Description to Fe-C Alloy Film Fiber Corrosion Sensors by Fractal Corrosion Images

Lihua Cai
School of Mechanical and Electrical Engineering, China University of Mining and Technology, Xuzhou, Jiangsu, China
Email: cailihua1984@163.com

Wei Li
School of Mechanical and Electrical Engineering, China University of Mining and Technology, Xuzhou, Jiangsu, China
Email: liweicumt@163.com

Abstract—**In this paper, the fractal dimensions are proposed for characterizing the optical fiber corrosion sensors, which is applied to measure the steel corrosion based on Fe-C alloy film. Because corrosion was a complex random phenomenon and the corrosion surface of Fe-C alloy film of the optical fiber corrosion sensor possessed fractal characteristics, the image fractal dimensions is as a quantitative index of the sensor corrosion degree. The experimental results showed that the complexity level of the sensor surface morphology increased and the optical output power increased along with the increase of fractal dimensions. And the sensing law of the thicker Fe-C alloy film is better than that of the thinner film. Therefore, it is feasible that the fractal dimension is used to characterize the Fe-C alloy film optical fiber corrosion sensors and it is also optimistic to the future prospect of the Fe-C alloy film optical fiber corrosion sensors.**

Index Terms—**Fe-C alloy film; fiber corrosion sensors; light output power; fractal dimensions**

I. Introduction

For the past few years, with the development of the optic fiber sensing technology, the optic fiber sensing technology is also researched continuously and deeply to monitor metal corrosion condition. And as one kind of which, the optic fiber corrosion sensor (OFCS) based on the Fe-C alloy film plated in the fiber core is used to monitor the corrosion condition of reinforcing steel bars and underground pipeline[1-3]. Many researchers have carried out large numbers of research to this kind of sensor and obtained some primary sensing performance[4]. However, the corrosion test of the plated film is not thorough and the test design is simplex.

Like most of natural phenomenon, metal corrosion in nature is complex and erratic, so the corrosion morphology and images could not be definitely identical even in the case of same material and environment. Meanwhile, corrosion images are irregular and unrepeatable. Therefore, fractal is one of the efficient ways to describe the corrosion behavior of the Fe-C alloy film. Now, many workers attempted to use fractal in corrosion researches. J.M. Costa presented a preliminary account on fractal properties of steel corrosion pitting[5]. And the fractal analysis of electrochemical noise helps to evaluate the inhibitor protection performance under the mild steel corrosion conditions tested[6]. Shaniavski and Artamonov calculated fractal dimensions for fatigue fracture surfaces[7]. Weng and Li use fractal dimension to modify the random fluctuation of average corrosion rate of car-bon steel in soils and obtain more accurate expression about soil corrosion[8]. Therefore the fractal dimension could be an important parameter to characterize the corrosion extent[9]. The author presented that the corrosion surface of Fe-C alloy film OFCS satisfied statistical fractal characteristics through plenty of testing data. In this article, the fractal dimension of corrosion morphology images of Fe-C alloy film OFCS is as a quantitative index of the sensor corrosion degree, which puts forward a new method for characterizing the OFCS.

II. The Principles of Fe-C Alloy Film OFCS.

The conductive optical signal through the fiber core generates an influence when Fe-C alloy film over the fiber core is corroded. Hence, we can obtain the corrosion information of steel materials by measuring real-time changes of output optical signal. The sensing principle is as follows:

According to the optical waveguide theory, when the angle of incidence θ meets the full-refraction condition with the incident light reflected by the interface of two mediums, the light beam occurs full-refraction phenomenon at the interface. The principle can be expressed as:

This work is supported by the National Nature Science Foundation of Jiangsu, China (No.BK2006037) and the Technology Support Project of Jiangsu, China (No. SBE201000378). Corresponding author is Lihua Cai (cailihua1984@163.com)

