Nanoparticle Shape Effects on Sintering Temperatures

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Introduction:

The objective for this project was to determine what effects nanoparticle shapes have on the sintering temperatures of silver. The metal in question is silver. Particle shapes examined include: cubes, plates, and rods. All particles involved were created using a polyol synthesis technique.(1)

Broader Impact:

In a broader focus, this research could potentially affect the marketability of precious metal clays. By isolating and determining which particle shape has the biggest effect on the sintering temperature, a synthesis can be refined to produce larger quantities of this shape and minimizing the size of particles. Lowering the sintering temperatures makes precious metal clays more marketable because an expensive furnace would not be needed to fire the clay products. A person could potentially fire the clay products in a conventional oven or a normal household microwave.(2)

Experiment:

A polyol process was used to synthesize the silver nanoparticles. The experimental setup is shown in Figure 1. Each synthesis was conducted in a solution of ethylene glycol with a small amount of dilluted hydrochloric acid. Hydrochloric acid was used to prevent the formation of twinned seeds. Twinned seeds are a defect that affect the growth and shape of the nanoparticles. They form when two seeds morph together. They grow into irregular shapes and growth rate is unpredictable. Poly(vinyl pyrrolindone) (PVP) was used as a surfactant in this process. The PVP bonds to the facets of the nanoparticles as they grow. This depends on the surface energy of the particles and the temperature at which the synthesis is conducted. Different synthesis temperatures cause the PVP to attach at different surface energies.(3) Syntheses were conducted at temperatures of 125, 130, 140, and 155 ^{o}C.

The nanoparticles were filtered from the solution of ethylene glycol and PVP using a vacuum pump and a nano-filter as shown in Figure 2. It is extremely important that all of the ethylene glycol and PVP is rinsed from the surface of the silver particles. If improperly rinsed the particles will continue to react.(4) Figure 3 shows an example of particles after improper rinsing. The results of improper rinsing can be seen in Figure 4 where the particle were originally cubes but have reacted to form shapes with less surface energy.(3)

A sample of each isolated shape was tested on a differential scanning calorimeter(DSC). Each test involved ramping the temperature at a rate of 10 C per minute from 40 C to 400 C two times and the samples were cooled at 10 C per minute twice and 20 C per minute once. The data produced from these tests were used to compare sintering temperatures of the different samples.

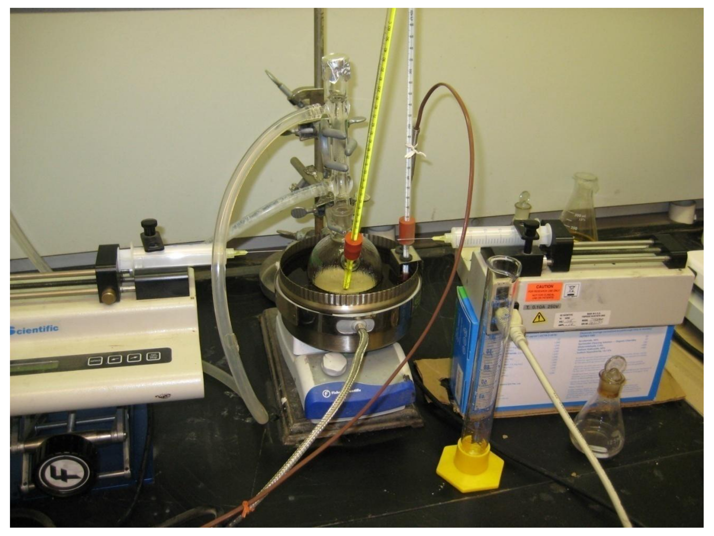

Figure 1. Image shows setup for synthesis.

Figure 2. Image shows filtering setup.

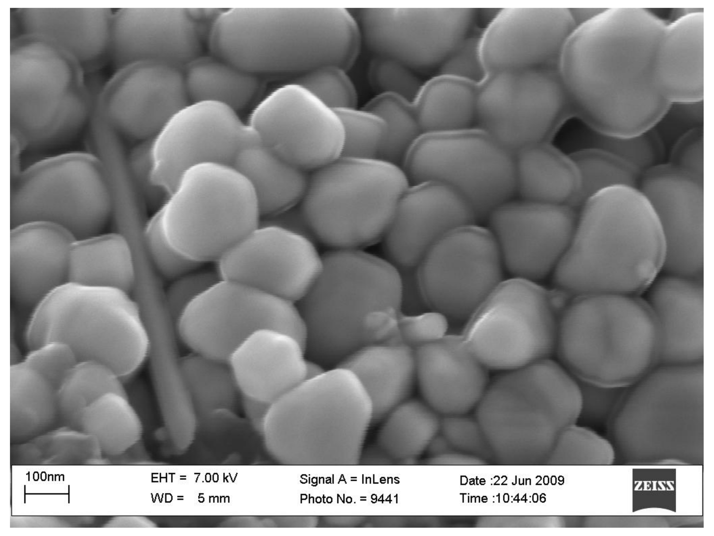

Figure 3. SEM image of silver particles with thin ethylene glycol and PVP layer.

