CHEMICAL ENGINEERING TRANSACTIONS

VOL. 65, 2018

A publication of AIDIC

The Italian Association of Chemical Engineering
Online at www.aidic.it/cet

Guest Editors: Eliseo Ranzi, Mario Costa

ISBN 978-88-95608-62-4; **ISSN** 2283-9216

DOI: 10.3303/CET1865099

Application of Image Analysis Technique in Pore Structure and Properties of Carbon Anodes

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This study purports to investigate the effect of image analysis technique on pore structure and performance of carbon anode. Methods: Through the introduction of image analysis methods, a fractal model is constructed to calculate and analyze the pores of carbon anodes, to understand the characteristics of their pore diameter distributions, and to explore the association rules between different aperture intervals and carbon anode resistivity based on gray theory. Results: The accuracy of research data on pore structure and performance of carbon anode is improved with the application of image analysis technique, which has certain application and popularization value.

1. Introduction

With the rapid development of China's economy, the electrolytic aluminum industry in China has also developed rapidly. The development of the aluminum industry has also raised higher requirements for the carbon anode used for aluminium. First, the demand is greatly increased, and then the quality requirements are becoming higher and higher. The gap accounts for 20% to 30% of the carbon anode, so improving the anode pore structure and performance is of great significance to reducing the carbon consumption and carbon dioxide emission, improving the anode quality and reducing the electric energy consumption. There are many factors affecting the pore structure distribution of carbon anode. From raw materials, kneading, molding to final roasting, the development and change of pores run through the preparation process of the whole anode, which has a very important influence on the quality and performance of carbon anode. Carbon anode for aluminum will undergo electrochemical process, physical process and chemical process in the process of electrolysis. The carbon anode is composed of aggregate carbon, carbon powder and binder asphalt. The failure of the carbon anode is the main cause of the failure of the aluminum electrolyzer. The common anodic faults in the electrolyzer include the uneven distribution of the current distribution, the partial overheating of the anode, the anodic drop off block, the anode "long package", the anodic block falling off, the anode appearing crack fault, the anode inclination, and the entry of a great number of carbon slag into the electrolyte and so on. There is a great relationship with the quality of the anode. When the anode failure occurs, the production process and technical conditions of the electrolyzer will be destroyed, the efficiency of the electrolyzer is reduced, the level of electricity consumption and the consumption of raw materials are increased, the difficulty of the electrolysis operation is aggravated and the grade of aluminum is reduced. The role of carbon anode in aluminum electrolysis is mainly affected by the temperature of electrolyzer, the influence of current distribution, the influence of conductivity and the consumption of anode.

Image analysis is based on image processing, using graphics and mathematical principles to process, identify, analyze and interpret image content. The image analysis method has been widely used in biomedicine, industrial testing, transportation, agriculture, military, space and other fields because of the advantages of visual, clear, efficient and quantitative (Elbeltagy et al., 2018; Han and Li, 2016; Luo and Jia, 2016; Fang, et al., 2016; Tang and Chen, 2016; Zi, 2017). In recent years, the combination of image analysis and quantitative metallography and stereology has made image analysis technology widely applied in many fields. The advantage of image analysis method is that it can express pore and particle shape and structure quickly and

Please cite this article as: Zhenghao Xi , Xiu Kan , Le Cao , Huaping Liu , 2018, Application of image analysis technique in pore structure and properties of carbon anodes, Chemical Engineering Transactions, 65, 589-594 DOI: 10.3303/CET1865099

