Generation of size-selected gold nanoparticles by spark discharge – for growth of epitaxial nanowires

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Abstract

One-dimensional semiconductor nanowires are a promising candidate for future electronic devices. The epitaxial growth of nanowires is often mediated by metal seed particles, usually gold particles. In this paper the setup of a simple and robust technique to generate nanometer-sized aerosol gold particles by spark discharge is described. Furthermore we demonstrate for the first time that particles generated by spark discharge can be used to design advanced nanoelectronic structures, namely nanowires. In order to obtain compact, spherical particles suitable for nanowire growth, the spark-generated agglomerate particles were reshaped in a special compaction furnace. The reshaped particles were used to seed the growth of epitaxial GaP and InP nanowires, by metal organic vapor phase epitaxy, which was shown to be a reliable and reproducible method. This work indicates the possibility of using spark-discharge generated gold particles for the creation of new electronic devices even at large scale processing.

Introduction

Electronic devices are soon reaching the physical limits of miniaturization and hence new concepts must be developed. To facilitate further down-scaling of devices one-dimensional semiconductor nanowires is a promising technology [1]. Several electronic and optoelectronic devices utilizing the properties of one-dimensional semiconductor nanowires have been demonstrated during recent years, such as light emitting diodes [2], single electron transistors [3], and nanobiosensors [4]. A variety of material systems have been investigated including group IV silicon and germanium, group III-V materials and combinations of group IV and III-V materials for growth of heterostructures [5]. Silicon has some advantages over III-V materials such as a better thermal conductivity and being a mature technology; however III-V materials offer interesting properties for high speed electronic and optoelectronic devices, including high electron mobility and, with the exception of GaP and Al-based compounds, direct bandgaps. In order to take full advantage of the unique properties of the nanowires utilized in such applications, optimized production of the wires themselves is required. The production of high quality epitaxial nanowires for different applications involves epitaxial growth mediated by nanometer-sized metal seed particles. Most often gold is used to mediate the growth of the wires either by the vapor liquid solid (VLS) mechanism [6], were the seed particle is in a liquid state, or by the vapor solid solid (VSS) mechanism [7], were the seed particle is in a solid state.

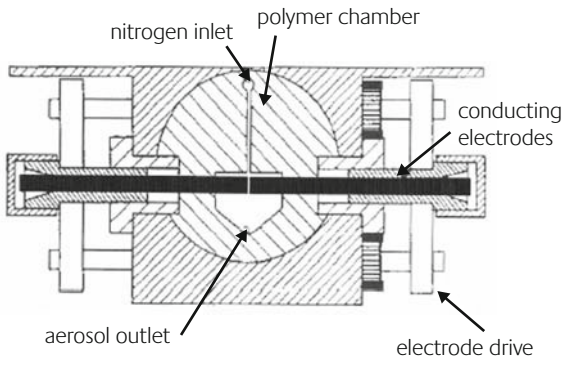
Size-selected gold nanoparticles can be produced by a variety of methods, including colloid chemistry [8] and aerosol technology [9]. The advantages of gas-suspended aerosol particles compared to liquid-suspended colloid particles include avoiding surfactants and residues on the surface, as well as simpler methods for achieving a uniform surface distribution and selected surface density. These benefits are highly desirable for the application of nanometer-sized gold particles as seeds for epitaxial growth of nanowires [10].

Here we describe the setup of a simple and robust technique to generate nanometer-sized aerosol gold particles by spark discharge, which can be integrated into an aerosol nanoparticle setup for size-selection and controlled deposition. We present size-distributions of the produced agglomerate particles, as well as morphological investigations of particle shape. In addition, the reshaping behavior of the generated agglomerate particles following heating in a special compaction furnace is reported, and compared with the reshaping behavior of aerosol gold particles produced by the evaporation/condensation method [9, 11, 12]. Finally the ability of the spark-generated particles to successfully function as seed particles for growth of epitaxial GaP and InP nanowires is demonstrated for the first time. Due to its simplicity this method seems highly promising for the creation of new electronic devices even for large scale production e.g. by using several spark generators in parallel enabling uniform deposition of gold particles over large wafers.