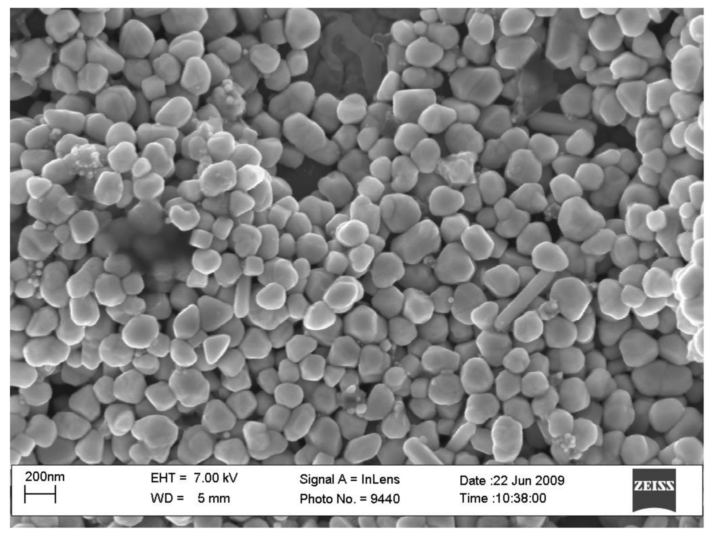

Figure 4. SEM image of cubes that have continued to react due to improper rinsing.

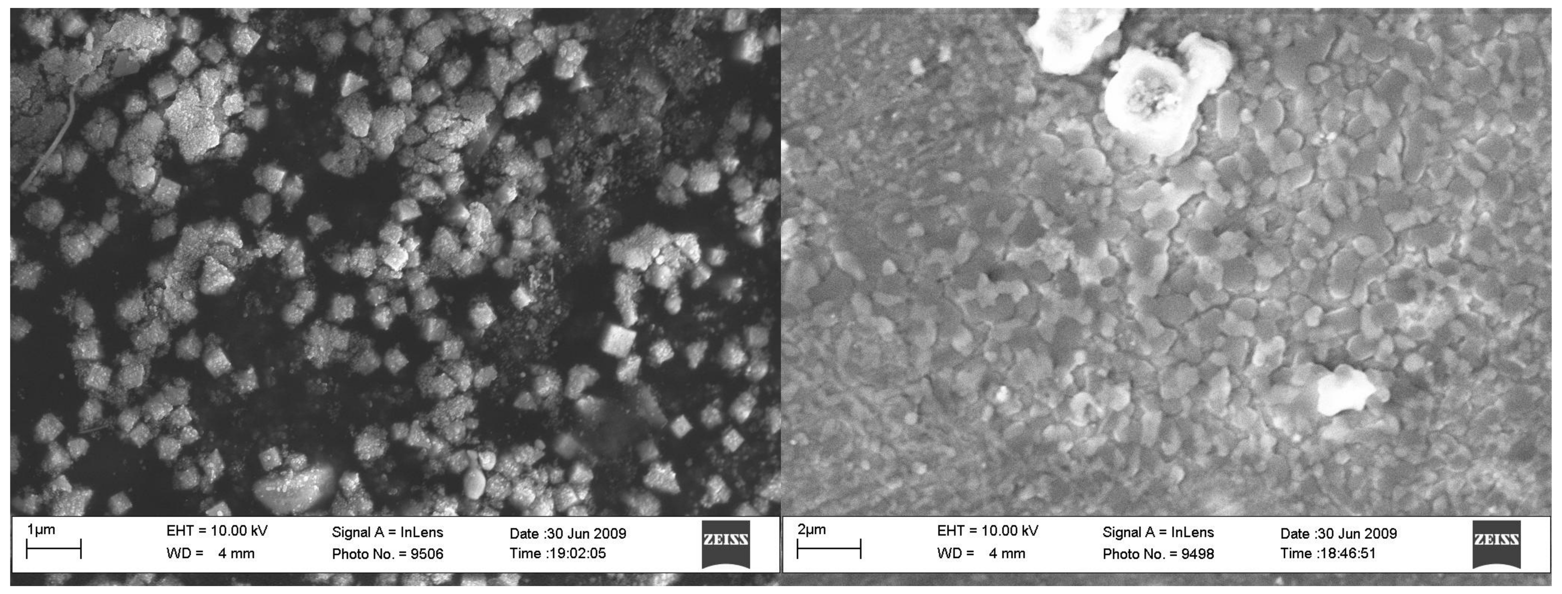

Figure 5. Left: SEM image of cubes imbedded in nano-filter. Right: SEM image of plates removed from the surface of the nano-filter.

