Research Progress on Microstructure Characterization of Silicon Carbide Fibers

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Abstract. Ceramic matrix composites have excellent properties such as high temperature resistance and oxidation resistance, and have good application prospects in the hot end parts of engines. In ceramic matrix composites, the microstructure composition of silicon carbide fibers determines their macroscopic properties. Therefore, this article takes silicon carbide fiber as the research object, summarizes the existing SiC fibers structure, composition, and morphology characterization methods, compares the development and advantages and disadvantages of XRD, SEM, TEM and other testing equipment analysis, and proposes the problems of sample preparation, surface defects and internal structure changes in the microstructure characterization of silicon carbide fibers are discussed, which provides a reference for the preparation of high-performance SiC fibers and the improvement of their characterization work.

Keywords. Silicon carbide fiber, microstructure, characterization method.

1. Introduction
Silicon carbide fiber has excellent properties such as high strength, high modulus, high temperature resistance, corrosion resistance, oxidation resistance and low density. It is often used as reinforcement of ceramic matrix or metal matrix composites. It has been preliminarily used in cutting-edge fields such as space shuttle and high-performance engine. It also has broad application prospects in various engineering fields. It is called a new material for aerospace and high-tech applications in the 21st century [1-2]. Since the successful development and application of precursor conversion preparation technology in the 1970s, and the continuous progress in recent decades, the development of SiC fibers in the world has experienced the following processes: first, the first generation of amorphous fibers with high oxygen and high carbon, represented by Nicalon and tyranno series fibers, in addition to a large number of carbon, oxygen and β- SiC microcrystalline phase, the latter also contains metal element Ti, which improves the microwave absorbing properties of the fiber, but their modulus and temperature resistance are far from enough; Later, the second generation of low oxygen and high carbon microcrystalline fiber, namely Hi-Nicalon fiber, was analyzed by TEM and electron microscope. It was found that the fiber contained a large amount of free carbon and amorphous silicon...
carbide, the oxygen content was still high, and other properties were not significantly improved except heat resistance; Finally, the third generation of polycrystalline fiber with near stoichiometric ratio introduces element B on the basis of the previous generation. After treatment in 1550 ° Ar for 10h, the fiber tensile strength can be maintained above 2.8Gpa, and the performance is excellent. At present, a large number of commercial development have been carried out. Domestically, after years of technical research, KD series and XD series fibers have been developed respectively, represented by National University of Defense Science and technology and Xiamen University. They have also become the two top SiC fibers platforms in China. Among the three fibers of KD series, through element and structure analysis, it is found that KD-II fiber has lower oxygen content than KD-I fiber, so the modulus and stability have been significantly improved. The latest KD-SA fiber has greatly improved the temperature resistance by adding Al, Fe, B, Ti and other elements. XD series fibers are mainly XD-II and XD-III fibers. According to the observation of their morphology and internal structure by transmission electron microscope, the content of free carbon is significantly reduced, and the purpose of high heat resistance of SiC fibers can also be achieved [3-6].

Therefore, it is necessary to study and analyze the microstructure of SiC fibers by adopting various material characterization means, making correct and appropriate use of various testing equipment, starting from the relationship between material composition and performance, find out the main structures and components affecting performance, and then optimize by improving process conditions to develop new and higher quality SiC fibers, so as to improve the performance of fibers [7]. In this paper, the microstructure characterization methods of SiC fibers are reviewed from three aspects: structural composition analysis, element composition analysis and micro morphology. Combined with the performance characteristics and application fields of various types of SiC fibers, the analysis principle, advantages, disadvantages and development status of several material detection technologies such as XRD, SEM and tem are summarized.

2. Microstructure Characterization of SiC Fiber

The results show that the microstructure of SiC fibers determines its properties. Therefore, studying the microstructure of SiC fiber is conducive to analyze its properties and lay a foundation for improving the properties of ceramic matrix composites.

