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A Review on Synthesis of Silicon Nanowires by Laser Ablation

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Abstract

Silicon (Si) is the most widely used semiconductor since many decades. Due to the developments in the synthesis and manufacturing technology new semiconductor materials have been introduced in semiconductor industry. Along with the newly developed semiconductors, Si is still considered the most reliable and durable semiconductor. The synthesis techniques of the Si have also been affected by the technological revolution and different Si based materials engineered at the nano-scale have been synthesized. Silicon nanowires (SiNWs) are one of the newly developed semiconductors of Si. SiNWs have interesting features due to their high aspect ratio and small size. The chemical, mechanical and electrical properties of SiNWs have opened new ways for the research in this field. Especially the charge carrying abilities and quantum confinement effects have gathered much attention of the scientific community. Different efforts have been made to synthesize SiNWs over the course of few years. Synthesis of SiNWs by laser ablation is one of the methods widely used due to its simplicity and good control over the parameters of the process. Different modifications have been made in this synthesis process by different researchers. The variations in laser ablation synthesis process and its outputs have been analyzed in this review paper. And effect of different parameters on the properties of SiNWs has been discussed.

Keywords: Laser ablation, Nanowires, Supersaturation, Vapor liquid solid mechanism, Lithography, Catalyst

1. Introduction

The nanostructures of semiconductors in the field of optics and electronics have stimulated the ongoing research in the respective area. Si is the most frequently used semiconductors in this field. The semiconductor devices which are based on silicon nanostructures have revealed novel charge carrying capabilities and quantum confinement effects due to their small size (Yang, Wu et al. 2003). Efforts have been made in last decades to produce structures made of nano-scaled engineered semiconductors fabricated by small building blocks such as quantum wells, quantum dots and nanowires. These nanostructures cause the fine tuning of the electronic band structures.

SiNWs which are one of the nano-scale semiconductors, have attracted attention of many researchers due to its appealing electrical, optical, mechanical and chemical properties due to their large aspect ratio (length to diameter ratio) (Zhang, Tang et al. 1998). SiNWs have found applications in solar cells field due to increased absorption in visible light region (Peng, Xu et al. 2005, Hu and Chen 2007, Tsakalakos, Balch et al. 2007, Lin and Povinelli 2009). They are also used as electrodes in batteries (Chan, Peng et al. 2008, Chockla, Harris et al. 2011), chemical sensors (Zhou, Hu et al. 2003, 2013) and thermoelectric materials (Boukai, Bunimovich et al. 2008, Hochbaum, Chen et al. 2008). SiNWs have also been used in devices such as biological sensors (Cui, Wei et al. 2001, Hahm and Lieber 2003, Li, Chen et al. 2004), field effect transistors (Duan, Huang et al. 2002, Cui, Zhong et al. 2003, Koo, Li et al. 2005) and integrated logic circuits (Huang, Duan et al. 2001). The term nanowire is used for the wires with diameter less than 100 nm, but this is not a strict rule for classification of nanowires (Schmidt, Wittemann et al. 2009). The SiNWs are quasi one dimensional crystalline materials having sp^3 hybridized bonding in between Si atoms. Different methods have been used to produce SiNWs which have been described below.

2. Synthesis Routes for SiNWs

The SiNWs have been produced by different methods to yield nanowires with different characteristics.

2.1 Synthesis by Chemical Vapor Deposition (CVD)

This method of synthesis relies on the substrate, CVD reactor and mixture of gases. The substrate consists of Si in form of a wafer. Mostly n-type Si substrate is used and to remove the native oxide from the surface of the wafer hydrofluoric acid (HF) is used (Cheng, Ren et al. 2012). A thin film of gold (Au) is deposited on the wafer and annealed afterwards to yield small particles of Au which act as a catalyst when the substrate is exposed to the mixture of gases. The gases used for this study included the mixture of SiH_4, B_2H_6 and HCl. Studies revealed that as the thickness of the Au film is decreased the catalyst droplet size decreases. The thickness of the Au film, annealing temperature and ratio of different gases in gas mixture affect the nanowires synthesized (Sharma, Kamins et al. 2005).

2.2 Synthesis by Chemical Etching

Chemical etching method has also been utilized to produce nanowires with desired diameters and orientations (Juhasz, Elfström et al. 2004). Si wafers have been used having peculiar properties such as purity levels or dopings. The nanowires obtained from the substrates carry the same properties as that of the substrate. The substrates with different resistivities have been used in different studies. The cleaning of the wafers is performed followed by dipping in $AgNO_3$ for Ag particles coating and then etching with HF, and H_2O_2 is done (Zhang, Peng et al. 2008). The nanowires obtained have the diameters as that of the Ag particles and have <111> growth direction.