© 2012 ACADEMY PUBLISHER
doi:10.4304/jcp.7.10.2599-2604

$$\theta > \theta_c = \sin^{-1}\frac{n_2}{n_1} \quad (1)$$

Where θ_c is the critical angle when the light waves are reflected by the interface of two mediums; n_1 and n_2 is the refractive index of two mediums, respectively. The light wave guide theory is applied to the OFCS. Fe-C alloy film which is similar with the composition of steel materials takes the place of the cladding of a certain part of the optical fiber, where is as a sensing field and became the metal waveguide. Because the absorption of Fe-C alloy film makes the optical energy loss larger inside the optical fiber in this field, which makes the emergent light energy lower. If the Fe-C alloy film is corroded, the sensing field is surroundded by corrosion medium and becomes a new waveguide environment, and the light beam satisfies the full-refraction condition in this sensing field, which makes the optical energy increase gradually. Therefore we can obtain the corrosion information of steel materials by measuring real-time changes of output optical signal. The optical fiber corrosion sensor is shown in Fig.1.

III. Experimental Set-Up

A. Fe-C Alloy Film Preparation

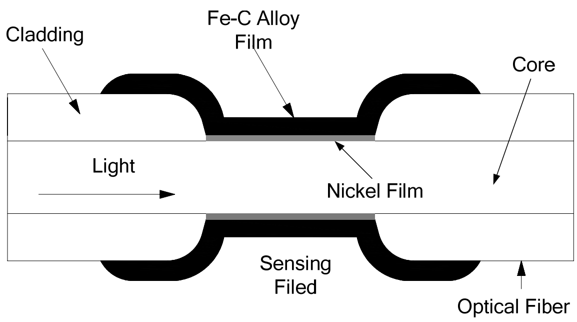

Fig.1 Optical fiber corrosion sensor with the corrosion-sensitive metal film

To plate Fe-C alloy film on the optical fiber surfaces, chemical plating nickel should be proceeded as a metal conducting layer. Before chemical plating nickel, surface treatment, such as removing fiber cladding, sensitization and activation, is prepared to obtain uniform and continuous nickel film.

(1) Surface treatment of optical fiber

Firstly, 15% hydrofluoric acid is selected to corrode the fiber cladding because of its strong volatile, and through repeated experiments, it is evidently linear relation between fiber diameter and corrosion time(the corrosion rate is 0.133 μm/min). In order to completely corrode the fiber cladding off, the corrosion time is about 480 min and the fiber diameter is 61 μm (the original fiber core is 62.5 μm), the process of which carries out fiber coarsening as well.

Then, the worked fiber is immersed into sensitizing solution to gain a layer of reduction substance on the fiber surface and offer catalytic activity metal ion for next activation. The recipe of sensitizing solution includes stannous chloride($SnCl_2 \cdot 2H_2O$ 10g/L) and hydrochloric acid(HCl 50ml/L).

Afterwards, optical fiber activation is that the Sn^{2+} on the fiber surface reacts with Pd^{2+} in the activation sulition to form precious metal crystal nucleus with catalytic activity for chemical nickel-plating. The recipe of activation solution includes palladium chloride($PbCl_2$ 0.6g/L) and hydrochloric acid(HCl 5ml/L).

(2) Chemical nickel-plating

In this experiment, sodium hypophosphite is as reducing agent and by repeated experiments, the optimum craft condition of chemical nickel-plating is shown in Table 1.

By observing the fiber cross section with scanning electron microscope(SEM), the nickel film is widely distributed out of the fiber around and the average thickness is approximately 0.2 μm .

Table I.
The Craft Condition Of Chemical Nickel-Plating

$NiCl_2 \cdot 6H_2O$	32g/L
C_2H_5COOH	25ml/L
$NaH_2PO_2 \cdot 6H_2O$	26g/L
H_3BO_3	25g/L
pH	3.5~3.8
temperature	77°C~79°C
time	20min

