

Facile, fast, and inexpensive synthesis of monodisperse amorphous Nickel-Phosphide nanoparticles of predefined size

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Initial experiments

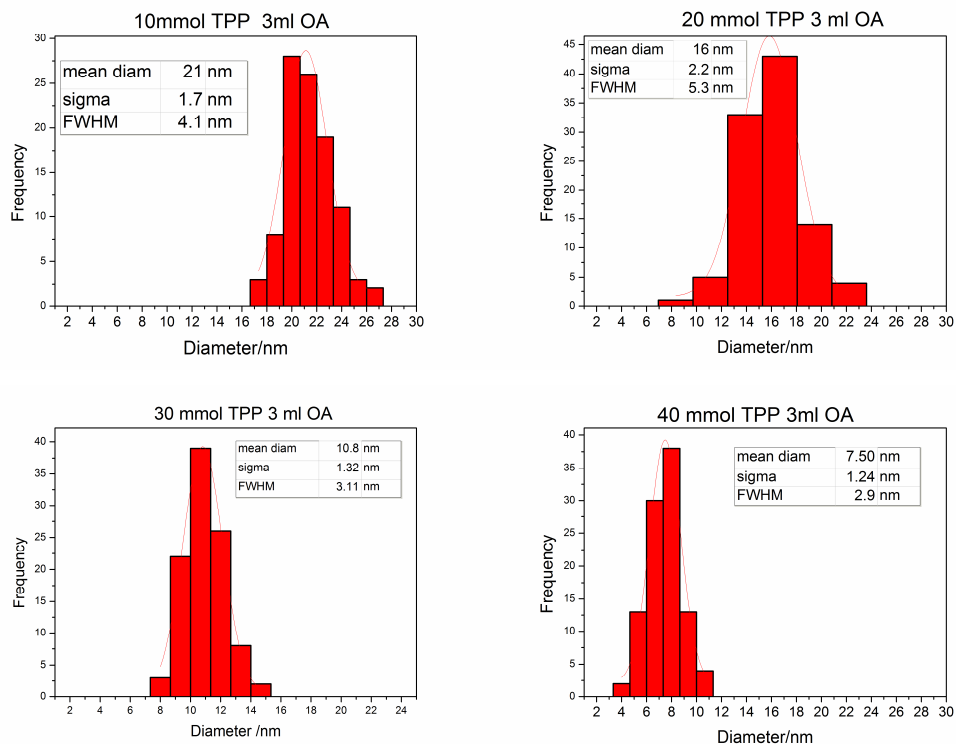
Initial experiments were performed using combinations of OA and oleic acid in order to produce nanoparticles. However, only macroscopic aggregates could be obtained for experiments where 10 ml (30 mmol) OA and 1 mmol Ni(acac)₂ were mixed with varying [0.95 ml (3mmol) - 9.5 ml (30 mmol)] oleic acid volumes. Hence, it is likely that neither oleic acid nor OA bind tightly enough to the surfaces of the Ni particles necessary to prevent agglomeration. Furthermore, the combination of Ni(acac)₂ and oleic acid appears to lead to unfavourable violent splashing reactions during thermal decomposition. Such strong reactions could successfully be avoided by using a combination of OA, TPP and Ni(acac)₂.

