A Case Study in Sizing Nanoparticles

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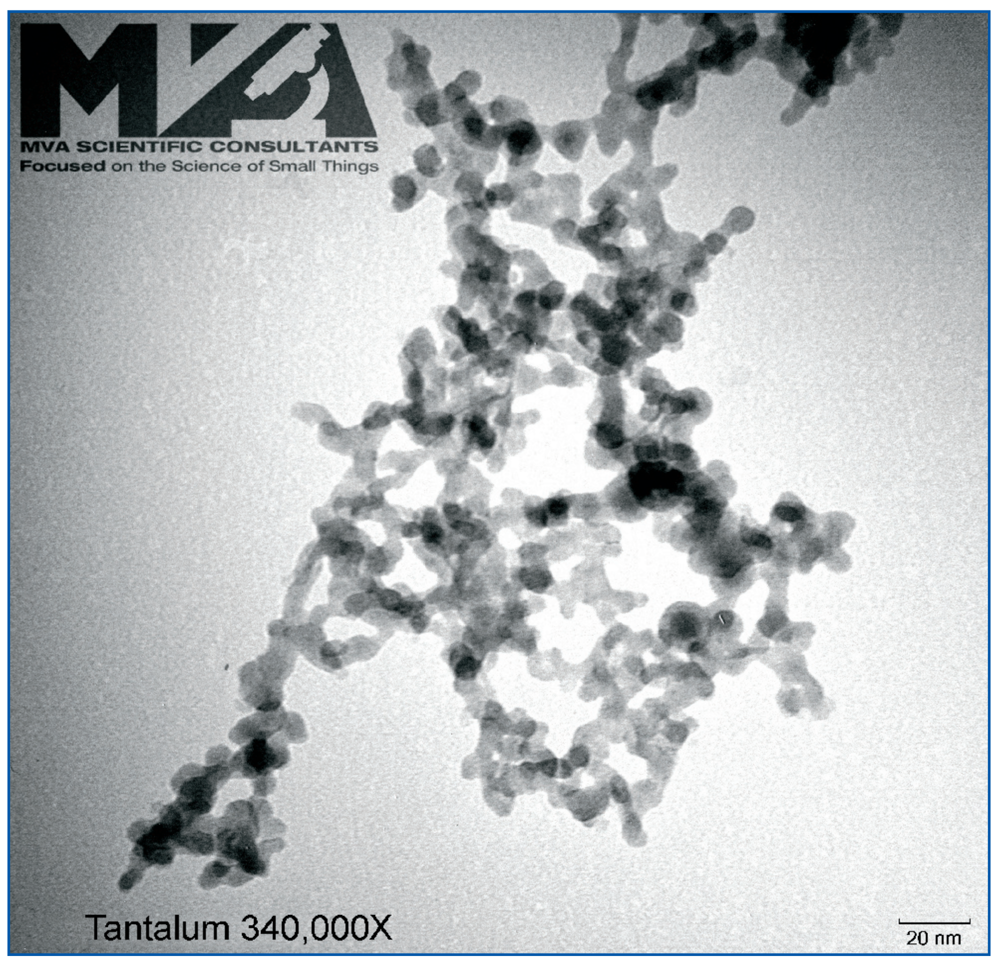
Background

Interest in nanoparticles has risen consistently and rapidly in recent years, but one problem has remained; what is the "best" way to determine the size of these materials? In other words, what is the particle size of my nano materials? In this study, we wanted to investigate what viable options were available to measure these types of materials. There are several techniques commercially available, but we choose to look only at dynamic light scattering (DLS), Microscopy (SEM or TEM), and finally a calculation of the average particle size from a BET surface area measurement.