(3) Electroplating Fe-C alloy film

Electroplating experiment adopts constant current electroplating and 20 steel is used as the anode. In order to obtain uniform clad layer, the anode is coiled into cylindrical shape, where is put into metallized fiber core, and the axile wire of the fiber keeps coincidence with that of the anode. A good deal of experimental grope is used to confirm the imposed current density of the cathode and the time of electroplating. the optimum craft condition of electroplating Fe-C alloy film is shown in Table 2.The plating solution temperature is controlled by high-accuracy constant temperature magnetic stirrers. Moreover, a small quantity of citric acid and ascorbic acid are mixed into the $FeCl_2$ sulution, and its equation[10] is as follows:

$$Fe^{2+} + \text{organic acid} + 2e = Fe(C)$$

© 2012 ACADEMY PUBLISHER

TABLE II.
THE CRAFT CONDITION OF ELECTROPLATING FE-C ALLOY FILM

$FeCl_2$	40g/L
citric acid	1.2g/L
ascorbic acid	3g/L
pH	2.5~3.0
temperature	35°C~40°C

Through a quantity of electroplating experiments, current density plays a role in the quality of the coating. Therefore, we selects $i = 0.5A/dm^2$ first and electroplates $20min$, then, we turns the current up to $i = 1A/dm^2$ and goes on eletroplating and the electroplating time decides the thickness of Fe-C alloy film. When viewed by SEM and shown in Fig.2, we

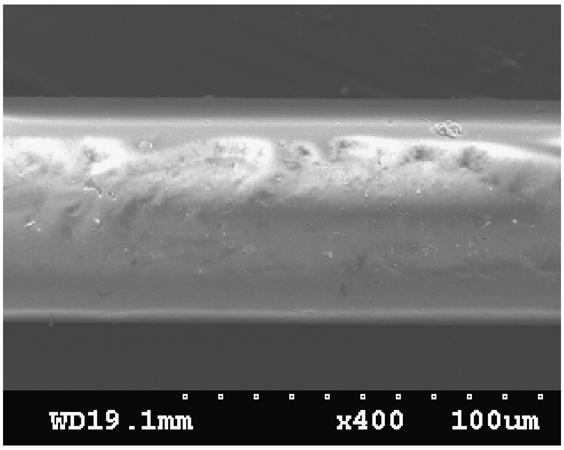

Fig.2 Surface morphology of fiber core electroplate with Fe-C alloy film

can see that the quality of coating is widely distributed and the surface of that is relatively flat, which is basically confirmed with the request.

B. Corrosion Sensing Experiments

After the optical fiber was plated with Fe-C alloy film, the sensing performance must be found out through corrosion test, which can be seen in Fig.3. The principle of the corrosion tests is very simple i.e., the optical fiber plated Fe-C alloy film is put into some kind of the corrosive medium and the change of the optical power transmitted in the fiber core is observed with the film layer gradually corroded. If we can find outthe accurate corresponding relation between the corrosion degree of the Fe-C alloy film and changes of output optical power, the corrosion degree can be judged with the change of output optical power.

In actual environment, different places have different corrosion condition with different corrosion rate. Therefore, because of the different corrosion rate, it is meaningless to record changes of output optical power with time as abscissas. And if we record changes of output optical power with percentage of corroded film as abscissas, although that would be not affected by corrosion rate, it is really tough to define the percentage of corroded film. Therefore, fractal dimension of metal-film corrosion morphology image is selected to express the corrosion degree. Corrosion is a complex random phenomenon, and at present, fractal dimension of the corrosion morphology image is used to express corrosion degree by many domestic and foreign researchers and the relationship is obtained among fractal dimension, corrosion morphology image and corrosion rate. The relationship is that the more complex the corrosion surface, the more irregular and the bigger the fractal dimension. And the faster the average corrosion rate, the deeper the corrosion surface hollow and the bigger the standard deviation, the bigger the fractal dimension. Therefore, the fractal dimension is an important parameter to characterize the corrosion degree. Author has analyzed that the corrosion surface of Fe-C alloy film OFCS satisfies with statistical fractal features by a large number of experimental data. In this paper, the fractal dimension of corrosion morphology image of