Experimental details

A commercially available aerosol generator (Palas, model GFG 1000), constructed for production of carbon soot particles, was used to produce nanometer-sized agglomerate gold particles. The particle manufacturing principle is based on electrode atomization by spark discharge, a mechanism that results in the production of nanometer-sized, highly charged, and agglomerated particles [13]. The generator contains two cylindrical electrodes positioned with their flat ends separated by a distance of 2 mm in the middle of a polymer chamber (Figure 1). The distance between the electrodes is varied automatically by an electric motor, and maintained during particle production to keep the breakdown voltage constant.

Figure 1

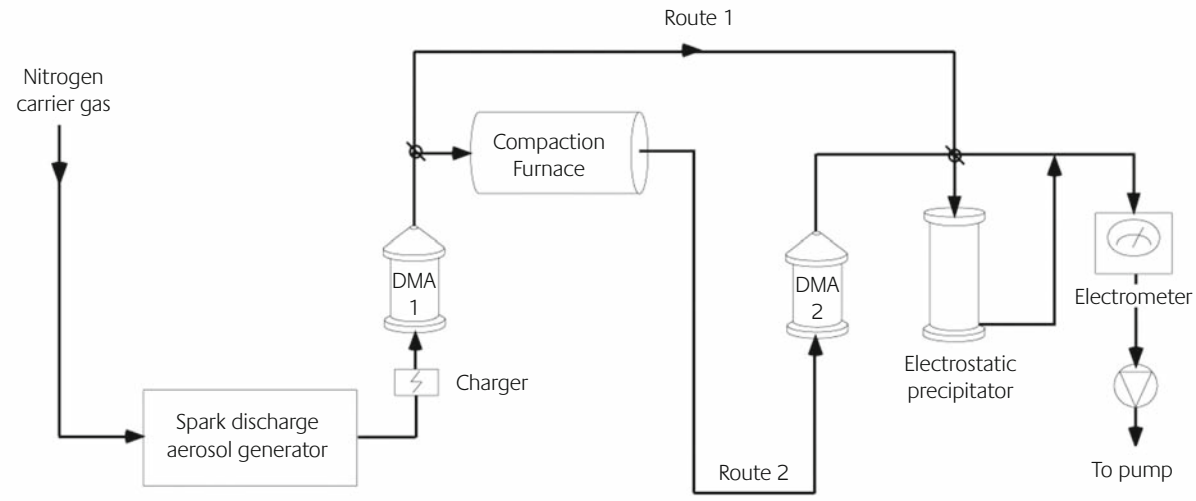


Schematic of the aerosol spark generator used to produce gold particles by spark discharge between electrodes

A 20 nF capacitor is connected to one of the electrodes, and charged by a high voltage supply with adjustable output current [14]. When the breakdown voltage of 2 kV is reached, the capacitor discharges instantaneously in a spark across the electrode gap, causing material from the electrodes to vaporize. Very small particles, primary particles, form from this vapor by homogenous nucleation which grow further by coagulation. In the original setup, a stream of argon gas or air focused between the electrodes transports the primary particles, formed due to homogenous nucleation of the vapor, through the polymer chamber towards the aerosol outlet about 15 mm downstream of the electrodes. For the production of gold nanoparticles we replaced the carrier gas with ultra-pure nitrogen in order to comply with cleanliness requirements of semiconductor industry.

Two parameters can be adjusted to optimize particle generation, namely the spark discharge frequency that can be varied between 0 and 300 Hz, and the carrier flow rate that can be varied between 2 and 8 l/min. An increase of spark discharge frequency corresponds to a higher number of discharges per second that evaporate electrode material, and hence a higher particle concentration. Furthermore, the increased particle number concentration leads to an increased coagulation rate. This most probably results in particles with a larger modal diameter, since coagulation is one of the processes responsible for further growth of the primary particles formed in each spark. The carrier flow rate is important in order to achieve a reproducible and constant particle generation. The flow rate needs to be large enough to remove all primary particles and ions between the electrodes before the ignition of the next spark. The effects of spark discharge frequency and carrier flow rate were investigated in this study.