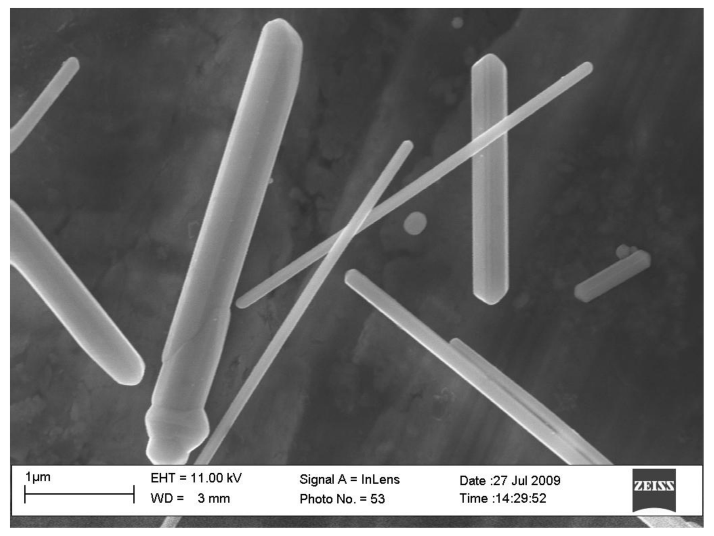

Figure 6. SEM image of rods produced at 155 degrees.

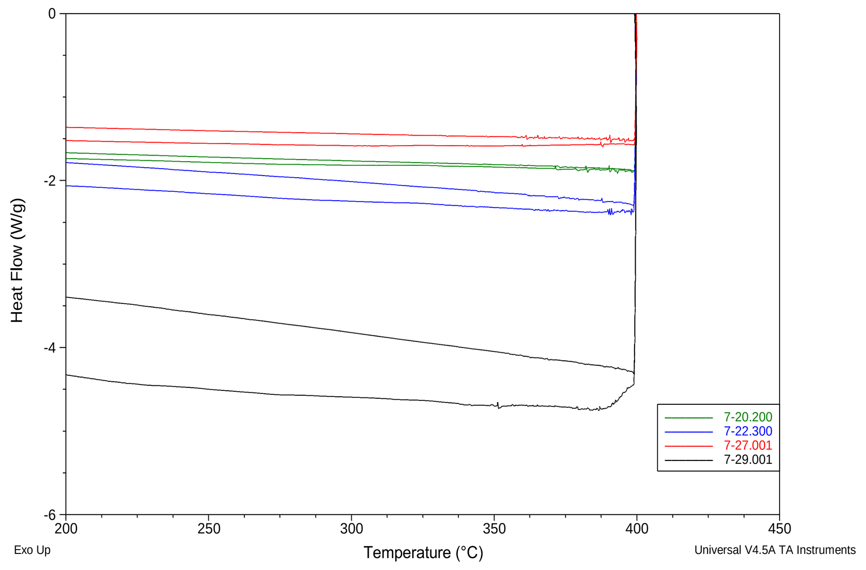

Figure 7. DSC graph from four nanorod tests.

Results:

125 ^{o}C Syntheses

- Particles were extremely jagged with a small amount produced
- There was no discernable size range for the particles

130 ^{o}C Syntheses

- Conducted at two different batch sizes
- Smaller batch produced varying results. Figure 5 shows an image of these particles. Plates and cubes were formed and then separated by the filtering process.
- Results from the small batch were never able to be duplicated

140 ^{o}C Syntheses

- Conducted at two different batch sizes
- Smaller batches produced product composed mainly of cubes, but none were formed with the distinct edges like the cubes shown in Figure 5.
- Larger batch sizes produced a very pure sample of rods.
- The main reason for the formation of rods is theorized to be a change in reaction kinetics from the smaller batch.

155 ^{o}C Syntheses

- Only conducted in large batches
- These experiments produced rods. Figure 6 shows an image.
- Monodispersion was shown with a diameter range of 75 – 125 nanometers

DSC Analysis

- Graphs from DSC analysis are shown in Figure 7.
- Tests seem to show little difference based on particle shape.
- Lines 7-20, 7-22, and 7-27 all had varying amounts of random undesirable particle shape content with 7-27 being almost pure rods and 7-20 being highly contaminated with the random particles.
- The peaks shown are assumed to be from PVP and ethylene glycol removal. No peaks are large enough to represent sintering.

References:

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Acknowledgements:

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