There is another type of etching called reactive ion etching which has also been used to produce SiNWs by using polystyrene beads on substrates (Huang, Fang et al. 2007). Polystyrene beads diameter determines the diameter of nanowires and to carry out the etching process. Ag is deposited by thermal evaporation process. The polystyrene beads are not covered by Ag, only the areas covered by Ag are etched when the substrates are exposed to reactive ion plasma. Studies have revealed that as the etching time is increased the length of the wires etched also increases (Huang, Fang et al. 2007).

2.3 Synthesis by Lithography

Lithography technique which is a top down method has been used to synthesize SiNWs. In this method the desired product is obtained by either reduction or electron lithography (Choi, Zhu et al. 2003, Juhasz, Elfström et al. 2004, Choi, Liew et al. 2008). Photolithographic technique has also been used (Za'bah, Kwa et al. 2012). In this method of synthesis the substrate of Si is oxidized and then subjected to different processing steps such as polysilicone deposition, low oxide formation, selective etching and then formation of the desired wires. The process parameters such as operating temperature, time of exposure, thickness and purity of the substrate effect the properties of the nanowires synthesized (Za'bah, Kwa et al. 2012).

2.4 Synthesis by Molecular Beam Epitaxy

This method of synthesis relies on a molecular beam which is an electron beam which melts the substrates and catalyst particles, and nanowires growth occurs by vapor liquid solid (VLS) mechanism which will be discussed later. The substrate with catalyst particles coated on it is placed in the chamber where molecular beam arrangements are made (Schubert, Werner et al. 2004). The experiment is carried in ultra-high vacuum conditions. This process is different from CVD process as the catalyst particle in this case does not act as seed. There are some modifications made to this process which include nanosphere lithography along with molecular beam epitaxy (Fuhrmann, Leipner et al. 2005).

2.5 Synthesis by Laser Ablation

This technique has been extensively used by different research groups. Different amendments have been made to the process by using different catalyst particles, substrates and synthesis temperatures (Fukata, Oshima et al. 2006). The details of this method have been discussed below.

3. Laser Ablation a Method to Synthesize SiNWs

Laser ablation technique is most widely used for the synthesis of SiNWs as it gives high yield and good control on the process parameters. Another advantage of this process is that it is chemically simple and clean process. In this review paper the synthesis of SiNWs by laser ablation is discussed. In this method for the synthesis of SiNWs, the targets made of pure Si along with catalyst particles are placed in the furnace and subjected to laser beam. The vapors of the Si are formed which are carried away by the carrier gas which is usually argon (Ar) and deposited on cold finger either made of copper or Si. The growth of the SiNWs occurs by the catalyst particles present in the form of wires. The schematic diagram of such operation is shown in Figure 1.

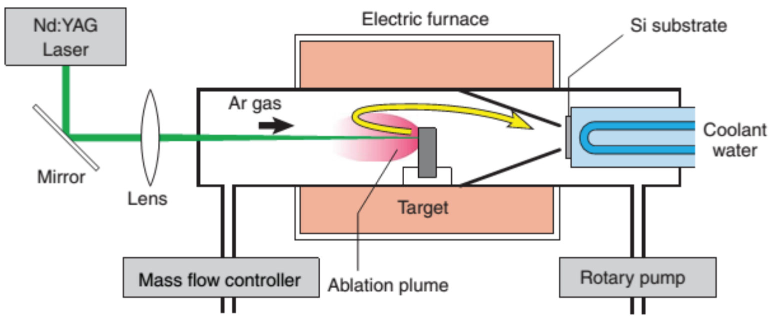

Figure 1. Schematic diagram for synthesis of SiNWs by laser ablation system.
Figure adopted with permission from(Fukata, Oshima et al. 2006)

The neodymium-doped yttrium aluminum garnet (NdYAG) laser is used in this particular case but the use of krypton fluoride (KrF) excimer laser has also been reported (Tang, Zhang et al. 1999). The targets are being ablated at ambient temperatures by laser beam and the formed vapors are carried away by the Ar gas flowing through the furnace chamber and mostly deposited at water cooled copper plate known as cold finger. The whole process is carried out in the vacuum. The laser ablation method for the synthesis of SiNWs is not new it has been already used to synthesize carbon nanotubes (Zhang and Iijima 1999, Guerrero, Puerta et al. 2008), iron nanowires (Mollah, Henley et al. 2010) and tin oxide nanowires (Liu, Zhang et al. 2003).