Figure 2



Schematic of the aerosol nanoparticle system setup. Gold particles are produced in the aerosol spark generator and carried by N_2 gas through the system. Following particle generation differential mobility analyzers (DMA) are used to size-select and measure size distributions of the particles and a special compaction tube furnace is used to reshape them. Finally the particles can be counted by an electrometer or fed into an electrostatic precipitator (ESP) for deposition

Particles were generated at spark discharge frequencies between 30 and 300 Hz, and nitrogen gas flows between 3.4 and 5.9 l/min. High purity (99.99%) gold rods with diameters of 3 mm mounted to cylindrical brass holders with diameters of 6 mm were inserted into the spark generator to replace the original graphite electrodes.

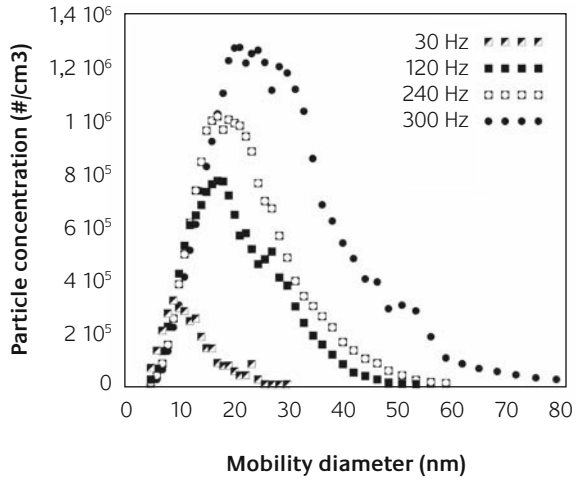
To enable size distribution measurements, reshaping of the agglomerate particles, and controlled particle deposition, the spark generator was connected to an aerosol nanoparticle system. The entire setup is shown in Figure 2. The agglomerate particles produced by the spark generator were passed through a neutralizer in order to achieve a reproducible and known charge distribution of primarily uncharged and singly-charged particles by collision of the particles with ions from a β-emitting ^{63}Ni source [15]. A differential mobility analyzer (DMA), here labeled DMA 1, was used to size-select the charged particles. The DMA, a standard instrument in aerosol science, classifies charged particles in a gas stream according to their mobility in an electric field [16]. This mobility is roughly inversely proportional to the particle diameter. Following size selection, particle concentration measurements can be directly performed using an electrometer knowing that each particle that passes the DMA carries one single charge (route 1 in Figure 2), or the agglomerated particles can be reshaped in a special compaction tube furnace (route 2 in Figure 2). Size distribution measurements of the agglomerate particles were obtained when route 1 was chosen, by stepwise scanning the voltage of DMA 1 and measuring the particle concentration with the electrometer. When instead route 2 was chosen, agglomerate particles of a fixed size selected in DMA 1 were reshaped at 300, 600, 900, or 1200°C into more compact particles. To obtain size distribution measurements of the reshaped particles the compacted particles were passed through a second DMA (DMA 2) that was stepwise scanned, and particle concentration were measured by the electrometer. By using this tandem DMA setup and scanning the reshaping temperature and measuring the peak value of the size distribution for each temperature [11], the compaction behavior of the particles was revealed.

In order to enable morphological investigations and nanowire growth experiments agglomerate particles, as well as reshaped particles, were deposited onto substrates placed inside an electrostatic precipitator (ESP) which focuses the charged particles onto a collector electrode [17] (see Figure 2). The ESP used in this study allows for the deposition of nanometer-sized particles with a very high efficiency up to a particle diameter of 100 nm onto a spot of about 1 cm in diameter. The time to reach a surface density of 1 μm^{-2} depends on the particle size and concentration and was for the experiments with the spark generator on the order of 3 minutes. For morphology, composition and structural investigations transmission electron microscopy (TEM) and X-ray energy dispersive spectroscopy (XEDS) were performed on particles directly deposited onto lacey carbon film Cu TEM grids. For nanowire growth experiments the particles were deposited onto GaP(111)B and InP(111)B substrates which subsequently were used as seed particles for growth of epitaxial GaP and InP nanowires by metal organic vapor phase epitaxy (MOVPE) [18]. MOVPE is a standard method for the fabrication of semiconductor devices, e.g. light-emitting diodes (LED). For the growth of GaP nanowires, particles reshaped at 300, 600, 900, and 1200°C with a particle density of 1 μm^{-2} of substrate and final diameter of 20 nm were used. Further details of GaP growth parameters are described elsewhere [19]. For InP nanowire growth, particles reshaped at 600°C with a particle density of 3 μm^{-2} of substrate and final diameter of 30 nm were used. Further details of the growth parameters for InP are described elsewhere [20].

