



Fractal dimension used for evaluation of oxidation behaviour of Fe-Al-Cr-Zr-C alloys



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ABSTRACT

The article describes the possibility of using a methodology for the quantification of oxidised surfaces. The methodology is based on determining the parameters on a dividing line between alloy and air which has been generated from images of a cross-section sample. The parameters are obtained from the methodology using fractal geometry, surface roughness characterisation and statistic tools. The methodology is presented on oxidised iron aluminides (Fe-29.7Al-3.8Cr-0.3Zr-0.2C and Fe-26.4Al-2.8Cr-0.2Zr-0.6C) at high temperatures (900–1200 °C). The results show that methodology can be used for easy quantification of alloy oxidation attacks and for easy comparison of alloy oxidation resistance.

1. Introduction

Iron aluminides have been studied as potential alloys for structural applications at high temperatures. The reason for choosing them was that binary Fe-Al alloys have very good high-temperature oxidation and corrosion resistance [1,2]. Binary Fe-Al alloys are frequently alloyed to improve mechanical properties, especially at high temperatures. However, alloying could have a detrimental effect on corrosion resistance, for example zirconium content above 0.1 at.% causes the formation of oxide protrusions due to preferential attack of Zr-rich precipitates [3–5]. In the case of alloying by tantalum, the spallation of oxide film was observed, possibly because Laves phase Fe₂Ta formed underneath the oxide scales at temperatures above 900 °C [6]. Therefore, the high-temperature oxidation and corrosion resistance of alloys should be tested and analysed. Measurements of mass changes, analysis of phases in and under the oxide layer, changes of chemical compositions depending on distance from the surface are usually applied to understand the corrosion mechanism and behaviour. Besides these “standard” methods, fractal dimension is increasingly being applied to describe the corrosion behaviour.

1.1. Fractal dimension

Fractal dimension is part of the wider theory of fractal geometry. Fractal geometry is closely connected to chaos theory. Furthermore, the obtained structures were produced by real dynamic systems, and the

obtained data was influenced by these systems [7–9]. The data can also be tested for chaotic properties and also simulated. A chaotic system can be identified by standard tools like Lyapunov coefficient, Hurst coefficient and also by fractal dimension.

The fractal dimension was defined by Benoit Mandelbrot [10], though scientists found some geometric problems with specific objects (e.g. the measurement of coastlines using different lengths of rulers by Richardson). A potentially powerful property of fractal dimension is the ability to describe complexity using a single number that defines and quantifies structures [7,11]. The number is mostly a non-integer value and the fractal dimension is higher than the topological dimension. For example, the Koch curve (one of the most famous mathematical deterministic fractals) has the topological dimension $D_T = 1$, but the fractal dimension $D_F = 1.2619$. In contrast, a smooth curve, as a line, has the topological dimension $D_T = 1$ and the fractal dimension $D_F = 1$. The fractal dimension can be computed or estimated for a set of points, curves, surfaces, topological 3D objects, etc. If the fractal dimension is higher than the topological dimension, we name the objects fractals. The fractal dimension of mathematical deterministic fractals can be calculated, while the fractal dimension of natural objects, series and sets can only be estimated using an appropriate method.

Even though applications of fractal dimension in industrial practice are quite rare and experimental [12], it is possible to find a promising test and applications [13–19]. However, the fractal dimension does not substitute other tools like statistics; the dimension provides additional information about the data. Previous research [15,20–24] shows that

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fractal dimension in conjunction with statistics can be used as a useful and powerful tool for an explicit, objective and automatic description of production process data (laboratory, off-line and potentially on-line).

A significant field of application for fractal dimension has been describing the morphology, roughness and texture of material surfaces. Fractal dimension was often used for evaluation or quantification of metallic corrosion previously by many authors. Most of the authors used the estimated fractal dimension for analyses of whole images of a corroded surface using the box counting method [25–31]. Analysis of a dividing line (boundary curve) can also be found [32], but authors also use the box counting method. The fractal dimension of a surface can also be estimated by the triangulation method [18] or using multifractal methods [33]. However, the box counting method has several problems, especially associated with repeatable image acquisition and processing. This method works with binary (black & white) images of fractals, however images of corroded structures are mostly displayed as grey-level or even colourized images. To ensure repeatability, transformation to a binary image must be made in the same way (using the same level of grey for thresholding). The transformation also requires the same image format (the same grey-level, colour mask, compression ...). These requirements can be fulfilled with strict adherence, which guarantees repeatability. Complications occur in determining the illumination and hardware conditions. The key issue is surface lighting to capture images: light position (especially angle), irradiance, wavelength of light, and the character of lighting (diffuse or directional). Furthermore, a camera has many parameters to be set, such as shutter speed (exposure time), aperture settings (f-number or f-stop), gain (ISO sensitivity) etc. Problems may occur in the comparison of surfaces with different reflection properties. All these problems make using the box counting method difficult to use for the comparison of several types of surfaces. Ensuring the same conditions independently on a device is impossible.

