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Scanning Electron Microscopy Testing Technology in Geotechnical Engineering

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Abstract
Geotechnical media itself is very complex, such as geotechnical structure, porosity, density, stress history, load characteristics, pore water and time effects. It is usually necessary to simulate the mechanical properties of geotechnical and simulate various aspects of geotechnical engineering including nonlinear stress-strain relationship, transient consolidation, steady-state rheology, well point precipitation, soil liquefaction analysis, construction process, geotechnical Stress-deformation and stability, slope stress and stability, analysis of slope and chamber anchorage effects, bearing capacity and subsidence analysis of subgrade, foundation, deep foundation pit, pile, etc., between soil and reinforced concrete road main body interaction. Due to the limitation of resolution and magnification, the traditional optical microscope is inaccurate for the qualitative analysis of fine particles. There is a certain error in the quantitative analysis of rock and soil. The observation of nano-micron mineral morphology and structural features is at a loss. With the deepening of oil and gas exploration and geological prospecting, it is necessary to provide information on all minerals, pores and trace elements in rocks. Therefore, the use of scanning electron microscopy to establish a rock mineral identification method based on large instruments is the need of current geological work. In this paper, the application of the sample and the test method, the dynamic monitoring method of the microstructure change, and the quantitative analysis technology of the scanning electron microscope are used to study its application in geotechnical engineering.

Key words: Microstructure, Scanning Electron Microscope, Test Method, Quantitative Analysis

1. Introduction

Geotechnical engineering is a new discipline closely integrated with environmental science. It mainly uses geotechnical perspectives, techniques and methods to manage and protect the environment [1-3]. Human production activities and engineering activities have caused many environmental hazards, such as the collapse of goaf caused by mining, groundwater overflow caused by regional land subsidence, industrial waste, municipal solid waste and other wastes. In particular, toxic and hazardous waste pollutes the environment, and the impact of construction disturbances on the surrounding environment [4]. The above environmental problems have been dealt with and prevented, and many new research topics have been proposed for geotechnical engineers. With the acceleration of urbanization and industrialization, environmental geotechnical research has received further attention [5]. From the perspective of maintaining a good ecological environment and maintaining sustainable development, people began to understand and pay attention to environmental geotechnical research [6-8]. However, geotechnical engineering itself is complicated. The pollution of air, water and land has become more complicated [9]. Under various environmental conditions, the principles and methods of soil mechanics currently used to analyze soil properties have been challenged [10]. Therefore, in recent years, geotechnical engineering testing technology has been greatly developed. Many new testing techniques, such as scanning electron microscopy, geotechnical centrifuge model testing and ground penetrating radar technology, have been applied to environmental geotechnical engineering. It provides an effective means for studying the microstructure of geotechnical media and the migration of pollutants in rock media [11].