Results and discussion

The production of nanometer-sized gold particles by spark discharge was found to be a reliable and simple method. Compared to other aerosol generation methods, especially the evaporation/condensation method [21], where a high temperature furnace is used to create particles by vaporization of gold followed by homogenous nucleation and subsequent coagulation, the spark generator is much less time-consuming and far easier to handle. It was possible to produce particles with fixed characteristics continuously over several hours, indicating the ability of the spark generator to operate in a stable manner during constant operation. This is another advantage compared to the evaporation/condensation method, where the particle number concentration drops over time due to clogging of the inner tubing transporting

Figure 3



Size distributions of the agglomerate particles generated at a nitrogen gas flow of 5.9 l/min and at spark discharge frequencies between 30 and 300 Hz, measured by DMA 1

the particles away from the furnace, a consequence of the high temperature gradient. The use of different spark discharge frequencies during generation of particles was observed to affect both particle number concentration and particle size, which is in good agreement with previous results on other materials [14, 22, 23]. With an increase of spark discharge frequency, the concentration of particles increased, and a shift to larger particle diameters was observed (Figure 3), for all nitrogen gas flows used.

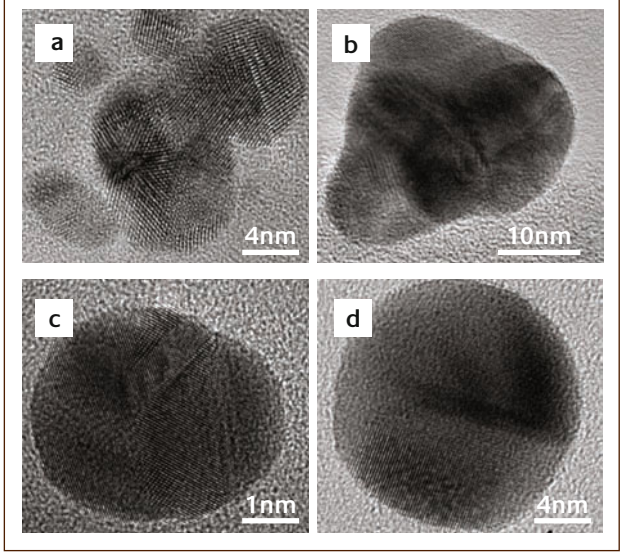
For a nitrogen gas flow of 5.9 l/min the peak value of the number concentration of particles increased from $3x10^5$ cm^{-3} to $1.3x10^6$ cm^{-3} when the spark discharge frequency was increased from 30 to 300 Hz. The particle number concentration was observed to scale linearly as a function of spark discharge frequency. This is in good agreement with the linear scaling of mass concentration per time with spark discharge frequency for graphite particles demonstrated by the manufacturers of the spark generator [14]. At the same time the peak particle diameter of the particles shifted from approximately 10 nm to 30 nm (Figure 3).

For different gas flows slightly different number concentrations were observed but the peak particle diameter was almost unaffected, indicating the influence of gas flow solely on concentration. The lowest gas flows were observed to generate a higher particle concentration compared to the highest gas flows, but for the medium gas flows no obvious trend was observed, and the results differed depending on spark discharge frequency only. It was however observed that certain gas flows together with certain spark discharge frequencies gave a more stable particle generation. The highest yield of particles at the most stable particle generation conditions was observed when combining a gas flow of 5.9 l/min with a spark discharge frequency of 300 Hz. Replacement of the nitrogen carrier gas with a gas having different viscosity coefficient could probably affect the size-distributions of the particles, but was outside the scope of this work.

