**Hot Wire Synthesis of Si Nanoparticles**

Manfred R Scriba\(^1,2\), Chris Arendse\(^1\), Margit Härting\(^2\) and David. T. Britton\(^2\)  

\(^1\) CSIR, Material Sciences and Manufacturing, P O Box 395, Pretoria 0001 South Africa.  
\(^2\) Dept. of Physics, University of Cape Town, Rondebosch 7701 South Africa

**ABSTRACT**

The viability of producing silicon nanoparticles using the HWCVD process is investigated. A system is assembled and particles are produced from silane at pressures between 3 – 48mbar, with hydrogen dilutions of 0-80%, at a total flow rate of 50sccm and with a tungsten filament maintained at 1650ºC. The as-prepared powder varies in colour from yellowish to dark brown and is deposited on all surfaces inside the reaction chamber. The material is a highly porous agglomeration of nanoparticles of primary size in the order of 40nm, with a narrow size distribution. The nanoparticles produced are mostly amorphous, hydrogenated and have a partially oxidised surface.

**1. Introduction**

Nanoparticles are considered as fundamental building blocks of nanotechnology. Likewise, silicon nanoparticles may form the basis for flexible electronics, including solar cells and luminescent materials, printed on paper [1].

Historically these particles have been manufactured by top-down approaches such as milling, laser ablation or etching, and bottom-up synthesis such as colloidal chemistry and gas phase pyrolysis. The chemical processes in the latter are generally equivalent to those in the chemical vapour deposition of compact films. In the case of silicon deposited by hot-wire CVD (HWCVD), powders formed by gas phase reactions and nucleation are often an unwanted by-product [2].

Furthermore, the charge cluster model that suggests nanometre size clusters are formed in the gas phase and become the deposition unit for thin films [3], leads us to believe that thermal catalytic pyrolysis, based on established HWCVD techniques, is a viable process for Si nanoparticle (powder) synthesis. The objective of this research is to produce Si nanoparticles in sufficient quantities while maintaining control of the important properties namely size, size distribution, composition and crystallinity.

**2. Experiment**

Si nanoparticles were synthesised using an in-house HWCVD system, which incorporates a water-cooled carousel that allows 6 depositions without compromising the vacuum. Particles were produced with silane as precursor and hydrogen dilutions ranging from 0 to 80% and at operating pressures of 3 to 48 mbar. A 0.5mm, coiled tungsten wire filament was fixed at 1650ºC, while the total flow rate was 50sccm. The temperature of the stainless steel substrates, in the carousel, did not exceed 70ºC. For each hydrogen dilution ratio, 5 substrates were loaded into the carousel and particles were produced with the same filament, but with the pressure being increased in 8 minute intervals. On completion the system is flushed with nitrogen and samples are stored at atmosphere. The
material was characterised for morphology, bonding configuration and crystallinity using scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD), respectively.

3. Results and Discussion

The as-prepared powder (Fig.1) varies in colour from yellowish to dark brown and is deposited on all surfaces inside the reaction chamber.

![Fig.1. a-Si:H nanoparticles produced at 48mbar and 40% \( H_2 \) dilution.](image)

The material is a highly porous agglomeration of nanoparticles. The primary size, determined from the micrographs, is in the order of 46nm with a size distribution of about 20nm. The production rate of powder deposited on the substrate is determined by weight per surface area and time (\( \mu g.mm^{-2}.min^{-1} \)). As expected, the production rate increases with decrease in hydrogen dilution and at first increases with increasing pressures, followed by a decrease after 8min of production. This decrease can be ascribed to the accelerated ageing of the filament under high pressure conditions [4].

Fig.2 shows the FTIR spectra of the material produced at different operating pressures. The absorption bands at wave numbers 2250, 1070 and 460 cm\(^{-1}\) indicate a partial surface oxidation [5], most likely caused by contact with air upon removing the powder after production [6]. The absorption bands at 2140, 2100, 880 and 640 cm\(^{-1}\) indicate the presence of Si-H mono- and polyhydride bonds, indicative of hydrogen surface passivation and micro voids [7], which can be interpreted as the voids between particles.

![Fig.2. FTIR spectra of a-Si:H nanoparticles produced at (a) 12mbar (b) 3 mbar.](image)

The XRD spectrum of commercial intrinsic silicon nanoparticles (Fig.3) displays clear crystalline peaks [8], with our darker brown material having a corresponding broad peak at the position of the (111) reflection at ~28.5°. There is also a second broad between 47° and 56°, with a

![Fig.3. XRD spectra of (a) commercial intrinsic silicon nanoparticles and (b) material collected from the shutter, produced at 6 mbar and \( T_{Sub}>120^\circ C \).](image)
suggestion of the (200) crystalline peak at 48º. Taken together these observations are indicative of a generally amorphous material with some crystalline order.

5. Conclusion

The experiments have demonstrated that thermal catalytic pyrolysis can produce a-Si:H nanoparticles in the size range of 20 to 60 nm. The particles are mostly amorphous, but small crystalline fractions can be detected in particles produced at substrate temperatures higher that 120ºC. The particles are hydrogenated and surface oxidisation is present. Although the production rate increases with increasing pressure and silane concentration, the filament deteriorates at an accelerated rate.

REFERENCES