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Si Nano-Particle Characterization by Atomic Force Microscopy

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Abstract- Nano-particles have been playing an active role in material science and technology for decades, however, there is a lack of efficient and direct method to characterize nano-particle sizes. In this paper, Si nano-particles were prepared by annealing a very thin amorphous Si layer (11nm in thickness) and atomic force microscopy (AFM) was used for investigation. In order to compare the AFM results with the values obtained by other methods, a standardized image processing flow was established to extract particle size and other information, which was applied to all the image results involved in this study. As expected, results extracted from AFM images are larger than those from e-beam imaging techniques due to the AFM tip/probe induced topography convolutions. In order to compensate the convolution, a simplified geometry model was created and applied to the current study. The AFM results after deconvolution are satisfying and this study also proved that AFM analysis with tip/probe deconvolution is a very suitable technique in characterizing Nano-particles with high efficiency and accuracy.

Keywords- *Si nano particle, AFM, deconvolution*

I. INTRODUCTION

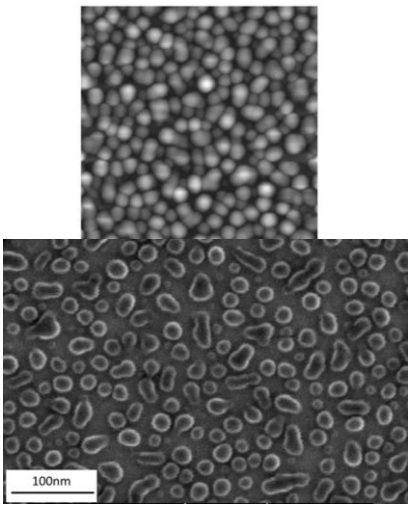
Nano-particles are used in many fields such as bioscience, materials science and semiconductor memory devices for decades [1-7]. Generally, particle size and its distribution are the major characteristics to evaluate the Nano-particle's quality. Traditionally, size characterization of sub-micron particles requires e-beam analysis technologies such as scanning electron microscope (SEM) and transmission electron microscope (TEM). Although results from these techniques are accurate, the sample preparation is time consuming (especially for TEM) and the inspected volume/area is limited. In this study, we focus on atomic force microscopy (AFM) analysis on Si nano-particles and compare the results to those obtained from SEM and TEM. The aim of this study is to establish a reliable and efficient nano-particle characterization method. The AFM tip convolution and its effect on the results are also discussed.

II. EXPERIMENTAL

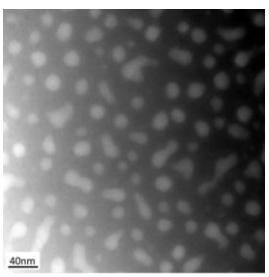
Si nanocrystal samples were prepared by annealing a layer (11nm) of amorphous Si on top of a Si wafer substrate, which has a thin oxide layer (6nm) at the wafer surface. The characterization experiments were performed on a Bruker Dimension V AFM, an FEI Sirion SEM and an FEI Titan TEM, respectively. The tapping mode was used for AFM, with a sharp etched Si probe tip (TESPA-V2 probe from Bruker [8]). For SEM and TEM analysis, the samples were taken at the same AFM inspected area as exactly as possible.

III. RESULTS AND DISCUSSION

Fig.s 1(a) to 1(c) show the Si nanoparticle images obtained from AFM, SEM and TEM, respectively.



(b)



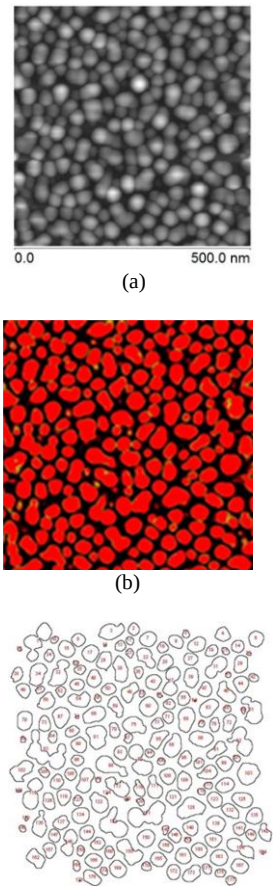
(c)

Figure 1. Si nanoparticle images obtained by (a) AFM, (b) SEM and (c) TEM.

