Abstract: The paper proposed a preprocessing method for scanning samples based on rinsing technology, and solved the effective fabrication problem of AFM scanning samples based on facilitated dispersing SWCNTs with SDS surface active agents. First, the ultrasonic oscillation method was applied, and the uniform alignment of SWCNTs in SDS was realized. Then, SDS solution of different concentrations was scanned and imaged with AFM, the influence of SDS solution on the imaging quality of SWCNTs was analyzed, and the method to effectively fabricate scanning samples after dispersing SWCNTs was found. The results of the experiments showed that the preprocessing of the SWCNTs solution scanning samples was the decisive factor to influence SWCNTs imaging quality, that the result of rinsing SWCNTs scanning samples for 5s at 0.1ml/s with di-ionized water was the best, and that with the same rinsing rate and angle, the rinsing di-ionized water quantity would influence the alignment degree of SWCNTs on the substrates and SDS macromolecules’ residual quantity on SWCNTs scanning samples.

1. Introduction
Since Iijima, a Japanese scientist, found carbon nanotubes (CNTs) in 1991, CNTs have received extensive attention due to the special physical structure and electrical characteristics[1]. CNTs have extreme potential in such areas of nano electrical devices and new functional materials as field-effect transistors, photoelectric near infrared spectral emitters, sensors and polymer compound materials[2]. As one-dimensional nano materials, due to the attraction of Van der Waals force, single-walled carbon nanotubes (SWCNTs) tend to twine with one another so as to gather easily and decrease the total surface energy of the system[3]. The gathering form usually destroys the excellent characteristics of SWCNTs in mechanics, optics, electricity and calorifics, which limits the use of CNTs in the fabrication of nano devices. For example, when SWCNTs are used in the assembly of carbon nanotube field effect transistor (CNT-FET), SWCNTs are required to accurately align and singly disperse between source and drain[4]. Thus, it’s of great necessity to study SWCNTs dispersion property. SWCNTs are lack of active genes, difficult to dissolve in organic solution and water, which makes the research on the chemical qualities hard[5]. Therefore, the research on how to improve SWCNTs dispersion property has become the precondition in the actual application of SWCNTs[6].

The key point in obtaining ideal dispersed SWCNTs is to solve the problem of uniform dispersion and dispersion stability of SWCNTs on substrates[7]. The surface active agent has special structure, which is formed by both hydrophilic groups and oleophilic groups, and thereby has good adsorption
and chemical reaction activity[8]. The SWCNTs surface activity can be changed by decreasing the surface tension of SWCNTs. The surface active agents adsorb on the surface of SWCNTs, forming colloids covering SWCNTs, and due to ionization or adsorption, the surface of colloids have charges[9]. Because of electrostatic repelling force, colloids can gather together, which makes nano particles in stable situation. Moreover, the long tail ends of surface active agents form space steric hindrance on the surface of nano particles, preventing SWCNTs from re-gathering. Due to adsorption, the surface active agents cover nano materials, forming a space barrier, keeping nano materials from colliding and combining with one another, which makes SWCNTs disperse in solution stably. Therefore, the dispersion, reaction and the surface structure of nano particles of SWCNTs in different dielectrics are improved. However, when the surface active agents reach a certain concentration, the surface active agent molecules would combine into colloid polymers from single ion or molecule, forming colloid groups, and the solution quality will mutate and form unimolecule film and make the adsorption ability decrease sharply. Meanwhile, the dispersion of SWCNTs will be influenced greatly[10].

Sodium Dodecyl Sulfate (SDS) is a comparatively long negative ion surface active agent. It can adsorb on the surface of solution and form diffusion layer, preventing SWCNTs from re-gathering. Thus, SWCNTs have good dispersion property. Whereas the macromolecules of SDS will influence the scanning and imaging of SWCNTs, even make it hard to obtain clear AFM images of SWCNTs. Against the influence of SDS on the scanning and imaging of SWCNTs, the paper proposed a post treatment method based on rinsing technology, and solved the effective fabrication problem of atomic force microscope (AFM) scanning samples based on facilitated dispersing SWCNTs with SDS surface active agents[11].