Since the advent of the first commercial scanning electron microscope in 1965, due to the development of electro-optical theory and optoelectronic technology and the improvement of low-energy electron detection technology, it has created favorable conditions for the improvement and application of scanning electron microscope [12-14]. It has been used more and more widely in the fields of biology, botany, medicine, geology, metallurgy, materials science and electronics [15]. It has a magnification of 100,000-200,000 times and a very high resolution. The imaging principle of a scanning electron microscope is different from that of an optical microscope and a general transmission electron microscope. It does not require an imaging lens, and its image is formed point by point in time and space order and displayed on the external microscope tube [16-19]. An electron beam emitted from an electron gun having an energy of up to 30 KeV or higher is reduced by a condensing mirror and an objective lens, and is focused on the surface of the sample to form a very fine electron beam having a certain energy. The surface of the sample is directly bombarded, interacting with the surface of the sample to excite secondary electrons in the sample, and the secondary electrons are accelerated to the scintillator by the acceleration pole. The optical signal is converted into an optical signal and then passed through a light pipe to a photomultiplier tube to convert the optical signal into an electrical signal. It is amplified by the video amplifier and sent to the cathode of the picture tube to modulate the brightness and contrast of the picture tube. Secondary electron images with different brightness and darkness and the same shape as the sample are displayed on the screen [20]. The results of geotechnical microstructure analysis and its quantitative techniques have made great progress in environmental geotechnical engineering. The United States, Japan and other countries have carried out preliminary research on the mechanism of geotechnical microstructure and the migration of pollutants in geomaterials, and proposed corresponding treatment measures [20-22]. For example, scanning electron microscopy is used to study the treatment of arsenic-contaminated sand. Considering the combined use of various reagents and reagent combinations, experimental studies on contaminated soil began with scale experiments [23]. The effect of the compound formed after the treatment and the test was examined using a scanning electron microscope. Tests have proved that various metal salts, strontium, manganese, magnesium and the like can effectively reduce the permeability of arsenic. Firstly, FeSO47 H20 is used, and then treated with portland cement, the anti-seepage effect is better. The results of the scale penetration test were faithfully reproduced in the full-scale test on site, and the effect was even better than the scale test. Scanning electron microscopy was also used to study the treatment of mercury in soil by sulfidation. The purpose of this test is to evaluate experimental techniques for converting large amounts of mercury into mercury sulphide [24]. The presence of two basic mercury sulphide and other compounds was determined by observing the product after the test with a scanning electron microscope. In addition, scanning electron microscopy was also used to analyze the cracking and healing process of sand and asphalt mixtures in order to investigate the reasons why the fatigue life of asphalt-silicon pavement measured by the laboratory was inconsistent with the actual observations. China is still basically lacking in these areas and is a very valuable research area [25].

In this paper, the application and test methods of the sample, the dynamic monitoring method of microstructure change, and the quantitative analysis technology of scanning electron microscope are used to study its application in geotechnical engineering. The rock mineral identification method established by large instruments has higher resolution and significantly improves the accuracy of rock identification [26]. It greatly broadens the scope of rock ore identification (such as identifying nano/micro minerals, different mineral varieties, etc.), which can provide mineral content and mineral composition of rock minerals comprehensively and accurately, objective and accurate diagenetic information, clear mineral microscopic morphology and Structural features, in addition, the functions of the instruments overlap each other, and the test results are mutually verified, ensuring the reliability of the test results[27].

2. Proposed Method

2.1. SEM Test Technology

1. Sample preparation method. Due to the particularity of the geotechnical material, in order to ensure that the observed microstructure scan image can truly reflect the original shape of the sample, the collected sample must remain in its original state without interference. Take a portion of the soil sample as needed, taking care to select the part that is representative of the structural characteristics of the soil sample. For example, when preparing a sample after mechanical testing, samples of different parts of the crack surface or inside or outside the shear band should be selected. It is also necessary to prepare samples in multiple directions. Typically, samples are applied by hand and mechanical cutting tools are used whenever possible. On the other hand, since the smooth surface of the clay is often present, smooth surfaces should be avoided during sample preparation to ensure that the observed microstructure can represent the overall condition of the soil sample [28]. According to the requirements of different types of SEM, make samples of a certain size and select the plane section under the magnifying glass [29]. To remove interfering particles from the surface of the sample, a thin layer of glue can be applied to the surface of the sample and the glue removed after the glue has dried. The fresh portion of the sample can be obtained by blowing loose particles with a rubber ball. However, for samples containing small clay minerals, the clay mineral flakes are pulled up when the rubber is torn off, so care should be taken when handling. The sample used for the scanning electron microscope must be a solid material to ensure the vacuum of the SEM vacuum system, so the sample containing moisture must be dried beforehand. In order to eliminate the charging phenomenon, the oily sample must be carefully cleaned with a solution such as acetone to ensure the surface of the sample is clean. Regarding the drying method of the sample, there are mainly an air drying method, a drying method, a humidity drying method, a displacement drying method, and a freeze vacuum sublimation drying method [30]. Among them, the air drying method is the most common and simple in the country, the sample is placed in the atmosphere, slowly losing water and drying. It is suitable when the soil moisture content is low and the shrinkage limit is less than 10%. The displacement drying method replaces water in the soil sample with a liquid having a low surface tension such as methanol, acetone, ethanol or isopentane. Multiple replacements were carried out with different concentrations of liquid and replaced with acetylene ketones, then air dried, dried or vacuum dried. The successful development of the freeze vacuum