quantitatively. At the same time, the geometric characteristics of individual pores and particles can be accurately obtained. To further discover quantitative information, we will further study and optimize the design principles of materials and provide a design plan for the production and preparation of materials.
There have been many studies on pore structure and properties of carbon anode at home and abroad (Grandfield, 2014), the CT technique was used to detect the inner pores and cracks in the anode. CT technology can detect the defects inside the anode without destroying the anode sample, and measure the porosity in the carbon anode, and the density distribution in the material can be obtained by computer tomography. The two value images of pore structure of carbon anode are obtained by dividing the threshold value, which provides a basis for further study of pore topology. Chevarin, et al., (2016) studied the variation of pore structure in the CO_2 reaction process of carbon anode specimens with different diameter and size. It was found that the opening pore with a diameter of more than 40 um has a great influence on the CO_2 reaction. This part of the pore is the key factor to determine the excess consumption of the anode. Reducing the gas transmission channel in the anode can reduce anode consumption. It is also found that the reactivity of the residue, coke and bituminous coke at the same calcination temperature has the same reactivity. The result of the study is a scientific doubt about the reason that the selective reaction of the classical theory is the cause of the carbon anode dropping. Chevarin et al., (2016) studied the variation of pore structure in the CO_2 reaction process of carbon anode specimens with different diameter and size. It was found that the opening pore with a diameter of more than 40 um has a great influence on the CO_2 reaction. This part of the pore is the key factor to determine the excess consumption of the anode. Reducing the gas transmission channel in the anode can reduce anode consumption. It is also found that the reactivity of the residue, coke and bituminous coke at the same calcination temperature has the same reactivity. The result of the study has a scientific doubt about the reason that the selective reaction of the classical theory is the cause of the carbon anode dropping. Azari et al., (2013) used CT technology to study the distribution of pinching temperature and kneading time on the porosity of carbon anode and the mixing effect of asphalt and petroleum coke. The results showed that the optimum kneading condition was 178 C, and the time was 10min. Azar and others studied on the extrusion process of paste found that the relationship between the density of the anode and the speed of the extrusion was smaller. The contribution of the extrusion speed to the densification of the anode is less than 2%, while the contribution of asphalt and petroleum coke is greater. Azari et al., (2014) also studied the extrusion process of the particle shape and porosity of petroleum coke and found that the shape of petroleum coke particles contributed more to the density of the green, and the shape of the particles could make up the shortcomings of the smaller petroleum coke density. Wang et al., (2013) found that the molding pressure and molding temperature have a great influence on the performance of the carbon anode when the aluminum electrolytic carbon anode is prepared by hot die pressure. With the increase of molding pressure and temperature, the pores in the anode carbon block first decrease and then increase. On the relationship between pore structure and density of petroleum coke particles, Sarkar et al., (2014) studied the influence of the temperature, the surface morphology of the petroleum coke surface, the surface morphology of petroleum coke and the influence of the inner pores of petroleum coke on the oil coke and the wettability of asphalt. The contact angle between green and petroleum coke decreases, and the existence of small pores impedes the penetration of asphalt into the pores of petroleum coke. To sum up, the above research is mainly about the role of carbon anode in aluminum electrolysis, the characterization method of pore structure of carbon anode, the relationship between pore structure and properties of carbon anode. At present, the traditional method is adopted to regulate the pore structure of anode in the production of carbon anode. The pore structure of the anode and control is studied by the image analysis method. The literature is not common. Therefore, based on the above research status, this paper studies the relationship between the pore structure parameters of carbon anode based on image analysis method and the anode raw density, resistivity, carbon dioxide reactivity and anode dropping mechanism, and explores the new pore structure performance and technical approach of reduction of carbon consumption without increasing the raw material cost.

2. Research Methods

This study systematically investigates the effects of pore structure on the raw materials, preparation and properties of carbon anodes with image analysis, which provides theoretical basis for reducing the production cost of carbon anode and improving the performance of anode. The image acquisition system is composed of metallographic microscope and digital camera. The obtained images are processed and analyzed by ImageJ software. A cylindrical carbon anode ($\Phi 25\times 50$ mm) is cut open and impregnated with the resin to increase the strength of the sample and prevent the pores from changing during the polishing process. The specimen is polished to 1,200 mesh subsequently with a number of sandpapers from coarse to finer, and then washed and polished with a metallographic polisher until its surface becomes like a mirror. Then the surface of the specimen is cleaned with deionized water to remove residual contaminants. The specific steps are as shown in Figure 1. Figure 1a shows the original color image. After the chroma and saturation is removed, the brightness is preserved, and then the original image is converted to a grayscale image (Figure 1b). Figure 1c is a brightness histogram of the grayscale image, where the pores and the carbon matrix correspond to one peak, respectively, and the brightness minimum is set as the threshold value to distinguish the pores from the carbon matrix. Figure 1d is the binarized image after threshold segmentation, where the black part is the carbon matrix and the white is the pores.