2. Experiment materials and methods

2.1 Experiment reagents and instruments

The nano materials used in this experiment are SWCNTs produced by Chengdu Institute of Organic Chemistry (model SH071011, diameter of SWCNTs 0.8-2nm, length 2-5 μm, purity >90%, surface area >380m²/g). SDS surface active agents were purchased from Beyotime Institute of Biotechnology Co., Ltd. Ultrasonic oscillator were purchased from Shanghai KeDao Limited Company of Ultrasonic Instrument (type: SK5210LHC). Atomic force microscope(AFM) was purchased from Veeco Company of United States (type: Dimension 3100).

2.2 The experiment flow

First, four kinds of 10ml SDS solution were prepared, whose concentrations were respectively 1%, 0.1%, 0.01%, and 0.001%. They were put in four test tubes and were sonicated for 10 minutes. Then 2 l solution of each was obtained with pipette to be scanned with AFM. The accurately matched SWCNTs solution (The quality percentages of the matched SWCNTs solution and SDS solution were respectively 0.1% and 1%). was put in test tubes and was sonicated for 4 hours. 2 l solution was obtained to be scanned with AFM, watched and compared. The experiment flow can be shown in Fig.1.

Fig. 1 The experiment flow
2.3 Observation of SDS solution samples of different concentrations scanned with AFM

The gathering activity of SDS surface active agents on the water solution interface was helpful to improve the dispersion stability of SWCNTs, whose concentration change played a key role in colloid system and the directed alignment of molecules. Meanwhile, when SDS facilitated the dispersion of SWCNTs, the coverage of SWCNTs prevented AFM scanning from imaging clearly, and part of SWCNT was covered and couldn’t show the superior electrical characteristics. So the study on the influence of SDS solution of different concentrations on the imaging of SWCNTs is also a key step in SWCNTs preprocessing.

10mg SDS solid powder was put in the test tube with 9.99ml di-ionized water, made into SDS solution whose concentration was 1%, sonicated for 10min to solve completely in the ultrasonication oscillator. Then 2μl reagent was dropped on a mica sheet with pipette, laid uniformly on the sheet with capillary quickly, weathered with N₂ immediately and scanned with AFM. 5ml SDS solution whose concentration was 1% was diluted 10 times into 0.1% solution with di-ionized water. All the same, SDS solution whose concentration was 0.01% and 0.001% was obtained and scanned with AFM. During imaging, the AFM system was working in tapping mode with frequency of about 300kHz, and the AFM probe used here was model MPP-11100 with rectangle Si cantilever, tip radius less than 10nm. The experiment process is showed in Fig. 2.

2.4 Observation of the scanning of SWCNTs with AFM
2.4.1 The SWCNTs dispersion in SDS surface active agents

The accurately matched SWCNTs solution (The quality percentages of the matched SWCNTs solution and SDS solution were respectively 0.1% and 1%) was put in a test tube, sealed with plastic film, sonicated for 2h with frequency of 59kHz in the ultrasonication oscillator, rested for 1h, sonicated again for 2h, rested for over 3h and disposed by precipitating. During ultrasonication, the location and the inclination angle of the test tube in the ultrasonication oscillator were regulated in the process of sonication so that SWCNTs solution in the test tube was “boiling” to mix completely uniformly. At the same time, the test tube was heated to 50°C to facilitate the dispersion.

![AFM scanning observation](image1)
![Ultrasonic dispersion](image2)

**Fig. 2** The experiment process under different SDS concentrations
2.4.2 SWCNTs rinsing disposition and AFM observation

2 μl SWCNTs solution was dropped on a mica sheet with pipette, laid uniformly on the sheet with capillary quickly, rested for 1m and weathered with N2. Sample A was obtained. All the same, 2 μl SWCNTs solution was dropped on a mica sheet twice, laid uniformly and rested for 1m. The mica sheet was down dipped 30 degree angle. Then the samples were rinsed with 0.5ml and 1ml di-ionized water at the rate of 0.1ml/s, and blew in very short time with nitrogen impulse gas flow of a certain pressure (about 0.15MPa). Sample B and C were obtained. The post treatment process is showed in Fig. 3.