2.1. Morphology Analysis

2.1.1. Scanning Electron Microscope (SEM). The principle of Scanning Electron Microscope (SEM) is to use a narrow focused high-energy electron beam to scan the sample, stimulate various physical information through the interaction between the beam and the material, and collect, amplify and reimage the information to achieve the purpose of characterizing the micro morphology of the material. The resolution of the new scanning electron microscope can reach 1 nm; The magnification can reach 300000 times or more and can be continuously adjustable; And the depth of view is large, the field of vision is large, and the imaging three-dimensional effect is good. Now the most widely used are tungsten filament and field emission Scanning Electron Microscope.

With the development of science and technology, Scanning Electron Microscope is the most commonly used characterization instrument to observe the micro morphology of materials. The conditions are not harsh and the operation is not difficult. The fiber surface and fracture morphology can be observed by scanning electron microscope. Due to the poor conductivity of SiC fibers, it needs to be sprayed with gold before observation, and according to the characteristics of small diameter, it needs to be enlarged by 6000-12000 times for observation. In addition, the X-ray analysis system (EDS) equipped by the Scanning Electron Microscope can also be used to analyze the micro region composition of the fiber [8-11].

As shown in figure 1, it is the SEM photo of KD-SA fiber surface observed by Yu[5] at 10000 times. It is found that many small particles (SiC grains) are densely and evenly arranged and tightly
combined, which shows that the tensile strength of the fiber is high and the mechanical properties are good.

![SEM morphology of KD-SA SiC fiber](image)

**Figure 1.** SEM morphology of KD-SA SiC fiber [12].

The fracture morphology of SiC fiber after tensile fracture is usually observed by Scanning Electron Microscope. The fatigue fracture source area, crack propagation area and instantaneous fracture area can be analyzed, so as to infer the fracture type and process. As shown in figure 2, it is the fracture morphology of Hi-Nicalon SiC fiber observed by Yao et al. [12] after high temperature annealing treatment. It can be seen that the fracture surface of the fiber is uneven, in feather shape, radiating from inside to outside. It is inferred that the fiber shows amorphous brittle fracture.

![Fracture morphology of Hi-Nicalon SiC fiber](image)

**Figure 2.** Fracture morphology of Hi-Nicalon SiC fiber [12].

2.1.2. Transmission Electron Microscope (TEM). TEM has higher magnification and resolution than SEM. At present, the highest resolution of transmission electron microscope can reach 0.2nm, so it can better and more clearly observe and analyze the micro morphology of fibers. Therefore, high-resolution transmission electron microscope TEM is also the most comprehensive and detailed characterization method with the best development prospect in the future. TEM can also be applied to phase structure analysis. Compared with SEM, it can further observe the crystal or atomic structure inside the material, such as lattice defects and structural changes. The current limiting conditions are harsh use environment and immature sample preparation methods, which is the biggest challenge faced by Transmission Electron Microscopy.

TEM sample preparation is difficult, and the methods are complex and diverse. At present, it is still in the development stage. The core idea is to make the sample into a film with a thickness of about 50~100nm, but the SiC fiber has a fine diameter and the material is hard and brittle. It is difficult to prepare appropriate TEM samples by conventional methods to meet the fine analysis of microstructure. Zhang et al. [12] Found that the cross-section TEM sample of fiber can be prepared by focused ion beam cutting (FIB), but this method requires professional instruments and high preparation cost. Therefore, the commonly used method is still embedding method. SiC fiber is implanted into a copper
tube with an outer diameter of 3mm and injected with epoxy resin. After curing, cut the sheet with a slicer and make the film sample after several grinding.

As shown in figure 3, it is the TEM image of a domestic high-strength SiC fiber observed by Shen et al. [12] with Transmission Electron Microscope. It can be seen that the grain of SiC fiber is relatively small, because the crystal SiC is the central structure of face centered cubic lattice, and there is co deposition of free Si during fiber preparation, which will cause lattice disorder of SiC.

![Figure 3. TEM image of SiC fiber](image)

2.1.3. Atomic Force Microscope (AFM). AFM is a new atomic level high-resolution instrument. Its principle is that when the distance between atoms is reduced to a certain extent, the force between atoms will rise rapidly. Therefore, the height of the sample surface can be directly converted from the force on the microprobe, so as to obtain the information of the sample surface morphology [13]. It can observe the sample surface at high magnification in the atmospheric environment, go deep into the nano scale and even atomic structure, and obtain the three-dimensional morphology of the sample surface. It has been widely used in the fields of semiconductors and nano functional materials. For SiC fibers, AFM can be used to analyze its surface morphology.