3.1 Growth Mechanism of SiNWs

The mechanism by which the growth of the nanowires occurs is well explained by *et al* Alfredo M. Morales (Morales and Lieber 1998). The growth of wires occurs by VLS mechanism by which the gas phase reactants are absorbed on catalysts particles or liquid metals. The vapors are supersaturated in liquid metal and then propagate in one dimensional wire from. This mechanism is illustrated in Figure 2.

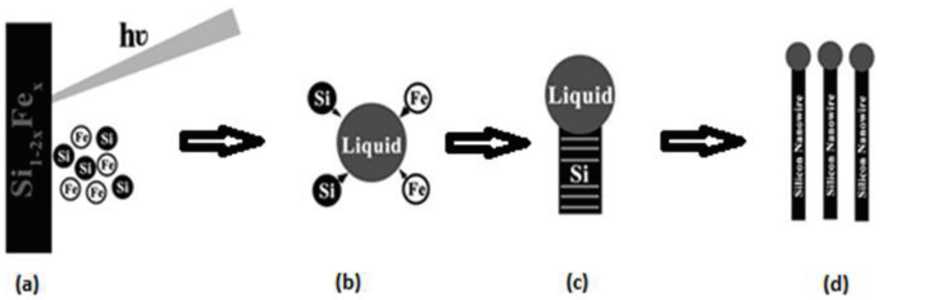

Figure 2. The VLS growth mechanism is illustrated in the following steps, (A) Targets are ablated by the laser beam and vapors of Si are formed. (B) The Si vapors are saturated in liquid metal. (C) After condensation due to collision of vapors with gas, super saturation of Si occurs and grows out of liquid metal particle. (D) Termination of growth occurs when the wires are out of the hot zone. Modified from (Morales and Lieber 1998).

3.2 Methods Employed for Synthesis

Different modifications in the synthesis of SiNWs by laser ablation have been made during last few years. We will discuss them one by one and try to compare the outcomes of the experiments, and the effects of different process parameters on the morphology and properties of SiNWs.

3.2.1 Laser Ablation using Fe Catalyst at High Temperature

The laser ablation apparatus is used here as shown in Figure 1 with slight variation. In this case excimer pulsed laser is used (Zhang, Tang et al. 1998). A quartz tube is placed in the furnace and a water cooled copper finger is inserted inside the quartz tube to collect the SiNWs. Ar gas flows through the chamber and vacuum is created by pumps. The laser ablates the targets of Si which are also placed inside the quartz tube. These targets are made of powders of Si 99.5% and iron 0.5%. The furnace temperature is maintained at 1200°C. The laser used has wavelength 248nm, frequency 10Hz, energy 400mJ and laser spot dimension of 1mm×3mm. The Ar gas flows at 50 standard cubic centimeters (sccm) with pressure of 500torr in evacuated conditions. The product is collected at the distance of 15cm from the target. The growth rate of 10-80µm/hour for the SiNWs has been observed. The analysis of the samples is done by transmission electron microscopy (TEM), x-ray diffraction (XRD) and Raman.

TEM analysis shows that the nanowires obtained have the diameters in the range of 16-21nm. TEM study also revealed that the nanowires were smooth and had stacking faults. Another interesting observation on high resolution TEM (HRTEM) has been made and that is the nanowires consist of crystalline cores and amorphous outer shells.

XRD measurements show that maximum intensity of the peaks occurs at (111) planes. The d spacing (the distance between the crystallographic planes) values obtained from XRD of SiNWs shows the same values as that of the bulk Si.

The Raman spectra of single crystalline nanowires, the produced nanowires and completely oxidized nanowires shows that the asymmetry of the peaks at low frequency. It also shows that as the size of the nanowires decreases the energy of the peaks decreases causing broadening in the peaks.

3.2.2 Laser Ablation using Au as Catalyst

In this case NdYAg laser with wavelength 532nm was used frequency 10Hz (Wang, Chung et al. 2004). Furnace temperature was maintained at 1000°C. The targets were made by mixing powders of Si 80% and Au 20% and compressing at 300MPa at room temperature and placed in the quartz tube. The laser spot 1.5mm and mixture of Ar and H_2 was flowing at the flow rate of 50sccm.