Regarding particle composition, XEDS measurements together with measurements of lattice fringes from high resolution TEM images confirmed that the generated particles were indeed pure gold, allowing for the general detection limit for XEDS of < 1 at%. Furthermore, TEM investigations showed that the use of different spark discharge frequencies and nitrogen gas flows had negligible effect on particle morphology. As can be seen in Figure 4(a), the agglomerate gold particles consist of collections of compact spherical primary particles with different diameters. Diameters of primary particles between approximately 1 and 8 nm have been observed. This morphology differs noticeably from the morphology of agglomerate gold particles produced by evaporation/condensation, where more chainlike agglomerate particles consisting of primary particles no larger than approximately 5 nm can be observed [9].

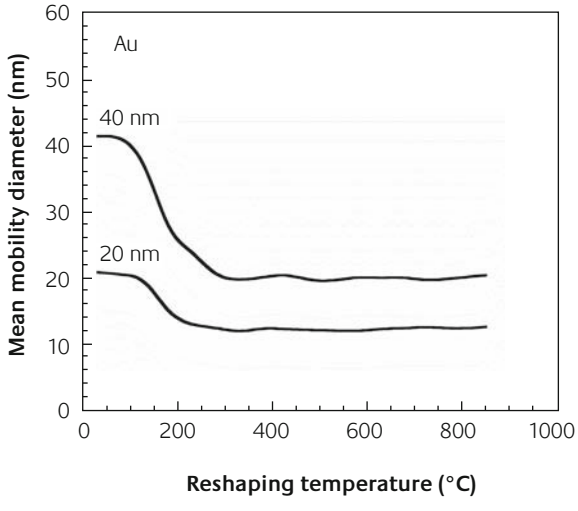
In order for the agglomerate particles to properly function as seed particles for nanowire growth, a reshaping step might be necessary, to yield compact spherical particles. Often monodisperse nanowires of a known diameter need to be produced, which in turn requires monodisperse particles of a known diameter [24]. The tandem DMA setup was utilized to achieve this narrow size distribution of particles [25]. A DMA, however, is constructed for size selection of spherical particles, which results in a less monodisperse particle distribution for non-spherical agglomerate particles which the untreated particles are as produced by the spark

Figure 4



TEM micrographs of (a) agglomerate gold particles and gold particles reshaped at (b) 300°C (c) 600°C (d) 1200°C in a special compaction tube furnace

Figure 5



The mobility diameter of the particles plotted as a function of the reshaping temperature in the compaction furnace. Particles with pre-selected diameters of 40 nm and 20 nm were reshaped at temperatures between 28 and 850°C

generator. In addition, it is possible that nanowire growth from less-compacted agglomerate gold particles shows unfavorable results, as will be described below. A thorough knowledge of the reshaping behavior of the particles is therefore crucial in order to achieve optimized gold nanoparticles and subsequent growth of nanowires with known diameters.

