Nanoparticle size and shape evaluation using the TSOM optical microscopy method

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ABSTRACT

We present a novel optical through-focus scanning optical microscopy (TSOM) method that produces nanoscale dimensional measurement sensitivity using a conventional optical microscope. The TSOM method uses optical information from multiple focal planes for dimensional analysis. The TSOM method can be used for nanoscale dimensional and defect analysis for a wide variety of target geometries and sizes. We present here an application of the method to analyze the size and shape of nanoparticles. We present the analysis based on simulations and also provide experimental data.

Keywords: TSOM, optical microscope, through-focus, nanoparticles, nanometrology

1. INTRODUCTION

There is great interest in using nanoparticles for biological and medical applications such as fluorescent biological labels, drug and gene delivery, bio detection of pathogens, detection of proteins, and probing of DNA structure [1]. It is important to determine size of nanoparticles for effective use in many of these applications. Several metrology tools are available for dimensional characterization of nanoparticles. A comprehensive review of techniques and tools for dimensional characterization is provided in reference 2. Each method has its own advantages and disadvantages. In spite of the availability of several instruments for dimensional analysis, there is a need for higher accuracy tools or methods that are also economical. We present here one such novel method developed at the U.S. National Institute of Standards and Technology.

It is a misconception that optical microscopes are not well suited for dimensional measurements of features that are smaller than half the wavelength of illumination (200 nm sized features in the visible region) due to diffraction [3]. It is true that diffraction dominated images make meaningful analysis of the targets difficult. However, we have circumvented this limitation by (i) considering the image as a “signal” that represents the target, (ii) using a set of through-focus images instead of one “best focus” image, and (iii) making use of highly developed optical models [4].

In conventional optical microscopy, it is usually deemed necessary to acquire images at the "best focus" position for a meaningful analysis, based on the belief that the most faithful representation of the target is rendered only at the best focus position. However, the out-of-focus images do contain additional useful information regarding the target. This information may be obtained using an appropriate data acquisition and analysis method. Based on this, and on the observation of a distinct signature for different parametric variations, we introduced a novel method for nano-scale dimensional analysis with nanometer sensitivity for three-dimensional, nano-sized targets using a conventional bright-field optical microscope [5-11]. The method is currently called the TSOM (pronounced as 'tee-som') imaging method. Here, we present one of the applications of the TSOM method for nanoparticle dimensional analysis.

2. THE TSOM IMAGE CONSTRUCTION

The first step is to construct a TSOM image from the images obtained using a conventional optical microscope. In Fig. 1 we demonstrate the method using a nanoparticle as a target. Optical images are acquired as the target is scanned at a predetermined step size through the focus of the microscope (along the Z-axis) as shown in Fig. 1(a). Each scan position results in a slightly different 2D intensity image. From these images, extracted optical intensity profiles passing through the location of interest on the target (through the center of the nanoparticle, for example) can be assembled and conveniently plotted as a 2D image resulting in a TSOM image as shown in Fig. 1(b), where the X (horizontal), Y (vertical) and Z (color scale) axes represent the spatial position across the target, the focus position, and the optical intensity, respectively.

NSTI-Nanotech 2010, www.nsti.org, ISBN 978-1-4398-3415-2 Vol. 3, 2010

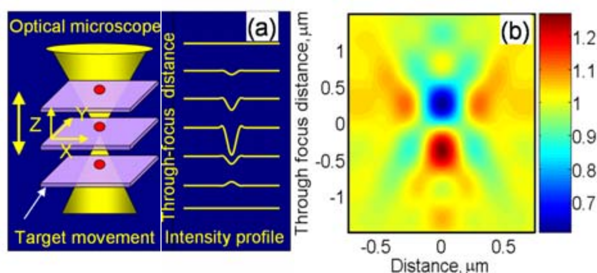

Figure 1. The method to construct TSOM images. (a) Schematic showing the image acquisition process for through-focus scanning of a gold particle. Schematic of the cross sectional image intensity profiles passing through the center of the gold particle at the various scan positions are shown on the right side. (b) The simulated two-dimensional TSOM image (X-Z plane) passing through the center of the 60 nm gold particle on quartz substrate. Wavelength = 365 nm, Illumination NA = 0.3, Imaging NA = 0.95.