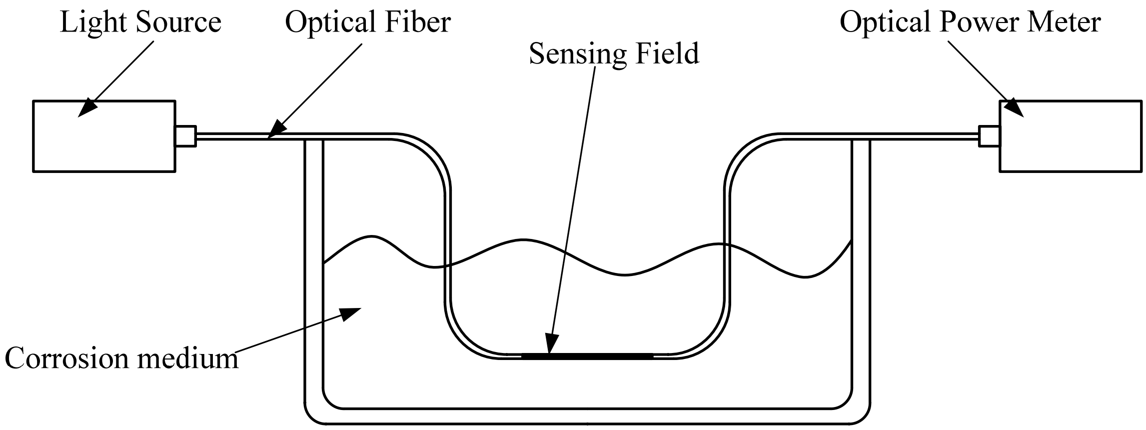

Fig.3 Principle of the corrosion test

© 2012 ACADEMY PUBLISHER

Fe-C alloy film OFCS is a quantitative measure, which puts forward a new method to characterize OFCS.

Given a long corrosion process in reality, some corrosive solution is generally used to accelerate corrosion process of the coated optical fiber in the lab. Compared with using different concentration corrosive solution, it is more significant that sensing law is found out with the same concentration corrosive solution and the different thickness metal films. Based on the above analysis, the corrosion experiment test is as follows:

(1) Firstly, nickel-film is plated in the core as the middle conductive layer. Then, Fe-C alloy film is plated outside the nickel layer with different thickness, which is put in the same concentration corrosive solution respectively.

(2) Fe-C alloy film of each thickness needs to proceed corrosion test more than once to obtain as many test data as possible and avoids some accidental data, consequently, the sensing law is found out.

Ⅳ. EXPERIMENTAL ANALYSIS AND RESULTS

A. Fe-C Alloy Film Structure Microcosmic Analysis

X-ray diffraction technique is used to assay the X-ray diffraction of Fe-C Alloy Film, comparing with that of 20 steel. The X-ray diffraction patterns are shown in Fig.4 and Fig.5.