The sample preparation of AFM is very simple. Just stick SiC fibers on stainless steel disc with double-sided adhesive, record the surface morphology of the sample through atomic probe contact scanning, analyze the data, calculate the fiber surface roughness and draw the corresponding image. Figure 4 shows Yu [13] applying atomic force microscope with a scanning area of 4μm² AFM 3D diagram of domestic KD-SA fiber surface observed. It can be found that the surface of KD-SA fiber is in the shape of undulating "peaks". Many small SiC particles or SiC columns are exposed on the surface of KD-SA fiber, which are neither isolated nor prominent. The "peaks" are low. These SiC particles are densely crowded together, their roots are interconnected, and the pores on the surface are relatively shallow.

![Figure 4. AFM 3D image of KD-SA fiber surface](image)
Compared with SEM and TEM, AFM has many advantages, such as providing the real three-dimensional surface of the sample, no special treatment of the sample, less restrictions on the use environment and strong software processing function; However, it also has the disadvantages of small imaging range and slow speed, and is greatly affected by the probe.

2.2. Component Analysis

2.2.1. Energy Dispersive Spectrometer (EDS) Analysis. EDS is used together with scanning electron microscope or transmission electron microscope. It is one of the main means of fiber micro region composition analysis. Various elements have their own X-ray characteristic wavelength, and the size of the characteristic wavelength depends on the characteristic energy $\Delta E$ released during the energy level transition. The energy spectrometer uses the characteristic of different X-ray photon characteristic energy of different elements for component analysis. Although the energy spectrum system attached to SEM can not be used for accurate quantitative analysis, but can only be used for semi quantitative analysis, it can be used to characterize the presence and relative content of elements on the surface of SiC fibers.

For sample preparation, SiC fiber needs to be sprayed with gold, and then fixed on the sample table with conductive adhesive for observation. It has the advantages of fast analysis speed, high sensitivity, good repeatability and stability of spectral lines, and there is no problem of focusing. It is suitable for the analysis of rough surfaces; The disadvantage is the low energy resolution and peak back ratio of spectral lines [14-16].

2.2.2. X-ray Photoelectron Spectroscopy (XPS) Analysis. XPS is a surface analysis method. Its principle is to radiate the sample with X-rays to excite and emit the inner electrons or valence electrons of atoms or molecules. The electrons excited by photons are called photoelectrons. The energy and quantity of photoelectrons can be measured to analyze the chemical properties and element composition of the sample surface. XPS analysis uses Al target as X-ray excitation source and monochromatic ALK at room temperature and vacuum $\alpha$. The surface of SiC fibers after drying was analyzed by XPS. The method is as follows: firstly, full XPS scanning is carried out on the fiber surface to detect all the elements on the fiber surface at one time, and then high-precision XPS narrow scanning is carried out on the C$_{1s}$, Si$_{2p}$ and O$_{1s}$ peaks of the fiber to determine the specific chemical existence state of each element. XPS sample preparation generally adopts powder tablet pressing method, and the sample is placed on clean aluminum foil. For SiC fibers, its conductivity is not very good, so it can be sprayed with gold first. In addition, sample pretreatment is also very important. Before sample preparation, it must be stripped with Ar$^+$ ions to remove sample surface pollution [17-18].

As shown in figure 5, it is the XPS full scan spectrum of the surfaces of four kinds of domestic SiC fibers observed by Zhu [18]. It can be seen that the XPS characteristic peaks of C$_{1s}$ and O$_{1s}$ appear on the outermost layer of the fiber, and faint Si$_{2p}$ and Si$_{2s}$ characteristic peaks are faintly visible. This shows that the element composition of the fiber surface mainly includes C, Si and O elements, and the relative content of C and O elements on the surface is higher than that of Si elements, that is, the fiber surface is mainly rich in carbon and a small amount of oxygen [18].
2.2.3. Auger Electron Spectroscopy (AES) Analysis. The principle of AES is to use Auger effect to conduct qualitative and quantitative analysis by detecting the energy and quantity of Auger electrons. AES is used to identify the chemical properties and composition of the sample surface. It is characterized by Auger electron source surface or even a single atomic layer, which only brings out the chemical information of the surface. It has the characteristics of small analysis area, shallow analysis depth and no damage to the sample. AES operation is relatively simple. After the fiber is fixed on the sample table, the AES energy spectrum of the fiber surface is directly measured to obtain the information of each element on the fiber surface. The pretreatment of samples, like XPS, also needs Ar\(^{+}\) ion sputtering technology to remove the pollution on the sample surface. At the same time, the fiber is peeled in the depth direction along the radial direction, and then the micro surface analysis can be carried out to obtain the distribution of elements from the surface to the interior of the fiber along the radial direction.