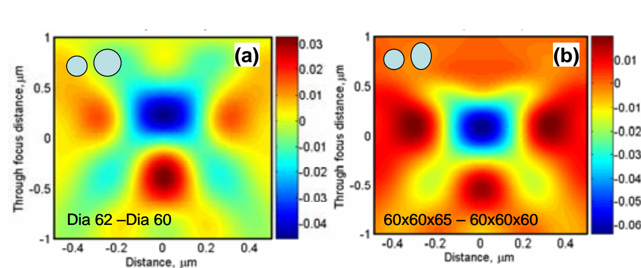

Figure 2. Simulated differential TSOM images for (a) the size difference of 2.0 nm in diameter (62.0 nm and 60.0 nm), and (b) the shape difference as a result of 5.0 nm elongation in the height. Illumination NA = 0.3, Collection NA = 0.95, Illumination wavelength = 365 nm, Gold particle on quartz substrate.

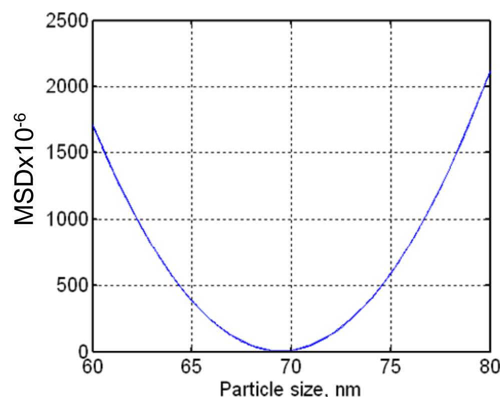

Figure 3. Determination of unknown particle diameter using the best match method from a library of simulations. Library range: 60 nm to 80 nm. Best match diameter: 69.45 nm. Illumination NA = 0.3, Collection NA = 0.95, Illumination wavelength = 365 nm, Gold particle on quartz substrate.

3. NANOPARTICLE DIMENSIONAL ANALYSYS USING THE TSOM METHOD

At present, we are exploring two ways of using the TSOM method for nanoparticle analysis. They are (i) to identify which dimensional parameter is changing (e.g. a change in diameter versus shape) and to estimate the magnitude of these changes, and (ii) to determine the absolute dimensions of nanoparticles.

3.1 Nanoparticle Shape determination

The first application is best examined by looking at the difference between the TSOM images produced by two different nanoparticles. As shown in Fig. 2, a difference in size (diameter) and shape produces distinctively different differential TSOM images. Larger differences produce similar differential images but with higher differential signal. As can be seen from the Fig. 2, this simple method using the TSOM images, enables determination of size (diameter) or shape analysis using the difference method with relatively less effort .

3.2 Nanoparticle Size determination

The second type of application requires accurate optical simulations with satisfactory experiment-to-simulation agreement. With the assumption that the TSOM images are unique [6] for a given target under a given experimental condition, we compare the experimental TSOM image of a nanoparticle with that of a library of simulated nanoparticle TSOM images. The best match (minima) provides the dimension (diameter) of the unknown particle.

A procedure for particle size analysis using the library-matching method is explained here using simulations. A typical TSOM image of a gold nanoparticle (60 nm) is shown in Fig. 1(b). The TSOM images for different sized nanoparticles change with size. We make use of this difference to determine the nanoparticle size. The first step for this is the simulation of a library of TSOM images for various sizes within the expected size range. For this work a finite difference time domain optical simulation program was used [4]. In the second step, we acquire the experimental TSOM images of the nanoparticle needing size determination. The third step is to compare the TSOM image of the nanoparticle of unknown size with that of the library to determine the mean square difference (MSD) [8] values of the differential images. A plot of the MSD values thus obtained is shown in Fig. 3. If the size of the unknown particle is within the size range of the library, in principle, the plot should show a well defined minima. The size corresponding to the minima indicates the size of the unknown particle. In this example, an "unknown" nanoparticle size of 69.5 nm produced the best-mach size of 69.45 nm.

In practice, this type of procedure using simulations may have limitations as there can be differences between simulated and experimentally obtained TSOM images that lead to inaccuracies in the fitting process. An alternative method is to create an empirical library from experimental TSOM images of particles with predetermined sizes and use this library to determine the size of an unknown particle. This nearly eliminates the issues arising due to mismatch between the simulated and the experimental TSOM images, although this introduces essential reference metrology measurements. In the following paragraphs, we present nanoparticle size determination using this later procedure.

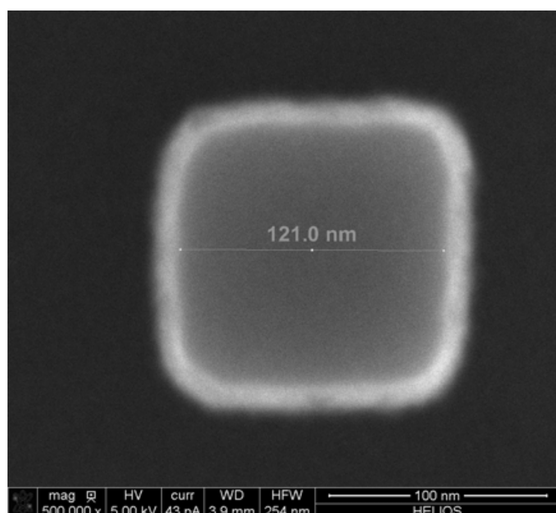

Figure 4. SEM image of a square nanodot showing its measured dimension.