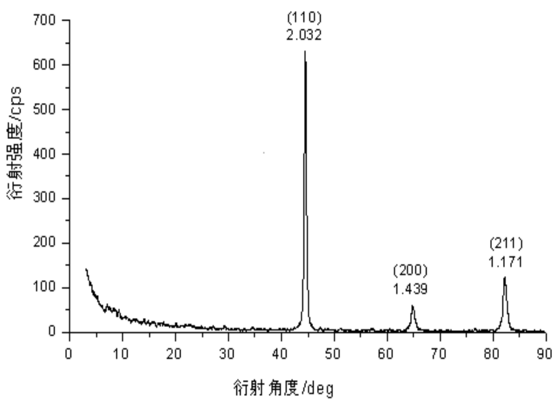

Fig.4 X-ray diffraction pattern of 20 steel

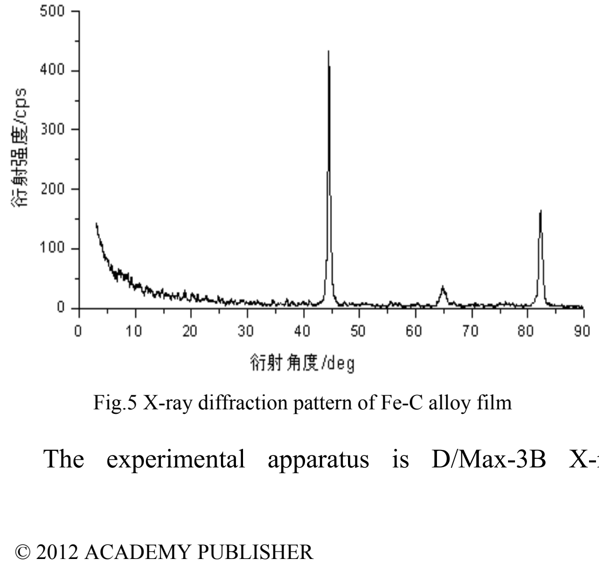

Fig.5 X-ray diffraction pattern of Fe-C alloy film

The experimental apparatus is D/Max-3B X-ray diffractometer from Japanese Rigaku company. And the experimental parameters are as follows: copper target, Kα radiation, graphite crystal bending monochromator, X-ray tube voltage is 35 KV, X-ray tube current is 30 mA, continuous scanning, scanning speed is $3°/\min$, and the sampling interval is $0.02°$.

From the X-ray diffraction patterns of 20 steel, we can perceive that the interplanar spacing is 2.032, 1.439 and 1.171 respectively, and the interplanar indices is 110, 200 and 211 respectively, which indicates that the crystal structure is α-Fe. And from Fig.5, it can be perceived that electroplating Fe-C Alloy Film forms amorphous microstructure and the diffraction peak position is consistent with that of 20 steel. However, the diffraction strength of Fe-C Alloy Film is weaker at the diffraction peak of 110 and 200 than that of 20 steel, and is stronger at the diffraction peak of 211 than that of 20 steel. That suggested that primary crystal structure type of Fe-C Alloy Film is basically consistent with that of 20 steel and is just different from the crystal growth direction, which is relative to some technology conditions of electroplating.

B. Corrosion Sensing Analysis

Fe-C alloy films with different thickness are electroplated. If the film thickness is very thin, the corrosion time is too short to satisfy corrosion condition. Howerer, if the film thickness is very thick, the fiber core is too thin to bear the weight of Fe-C alloy films. Hence, to obtain the preliminary sensing law of different thickness, optical fibers Plated Fe-C alloy film, with different thickness (3.0 μm, 7.6 μm and 12.8 μm, respectively), is put into 10% nitric acid solution to conduct corrosion test, which is connected between light source and optical power meter. In the process of the corrosion, variation of the light output power and corrosion image is observed. When the light output power stops to change and stabilize for a period of time, we can terminate the experiment. The corrosion image and corresponding fractal dimension of Fe-C alloy film are shown in Fig.6. We can see that the corrosion images are more complex and the Fe-C alloy film corrodes more severely, the fractal dimension is bigger. Afterwards, the light output power was recorded with the variation of the fractal dimension.

Fig.7 shows that the relation is described between the three Fe-C alloy film with different thickness and the light output power. The results indicate that the curve of the 3.0 μm Fe-C alloy film is not better than that of the 7.6 μm and 12.8 μm Fe-C alloy film. And the 3.0 μm Fe-C alloy film is corroded plot by plot when the surface morphology is observed (the other two Fe-C alloy films are corroded gradually). The reason of that phenomenon should include two aspects: one is that the 3.0 μm Fe-C alloy film is too thin, the other one is that the bond between the alloy film and the fiber core is too weak. From the curve of the 7.6 μm and 12.8 μm, we can perceive the corrosion process is basically smooth at the