sublimation dryer greatly improved the level of microstructure preparation. The basic principle is that the sample is rapidly frozen at the refrigerant temperature to rapidly freeze the water into tiny ice crystals (to prevent the formation of ice cores). Then, under vacuum, the temperature of the sample was raised to between -50 ° C and -100 ° C, and the minute ice crystals were directly sublimated. The soil sample is dry. Commonly used refrigerants are: (1) liquid alkane (-196 ° C); (2) frozen isopentane with liquid nitrogen; (3) frozen propane with liquid nitrogen (-190 ° C); (4) frozen fluorine with liquid nitrogen Lyon, the freezing speed is 100 ° Cs; (5) the liquid nitrogen is formed by mechanical pumping to form nitrogen ice (-230 ° C), and the nitrogen ice is dissolved, which can greatly accelerate the freezing speed. In the 1980s, China successfully developed a vacuum vacuum sublimation dryer to fill the gap in microstructure preparation technology. The use of this technology to study the microstructure of frozen soil has achieved good results. It is especially suitable for the preparation of high water content soil samples and is a promising sample preparation technique. In addition to the widely used drying methods, the complex method is also a very effective method of sample preparation. The basic practice is to fill and infiltrate the sol into the sample to be observed and replicate the surface traces of the sample onto the film. Obviously, the prepared film and sample have opposite relief characteristics [31]. After standing for a while, after the solution was completely dried, the sample was dissolved in water, the floating dirt on the film was carefully washed, coated, and subjected to scanning electron microscope observation. In order to observe the microstructure of the frozen soil, a chloroform sol sample having a concentration of 4% to 5% was used to obtain good results. Preparing samples by complex methods is simple and convenient, and truly reflects the surface structure and other characteristics of the sample [32]. However, chloroform solutions are more toxic, less permeable, and do not readily reflect the fine structure of the sample. For samples with good electrical conductivity, scanning electron microscopy can be performed without treatment. For geomaterials with poor conductivity, samples tend to accumulate charge under the incident electron beam and affect image quality. Therefore, a thin gold (or silver, carbon) film layer is sprayed on the surface of the sample. It is more effective to use platinum, palladium or an alloy thereof as a coating [33].

2. Microstructure of soil and its analysis method. The microstructure of the soil includes three aspects: (1) morphological characteristics, that is, the size, shape, surface characteristics and quantitative proportional relationship of the structural unit; (2) geometric features, that is, the spatial arrangement of each unit; (3) energy Characteristics, that is, the connection characteristics between unit cells. Since Terzaghi first proposed the concept of soil microstructure, it has a comprehensive understanding of the microstructure and morphology of the soil. In addition to the general viscous soil, the research object also includes loess, expansive soil, frozen soil, frozen soil and artificially prepared structural clay. The Tan Luorong system summarizes the possible microscopic morphology of the soil. Shi Bin summarized the microstructure of several typical compacted expansive soils through a large number of scanning electron micrographs. In general, the study of soil microstructure should start from the following aspects: (1) The shape and size of the basic unit of the microstructure. Under the microscope, the basic unit body with obvious physical boundaries is called a microstructure and can be divided into a first unit and a second unit. The main unit refers to a microaggregate having strong original cohesive force and being difficult to separate. Secondary unit refers to aggregates assembled from primary microaggregates, such as flakes and granules. These secondary microstructure units can be classified into agglomerates (10 μm) and fine particles (5.0-10) depending on the size of their aggregation. 0 μm), microparticles (1.0 to 5.0 μm) and ultrafine particles ([1.0 μm), (2) Contact state between basic unit bodies, In the form of sheet or plate-like aggregates, it is usually in the form of face-to-face, side, side, etc., in the case of granular aggregates, in the form of direct contact or inlaid contact. Some are connected by cemented materials. (3) The form of connection between the basic unit bodies. The interaction between the basic unit bodies is very complex and the forms of action include the presence of charge, water film and bonding material. Scanning electron microscopy can only observe the shape, and the interaction force test must be combined with probe analysis, chemical analysis, etc. for comprehensive measurement [34].