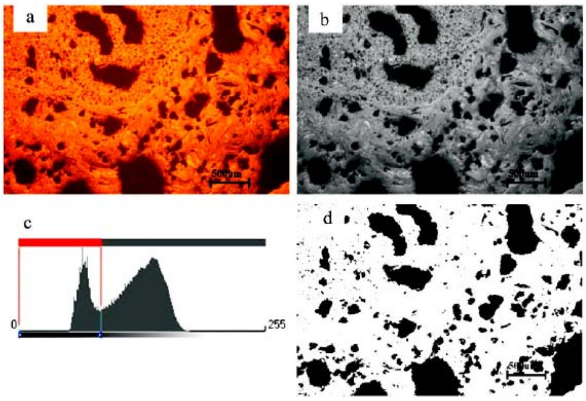

Figure 1: Analysis and processing of pore microscopic image of carbon anode sample

3. Results and Discussions

3.1 Principle of image analysis

The image analysis is to use graphics and mathematical principles to process, identify, analyze, and interpret image content based on image processing. The whole image system is divided into H (means number) main modules according to functions, input module, processing module W and calculation and output module. These H modules are relatively independent in function, but they are closely related and indispensable. A data flow communication mechanism is adopted between each module to perform data connection by sharing the common data area of the memory. The input module is mainly responsible for the collection of digital images, and converts the topography of the analyzed object into digital images. The processing module is used to complete the reading and transformation of the digital image files by converting the processing of the spatial domain into the processing of the transform domain, to obtain clearer features of the object and reduce the error of the extraction parameters of the object. According to different application fields and extraction target bodies, the image processing module usually includes: image preprocessing, image segmentation, morphological processing, and impurity removal. The output module performs parameter setting, calculation, statistics and derivation of the segmented binary image through a corresponding measurement method.

3.2 Characterization of pore structure of carbon anode based on image analysis

In recent years, image analysis is combined with the principles of quantitative metallography and stereology, which has enabled it to be widely used in many fields. Its advantages lie in that it can quickly and quantitatively characterize the pore structure and the shape of the particles, accurately obtain the geometric characteristics of individual pores and particles, and provide design schemes for further discovery of quantitative information, deepened research and optimization of design principles of materials, and material production. Based on the results of previous researches, this study optimizes the sampling and image processing of the image analysis method, extracts the petroleum coke particle characteristic parameters and the internal pore structure parameters of the anode according to the stereological principle, establishes a relationship model between petroleum coke particle characteristic parameters and petroleum coke loading performance to predict the loading performance of petroleum coke particles, and explores the relationship between the anode pore structure parameters and the anode resistivity and CO_2 reactivity, which provides theoretical guidance for the production of high quality anodes.

3.3 Preparation and calcination of carbon anode samples

The petroleum coke particles with different sizes are used as raw materials, in which the large granule petroleum coke accounts for 30%, and the mass ratio of added asphalt to petroleum coke is 18: 100. The mixture is kneaded under different temperature and time conditions (as shown in Table 1). The kneaded paste is molded at 20 MPa into an anode green sample (Φ25 mm × 50 mm), and then calcined in a reducing atmosphere at a maximum firing temperature of 1100 ℃.