Characterisation

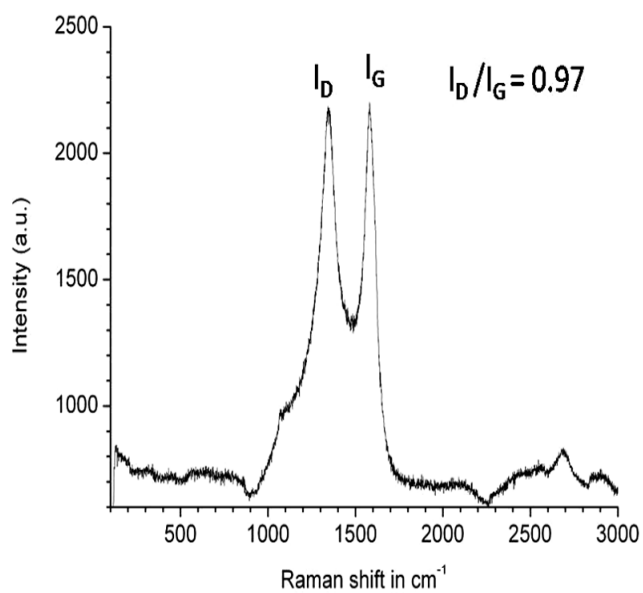
Particle sizes were analysed by counting 100 particles from transmission electron microscope (TEM) images taken with a Jeol4000HR and a Jeol2010 TEM. For non spherical particles, the longest axis of the particles was measured. The nanoparticles were counted manually without any automated particle counting software in order to avoid computer pattern recognition errors. X-ray diffraction (XRD) was carried out to obtain information about the crystalline state of the particles. Drops of the solvent containing the analyte were applied to a single crystal of silicon. Analyses were carried out at room temperature, using a fully automated Siemens D5000 powder diffractometer employing copper K α radiation ($\lambda=0.15406$ nm) and a secondary monochromator. The samples were continuously spun during data collection and were scanned using a step size of 0.05 2 θ in the range of 5-75 2 θ and a count time of 12 seconds per step. A VG Clam X-ray photoelectron spectrometer was employed in order to investigate the chemical composition of the nanoparticles. In order to remove the organic capping of the particles originating from the TPP, the nanoparticles were exposed to ion-bombardement using 4keV Ar⁺ ions from a Leybold IQE 12/38 rastered ion gun. An initial confirmation of the chemical composition of the particles was obtained from EDX analysis using a Jeol-JSM-6480LV SEM.

Ni nanoparticle catalysed carbon nanotube growth and characterisation

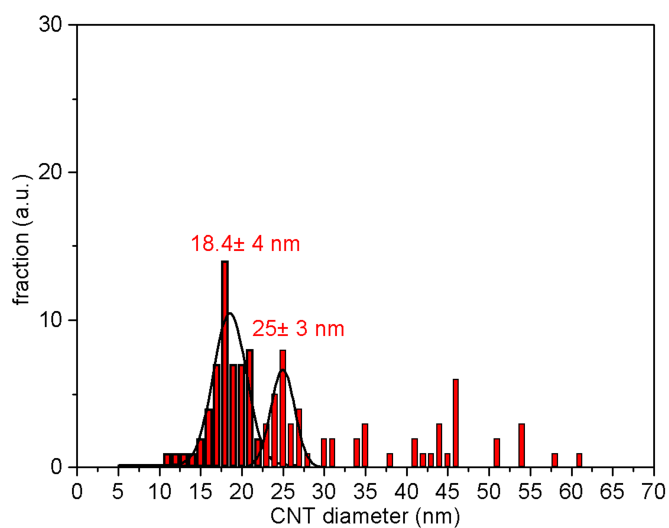
For carbon nanotube growth Ni-based nanoparticles in cyclohexane were deposited directly on the inner walls of a quartz tube (900 mm long, 25 mm inner diameter). Typically 1 mmol of nanoparticles dispersed in 5 ml of cyclohexane or toluene were poured and deposited into a rotating quartz tube. The quartz tube was rotated manually during that process and compressed air was blown through the tube in order to evaporate the solvent. As a result, the quartz tube showed a brownish coating, indicating a film of deposited nanoparticles. The coated quartz tube was placed inside an Elite 1200°C Single Zone vertical tube furnace at the bottom of which was a three neck flask containing a reservoir of cyclohexane as a carbon source. Whilst heating the furnace to 750°C the quartz tube, of which 500 mm was in the hot zone of the furnace, was flushed with 200 sccm Ar. At 750°C 1000 sccm Ar was bubbled through the precursor solution carrying it into the furnace. After 30 min the furnace was cooled down and the grown carbon nanotubes were mechanically removed from the quartz tube walls. TEM images of the as synthesised nanotubes were taken with a Jeol4000HR TEM. SEM micrographs from carbon nanotubes were taken with a JEOL Scanning Microscope 840F. Raman analysis was carried out using a Horiba Labram Aramis Raman spectrometer with a 532 nm green laser.



Ni-P nanoparticle size distributions



Raman spectrum of the raw CNTs material produced using toluene in conjunction with Ni-P nanoparticles.



Size distribution of CNTs produced using 18nm Ni-P nanoparticles. There appears to be good correlation between the nanoparticle diameter (18 nm) and the nanotube diameters (18.4 nm), although also an accumulation of nanotube diameters around 25 nm and a few larger diameters were also found.