Application of Scanning Electron Microscopy in Metal Material Detection

Ting Yu¹, Fengping Zhong¹,², Fang Zhang¹, Chenkai Ying¹ and Guihong Geng³*

¹Zhejiang Academy of Special Equipment Science, Hangzhou 310000, China
²Key Laboratory of Special Equipment Safety Testing Technology of Zhejiang Province
³North Minzu University, Yinchuan 750021, China
Email: 342640267@qq.com (Ting Yu), zjupec@foxmail.com (Fengping Zhong), zhangfang@zjtj.org (Fang Zhang), yingchenkai@hotmail.com (Chenkai Ying), gengguihong@163.com (Guihong Geng)

Abstract. Scanning electron microscopy (SEM) plays a very important role in the process of microstructure, fracture analysis, qualitative and quantitative analysis of micro-area composition, microstructure analysis and so on. With the rapid development of material science and technology, various industries also put forward higher and higher requirements on the technical level of testing. Based on the development background of Scanning Electron Microscopy (SEM), this paper introduces the application of scanning electron microscope in the observation of metal microstructure. Mainly about the characteristics of scanning electron microscope to continuous zooming, equipped with advantages of backscatter diffractometer, large depth of field, is advantageous to the samples from the macroscopic characteristics by combining with the analysis of microstructure, and quantitative analysis to test samples, also can research the fracture morphology of the sample, the recrystallization organization, grain orientation and texture characteristics, etc.

1. Introduction
For a long time, researchers analyze the properties of materials by observed microstructure, especially for metallic materials, which is closely related to their properties. The scanning electron microscope was used to observe the macrostructure of the metal sample, and the overall morphology of the material was preliminarily understood. Through the phase distribution, grain size, the degree of dispersion of the additive, whether there are loose shrinkage cavities on the surface, etc., the degree of segregation, hardness, plasticity and toughness of the material can be analyzed. By observing the fracture morphology of material and the on-site failure analysis, crack morphology of fracture zone and growth direction can be observed. At the same time, pollution degree of break source position and whether is corrosion can also be detected. Finally, the fracture type can be inferred and the cause of fracture can be obtained. Electron backscatter diffraction (EBSD) technique was used to analyze the microtexture of metal materials. After processing the original data, the information of grain size, phase distribution and proportion, grain boundary type and recrystallization proportion of the sample could be calculated.

In this paper, development and application prospect of scanning electron microscope are introduced, and application of scanning electron microscope in metal alloy detection is summarized. The microstructure, texture, fracture morphology and energy dispersive spectrometer of metal alloy were
carried out by scanning electron microscope, which provides a reference for the application of scanning electron microscope in the field of metal alloy.

2. The Development and Application Prospect of Scanning Electron Microscopy

Scanning electron microscopy produces images by focusing an electron beam on the surface of a sample. The atoms and electrons in the sample interact with each other to produce a variety of signals of surface morphology and composition information, and image can be output by combining scanning path of electron beam with intensity of the detection signal [1-3]. In the development history of scanning electron microscope, the first person who proposed and used scanning electron microscope was McMullen of Cambridge University [2,4]. In 1953, McMullen developed a new scanning electron microscope, which was improved by Smith, and the first high-quality scanning electron microscope was successfully produced in Canada in 1958. Since then, the scanning electron microscope has officially become a very practical scientific research tool [5-7]. Scanning electron microscopy was developed in China after the 1970s. Although it started late, it developed rapidly. By the 21st century, China's SEM has taken a place in the world.

Scanning electron microscopy is widely used in various scientific researches because of its advantages such as high resolution, large depth of field, no restriction on the shape and thickness of samples, large field of view, dynamic observation, no damage and pollution degree of samples. Wang Bo et al. [8] observed the ultrastructure in intestinal tract of Drosophila using scanning electron microscopy and studied influence of ulcerative colitis on it. It was found that the membranous structure of internal organelle was destroyed and intestinal microvilli were damaged in the group, the number of stem cells was increased, the number of microorganisms in the intestinal tract was increased, and number of unsecreted intestinal cells was increased. It is concluded that ulcerative colitis causes changes in intestinal ultrastructure, so it has adverse effects on enteritis. Wang Kunyang et al. [9] studied pore structure characteristics of shale by using scanning electron microscopy and observed distribution of organic and inorganic pores in shale by combining scanning electron microscopy and atomic force microscopy (AFM), found that the non-mean properties of pores lead to changes in pores and throat, thus affecting reservoir performance and physical property characteristics. Gao Xueping et al. [10] summarized development and application prospect of scanning electron microscopy technology, and indicated that SEM plays an important role in the research of material microscopic field, and further development of its characterization technology and microscopy technology will greatly promote the progress of scientific research.