Samples A, B and C and the SWCNTs solution sample that was not dispersed were scanned and compared with AFM in tapping mode.

3 The experiment results and analysis

Fig. 4 is the AFM characterization observation figure of SDS of four different concentrations: 1%, 0.1%, 0.01% and 0.001% in micron scope. From fig. 4(a), we can see that SDS surface active agent molecules form colloid, so that the contact of water and air reduces and the surface tension of the solution decreases. In Fig. 4(b), some SDS molecules gather and the hydrophobic groups of these SDS surface active agents rely on one another, forming small colloid groups, so the surface tension of the solution increases. SDS colloid groups in fig. 4(c) are much less than those in (b) and (c), and the surface tension is bigger. The surface energy of SWCNTs decreases with it. In fig. 4(d), the concentration of the SDS surface active agents is so low that air and water contact directly, there are a few surface active agent molecules in water, SDS has no colloid groups, and the electrostatic repelling force between colloids becomes smaller and the adsorbency decreases with it.

Fig. 3 SWCNTs post treatment principle

Fig. 4 The figure of SDS of different concentrations scanned with AFM

SWCNTs were dispersed by the method in 1.4 and the dispersed reagents were light black uniform
transparent bodies. There were no graininess suspended substances in the reagents through naked eye observation. Such SWCNTs sample was characterized with AFM. From fig. 5(a), it can be seen that SWCNTs which was dispersed only by ultrasonication oscillation and not added into SDS surface active agents gather seriously. Fig. 5(b) is SWCNTs solution that has not been rinsed. Some SWCNTs exist singly and don’t twine with one another, but seen from the AFM height characterization value, the height of single SWCNTs is less than 2nm and the dispersion situation is not ideal. Compared with fig. 4(a), because SDS colloid groups are too many and the length and the diameter of SDS molecules are far larger than SWCNTs, SDS surface active agents form saturant adsorbency on SWCNTs and cover most SWCNTs, so that more SWCNTs can’t be characterized with AFM. And just because of the influence of macro molecules of SDS active agents on the scanning and imaging of SWCNTs, SWCNTs solution can be post treated by rinsing. Fig. 5(c) is the figure of SWCNTs solution scanned with AFM that is washed with di-ionized water at 0.1ml/s for 5s. From the AFM height characterization value, it can be seen that the height of single SWCNTs is less than 2nm. And compared with fig. 5(b), some SDS molecules adsorbed on SWCNTs are flushed out with water, thus the adsorbency quantity decreases, but the single SWCNTs are comparatively more. Therefore, SDS is good for decreasing the surface energy of SWCNTs and increasing the mutual repelling force, and the macro molecules of SDS surface active agents also have less influence on the scanning and imaging of SWCNTs. Fig. 5(d) is the figure of SWCNTs solution scanned with AFM that is washed with di-ionized water at 0.1ml/s for 10s. It is showed in the figure that the SDS concentration in the solution is low, but because of rinsing, most SWCNTs lose and the SWCNTs stability isn’t good.

Fig. 5 The figure of before and after rinsing SWCNTs scanned with AFM
4. Conclusion

The results of the research show that the preprocessing for scanning samples method based on rinsing technology can solve the effective fabrication problem of AFM scanning samples based on facilitated dispersing SWCNTs with SDS surface active agents. Before centrifugating purification, the research on the influence of the macro molecules of SDS surface active agents on the scanning and imaging of SWCNTs is a meaningful step in the preprocessing of SWCNTs. In the fabricated SWCNTs solution, besides foreign substances brought by self growth of SWCNTs, such as carbon nano particles, amorphous carbon\cite{ref}, fullerene particles and metal catalysts. SDS surface active agents can block the imaging of pure SWCNTs. From the SDS characterization figure obtained by observing AFM scanning, the SWCNTs characterization figures before and after rinsing, the influence degree of SDS molecules on the scanning and imaging of SWCNTs can be clearly shown. What’s more, the factors influencing the imaging of SWCNTs are properly considered, which will provide important foundation for how to obtain ideal dispersion results of SWCNTs.

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