The nanowires are deposited inside the tube of quartz, samples are taken out at the different distances from the target like 2, 4, 6-8 and 10-12cm. SEM observation showed that the sample taken at the distance of 2cm were consisting of curved nanowires while 4cm sample had curved nanowires having embedded Au nanoparticles in nanowires. The samples taken at the distance of 6-8cm showed straight nanowires with no Au nanoparticles embedded in. The samples at the distance of 10-12cm samples showed the straight nanowires with embedded Au nanoparticles. An explanation has been proposed for the presence of Au particles in SiNWs. During ablation the collision of the vapors occurs with the carrier gas, there is a possibility that nanoparticle are formed due to that collision so when the SiNWs grow there is a chance that nanoparticle may go into the nanowires and sink there (Wang, Chung et al. 2004). The diameters of nanowires which are produced by this method can be seen in Figure 3 showing that most of the nanowires have diameter in the range of 27-32nm.

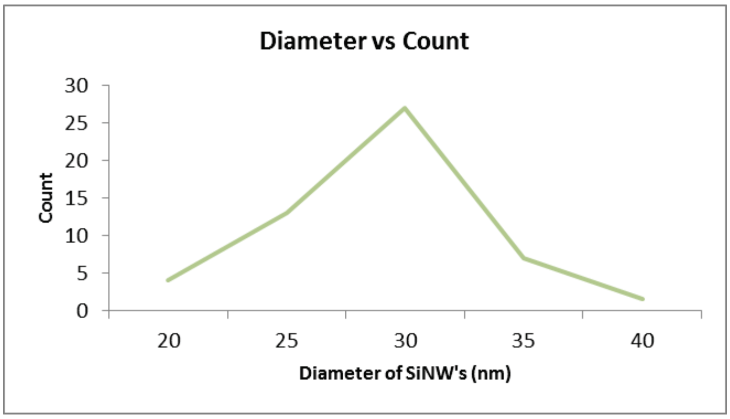

Figure 3. Diameter distribution of SiNWs. Modified from(Wang, Chung et al. 2004)

The diameters of nanowires which are produced by this method can be seen in Figure 3 HRTEM shows that the SiNWs obtained by this process also have crystalline Si core and amorphous silicon oxide sheath. HRTEM observation also shows that growth of SiNWs occurs along <111> direction.

3.2.3 Laser Ablation of Mixture of SiC and SiO_2

Targets consisting of SiC 90wt.% and SiO_2 10wt.% have been used (Tang, Zhang et al. 1999). Powders were pressed under hydraulic press for 24 hours at 150°C. The carrier gas used was Ar which flows at 50sccm in evacuated quartz tube under pressure of 700torr. The KrF excimer laser has been used with wavelength 248nm, frequency 10Hz and pulse duration 34ns. The temperature of the chamber was set at 1400°C. The laser ablation process was started when the whole chamber had the homogenous temperature. The product was obtained in the form of two sponge webs. The quantity of material in first web which was close to the target was more than then other second web.

Scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and TEM analysis was made. The results of SEM observation showed that SiNWs with smooth surfaces were obtained which have same diameter throughout the length. The average diameter of SiNWs obtained was 14nm. The results of EDS and TEM showed that the core of nanowires was of crystalline Si and outer layer of amorphous SiO_2.

The analysis TEM analysis of the first web showed that it contain SiNWs but the second web had SiNWs along with β-SiC nanoparticles. Oxide assisted growth mechanism has been suggested for the growth of nanowires. The following reaction was proposed

$$Si_{(s)} + SiO_{2(s)} \rightarrow Si_{(s)} + SiO_{(g)} + CO_{(g)} \quad (1)$$

The carrier gas carries SiO vapors and its deposition results in SiNWs. β-Sic nanoparticles are only found in the second web illustrating that they are not formed as the result of the laser ablation. β-SiC nanoparticles are formed as the result of the chemical reaction and not by the growth mechanism as SiNWs do. Another method utilizes laser ablation of SiO powders (Tang, Zhang et al. 2001) and growth of SiNWs occurs by the same mechanism proposed now in which the carrier gas carries SiO vapors and causes growth of SiNWs after deposition. The diameters of nanowires obtained by this were in the range of 10-20nm.