© 2012 ACADEMY PUBLISHER

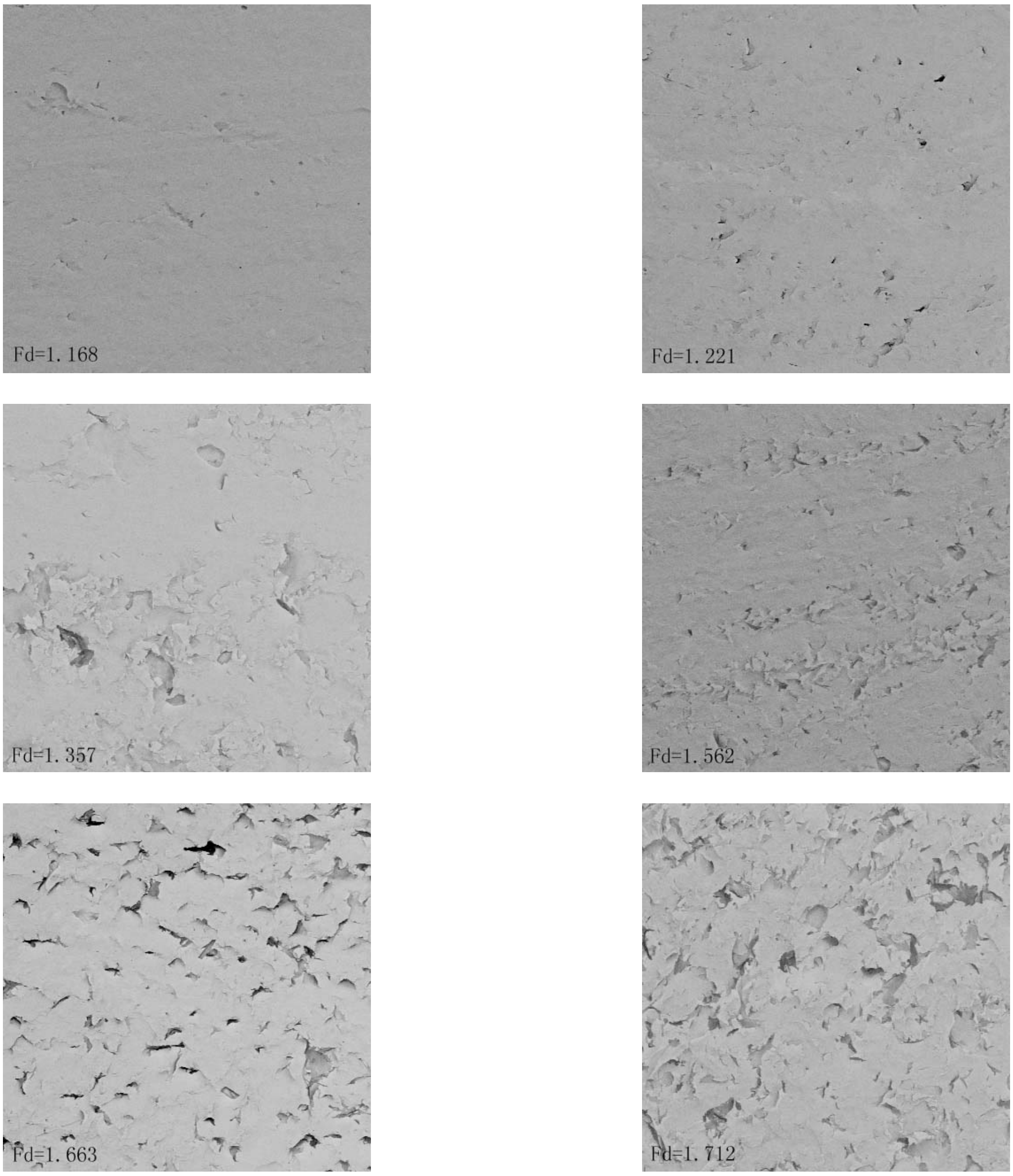

Fig.6 Fractal dimension of different corrosion images

beginning of corrosion, after that, the light output powerstarts growing. At last, the light output power increases rapidly because the Fe-C alloy film begins to drop plot by plot.

