

Study on Nano-SiO₂ measured by Atomic Force Microscope

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Abstract. The morphology and structural evolution of nano-SiO₂ powders obtained by pressuring samples and sintering in muffle at different temperatures were studied by X-ray diffraction (XRD) and atomic force microscope (AFM). The experimental results show that the particle sizes of nano-SiO₂ increase with temperature rising, and it meets the physical mechanism of particle growth.

Introduction

As one of the most widely used nanomaterials, the nano-SiO₂ with its unique small size effect, surface and interface effect, volume effect, etc., have broad application prospects^[1-2]. Nano-SiO₂ is added to other materials to improve certain characteristics of the material. When the core of Al₂O₃/SiO₂ nano-composite materials is prepared, adding the right amount of SiO₂ powder to make of nano-ceramic cores sintered, the composite density increased and sintering temperature decreased, the strength improved^[3]. In this paper, the AFM is used to scan the nano-sio₂ samples sintered at different states at normal temperature and pressure condition in the contact mode to obtain the surface imaging, then to analyze the particle size through the post-processing software to study particle size variation with sintering temperature.

Experimental methods

Nano-SiO₂ Powder (purity: 99.9%, average particle size 20 nm, surface area 600 m² / g). The proper powder will be pressed into the sample in the mold ($\Phi=6\text{mm}$) of tablet machine (DY-20) under 90MPa. The samples are sintered 2 h in muffle furnace at 200 °C, 900 °C, respectively. The membrane-surface morphology, in terms of the mean surface roughness (Ra), was studied by atomic force microscopy (CSPM-3000) using the contact mode. The mean roughness was defined as the average value of the surface relative to the center plane for which the volumes enclosed by the images above and below the plane were equal.

Result and discussion

Fig.1 shows XRD spectra of SiO₂ original powder. The diffraction peak is a crystal package. The crystalline state of original powder is amorphous.

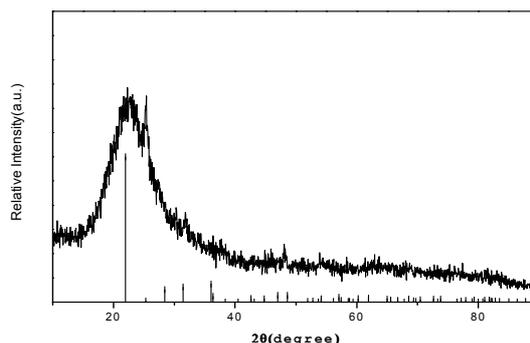


Fig.1 XRD patterns of SiO₂ original powder

Fig.2, Fig.3, Fig.4, Fig.5, Fig.6 show that the particle size distribution of the surface is uneven, and the particle sizes of original powder and the sample sintered at 200°C are smaller; the particle sizes of the sample sintered at 900°C are larger. The particle size increased significantly as the sintering temperature increased.