Pursuant to the given problems, we carry out research on the application of other methods for fractal dimension estimation. The compass method for a dividing line (boundary curve) seems to be suitable for estimating the fractal dimension [15–17,20–22]. Information on roughness or corrosion attack must be reduced to a 1D representation, into the dividing line between alloy and air, or glass and other environments. Cross-section samples must be prepared for image obtaining that make the analysis more time consuming. However, conditions for obtaining the dividing line are not as strict as for the Box-Counting method. It is important to have a sufficient visible contrast between the alloy and the surroundings, a well-focused image, and to use the same scale for comparing a given set of samples. Furthermore, cross-section sample images are used commonly while investigating alloy corrosion behaviour.

In general, cross-sections of corroded samples have different levels of surfaces, Fig. 1. The surfaces are represented by a dividing line between the alloy and oxide layer. The dividing line can be smooth, corrugated or complex on a different level and the line can be compared or described by words or using suitable photos. However, objective quantification can give additional information or can substitute some parameter of the corrosion obtained from other methods.

The selected parameters of fractal dimension and statistical tools were used in [16,17] for the quantification of corrosion resistance against molten glass. Iron aluminides with different chemical compositions and austenitic steel were tested in lead molten glass and in molten soda-lime glass. The results revealed the detrimental effect of zirconium on the corrosion resistance of iron aluminides and the comparable or better corrosion resistance of Fe-25Al-5Cr compared to austenitic steel. Nevertheless, these parameters were evaluated only for comparison between the state of samples surface before and after the corrosion test. The paper [15] described the methodology application for corrosion attack of Fe-14Al-6Cr in molten soda-lime glass depending on time. The basics of the methodology were described earlier in [20,21].

The purpose of this paper is to present a developed objective methodology for quantification of the surface roughness of oxidised alloys for easy comparison of oxidation resistance of alloys, which have different chemical compositions. The methodology, which takes advantage of fractal geometry, was developed and applied on alloys based on Fe-Al-Cr-C-Zr. The high-temperature oxidation behaviour of these alloys were investigated using “standard” methods such as light optical microscopy (LOM), X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive X-ray spectrometry (EDS) previously in [3].

2. Experiment

2.1. Samples and oxidation test

Two Fe–Al–Cr–Zr–C alloys were melted in a vacuum furnace and ingots were produced by investment casting [3]. The composition of the alloys (in at.%) is given in Table 1. The ingots with dimensions $30 \times 40 \times 350 \text{ mm}^3$ were rolled at $1200 \text{ }^\circ\text{C}$ to sheets with a final thickness of 13 mm. For oxidation tests rolled samples with dimensions $10 \times 10 \times 1 \text{ mm}^3$ were cut by electrical discharge machining (EDM) and then the surfaces of the samples were finely ground to 1200 grit (initial state).

The oxidation tests were carried out using a Setaram SETSYS 16/18 thermobalance with continuous recording of the mass gains and a heating rate of $9 \text{ }^\circ\text{C}/\text{min}$. Samples of both alloys were oxidised in synthetic air (20.5% O_2 , 79.5 N_2) at a flow rate of $1.54 \times 10^{-6} \text{ m}^3/\text{s}$ at temperatures ranging from 900 to $1200 \text{ }^\circ\text{C}$.

2.2. Methodology

After the oxidation test, samples were carefully cut and encapsulated in the conductive resin. Then the samples were ground and polished with diamond suspensions, the last step was polishing with $0.05 \text{ }\mu\text{m}$ of colloidal silica. The cross section of the surface was investigated using light optical microscopy, see Figs. 1 and 2 and Fig. 2a. The images were evaluated and described by image analysis using a software tool developed in Matlab (release R2009a and R2013b). The objective evaluation of a dividing line must solve three key steps:

- repeatable and reliable determination of the dividing line (pre-processing, Fig. 2b),
- evaluation of the dividing line by appropriate parameters (processing, Fig. 2c–f) and
- selection of useful information from the results (post-processing, Fig. 2g).

2.3. Repeatable and reliable determination of dividing line

The same conditions of image acquisition for the determination of the dividing line were provided. The images used were sharp and had a sufficient contrast between the alloy and oxides (scales) or products of corrosion. The images were in the same magnification and the same grey-level, also captured under the same light conditions, using the same exposure setting (shutter speeds, lens aperture, gain) and using the same equipment for image capturing. The borderline (dividing line) between the alloy and oxide layer must be continual from left to right in captured images, without a break, because the aim was to simplify and to objectify the image processing and also to strictly use only the surface for image analysis.

Matlab was used for determining the dividing lines low-level contour plot computation *contour* in software. A toolbox for finding and verifying dividing lines was developed. The toolbox is able to automatically determine the dividing line.

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