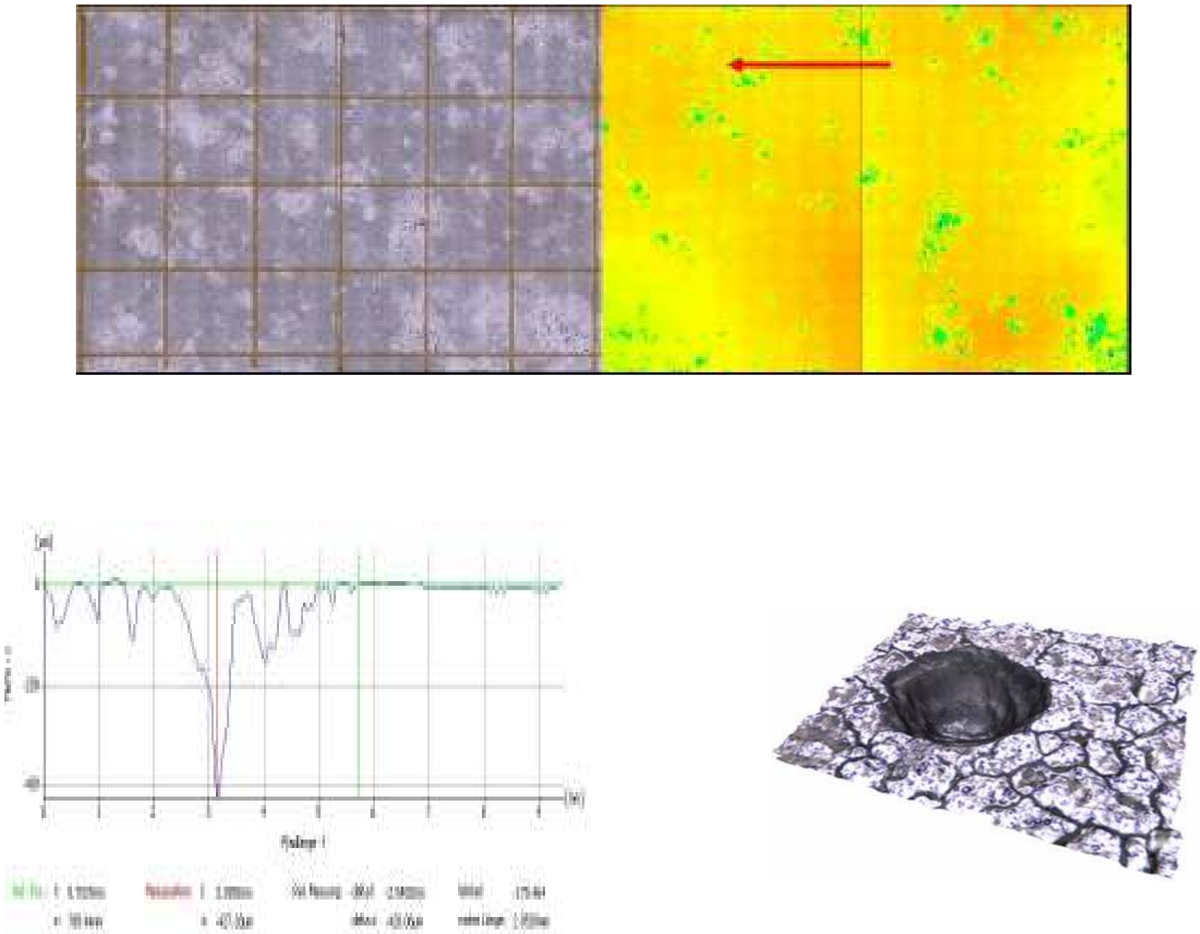

Figure 5: Measurement of pit depth with 3D microscope [3]

Conventional Optical Microscopy:

The use of conventional optical microscope to measure pit depths is one of the well known oldest method and still not exhausted [4]. During this study the pit depth distribution was measured by using an optical microscopy with a calibrated eyepiece. The sample was marked into equal parts and pit depths were measured in each section. In a first step a single pit was located on the specimen's surface. The pit depth was a difference in height of a sample surface and bottom of a pit measured by focusing microscope's knob as shown in figure 6. Depths of pits were measured one by one with this technique and normally results were interpreted in terms of the maximum pit depth or the average of the ten deepest pits. High personal efforts are required to locate and measure depths of pittings on a whole surface of a specimen. The rate of accuracy in measurements is also not very high due to less resolution and personal errors. Optical microscope (Olympus BX51M) was used in this study to measure pit depths on steel coupons after corrosion tests.