3. Dynamic monitoring methods for microstructural changes. The change of microstructure of geomaterials under stress is an important aspect of theoretical research on geotechnical mechanics. Traditional SEM analysis can only give the shape of a microstructure under certain conditions or after the sample is destroyed. Zhang Meiying et al. designed a special loading device that was placed on a stretching table of a scanning electron microscope. It can perform direct shear and compression tests to visually observe the stress-strain process of the geotechnical medium under stress and the corresponding microstructural changes. At the same time, you can also record and take photos. The size of the compressed specimen is rectangular 15 mm × 7 mm × 5 mm and 16 mm × 11 mm × 4 mm. The size of the direct shear specimen is cylindrical, 12 mm in diameter and 3 mm in height. The equipment was tested on loess, weathered sandstone and air-dried clay and compared to conventional direct shear tests. It is believed that the stress-strain curves obtained by the two are basically the same, which can well reveal the internal mechanism of the microstructure changes in each stage of the shearing process. However, due to the small size of the sample, it also causes mechanical differences between it and conventional shear tests. Later, Zhang Meiying and others designed another small shear meter and assembled it under an optical stereo microscope. It can obtain the relationship between the stress and displacement of the

geotechnical medium and observe the whole process of the change of the microstructure of the geomaterial to the damage[35]. The instrument is mainly composed of two parts, namely an afterburning combustion system and a shearing device. The afterburning system is applied by two hydraulic systems, one for positive pressure and the other for tangential shearing. The shearing device comprises a shearing base, a shear box, two gauges or displacement sensors for measuring displacement. The inner wall of the shear box is 12 mm long, 12 mm wide and 5 mm high, and the size is still small. Preliminary testing of various soils using the instrument showed good performance and was different from previous equipment, and the samples were not limited by dry and wet conditions. It should be noted that due to the inherent shortcomings of scanning electron microscope testing techniques, it is difficult to monitor the dynamic changes in the internal structure of the sample. The newly rapidly developing computed tomography X-ray technology provides a better way to solve this problem [36].

2.2. SEM Quantitative Analysis Technology

Due to the complexity of the microstructure of geomaterials and the limitations of technical means, most of the researches on microstructures in the past have been qualitative analysis. With the development of computer image processing technology, the quantitative research on the microstructure of geomaterials has made a breakthrough [37]. Typical work such as Tovey, Osipov, Wu Yixiang, Shi Bin, Wu Yanqing and so on. The Videolab image analysis system developed by Moscow University is very effective in extracting the quantitative index of clay soil microstructure, especially in the quantitative research on the orientation of microstructure unit. The system is directly connected to the scanning electron microscope and the energy spectrometer, and the microstructure image obtained by the scanning electron microscope can be directly input and stored and processed without image taking. After the image is processed by the system, two kinds of quantitative information, that is, the microstructure information and the spatial distribution (directionality) of the microstructure unit body can be obtained. The microstructure information includes the total area of the particles or pores, the total perimeter, the average area, the average perimeter, the average shape factor, the average particle size or pores, and the interrelationship therebetween. The shape factor of a particle or pore is defined as:

$$F_i = C / S \quad (1)$$

Where C is the circumference of the area of the particles or pores, and S is the actual circumference of the particles or pores. The average shape factor is defined as:

$$F = \sum_{i=1}^{n} F_i / n \quad (2)$$

Where n is the number of statistical particles or pores. The orientation of the microstructure unit body includes the orientation distribution, the main orientation angle, and the anisotropy rate. Since the body orientation directional unit are mirror symmetrical in the range of 0 ~ 360 °, so that only the statistics unit body orientation 0 ~ 180 ° in it. Video lab image analysis system in the range of 0 ~ 180 ° to 10 ° azimuth is divided into 18 units, which can analyze the intensity directional microstructure units in each partition member, whereby the entire structure of the unit body image The directional distribution (generally elliptical distribution) and the main orientation angle. In order to reflect the overall orientation of the clay soil microstructure, the Videolab image analysis system introduces the concept of anisotropy rate, which can be expressed as follows:

$$I_n = \frac{R - r}{R} \times 100\% \quad (3)$$

In the formula, In an anisotropic ratio, R is the length of the major axis of the ellipse, r is ellipsoid minor axis length, In the visible range of between 0 and 100%. When In = 0, the particles or pores are randomly distributed and isotropic; and when In = 100%, they are fully anisotropic. In order to characterize the order of structural units, Shi Bin introduced an orderly indicator by means of the relevant ideas of modern information system theory. That is to say, the probability entropy is considered to reflect the order of the structural unit bodies better than the anisotropy rate. In view of the fact that computer image processing systems are relatively expensive, it is still difficult to popularize. The method basically obtains corresponding quantitative information by manual drawing (image preprocessing can also be performed by a computer). Using this technique, quantitative studies have been carried out on sedimentary soils and compacted expansive soils of the Pacific honeycomb structure. Compared with the results of the Videola b image processing system, the error of the two methods is about 5 % to 10 %. The results show that simple quantitative analysis methods are feasible and effective. Bai and Smart define a set of direction indicators for analyzing SEM images by digital image processing techniques. The orientation of clay particles during consolidation and undrained shearing was investigated.

3. Experiments

Instrument working conditions: The scanning electron microscope used in the test was a FEI Quanta 250 FEG field emission scanning electron microscope with a resolution of high vacuum of 3.0 nm (secondary electron imaging, SE) at 30 kV, and 4.0 nm (back scattered electron imaging, BSE) at 3 kV. 8.0nm (SE); 3.0nm (SE) at 30kV under low vacuum, 4.0nm (BSE) at 30kV, 10.0nm (SE) at 3kV; environmental scanning electron microscope (SEM): 3.0nm at 30kV (SE)). Spectrometer model: EDAX GENESIS Apex.

Working environment field: Based on the difference of the types of elements contained in minerals, most of the conductive properties are not good. The observation is carried out in a low-vacuum environment. For the minerals and slags with poor conductivity of the conductive electrodes, a small ion sputter is required to spray the gold. Treatment, samples with good electrical conductivity can be observed under high vacuum.

4. Discussion

Samples were taken from a batch of bulk ore imported from Australia, Singapore, South Africa, Malaysia, India and Brazil. Small pieces of iron ore and its powder were taken directly as SEM samples. In order to achieve better observation results, a 10 nm gold film was sprayed on the surface of each sample by SBC-12 ion sputter. The observation was carried out under the conditions of 20kV accelerating voltage, beam spot 4.0, working distance 10mm and magnification of 10000 times. The surface topography of the ore samples from different countries is shown in Fig. 1.