3. Application of Scanning Electron Microscope in Metal Alloys

Scanning electron microscopy can continuously adjust the magnification, which is conducive to the combination analysis of macroscopic characteristics and structure of the samples. At the same time, the energy dispersive spectrometer was used to carry out qualitative and quantitative analysis of the detected samples by spot, line, and plane scanning. The electron backscatter diffractometer can also be used to study the recrystallization structure, grain orientation and texture characteristics of the sample [11-14].

3.1. Metal Microstructure Analysis

Due to the limited resolution of optical microscope, the observation of metal microstructure has certain limitations, but when scanning electron microscope is used to analyze microstructure of samples, its advantages can be fully utilized [15]. Scanning electron microscopy can not only observe the overall morphology of the sample, but also observe the microscopic structure of a particular area. The following image is the scanning electronic image of the copper lead monotectic alloy, as shown in figure 1.
Figure 1(a) SEM picture of Cu-Pb monotectic alloy observed at 100 times. It can be seen from the figure that the gray part is matrix structure and bright-white structure is second phase, and there are a few segregation structures in the picture. The white circle range in figure 1(a) was selected for magnifying observation, as shown in figure 1(b). The homogeneity of this part is good, matrix is smooth and flat, and there is no loose shrinkage cavities, and microstructure of the second phase is uniformly.

Figure 2 is the SEM picture of AZ91D magnesium alloy with SiCp added. According to figure 2(a), it can be seen that the black-gray part is matrix structure, the white-gray vermicular structure is the second phase, and there is bulging block third phase structure on the surface of the matrix. Select the part with these three phases (white circle in figure 2(a)) to enlarge for further observation. This is shown in figure 2(b).

In summary, the scanning electron microscope images can preliminarily determine which phases exist in the alloy, and select appropriate part for next phase analysis with the energy dispersive spectrometer.

3.2. Analysis of Micro-area Composition of Metal Materials with Energy Dispersive Spectrometer
The energy dispersive spectrometer can detect phase of sample. It is necessary to analyze the microstructure and composition of second phase and inclusions by means of energy dispersive spectrometer. However, ordinary optical microscope neither accurately observe the submicroscopic morphology of inclusions, nor analyze their composition, so it is difficult to determine the type of
inclusions or the phase. Surface scanning was performed on the white circle areas in figure 1 and figure 2 to detect the type and distribution of phase, as shown in figure 3.

Figure 3. Scanning electron microscopy-energy dispersive X-ray analysis of metal alloy.

Figure 3 is the EDS scanning image of Cu-Pb monotectic alloy and Cu-SiCp/AZ91D magnesium matrix composites. According to the spectrum of Cu-Pb monotectic alloy, the purple structure in the electronic image is Pb, the green structure is Cu, corresponding to the gray matrix structure in the SEM image is Cu, and the bright-white structure is Pb. The point scanning spectrum further proves this view. According to the energy spectrum of Cu-SiCp/AZ91D magnesium matrix composites, the black-gray part is Mg matrix structure, the white-gray vermicallike structure is mainly composed of Al and Mg as second phase, which is covered with a small amount of Zn, and the bulging block third phase structure on matrix surface composed Mg and Si.

Scanning electron microscopy-energy dispersive X-ray mainly includes "point", "line" and "surface" three analysis methods. Through the point-scanning test, element composition can be determined. Selecting the place (Spectrum 1 and Spectrum 2 in figure 3) by the energy spectrometer, the mass percentage or atomic percentage of each element in this place can be obtained. The line-scanning test can be established by collecting a line. Each color corresponds to the elements contained in the sample, and the fluctuation degree of the spectral line represents element content. Through surface scanning, the distribution characteristics of the element in the whole part can be seen, specifically including distribution position and relative number of elements. Scanning electron microscopy combined with energy spectrometry can simultaneously realize the detection of the material microstructure and micro-composition, complete the qualitative and quantitative analysis of the chemical elements in the observed area, and determine the main chemical elements. Scanning electron microscopy combined with energy dispersive spectrometer can analyze the microstructure and chemical elements of metal materials, guide the technological production and solve the quality problems of products [13-14].
3.3. Fracture Analysis of Metal Materials

A very important controllable performance index of scanning electron microscopy is the depth of field, which can simultaneously focus on observation of all parts of uneven sample [2, 13]. The most important is the depth of field ability of the microscope when analyzing experimental fracture or on-site failure fracture. When using scanning electron microscopy to analyze the fracture, don’t necessary destroy the sample, directly put into specimen chamber for observation, and the fracture type of the metal material can be analyzed. The following figure shows the tensile fracture morphology of SiCp/AZ91D magnesium matrix composites, as shown in figure 4 [16].