3.2.4 Laser Ablation of Mixture of Si and SiO_2

An excimer laser with wavelength 248nm and frequency 10Hz is used and Ar gas flows through the ceramic tube, while the temperature of the tube where target is placed is maintained at 1200°C (Wang, Zhang et al. 1998). The targets are made with different compositions and different growth rates of nanowires are observed for them which are shown in table 1. The results reveal that the yield of the targets containing SiO_2 is higher than the metal containing targets.

Table 1. Yield of SiNWs by using different target materials, adapted with permission from (Wang, Zhang et al. 1998)

Target	Temperature ( )	Yield (mg)
Si + 1% Fe or 1% Ni	1200	0.1
Si + 1% Co	1200	0.1
Si + 0.5% Co + 0.5% Ni	1200	0.1
Si wafer	1200	Very little
Si powder	1200	Very little
Si + 5% SiO_2	1200	0.3
Si+ 10% SiO_2	1200	0.5
Si+ 30% SiO_2	1200	2.5
Si+ 50% SiO_2	1200	3.0
Si+ 70% SiO_2	1200	1.5
Si+ 90% SiO_2	1200	0.5
SiO_2	800-1200	0

The SEM and TEM observation reveals that nanowires of the diameter 9-12nm are obtained and no evidences of the metals at the tips were found. HRTEM analysis showed that the SiNWs had crystalline core with amorphous shell of silicon oxide. However the growth mechanism of nanowires by ablation of SiO_2 containing targets is explained afterwards by Yi-Han (Yang, Wu et al. 2003). But it has been found that the silicon oxide sheath around the SiNWs prevents the lateral growth.

Yi- Han Yang conducted the same type of experiment but varying the targets compositions. He utilized the targets mixtures of the powders of Si + Fe, Si + Ru (ruthenium), Si + Pr (praseodymium), Si + SiO_2 + Fe, Si + SiO_2 + $RuCl_3$, and Si + SiO_2 + Pr_6O_{11} respectively. The targets were pressed in the form of pallets. The weight ratio of Si 90% + metal 10%, and Si 45% + SiO2 45% + metal 10% was maintained in the pallets. The NdYAg laser with 1064nm wavelength at frequency of 30Hz was used. The temperature of the chamber was maintained according to the eutectic point of mixtures used.

SEM analysis showed difference in the morphologies of the SiNWs prepared by Si + Fe and Si + SiO_2 + Fe targets; this can be seen in the Figure 4.

Figure. 4 (a) shows the SiNWs prepared with the help of SiO_2 showing that nanowires are in the range of 50-300nm diameters. (b) Shows the nanowires obtained without SiO_2 showing very thin nanowires. (c) Shows that there are beads of nanoparticles in nanowires. The composition of the beads illustrates that they are mostly consisting of Si, oxygen and Fe, this means that the growth of the nanowires is dominantly occurring due to SiO_2 catalyst particles and not be Fe particles. In the inset XRD measurement shows the presence of different elements in nanowires, Cu element detected due to deposition of SiNWs on cold Cu finger. (d) The growth occurs only by VLS mechanism and metal tips are found at one end having composition $FeSi_2$. (e) HRTEM shows that the core of the nanowires is of crystalline Si surrounded by amorphous silicon oxide. The spacing d of crystallographic planes is 0.32nm and growth occurs in <112> direction while in (f) the growth occurs in <111> direction.

Same observations were made for Pr ad Ru containing targets, the growth of the nanowires was controlled by SiO_2 catalyst and by metals when SiO_2 was absent in the targets. The diameters of SiNWs obtained in the SiO_2 containing targets were more than that of metal containing targets. The nanowires had crystalline cores with diameter smaller than the amorphous SiO_2 oxide. The growth of the nanowires was observed by HRTEM showed the same results obtained in previous case illustrating that in presence of SiO_2 as catalyst growth occurred in <112> direction and in case of only metal particles as catalyst growth occurred in <111> direction. In the case of targets containing Si and metal, the tip of the nanowire had the eutectic composition. The Pr containing targets had $PrSi_4$ and Ru containing targets had $RuSi_3$ at the tip of the nanowires. This purposed growth observation can be seen in the figure. 5

