

GROWTH STUDY OF SILICON NANOWIRES USING GOLD AND GALLIUM AS CATALYST BY IN SITU SCANNING ELECTRON MICROSCOPY

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We present a method that allows the synthesis, in a scanning electron microscope (SEM), of silicon nanowires (Si NWs) using Au or Ga as catalyst solvent. In situ SEM nanowire growth offers the ability to observe, film, and record events as they occur in real time. To get in situ growth conditions, we induced a localized heating by flowing current through a tungsten submicronic wire. The Vapor-Liquid-Solid (VLS) Si NWs synthesis is obtained by exposing the heated catalyst solvent to an atmosphere of silane¹⁻⁴. For the two different catalyst solvents used (Au and Ga), we have studied the growth mechanism and determined the structure and the composition of the Si NW's by electron microscopies characterizations.

Experiments were carried out in a nanofabrication station and in a transmission electron microscope (TEM- 2000FX – Jeol) equipped with an energy dispersive x-ray (EDX) analyser. The nanofabrication station is a vacuum chamber containing a cross beam system coupling a scanning electron microscope (SEM -JSM 5910 - Jeol) and a focused ion beam (FIB – Canion 31⁺ - Orsay Physics). The nanofabrication station is also equipped with a gas injection system (GIS) and an in-situ electrical module (current-voltage source, picoammeter). For this study, we have developed two original sample devices suitable for in situ SEM localized growth of Si NWs. The first device is based on an Al pattern deposited on a SiO₂ substrate using conventional microelectronic processes (Figure 1.a.). The second device is based on four Au electrodes thermally evaporated, through a mask, on a Si₃N₄ auto supported membrane (Figure 1.b.c.). This membrane is transparent to high energy electrons (thickness 50nm) allowing subsequent analysis in a TEM. Conductive micronic tungsten wires are fabricated, on both sample devices between the macroscopic electrodes, by Focused Ion Beam Induced Deposition (FIBID) from organometallic precursor W(CO)₆. Structural, chemical and electrical properties of the tungsten wire deposited by FIBID as fully described in previous works^{5,6}. The devices can be mounted on the in-situ electrical module allowing the heating of the tungsten wires by Joule effect. The local pressure of silane in the vicinity of the heated wire is obtained using the gas injection system while the base pressure in the chamber is in the 1.5.10⁻⁵ Torr range.

The reaction temperature is a key parameter in the VLS Si NWs synthesis. Based on recent results on nanoscale thermal properties in solid⁷⁻⁹, we have developed a simulation method to get the temperature profile for different heating conditions of the tungsten wire (Figure 2). The results of temperature simulation can be compared with our experimental results obtained by SEM and TEM observations of Au nanoparticles evaporation. These profiles were useful to estimate the temperature condition for VLS Si NWs synthesis.

We have followed the nucleation and the growth of Si NWs and observed that two very different growth mechanisms are obtained depending on the catalyst solvent (Au or Ga). For Au as solvent (Figure 3-4), the growth mechanism is the classical VLS process, the catalytic droplet is on the top of the Si NW and the growth stops when the temperature of the droplet is below the eutectic temperature. For Ga as solvent (Figure 5-6), only one nanowire grows from its bottom in the vicinity of the gallium droplet, the decomposition of silane is induced by the high temperature of the tungsten wire providing silicon for the VLS synthesis. We have observed that the catalytic droplet remains at the base of the Si NW so that this particular growth mechanism leads to the formation of a unique and very long nanowire.

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Figures:

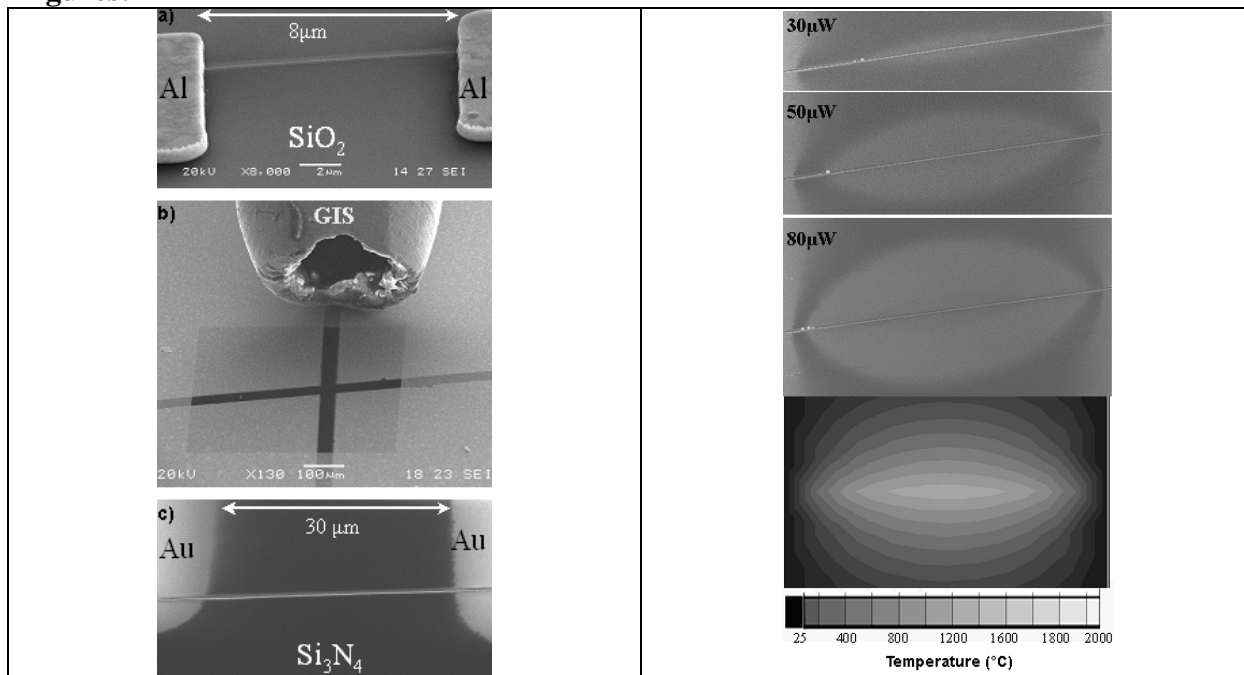


Figure 1. SEM images of the SiO₂ (a) and Si₃N₄ (b,c) sample devices

Figure 2. SEM images for different heating conditions and the associate temperature profile for a heating at 80µW.

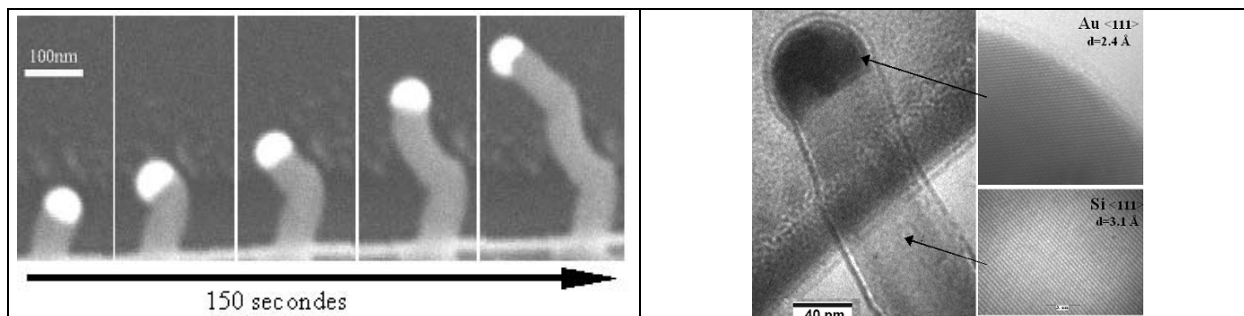


Figure 3. Successive SEM images of Si NWs on Si₃N₄ sample device using Au as catalyst solvent.

Figure 4. TEM images of Si NW on Si₃N₄ sample device

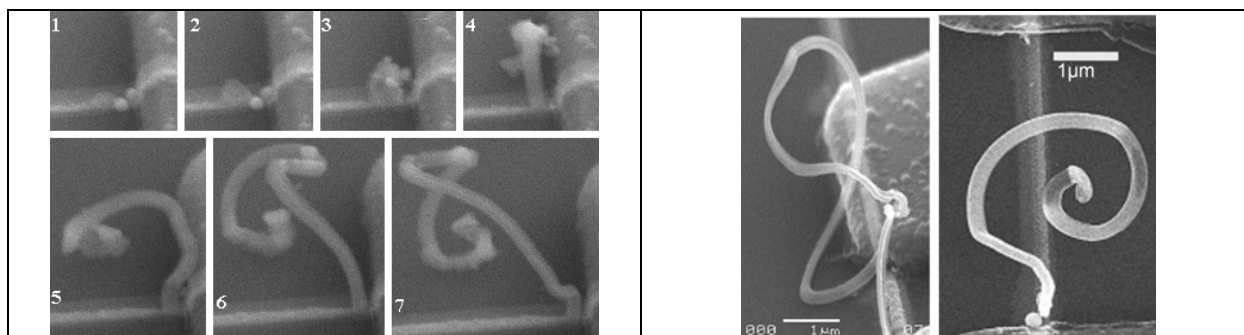


Figure 5. Successive SEM images of Si NW growth on SiO₂ sample device using Ga as catalyst solvent.

Figure 6. SEM images of Si NWs on SiO₂ sample device using Ga as catalyst solvent.