It can be seen from Fig. 1 that the Si nano-particles are much closer to each other in Fig. 1(a) than in Fig.s 1(b) and 1(c).

In order to extract particle size information, a standardized image processing and analysis method was established based on ImageJ software [9].

Fig. 2 depicts how the images are processed to extract the particle size and other information in the present study by using AFM images as an example. The raw images obtained from AFM/SEM/TEM were firstly adjusted to show a proper brightness/contrast level, followed by applying a suitable threshold grey value to color the area with nano-particles and finally the software's particle analysis function was used to extract the information needed.



(a) (b) (c)

Figure 2. Image processing flow used in this study: particle analysis by ImageJ freeware was performed on the raw image (a) and the identified particles were shown in color-filled image (b) and edge image (c).

Table I summarizes the data extracted from AFM, SEM and TEM images. The particle density values from all three methods tally with each other while AFM shows the highest values in particle size (diameter $2r_{AFM}$).

TABLE I. PARTICLE DIAMETER EXTRACTED FROM AFM/SEM/TEM IMAGES

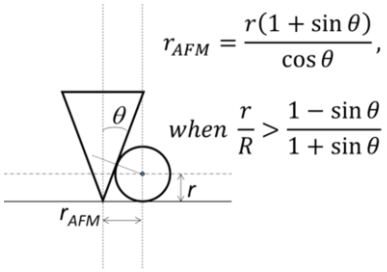
Type of Analysis	Diameter $2r_{AFM}$ (nm)	Density (10^{10}/cm^3)
AFM	27.28	9.52
SEM	20.81	9.11
TEM	19.92	9.05

The AFM tip convolution will make the measured particle diameter ($2r_{AFM}$) appear to be larger than the actual size. Fig. 3 shows how this tip convolution will affect the measurements, with relative to the ratio of actual particle radius, r, and tip's geometry (radius R and tip end angle 2θ). To simplify the calculations, a triangle with a circular top end were used to construct the tip end, which can be clearly seen in Fig. 3(b).

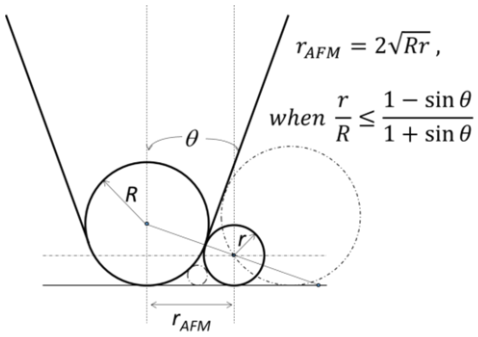
Fig.s 3(a) and 3(b) are used for two different occasions: tip size (R) is much smaller than particle size (r), which is shown in Fig. 3(a), and tip size R is comparable to particle size r, as in Fig. 3(b). A geometry analysis will give the following two equations:

$$r_{AFM} = \frac{r(1+\sin\theta)}{\cos\theta}, when\ \frac{r}{R} > \frac{1-\sin\theta}{1+\sin\theta} \quad (1)$$

$$r_{AFM} = 2\sqrt{Rr}, when\ \frac{r}{R} \leq \frac{1-\sin\theta}{1+\sin\theta} \quad (2)$$



(a)



(b)

Figure 3. Relationship of AFM tip's geometry θ, actual particle size r and the measured partice size r_{AFM}.