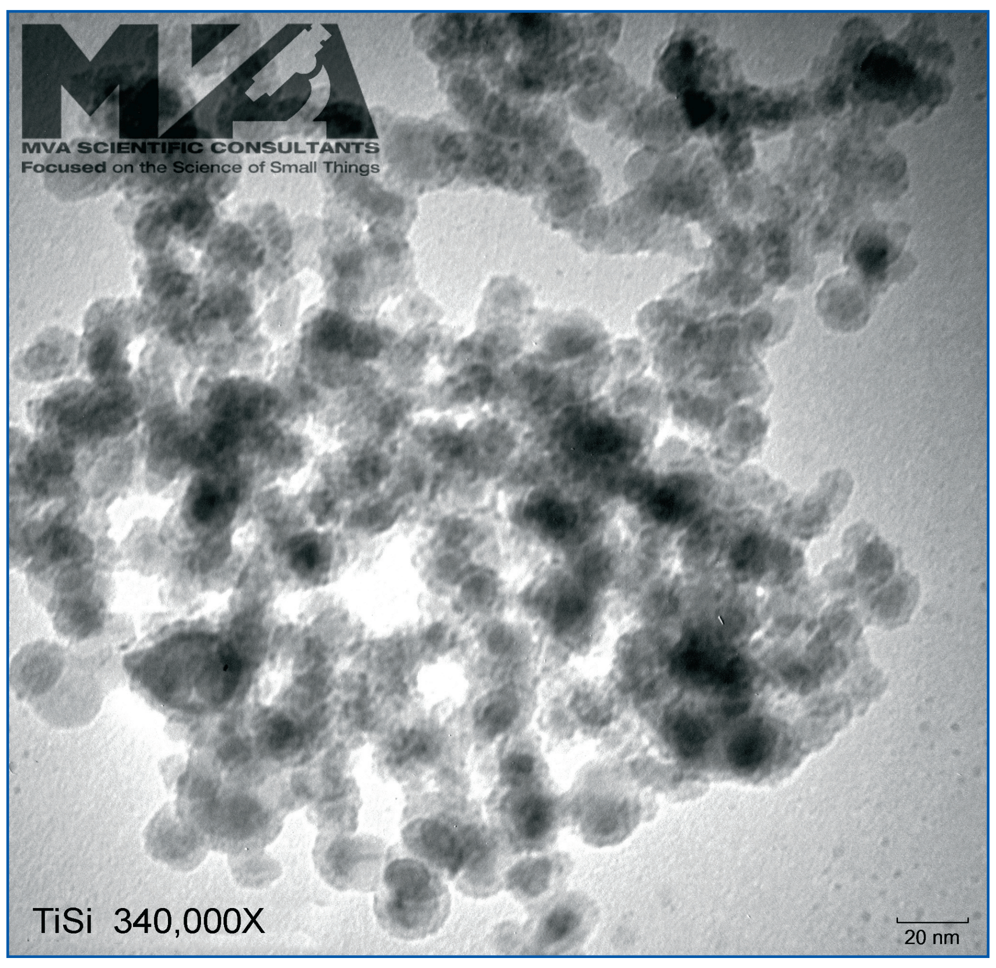
Case Study

Samples of nanoparticles were chosen from a variety of industries and were simply identified as Nickel, Carbon, Titanium di-silicide ($TiSi_2$), and Tantalum. Samples were analyzed using a Micromeritics TriStar 3000 gas adsorption instrument which calculated a BET surface area. From this, an average particle diameter was calculated assuming nonporous spherical particles and a theoretical density of the individual materials. Without going through the entire derivation, the equation for calculating the average particle diameter in nanometers is 6000/(BET surface area in m^2/g) x (density in g/cm^3). The BET surface area and the average particle size results are found in the table below.

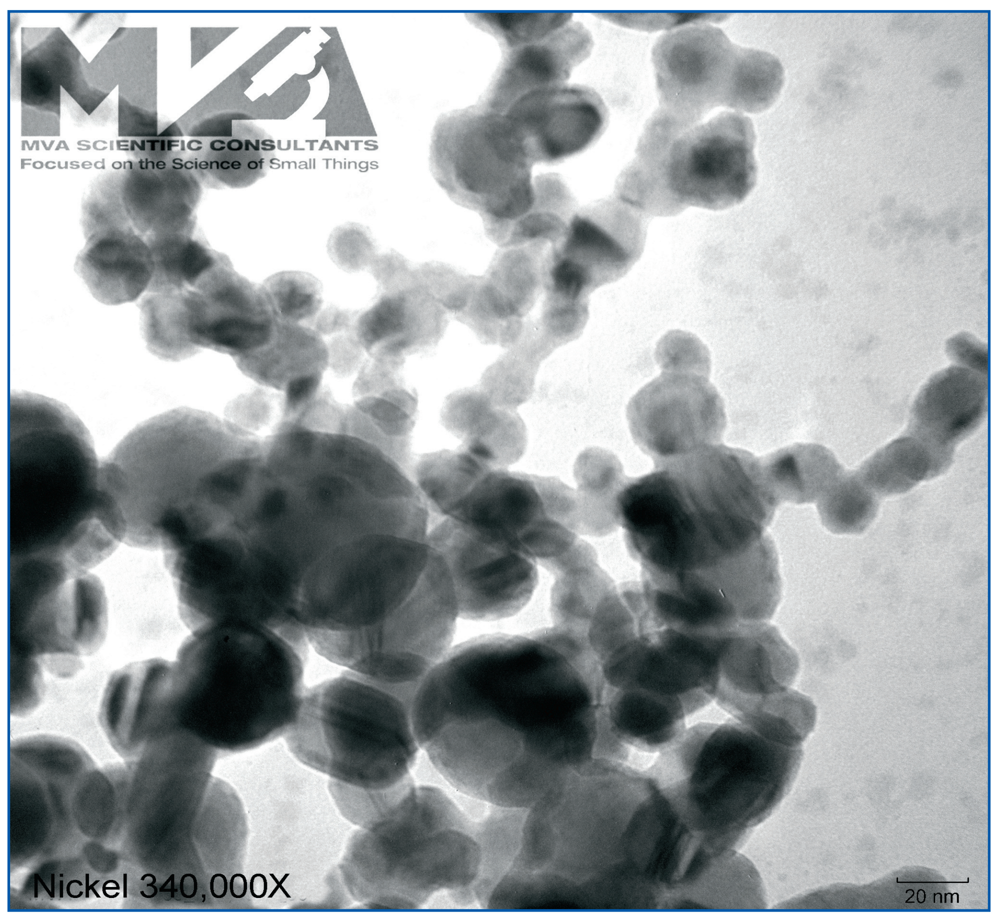
These samples were submitted to MVA Scientific Consultants for particle size analysis by TEM. Samples were also sent to various manufacturers of DLS instruments. Results varied with the energy applied in an attempt to disperse these samples. The results shown in the table below were obtained after sonicating for at least two hours prior to analysis.