Table 1: Mixing time and temperature of carbon anode paste

Sample category	A	B	C	D	E
Kneading time, min	3	5	7	7	7
Kneading temperature, °C	160	160	160	150	170

3.4 Measurement of anode sample resistivity

According to the standard (YB/T120-1997) principle, the resistivity measurement of the carbon anode sample is performed at room temperature. In order to improve the measurement accuracy, the voltage values of the same anode sample are measured at different currents of 0.2, 0.4, 0.6, 0.8 and 1.0 A respectively. With the current plotted on the abscissa, the voltage plotted on the ordinate, and the slope taken as the resistance value of the anode sample, the resistivity value of the anode sample is calculated according to the following equation: ρ=USIL (1), where, ρ--resistivity of materials, μmΩ; S--cross-sectional area of materials, mm2; L--length of materials, m; U--voltage applied across the conductor, mV; I--current that is through materials, A.

3.5 Image extraction

To analyze the internal pores of the anode, firstly, image shall be collected and cross-section information obtained. Computers can only recognize digital images, so image acquisition plays an important role in image analysis. In this study, a metallographic microscope is used to collect the image of the carbon anode sample, and the camera is connected to the microscope through a digital adapter. The collected images are transmitted to a computer for processing and analysis to obtain the desired information.

3.6 Discussions

3.6.1 Porosity and measured resistivity of carbon anode

With the extension of the kneading time, both the porosity and the resistivity of the anode are decreased. When the kneading temperature is increased, the two tend to decrease first and then increase. Under the experimental conditions, the kneading time for obtaining the lowest resistivity is 7minutes and the kneading temperature is 160 °C.

3.6.2 Porosity and pore diameter distribution of carbon anode

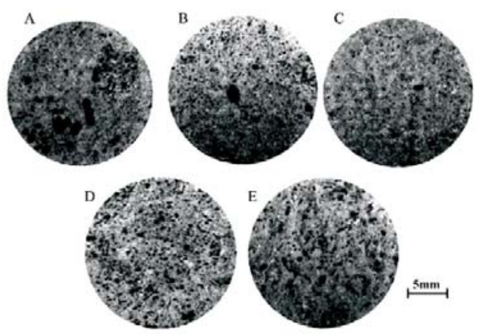

Figure 2: Section pore morphology of different carbon anode samples

Figure 2 shows the grayscale images of the cross section images of different anode specimens, where the darker parts are the pores and the lighter parts are the carbon matrix. The distribution of the pore diameter inside the anode can be obtained by image analysis. Figure 3 shows the porosity-pore diameter distribution curve of the sample, from where it can be seen that each anode sample curve has a peak of porosity at a pore diameter of 100 μm, which may be due to the fact that it is difficult for the asphalt to penetrate inside the pore diameter of petroleum coke during kneading. With the increase of pore diameter, the porosity of carbon anode decreases. Samples A, B, D, and E have multiple peaks at 500, 200, 250, and 300 μm, respectively, while sample C has fewer peaks. Referring to Table 1, the peak porosity of Samples A and B may be due to the relatively short kneading time and insufficient tightness of asphalt and petroleum coke.

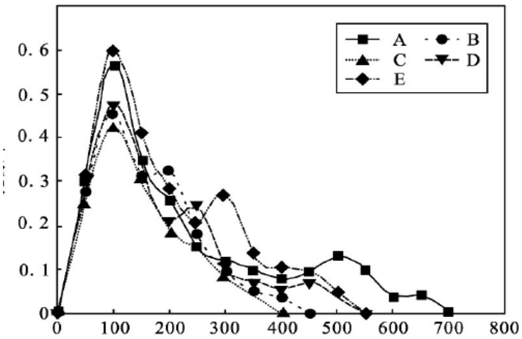

Figure 3: Porosity and pore diameter distribution of different carbon anode samples