Therefore, compared with XPS, AES has higher sensitivity of chemical analysis and faster speed of data analysis. Both are mainly applied to the surface element analysis of fibers, but XPS has better quantitative analysis ability and is more widely used, but its disadvantages are difficult to focus and large irradiation area [19]; In contrast, AES has high micro area analysis ability and strong depth profile analysis ability, but its problem is that it can only determine samples with good conductivity, and can not spray gold on insulated samples.

Zhu [19] scanned the surface and interior of a domestic SiC fiber through AES, and obtained the full scan diagram as shown in figure 6. It can be found that the element composition is only C, Si and O. at the same time, comparing the two spectra, it can be seen that the C and O peaks on the surface are higher than the internal C and O peaks, while the Si peak on the fiber surface is much lower than the Si peak inside the fiber, indicating that the fiber surface is rich in carbon and oxygen compared with the interior [20].

**Figure 5.** XPS full spectrum of SiC fiber surface [18].

**Figure 6.** Full sweep of Auger electron spectroscopy of SiC fiber: (a) surface, (b) interior [19].
2.3. Structural Analysis

2.3.1. X-ray Diffraction (XRD) Analysis. The principle of XRD analysis is Bragg equation: \(2d\sin\theta = n\lambda\). When a beam of X-ray passes through the crystal, diffraction will occur. The superposition of diffraction waves will strengthen the intensity of the ray in some directions and weaken in other directions. The crystal structure can be determined by analyzing the diffraction pattern obtained on the photographic negative. The sample preparation of X-ray diffraction analysis is relatively simple. A bundle of fibers to be tested can be placed on the sample table orderly, and its surface can be tested directly; For phase analysis, the sample needs to be ground into a powder suitable for diffraction experiment, and then the powder is made into a very flat test piece. In the whole process, it is necessary to maintain the stability of the sample, so that the diffraction data obtained can be meaningful. For the test of fiber samples, it should also be pointed out whether the irradiation direction of the test fiber is parallel irradiation or vertical irradiation, because different orientations will lead to different diffraction intensities [20-23].

XRD is an effective means to analyze the phase structure and preferred orientation of SiC fibers. It is known that SiC fibers are generally composed of \(\beta\)-SiC grain, graphite and certain content of amorphous phase sicxoy. As shown in figure 7, it is the XRD pattern of a typical Continuous SiC fiber obtained by Zhang [21]. It can be seen that in addition to the extremely strong diffraction peaks of SiC (111), there are also SiC (220), (311) and (222) diffraction peaks in the spectrum, indicating that SiC fibers are mainly composed of \(\beta\)-SiC composition. SiC (111) diffraction peak is high and sharp, indicating \(\beta\)-SiC has high crystallinity and large grain size [22]. \(\beta\)-SiC is a face centered cubic structure, (111) is its closely arranged surface. After the formation of crystal nucleus, it begins to grow in its preferred <111> orientation. According to the competitive growth mechanism, the crystal nucleus with <111> orientation parallel to the radial direction of fiber has the fastest growth rate, and finally develops into columnar crystal with the continuous growth of grain size, showing obvious <111> preferred orientation [23].