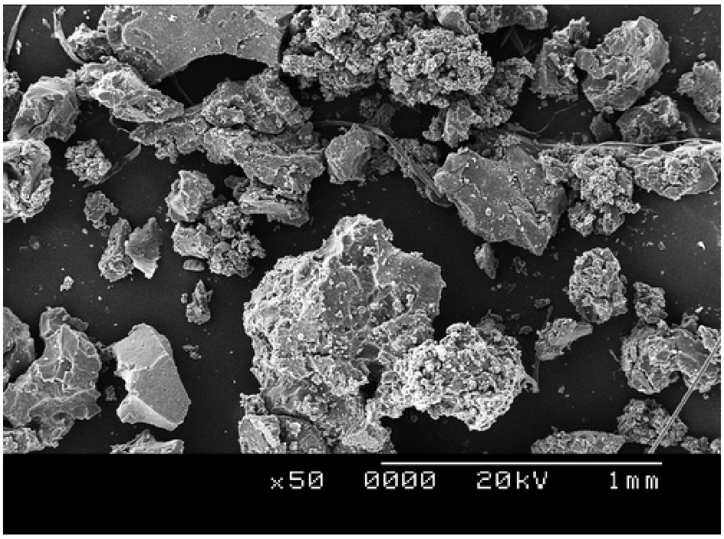

Figure 1. Sample surface microtopography

Differences in the growth environment will result in different forms of iron ore, even iron ore in different layers of the same type. Irregularities are the basic surface morphology of iron ore. As can be seen from the naked eye, each sample is in the form of a block. ,rough surface. Analysis of several ores found that the samples were different and generally irregular, and the samples were more pronounced in Australia, South Africa ore and Indian ore. However, the shape of iron ore entering from Singapore and Malaysia varies widely, and the growth state of the ore in Figure 1b can be seen. The color of the various ores is different under an electron microscope. According to the energy spectrum observation, the color is mainly iron oxide, and the shallow material is mainly a combination of elements such as Si, O, and Al. There are differences in the macro and micro characteristics of the ore, and the composition of the materials is also different. Scanning electron microscopy observation and analysis show that its formation is related to mineral crystallization and is closely related to its formation conditions. After the ore is sorted and integrated, the residue is called tailings. The content of its main value component is the tailings grade. The selection of an economically sound beneficiation scheme to evaluate the selectivity of the ore is an important parameter. Figure 2 is a batch of lead tailings from India. The element content table is shown in Figure 2. The lead tailings sample was observed under a scanning electron microscope. The results of the chemical methods after pulverization cannot be one-to-one. Combined with its energy spectrum analysis, the main elements contained in Figure 2a are O, Al, Si, Fe, Pb. The spectrum in the direct response element in the content of the table element helps to quickly detect substances consisting essentially of clay minerals such as kaolin Al, Si, O. Figure 2b is blocky with a black and bright surface. The energy spectrum shows that the Si element in this region is significantly smaller than the Si element in Figure 2a and contains a small amount of Mg and Ti. The Pb content in this area is less than that in Figure 2a. The chemical method after powder is more comprehensive. Figure 2 shows the energy spectrum of a batch of lead tailings. Bar = 100 μma, White area; b, Black area.

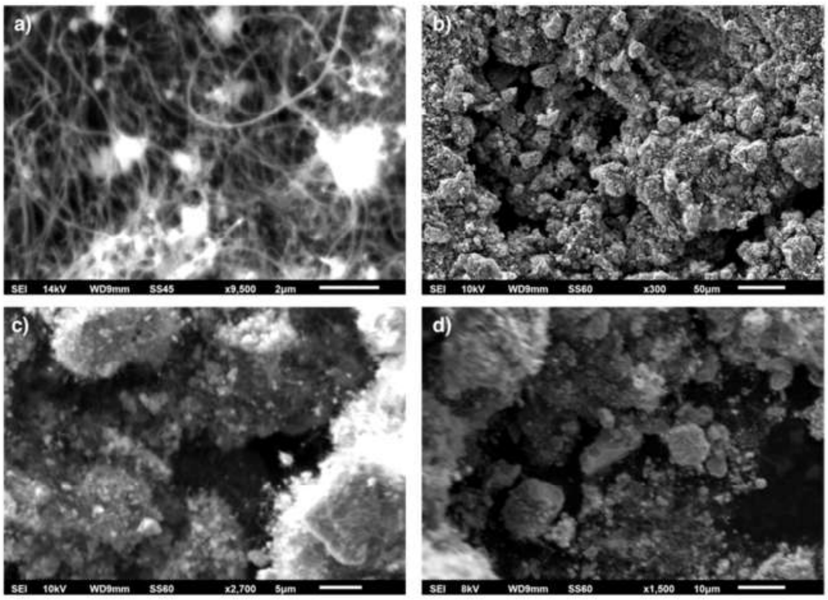

Figure 2. SEM image of a batch of geotechnical solid waste

In the analysis and detection, due to the limitation of the chemical detection on the sample preparation, the sample was analyzed under the electron microscope and the energy spectrometer, and the characteristic samples in the solid waste of the batch were grayish white and light in color, and the observation was The continuous discontinuous annular sample has a firm surface and is characterized by a cast-like surface. It is a random spherical and granular sample, which is blocky and shiny under electron microscope. The basic geometric parameters of the obtained particle shape were calculated by image processing software to calculate the elongation, and the calculation results of all the particles having a particle diameter larger than 25 μm were counted as shown in Fig. 3.