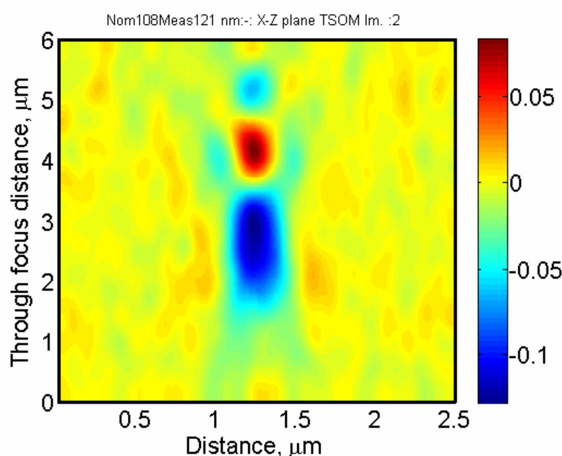

Figure 5. Experimental intensity normalized TSOM image of the nanodot shown in Fig. 4. Wavelength = 546 nm, TE Polarization, Illumination NA = 0.1, Collection NA = 0.8, Si nanodot on Si substrate.

For this purpose, approximately square Si nanodots on Si substrate were fabricated with nominal sizes ranging from 40 nm to 150 nm, with a fixed height of about 70 nm. The SEM lateral dimension reference measurements were always larger than the nominal designed dimensions. Even though the nanodots are not exactly the same as nanoparticles, the measurement procedure remains the same. Lateral dimensions of the nanodots were measured using an SEM, that has a nominal measurement uncertainty of about 5%. An SEM image of a 121 nm nanodot is shown in Fig. 4. Following the SEM measurements, the TSOM images were acquired for the selected nanodots using polarized illumination at a wavelength of 546 nm. A typical intensity normalized TSOM image for TE polarization is shown in Fig. 5. Using the experimental TSOM images thus created, integrated mean square intensities (MSI) for the selected nanodots were evaluated and plotted as a function of the nanodot size as shown in Fig. 6. Under the current experimental conditions, the curve nominally follows a linear trend. This is treated as the library or the calibration curve for dimensional analysis of nanodots of unknown size.

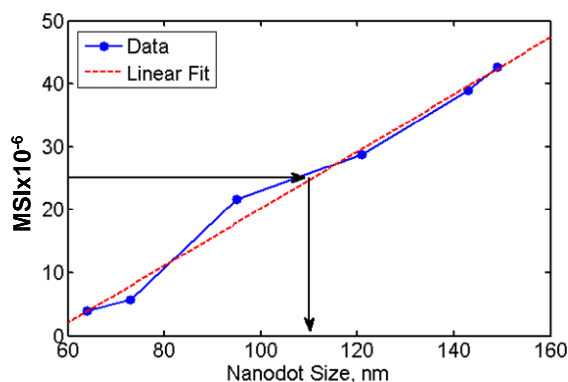

Figure 6. Experimental mean square intensities of the normalized TSOM images of the selected square nanodots showing a linear trend with size. Arrow marks indicate experimental size determination of nanodot of unknown size using the library.

Using the calibration curve an attempt was made to measure the "unknown" size of a nanodot, the size of which was measured to be 103 nm using the SEM. The integrated mean square intensity of the TSOM image of the "unknown" nanodot was then compared with the calibration curve, producing a measured size of 108 nm. Considering this as an initial attempt, the measured size appears to be satisfactory. However, further study is required to refine and optimize the measurement process.

Advantages of using the TSOM method are: relatively inexpensive conventional optical microscopes can be used for nanoscale dimensional metrology, provides up to one nanometer dimensional sensitivity, distinguishes between

different types of dimensional variations and different magnitudes, a wide variety of target geometries can be used, and the requirement for defining the "Best Focus" is eliminated [5-11].

4. SUMMARY

We have presented a method to create a TSOM image using conventional optical microscopes. Using both simulations and experiments, we demonstrated the determination of the size and shape of nanoparticles by employing the TSOM method.

Acknowledgements: Financial support through an EXPLORATORY project from the Manufacturing Engineering Laboratory of NIST is gratefully acknowledged. We thank the Center for Nanoscale Science and Technology Nanofab of NIST for help with the sample preparation.

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