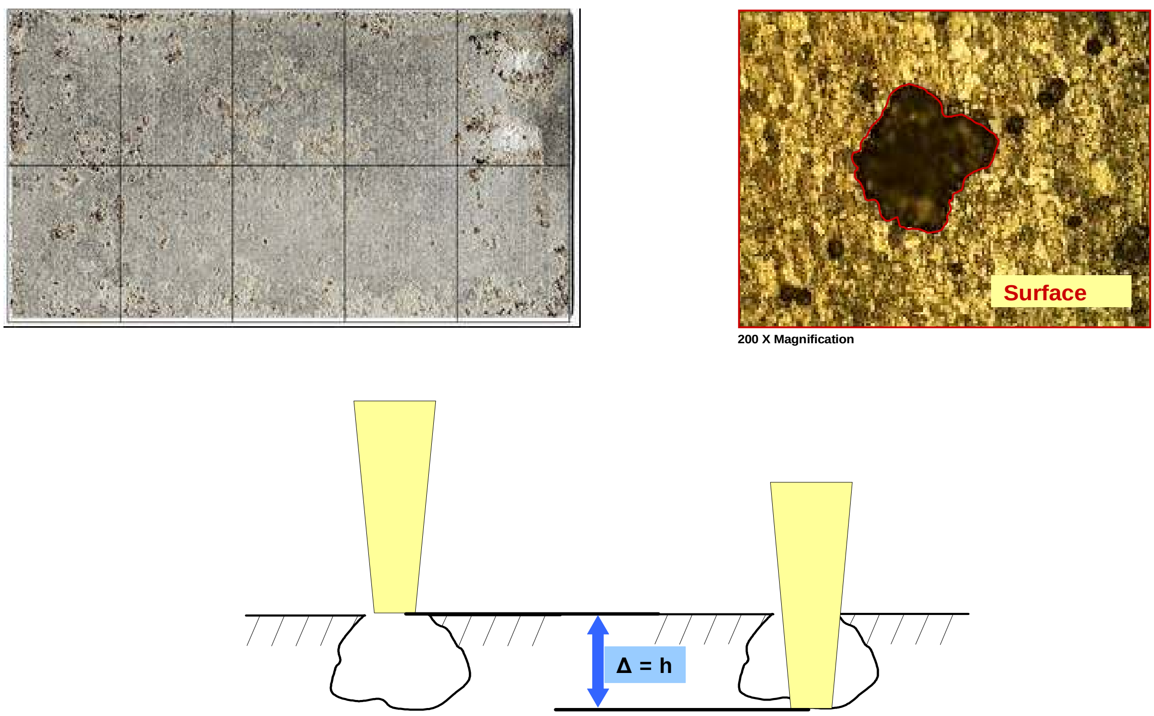

Figure 6: Schematic approach of measuring pit depth with optical microscope

Conclusion:

The following conclusions can be drawn from this study that each of the explained methods has its own characteristics and advantages depending on the type and purpose of required information. The comparison has revealed the fact that none of the discussed method fulfils all the desired requirements. Every technique has its own merits and demerits but X-ray radiography appears amongst one of the suitable especially in terms of personal efforts and costs. The summary of all three methods in a form of comparison are explained in a table 1.

Size of specimen (700X700 mm)	**Light Microscopy**	**3D Microscopy**	**X-ray Radiography**
Costs	Low	High	Medium
Time required	Medium (1hr, dependent on corroded surface)	Medium (1hr, dependent on corroded surface)	Less (30 mins, independent on corroded surface)
Possible Analysis	Pit depth measurement 2D photographs	Percentage affected area 2D & 3D photographs	Percentage affected area 2D photographs
Precision & Accuracy	Pit depths: Medium (Till 20 µm exactness)	Pit depths: High (Till 1µm exactness)	Pit depths: High (Till 5 µm exactness)
Reproducibility	Bad (Examiner dependent)	Very Good	Good

Table 1: Comparison of light microscopy, 3D microscopy & radiography

Low cost and time are obvious advantages of this technique however it has some limitations also. Sometimes, precise estimation of a pit is also difficult because of alignment of the pit relative to radiation beam. The most significant limitation of this technique is accessibility of both sides of the sample to X-ray radiations. The results of this work have shown that radiography is a very promising way for quantification of pitting corrosion both in terms of maximum pit depth and percentage of infected area due to corrosion.

The results obtained by this method are fully satisfactory and on the basis of them susceptibility to pitting corrosion for each material can be ranked. The quantification of corrosion allows comparing and ranking different grades which lead to improve the selection of the right material.

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