![Figure 7. XRD pattern of SiC fiber [21].](image)

In addition, XRD can also be used to calculate the grain size of fibers. After obtaining the XRD spectrum of the fiber, the phase analysis and measurement were carried out by using the analysis software The half height width \(B\) of the diffraction peak of the (111) crystal plane of \(\beta\)-SiC grain is calculated by using equation (1) Scherrer formula to calculate the average thickness \(L_{111}\) perpendicular to the (111) crystal plane, where \(L_{111}\) can approximately represent the average size of the grain [24].

\[
L_{111} = \frac{K\lambda}{B\cos\theta}
\]  

(1)
2.3.2. Raman Spectroscopy (RMS) Test. Raman Spectrum is a scattering spectrum based on Raman Scattering effect. Because the frequency difference between scattered light and incident light is independent of the frequency of incident light and only related to molecular structure, it can be used for the identification of molecular groups and the analysis of material structure, and is particularly sensitive to carbon containing materials. The method is to directly measure the surface and cross section of SiC fiber samples at room temperature. The wave number range is 500–2000 cm\(^{-1}\) and the resolution is 1 cm\(^{-1}\) [25]. The sample preparation of Raman spectrometer is not too difficult. Like XRD method, the fiber surface can be measured directly at room temperature. To determine the phase composition, the fiber needs to be cured, mechanically ground, made into a thin film sample, and then polished and polished.

The fast and convenient Raman Spectrum and the wave number are very sensitive, suitable for analyzing the phase structure of SiC fibers, especially for characterizing the free carbon in the material. Figure 8 shows the Raman Spectrum of a typical SiC fiber polishing sample observed by Zhang [26] from the W/SiC interface reaction layer to the surface along the fiber direction. The number marked on each spectrum line is the distance between the test point and the reaction layer, 0 μm is the nearest test point near the reaction layer. β-SiC is a face centered cubic structure, and the C atoms and Si atoms in the crystal cell form a non central symmetrical structure similar to diamond by covalent bond [27-31]. Therefore, two scattering peaks separated from each other are shown in the Raman Spectrum, located at 796 cm\(^{-1}\) and 972 cm\(^{-1}\). Combined with the Transmission Electron Microscope bright field image and high-resolution lattice image, it can be deduced that the SiC crystal particle size change, low-scale disorder and Si co deposition defects in the process of fiber preparation by chemical vapor deposition lead to the disordered microstructure of SiC layer. These defects are sensitively reflected in the Raman Spectrum by the frequency shift and intensity change of scattering peak [26-27].

![Figure 8. Laser Raman Spectra of SiC fibers at different positions [26].](image)

Raman Spectroscopy has been more and more widely used in the analysis of SiC fibers in recent years. Compared with XRD analysis method, its advantage is that the bulk phase signal is deeper, up to hundreds of nanometers, its laser beam focusing radius is smaller, and the samples with smaller area can be analyzed. Based on different working principles, their application fields are not exactly the same. XRD is only suitable for measuring substances with lower concentration. At the same time, RMS also has the following shortcomings: (1) Raman scattering area; (2) Different vibration peak overlap and Raman scattering intensity are easily affected by optical system parameters; (3) The interference of fluorescence phenomenon on Fourier transform Raman spectroscopy; (4) In Fourier transform spectral analysis, the problem of curve nonlinearity often occurs; (5) The introduction of any other substance will bring some degree of pollution to the measured body system, which is equal to the possibility of introducing some errors and will have a certain impact on the analysis results [9]. However, if Raman Spectroscopy and X-ray diffraction are used to analyze the fiber surface, the surface and cross section can be measured directly at room temperature, which is a great convenience and advantage.
2.3.3. *Transmission Electron Microscope (TEM).* The principle of TEM has been explained in the above morphology analysis. It can not only observe the micro morphology, but also be used for the internal structure analysis of SiC fibers. As shown in figure 9, it is the high-resolution lattice image of a micro region of SiC fiber observed by Zhang [28]. The phenomenon of "tailing" of image diffraction spots indicates that there are a large number of growth defects such as twins and stacking faults in the growth process of SiC fibers [13-19]. TEM analysis can be combined with Raman Spectrum to determine the grain boundary, stacking fault and other defects of SiC [28-29].