![Figure 4. SEM morphology of fracture of matrix alloy and composite material [16].](image)

Geng Guihong et al. [15] prepared and studied magnesium matrix composites, analyzed fracture by scanning electron microscopy. The results showed that fracture morphology of AZ91D magnesium alloy matrix was relatively loosened and flakelike, still flakes in local parts, showing the characteristics of brittle fracture. Compared with AZ91D magnesium alloy, the fracture of SiCp and Cu-SiCp reinforced magnesium alloy is denser and similar in morphology. There are a large number of dimples (the dimples are smaller after the addition of Cu-SiCp), partial cleavage steps. The fracture modes are ductile fracture and partial quasi-cleavage fracture.

Compared with light microscope and transmission electron microscope, scanning electron microscope has an important characteristic of the depth of field, scanning electron image has three-dimensional sense. Scanning electron microscopy plays an important role in fracture analysis in the following three aspects. First, it can make macroscopic observation of fracture failure parts, comprehend crack morphology of the fracture source area, and analyze the propagation direction of the fracture source area. Secondly, Scanning electron microscopy (SEM) can also be used to observe contamination degree of fracture source area and whether there is corrosion or not, judged fracture type and radiation pattern in the extension area [8]. Thirdly, the crack growth zone and morphology of inclusion were observed in the original condition to comprehensively infer the type of fracture and analyze cause of fracture [14, 17].

3.4. EBSD Analysis

The Electron Back Scatter Diffraction (EBSD) technique is based on analysis of diffractoma bands formed by electron beams excited on surface of inclined sample in scanning electron microscopy to determine crystal structure, orientation and related information [18]. EBSD transformed traditional method of texture analysis (X-ray diffractometry) and created a whole new field of science called “microtexture”, which combines microstructure and crystallographic analysis. EBSD orientation information process is: first, directly corresponding to different directions is obtained by electron diffraction kikuchi diffraction pattern, and then, using the polar diagram and analysis of inverse pole figure software characterized samples grain orientation, and finally, by orientation distribution can get specific information, including grain size, texture, grain boundary, phase distribution and recrystallization fraction [18]. Cu-Pb monotectic alloy was photographed used EBSD technology, as shown in figure 5 [19].

Cu-Pb monotectic alloy was photographed used EBSD technology, as shown in figure 5 [19].
In order to measure and analyze grain size of Cu-20wt.%Pb alloy, Yu Ting et al. [19] carried out EBSD shooting on the sample, results showed that: Under conventional conditions, the average grain size of Cu-20wt.%Pb alloy is about 0.826mm, and the average grain size of Cu-20wt.%Pb alloy is about 0.247mm after composite field treatment, which decreases by 70.1%.

For alloy treated by deformation, heat treatment and applied field, EBSD technology is needed for more comprehensive analysis. Cao Yanan et al. [20] used EBSD to analyze speckled microstructure of Ag after current-carrying friction experiment when studying the speckled microstructure and wear resistance mechanism of Ag film by magnetron sputtering. The results show that there are (012) and preferred orientations (111) in the speckle microstructure, the average particle size is 582nm, and there are a lot of very fine grains. At the same time, there are a large number of twins, (111) orientation twins accounted for 93.5%, and a large number of {111} plane family stacking fault structures were found at twin boundary. Su Yuanming et al. [21] explored influence of cold rolling deformation and annealing regime on the grain size of 5083 aluminum alloy under ultra-fast heating, and observed by electron backscattering diffraction (EBSD) technology, and found that the average grain size gradually refined with increase of heating rate after cold rolling deformation. The recrystallized grain size is influenced by the coupling effect of grain boundary migration rate and nucleation rate.

EBSD technology is the different effects produced by the interaction between sample and electron beam, namely, the "diffraction pattern" formed by diffraction in crystal or lattice plane with regular arrangement, which also represents crystal structure information of material. EBSD technique is by express kikuchi diffraction patterns of micro information, according to information to analysis structure of crystal materials and processing, widely used in metal materials microstructure analysis, such as grain orientation difference analysis of dual phase steel, nickel base alloy in the identification of precipitate phase in the grain boundary type statistic, alloy steel, etc., on the other hand, with the development of scanning electron microscopy analysis technology in recent years, combination of EBSD and energy dispersive spectrometer on scanning electron microscopy can not only observe the microscopic morphology, but also analyze the crystal structure and composition of the unknown phase.

4. Conclusion
Scanning electron microscopy plays a key role in fracture morphology, micromorphology, qualitative and quantitative analysis, failure analysis and other aspects. Combined with specific research direction, the relationship between microstructure of various metal materials, process conditions and properties can be studied. The diversification and inteligentization of SEM analysis function will play an important role in the exploration and research of new metal materials and technology. With rapid development of material science and high technology, requirements of various industries to level of detection technology are increasing day by day. It can be predicted that scanning electron microscopy will play a further role with its advantages.
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