TABLE II. PARTICLE DIAMETER OBTAINED FROM AFM (BEFORE AND AFTER TIP CONVOLUTION CORRECTION), SEM AND TEM ANALYSES ON VARIOUS SAMPLES

Sample	AFM (nm)	AFM after correction (nm)	SEM (nm)	TEM (nm)
A	27.28	19.10	20.81	19.92
B	20.81	13.46	13.93	N/A
C	19.92	14.15	13.84	N/A

AFM probe tips usually have sharp ends, thus, 2θ should be in the range of 0~90°, which means θ should be 0°~45°. Therefore, (2) is always less than 1, which means (2) will only be valid when the actual particle size, r, is smaller than tip radius R.

Based on the analysis above, a correction was performed with tip radius R equals 8nm and θ equals 20° [8]. As the measured particle size $2r_{AFM}$ is 27.28nm (see Table 1), (1) will be used for size correction (deconvolution), which leads to:

$$r_{AFM} = \frac{r(1+\sin\theta)}{\cos\theta} = 1.428r \quad (3)$$

Applying (3) to Table I, we can have a corrected AFM particle diameter values after tip deconvolution, which are listed in Table II.

Equation (3) was applied to two other similar AFM measurements on Si nanoparticle samples and the results are also showed in Table II (samples B and C). It can be seen that the corrected AFM results show a very good agreement with SEM/TEM results. As AFM sample is the easiest to prepare compared with SEM and TEM and the latter two have limited inspection volume when imaging at very high magnification, AFM will be the most suitable method due to its efficiency and convenience in sample preparation.

The deconvolution discussed in this study is based on simplified tip geometry and the sample has a monolayer of particles, which are very close to the situation of the samples and tip used in the current study. Extended explorations and discussions will be needed if the sample is multi-layered and/or AFM probe tip has a different geometry.

The particle volume fraction can also affect the deconvolution method used in the current study. There should be enough distance between particles to allow the AFM tip to move around. More study will be needed if the volume fraction is too high.

As the AFM sample preparation is simple (compared with those for SEM and TEM) and the view of field under AFM scope can be easily adjusted and move around in macro scale, it can be concluded that AFM with deconvolution is highly suitable for Nano-particle characterization.

IV. CONCLUSIONS

In conclusion, an improved AFM nanoparticle characterization method has been established by our current study, which including image process flow and results correction, after taking the tip convolution effect into account. Our study shows that the AFM measured results can be corrected to a level matching those obtained with e-beam methods with a proper tip-particle deconvolution model.

REFERENCES

[1] A. Balamurugan, K. C. Ho, and S. M. Chen, "One-pot synthesis of highly stable silver nanoparticles-conducting polymer nanocomposite and its catalytic application," Synth. Met. vol. 159, pp. 2544-2549, 2009.

[2] I. Brigger, C. Dubernet, and P. Couvreur, "Nanoparticles in cancer therapy and diagnosis," Adv. Drug. Deliv. Rev. vol. 54 pp. 631-651, 2002.

[3] T. Pradeep and Anshup, "Noble metal nanoparticles for water purification: A critical review," Thin Solid Films. vol. 517, pp. 6441-6478, 2009.

[4] L. Lee, C. Zeng, X. Cao, X. Han, J. Shen, and G. Xu, "Polymer nanocomposite foams," Compos. Sci. Technol. vol. 65, pp. 2344-2363, 2005.

[5] W. Liu, "Nanoparticles and their biological and environmental applications," J. Biosci. Bioeng. vol. 102, pp. 1-7, 2006.

[6] C. J. Murphy, "Nanocubes and nanoboxes," Science, vol. 298, pp. 2139-2141, 2002.

[7] C. Bonafosa, M. Carradaa, G. Benassayaga, S. Schamm-Chardona, J. Groenena, V. Paillarda, B. Pecassoua, A. Claveriea, P. Dimitrakisb, E. Kapetanakisb, V. Ioannou-Sougleridisb, P. Normandb, B. Sahuc, and A. Slaouic, "Si and Ge nanocrystals for future memory devices," Mater. Sci. in Semicon. Proc. vol. 15, pp. 615-626, 2012.

[8] Website: http://www.brukerafmprobes.com/p-3844-tespa-v2.aspx.

[9] Website: http://imagej.nih.gov/ij/.