3.6.3 Fractal dimension of pore distribution of carbon anode

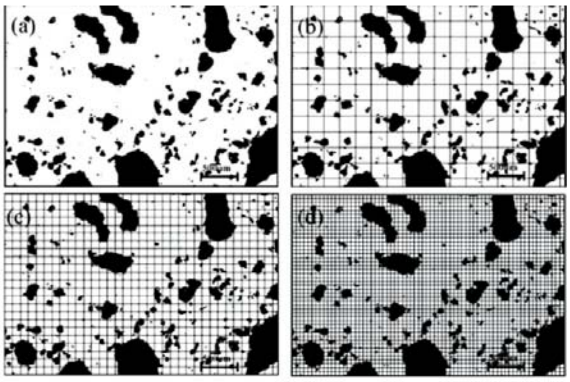

Figure 4: Pore mesh coverage of anode sample

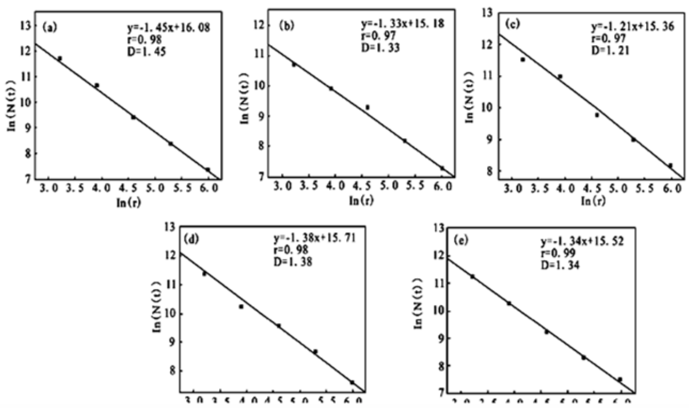

Figure 5: Fractal dimension of pore distribution of different carbon anode samples

The fractal dimension of the pore distribution is the fractal dimension of the original area distribution, which is determined by the box-counting dimension method. In the binarized image, the square uniform grid is added according to different size (side length RI), and part of the grid is blank, while another part of the grid is covered with pores, as shown in Figure 4. By counting the number of blank grids, N (ri) is obtained and the counts are made using ImageJ software. The original image (Figure 4a) is divided into different units using black grid lines (Figure 4b: the grid with pores and the blank grid). After calculating the area of the white part in each grid, it is known that the area in the blank grid is constant and maximum, and the area of the white part in the grid with pores is bound to be smaller than that of the blank grid. Then gradually reduce the grid size (Figure 4c and Figure 4d) to get different N (ri) values. Figure 5 shows the fractal dimension of the pore distribution of different anode samples, from where it can be seen that correlation coefficient of the straight line fitting results is from 0.97 to 0.99, indicating that the internal pore distribution of the carbon anode conforms to the fractal rule. The fractal dimensions of pore distribution in samples A, B, C, D and E are 1.45, 1.33, 1.21, 1.35 and 1.34 respectively, wherein it can be seen that sample A has the largest fractal dimension, while sample C has the smallest fractal dimension. The larger the fractal dimension, the more uneven the pore - pore diameter distribution will be in the anode.

4. Conclusions

With image analysis technique, this study defines the fractal dimension, orientation factor, porosity, connectivity ratio, shape factor and coordination number of contour as the characterization of pore structure of carbon anode, combines with the characteristic parameters of petroleum coke particle morphology to put forward a method for characterizing the overall pore structure parameters of carbon anode by analyzing and processing image data, uses the analytical data to characterize the pore structure of carbon electrode during its service, and adjusts and modifies the parameters of pore structure appearing in the process. The image analysis technique not only enables us to grasp the characteristics of pore parameters inside the carbon anode and the characteristics of the pore diameter distribution, but also enables us to calculate the fractal dimension so as to clearly and accurately show the distributions of different pores and pore diameters. In addition, through the application of image analysis, the correlation between internal porosity and resistivity of carbon anode can also be determined during the study process of pore structure and performance of carbon anode, and it is found that the resistivity of carbon anode increases when the porosity increases, but the value does not increase monotonically. Therefore, the application of image analysis technology to the investigation of the pore structure of carbon anode and its performance has significant application effects.

Acknowledgements

This work is jointly supported by the National Natural Science Foundation of China (Grants Nos: 61701295 and 61703270) and Natural Science Foundation of Shanghai (17ZR1411900).

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