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A novel strategy for the synthesis in supercritical fluids of silicon particles for near-infrared and optical metamaterials

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1. Introduction

The synthesis of silicon micro- and nanoparticles is a central topic in the field of material chemistry. This material is particularly well suited for many different applications, from the realization of luminescent labels for in vitro and in vivo imaging ¹ to the production of cathodes for lithium batteries ². Recently, it has been reported that sub-micrometer Si particles are ideal for the production of optical and near-infrared metamaterials ³.

Today, the production of silicon particles is mainly addressed via soft chemistry routes, such as the reduction of silicon tetrachloride with strong reducing agents ^{4,5}. These strategies typically yield low quantities of small silicon particles, and tedious post-synthesis purification steps are required to remove the by-products. These processes are not suitable for the continuous synthesis of Si particles in large quantities, and their scale-up from laboratory to industrial production is impaired.

In this oral communication, we propose a novel strategy for the production of large silicon beads, using supercritical fluids. In this oral communication, we propose a novel strategy for the production of large silicon beads, using supercritical fluids.

2. Results and discussion

The synthesis is readily available, employing non-pyrophoric commercial precursors, and no post synthesis purification, thus it can be easily adapted to a continuous process.

Large silicon spheres could be produced via this novel synthesis. Electron transmission microscopy (TEM) shows that the size of the spheres ranges between 500 nm and 2 μm (figure 1).

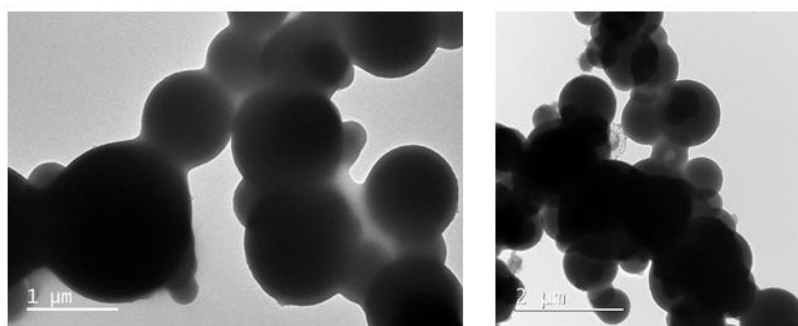


Figure 1. TEM picture of large Si spheres

Scanning electron microscopy associated to energy dispersive X-ray spectroscopy indicates that the spheres are composed by Si.

Raman spectroscopy (figure 2) shows a peak at 480 cm^{-1} , corresponding to the optical vibration mode of amorphous Si⁶.

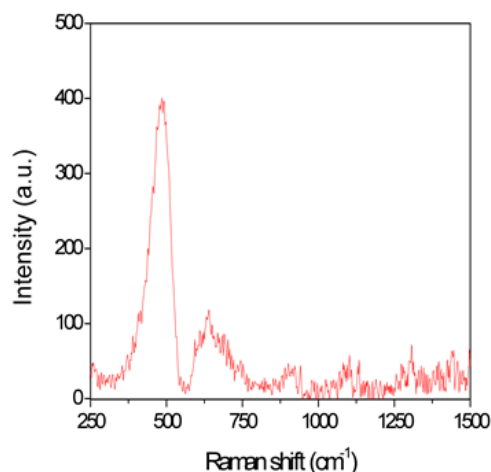


Figure 2. Raman spectrum of silicon spheres

A parametric study has been carried out to understand the mechanism underlying the formation of Si spheres, which is likely to be radical. Due to their intense Mie scattering, large Si spheres could be suitable for the assembly of metamaterials active in the infrared and visible spectrum. Therefore, the scattering behavior of the particles has been characterized by static light scattering (SLS).

3. Conclusions

Large, amorphous silicon spheres have been produced according to a new synthesis strategy. Contrary to common bottom-up techniques, which usually yield very small particles (2-10 nm), this supercritical approach permits to produce particles with size between 500 nm and 2 μm . The production of large Si particles by bottom-up synthesis has been a long standing goal in the field of nanomaterials. Moreover, this supercritical synthesis is quite straightforward and could be easily scaled-up or adapted to continuous flow. This result is not only important because of its potential technological impact, but because it permits to investigate a new reaction mechanism occurring in the supercritical domain.

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