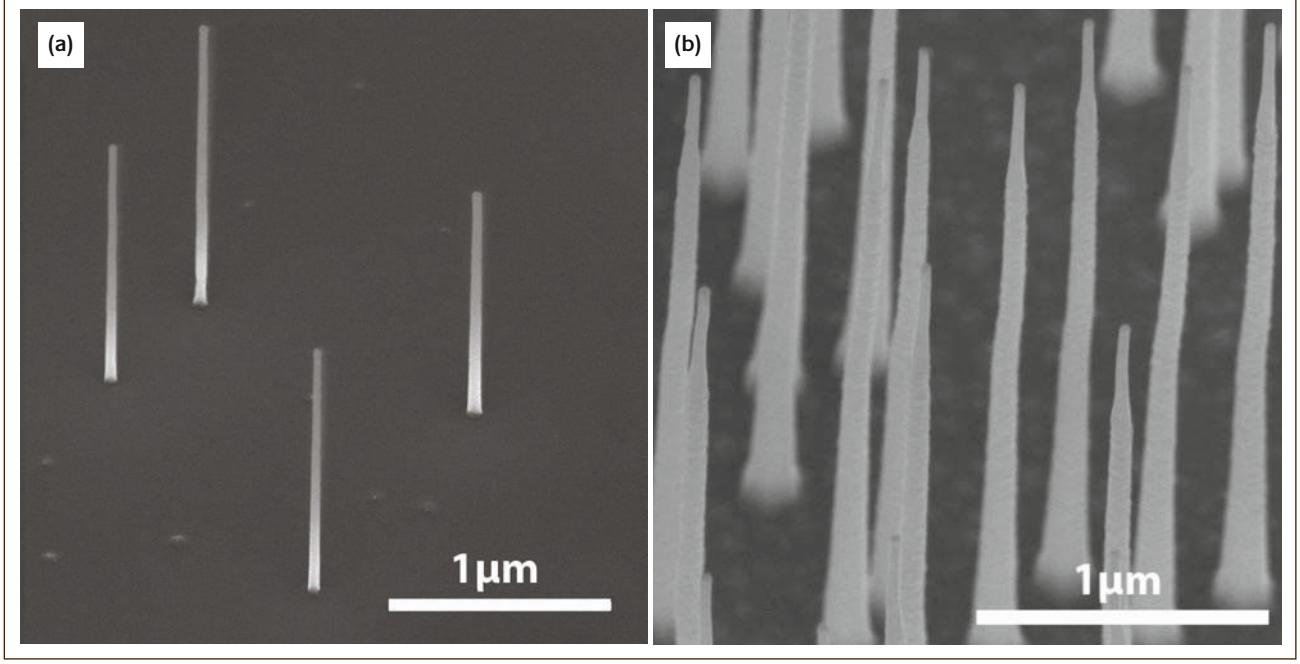
The reshaping behavior of gold particles with pre-selected diameters of 20 nm and 40 nm is presented in Figure 5, where the peak particle diameter of the particles obtained by the second DMA is plotted as a function of the reshaping temperature in the compaction furnace. From such graphs the two different steps of particle reshaping, namely compaction and internal rearrangement, are easily identified [11]. Compaction is said to occur as long as a decrease in particle diameter with an increasing reshaping temperature can be observed, and corresponds to the region in the graph where the particle diameter decreases. The compaction is complete at the so-called compaction temperature, T_C, where no more shrinking of particle diameter takes place. Following T_C internal rearrangement will occur, corresponding to the region in the graph where the diameter is almost constant. The compaction temperatures for particles with pre-selected diameters of 20 and 40 nm were observed to be approximately 500 K and 525 K respectively. This is in good agreement with observations by Karlsson *et al.* [11] stating that particles with a larger diameter have a slightly higher compaction temperature compared to particles with a smaller diameter. Furthermore, the compaction temperature for metal particles should be in the range of 1/3 to 1/2 of the bulk melting point according to the same investigation by Karlsson *et al.* [11]. Since the bulk gold melting point is 1337 K, the compaction temperatures of 500 and 525 K agree with that prediction as well.

On the other hand, the spark-generated particles produced in this study showed a significantly larger decrease in size during compaction than particles produced by evaporation/condensation, even though the compaction temperature is almost identical. Magnusson *et al.* [9] reported final diameters of around 30 and 18 nm from agglomerate particles of initially 40 and 20 nm respectively. From Figure 5 it is clear that the shrinking is larger in our case, with final diameters of around 20 and 12 nm from agglomerates of initially 40 and 20 nm. This is most likely due to slightly different compaction mechanisms caused by the different morphology of the agglomerate particles. Depending on the size of the primary particles the compaction can occur either by the "surface area change mechanism", where the primary particles coalesce with each other, or by the "rearrange mechanism", where the primary particles will undergo a rearrangement process to compact [12]. For agglomerates containing larger primary particles the rearrange mechanism will dominate, leading to a larger amount of shrinkage, whereas for agglomerates containing smaller primary particles the surface area change mechanism will dominate, leading to a smaller amount of particle shrinkage. As mentioned above the agglomerate particles produced by spark discharge consist of a greater number of larger primary particles compared to the chainlike agglomerate particles produced by evaporation/condensation. Therefore the rearrange mechanism will dominate during compaction, giving a larger amount of particle shrinkage.