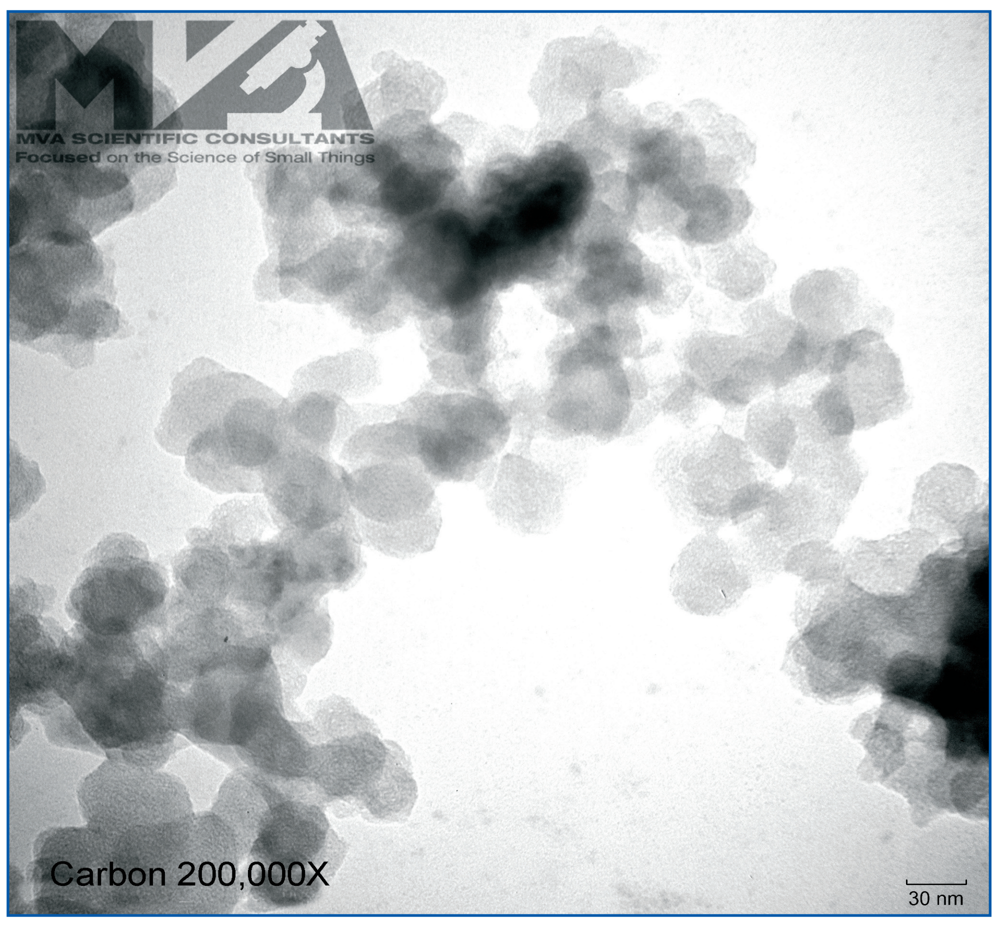
Tantalum



$TiSi_2$



Nickel



Carbon

The images above are the results from the analysis using the transmission electron microscopy technique (TEM).

Summary

If the nanoparticles exist in a dry powder form and have formed aggregates such as the materials used in this study, then a reasonable primary particle size may be obtained by using either TEM or by calculating an average particle diameter using a measured BET surface area. It is important to point out that the difference observed between the calculated value from BET surface area and the TEM results is due to a small amount of surface being lost due to the primary particles forming aggregates.

If the aggregate particle size is preferred, a combination of microscopy or light scattering method, either dynamic or static light scattering, may be appropriate. So much energy is required to disperse the aggregate nanoparticle materials tested, that a primary particle size was not obtainable using DLS technique.

If your particles are suspended in a stable solution and have not formed aggregates, either TEM or dynamic light scattering method would provide good primary particle size information.

The table below summarizes the results obtained from each technique.

Sample Description	BET S. A. (m^2/g) (Micromeritics Analytical Services)	Calculated Avg. Particle Size from BET	Mean Particle Size Diameter by Volume Measured using TEM (MVA Scientific Consultants)	Mean Particle Size by DLS
Tantalum	89.73	8 nm	6.8 nm	316 nm
$TiSi_2$	97.25	19 nm	12.6 nm	157 nm
Nickel	26.3	35 nm	23.6 nm	1.3 micrometers
Carbon	62.05	45 nm	31 nm	Not Available

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