![Figure 9. high resolution lattice image of a micro region of SiC fiber [28].](image)

2.3.4. *Small Angle X-ray Scattering (SAXS).* SAXS analysis is to calculate the aperture distribution of the sample according to the X-ray scattering spectrum of the sample [30]. As the name suggests, the difference between SAXS analysis and X-ray large angle diffraction method is that the angle is very small. Because X-ray can penetrate the sample, the pore size distribution of all pores in the sample can be obtained, and the pore size distribution of micropores in SiC fibers can be obtained. Because there are a certain amount of micropores in SiC fibers, the determination of micropore content is of great significance for the preparation of high-strength fiber, which also promotes the application of small angle X-ray scattering in the study of material microstructure, and its research trend is increasing year by year. Although it is still in the development stage, it has shown its advantages in quantitative study of fiber pores compared with SEM, TEM and other methods. The advantage of SAXS is that there is no need for additional pretreatment of samples, which can be carried out directly in the laboratory.

The sample preparation method is to carry out temperature rise inorganic treatment at different temperatures under the protection of N₂, and then naturally cool to room temperature to prepare fiber samples [30-31]. Before the test, the sample needs to be ground into powder and made into sample pieces. Figure 10 shows the pore size distribution of SiC fiber at a certain temperature calculated by he [31] and others with SAXS. It can be seen from the figure that the pores of the fiber in the range of 10–15 nm account for a large proportion. At this time, the fiber begins to decompose and gas (such as H₂, CH₄, etc.) is generated inside the fiber. The overflow amount of gas is less. Due to volume expansion, more micropores are generated in the fiber [32]. The research shows that the micropore content in SiC fiber will increase with the increase of temperature. In the pores of the fiber, the proportion of micropores is high, but the pore volume is very low, indicating that SiC fiber is not suitable for direct adsorption fiber. As for the extent to which the strength of SiC fiber is affected by micropores, further research is still needed.
Figure 10. Pore size distribution of domestic SiC fiber at a certain temperature [31].

In addition to the detection technologies mentioned above, there are the following characterization methods applied to SiC fibers: (1) Secondary Ion Mass Spectrometry (SIMS), which can analyze the elemental characteristics of the sample surface by using the secondary ions generated after high-energy electron beam bombardment with the sample [33]; (2) Fourier transform infrared spectroscopy (FT-IR) can qualitatively analyze the internal chemical composition and structure of SiC fibers by using the absorption characteristics of substances to infrared radiation of different wavelengths [34]; (3) Electron Energy Loss Spectroscopy (EELS), by measuring the kinetic energy change of electrons after interaction with samples, the atomic structure and chemical characteristics of fibers can be determined [35]; (4) Nuclear Magnetic Resonance (NMR) generally selects organic solvent and uses Tetramethylsilane (TMS) as internal standard to determine different elements in fiber [36-37]; (5) High Resolution Electron Microscopy (HREM), SiC fibers morphology characterization instrument, higher resolution than SEM and higher acceleration voltage [38]. However, due to the harsh external objective conditions, the limitations of use characterization and the difficulty of operation, the scope of application is still limited.

3. Conclusion
In this paper, the characterization methods of the microstructure of silicon carbide fiber are summarized from three aspects: micro morphology, element composition analysis and structural composition analysis. SEM, TEM, XRD and other methods are explained from the aspects of operation principle, sample preparation, comparison of advantages and disadvantages, development status and so on. From the current research results of SiC fibers, SEM has become the most common method to study the microstructure of SiC fibers because of its simple operation, while TEM and AFM have not been widely used to observe the surface morphology of SiC fibers due to sample preparation problems and application limitations; In the aspect of composition analysis, several methods have their own advantages and disadvantages, so EDS, XPS and AES are generally used to analyze the element content and distribution of SiC fibers in an all-round way; In terms of structure, XRD and RMS are the two most classical analysis methods. For the microporous defects in SiC fibers, SAXS is a characterization method for measuring pore size distribution, which has sprung up in recent years, but it is not mature and needs further development and research.

In terms of fiber characterization, although some research has been carried out on various characterization methods at home and abroad, the problem of sample preparation has been accompanied by the small size of fiber and easy brittle fracture, and there is little research on the mechanism of surface defects and internal microstructure changes of SiC fibers. Therefore, a more systematic SiC fibers characterization system needs to be established, to further improve the related research on the microstructure of SiC fibers, and then optimize the process conditions to improve the properties of SiC fibers. It provides theoretical support and help for the research in the field of silicon carbide fiber in the future.
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