In order to further investigate the reshaping process, high resolution TEM analysis of deposited particles reshaped at 300°C, 600°C, and 1200°C was performed (Figure 4(b-d)). During the second reshaping step, internal rearrangement, the particles become even more spherical as well as less polycrystalline, but without significant change in mobility diameter. As can be seen from Figure 4(b), although the gold particles have already become compact at 300°C, they are still polycrystalline and not completely spherical. With increasing temperature they become more and more spherical (Figure 4(c)), but are still polycrystalline even if less so than in figure 4b. If high enough temperature is reached there is actually a third reshaping step, evaporation, which is normally observed above the melting point of the bulk material. The particle shown in Figure 4(d) reshaped at 1200°C, a temperature well above the melting point, is almost completely single crystalline and has presumably been molten. The morphology of these particles is fairly similar to the morphology reported by Magnusson *et al.* [9] for particles produced by evaporation/condensation. The major difference in reshaping behavior thus appears to occur during the first reshaping step until the compaction temperature is reached.

Following particle characterization, the ability of the particles to function as seed particles for growth of nanowires was examined. Gold particles with a diameter of 20 nm after reshaping at 300°C, 600°C, 900°C, and 1200°C were used for growth experiments of GaP nanowires. Regardless of reshaping temperature, all particles were observed to work well as seed particles for epitaxial GaP nanowires, which can be seen in Figure 6(a) for nanowires seeded with particles reshaped at 600°C. Nanowire characteristics such as surface density and growth rate corresponded well with what was expected from previous growth of GaP nanowires seeded by aerosol gold particles produced by evaporation/condensation [19], with the exception of wires seeded by particles reshaped at 300°C. For this sample, containing particles reshaped at 300°C, surface density was slightly lower than expected from the electrometer readings of the amount of particles in the aerosol. This might be due to incomplete compaction of a small amount of the particles, which may make them less suitable as seed particles for nanowires. The growth rate of the resulting nanowires was however the same as the growth rate observed for the nanowires seeded with particles reshaped at the higher temperatures. In order to achieve the highest yield of particles reshaped at a sufficient temperature for optimum nanowire growth, the use of particles reshaped at 600°C is recommended.

Figure 6



SEM micrograph of (a) epitaxial GaP nanowires seeded with 20 nm gold particles, reshaped at 600°C, at a growth temperature of 460°C. The particle density was 1 µm^{-2}. (b) epitaxial InP nanowires seeded with 30 nm gold particles, reshaped at 600°C, at a growth temperature of 420°C. The particle density was 3 µm^{-2}. Images were acquired with the sample tilted (a) 52° (b) 30° towards the e-beam

Particles reshaped at this optimum temperature were also used as seed particles for the growth experiments of InP nanowires. The particles were observed to mediate the growth of these nanowires in a reliable and reproducible way as well, demonstrated in Figure 6(b). The expected surface density was achieved and the growth rate agrees reasonably well with previous growth experiments on InP nanowires.

Conclusions

We have described the setup of a simple and robust technique to generate nanometer-sized aerosol gold particles by spark discharge. Agglomerate particles were produced, which consisted of collections of compact fairly spherical primary particles with diameters between approximately 1 and 8 nm. By increasing the spark discharge frequency the concentration of particles increased and a shift to larger particle diameters was observed. After reshaping these agglomerate particles at higher temperatures in a special compaction furnace, the particles became almost spherical and nearly single crystalline. The reshaped particles turned out to seed the growth of epitaxial GaP and InP nanowires in a reliable and reproducible way. Since the reshaping behavior of the particles and the nanowire growth parameters were similar to those reported for aerosol gold particles produced by other methods, it seems highly likely that the particles can seed nanowires of other materials as well. In addition, the simplicity of this setup indicates the capability of using it for large scale processing of nanoelectronic structures were the use of several spark generators in parallel would enable uniform deposition of gold particles onto large wafers.

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