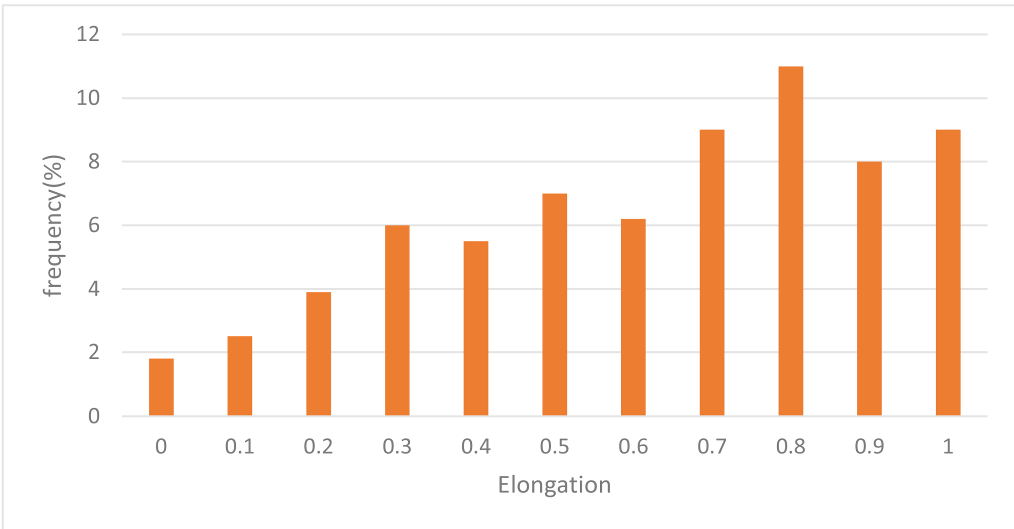

Figure 3. Statistical distribution of particle shape elongation

The energy spectrum test showed aluminum ingots, zinc ingots, zinc oxide powder and a small amount of graphite with different morphologies. Among them, the ratio of solid aluminum ingot and graphite in the sample is very small, which is consistent with the inspection. The application of electron microscopy combined with spectrometer can characterize these solid wastes in the shortest time. In the daily identification, timely report and false report are found, and the information base of imported waste and its corresponding goods is established. It provides a reliable means for exploring accurate, fast and intuitive methods for identifying the attributes of imported solid waste, and more effectively improving customs clearance services for geotechnical import mineral enterprises.

5. Conclusions

As a basis for geological work, rock engineering identification is a comprehensive analytical work. Only the correct analysis of the type, content, structure and structural characteristics of the minerals in the rock can be accurately analyzed. To provide accurate information about rock sedimentary environments, rock genesis, mineral composition and diagenesis. With the development of instruments, the combination of instruments has become a common method in analytical testing. The application of its application to the identification of rock minerals is the development direction of large-scale instruments and the main content of rock mineral identification research. A modern rock mineral identification method based on scanning electron microscopy, which provides comprehensive and accurate information on rock minerals, accurate mineral content, objective and accurate diagenetic information, and clear mineral morphology and structural features. In addition, the functions of the instruments overlap each other, and the test results are mutually verified, which ensures the reliability of the test results and provides comprehensive, accurate and reliable analysis and test results for oil and gas exploration and geological exploration. Compared with traditional optical microscopy methods, these modern large-scale instruments have not only greatly improved the accuracy of rock mineral identification, but also greatly expanded the scope of rock ore identification (such as nano/micro minerals, different mineral varieties, etc.)), capable of accurately, quickly and objectively identifying minerals in rocks.

The rock mineral identification method established by large instruments has higher resolution and significantly improves the accuracy of rock identification. It greatly broadens the scope of rock ore identification (such as identifying nano/micro minerals, different mineral varieties, etc.), which can provide mineral content and mineral composition of rock minerals comprehensively and accurately, objective and accurate diagenetic information, clear mineral microscopic morphology and Structural features, in addition, the functions of the instruments overlap each other, and the test results are mutually verified, ensuring the reliability of the test results. Compared with traditional optical microscopy methods, modern large-scale instrument rock mineral identification technology provides the basis for revealing symbiosis, reaction, evolution, rock genesis, sedimentary/diagenetic environment, providing accurate and comprehensive information on the quantitative, structural and diagenetic properties of geological work, laying a solid foundation for the successful completion of geological work.

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