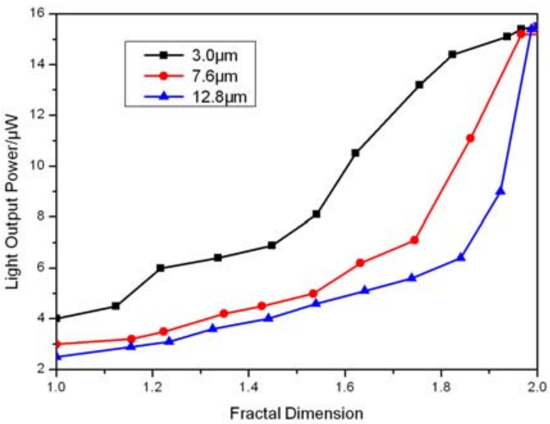

Fig.7 Variation of the light output power with fractal dimension of Fe-C alloy film corroded(10%HNO3)

Ⅴ. CONCLUSIONS

In this paper, we describe a novel method to characterize the Fe-C alloy film OFCS based on fractal corrosion images. Through reduplicative corrosion tests, the corrosion law of Fe-C alloy film OFCS is obtained basically. When the film thickness is larger, the corrosion process is smooth at the beginning and the light output power is increasing sharply at the end of corrosion, the phenomenon of which is because Fe-C alloy film begins to drop plot by plot at the end of corrosion.

In addition, we can find that the repeatability of experimental result is well in acid solution, and in the same conditions of the corrosion solution, the law between the light output power and the fractal dimension with the same thickness Fe-C alloy film is basically similar. Therefore, it is feasible that the fractal dimension is used to characterize the Fe-C alloy film OFCS and it is

© 2012 ACADEMY PUBLISHER

also optimistic to the future prospect of the Fe-C alloy film OFCS.

ACKNOWLEDGMENT

This work is supported by the National Nature Science Foundation of Jiangsu, China (No.BK2006037) and the Technology Support Project of Jiangsu, China (No. SBE201000378).

REFERENCES

[1] M. W. Woodruff, J. S. Sirkis: SPIE, 2191, 1994, pp. 511-515.
[2] P. Rutherford, R. Ikegami, J. Shrader: SPIE, 3042, 1997, pp. 248-259.
[3] P. L. Fuhr, D. R. Huston, A. J. McPadden, R. F. Cauley: SPIE, 2719, 1996, pp. 229-237.
[4] Dong.Saying,Peng.Gangding,Luo.Yanan.Preparation techniques of metal clad fibres for corrosion monitoring of steel materials.Smart Materials and Structure, 16 , 2007,pp. 733-738.
[5] J.M. Costa, F. Sagues, M.Vilarrasa.Fractal patterns of corrosion pitting. Corrosion science, 32, 1991,pp.665-668.
[6] E. Sarmiento, J.G. González-Rodriguez , J. Uruchurtu. A study of the corrosion inhibition of carbon steel in a bromide solution using fractal analysis. Surface & Coatings Technology,203,2008,pp.46-51.
[7] Shaniavski, A.A., Artamonov, M.A.. Fractal dimensions for fatigue fracture surfaces performed on micro-and meso-scale levels. Internet J. Fract. 128, 2004,pp.17–24.
[8] Weng, Yongji, Li, Xiangyi. Study on characterization for soil corrosion rate fluctuation of carbon steel. Chemistry, China, 7, 2005, pp.547–550.
[9] S. J. Xu, Y. G. Weng: Pattern Recognition Letters, vol. 27,2006.
[10] X. M. Li, W. M. Wei, Z. Q. Huang,et al. A new type of fiber optic corrosion sensor. Chinese Journal of Sensors and Actuators, 32,1999,pp.176-180.

Lihua Cai was born in 1984. She is a Ph.D. candidate of China University of Mining and Technology at Xuzhou city in Jiangsu Province China. From Sept. 2003 to Jul. 2007, she was Mechanical Engineering and Automation major at China University of Mining and Technology. After finished her bachelor, she was admitted to a ph.D. student without examination by China University of Mining and Technology in the field of Artificial Intelligence under the supervision of Prof. Wei Li. Her main research interests include automated reasoning and intelligent planning.

Wei Li was born in 1964. He got M.S degree in 1989 and Ph.D degree in 2004 at China University of Mining and Technology at Xuzhou city in Jiangsu Province China. He is a professor and doctoral supervisor at China University of Mining and Technology and he is the dean of school of Mechanical and Electrical Engineering at China University of Mining and Technology. His main research interests include distributed artificial intelligence, knowledge engineering and intelligent instrument,etc.

© 2012 ACADEMY PUBLISHER