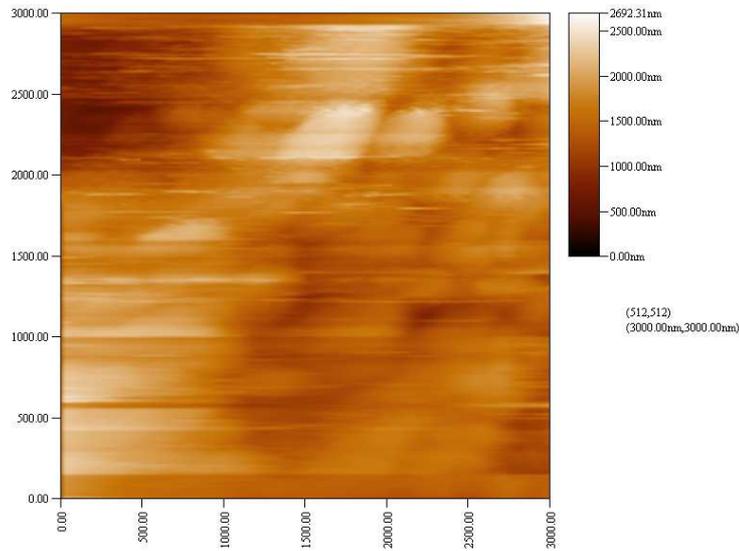


Fig.2 The AFM image of SiO₂ original powder

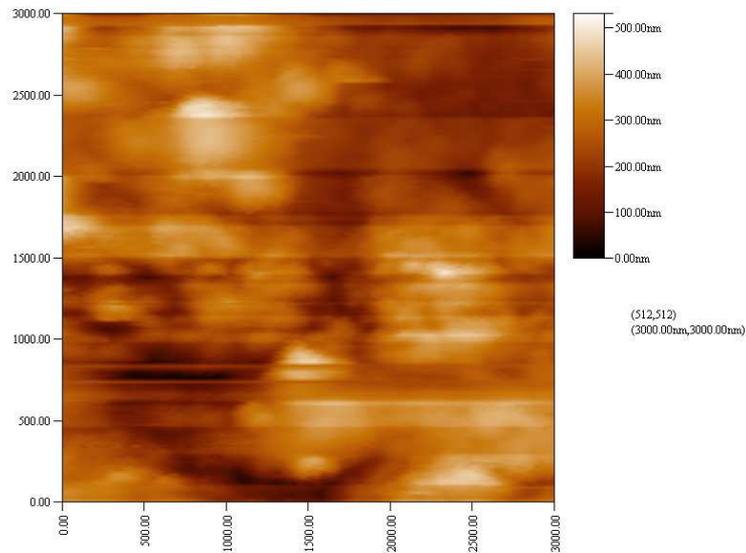


Fig.3 The AFM image of SiO₂ sample sintered at 220 °C for 2 hours

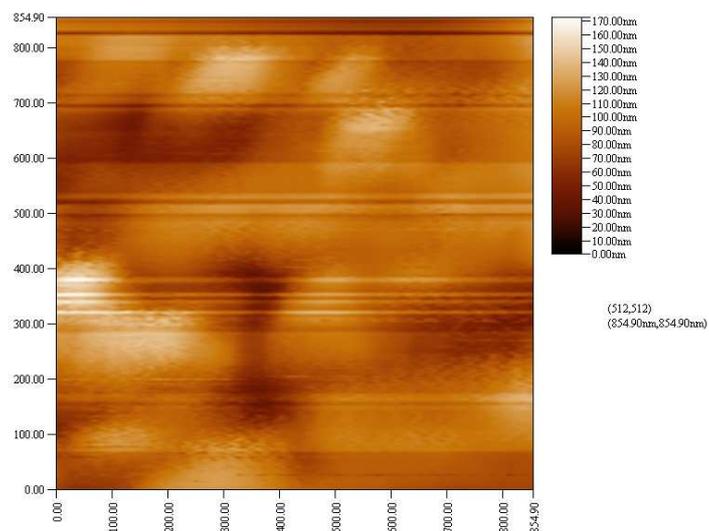


Fig.4 The AFM image of SiO₂ sample sintered at 220 °C for 2 hours

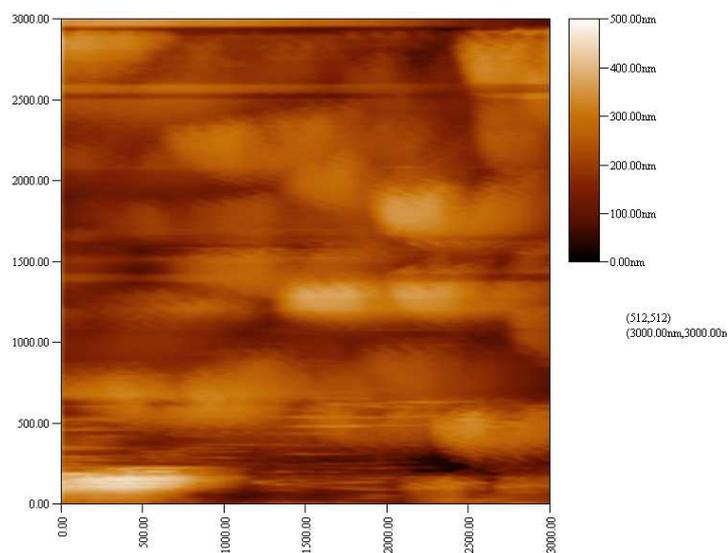


Fig.5 The AFM image of SiO₂ sample sintered at 900 °C for 2 hours

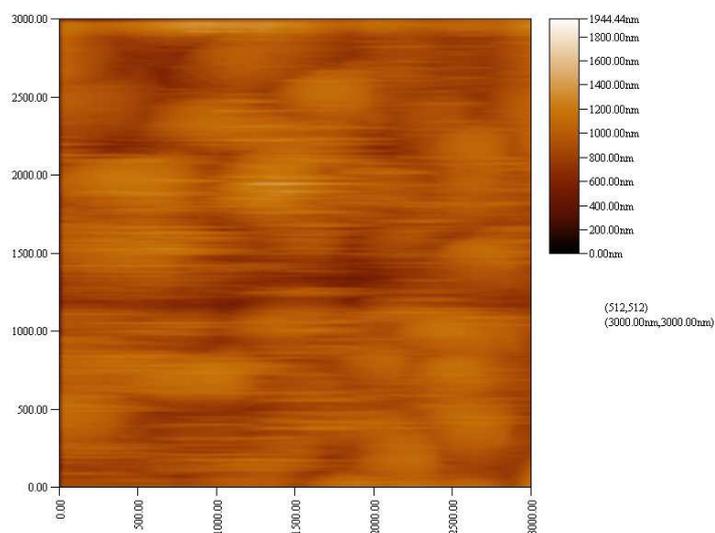


Fig.6 The AFM image of SiO₂ sample sintered at 900 °C for 2 hours

Table 1 show that the average particle size of the original powder is 110.87nm, the average particle size of samples sintered at 200 °C is 96.63nm, similar to the original powder, the average particle size of samples sintered at 900 °C is 184.70nm, increased by 66.59% than original powder. The particle size of the original powder obtained by AFM scanning is much larger than the calibration value (20nm), probably due to the particle size of SiO₂ nano-crystalline at initial state is very small and SiO₂ nano-crystalline is reticular structure, the cold forming results in particle increasing. The average sizes are obtained by the images analyzed on particle size. Then the final results are acquired by averaging the mean size of all images of the same sample, shown in Table 1:

Table 1: Average particle size of SiO₂ samples in different states (AFM test)

| Temperature(°C)/Time(h) | Original powder | 200°C/2h | 900°C/2h |
|-------------------------|-----------------|----------|----------|
| Average size (nm) | 110.87 | 96.63 | 184.70 |

Conclusions

- (1) AFM is a very effective tool to analyze the particle morphology and estimate particle size from the above experimental analysis.
- (2) The experiment results show that the particle size of SiO₂ increased as the sintering temperature increased.
- (3) In general, the above experiment results agree well with the physical mechanism of particle size growth.

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