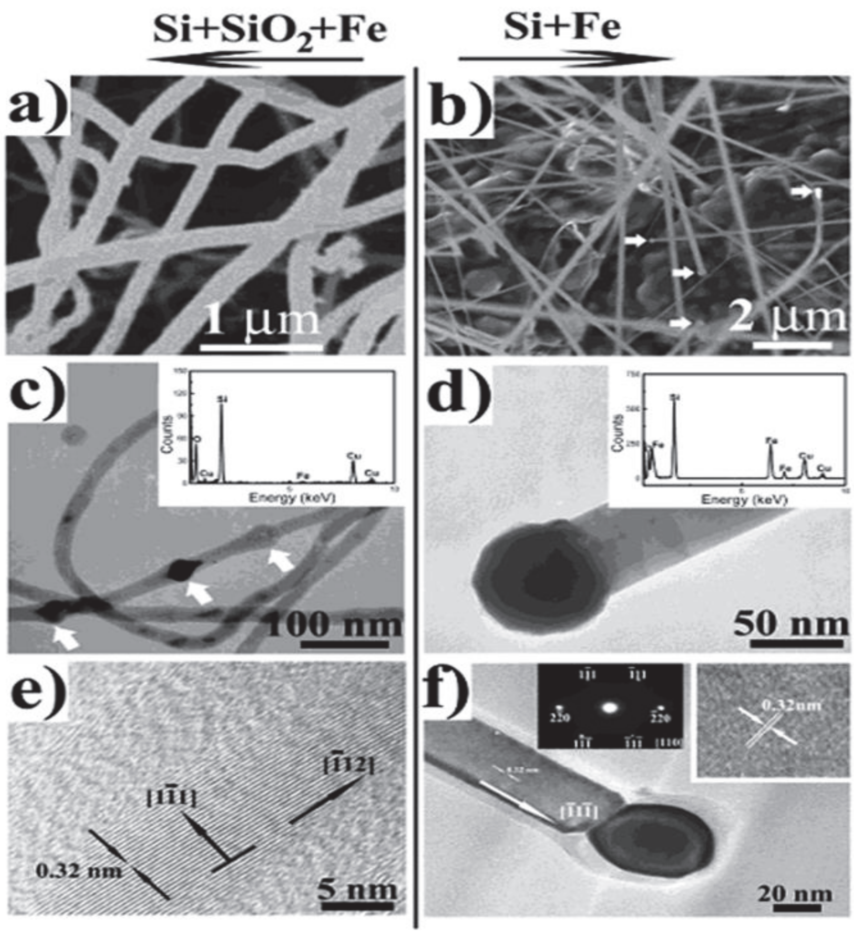

Figure 4. SEM images of SiNWs obtained by Si + Fe and Si + SiO_2 + Fe targets, adapted with permission from (Yang, Wu et al. 2003)

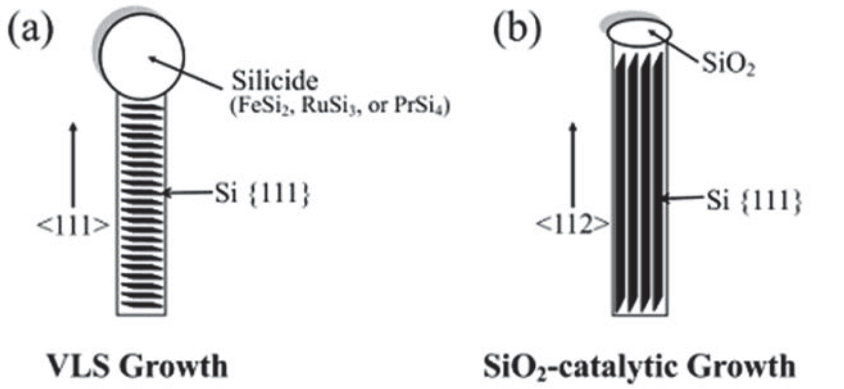

Figure 5. Growth of Silicon nanowires by VLS mechanism (presence of metal as catalyst) and SiO_2 as catalyst, adapted with permission from (Yang, Wu et al. 2003)

3.3 Effect of Different Parameters on the Properties of SiNWs

The parameters which can affect the properties of synthesized SiNWs are the amount of catalyst particles, power of the laser used for ablation and synthesis temperature. The effects of these parameters have been discussed in detail below.

3.3.1 Effect of Varying Amount of Catalyst Particles

In order to carry out this experiment targets for laser ablation were made of Si and Fe in $Si_{99}Fe_1$, $Si_{98}Fe_2$, $Si_{95}Fe_5$, $Si_{90}Fe_{10}$ atomic ratios respectively (Fukata, Oshima et al. 2005). Sintering of these powders was done at 900-950°C and then placed in ceramic tube which was heated up at 1200°C (the eutectic point of Si-Fe). NdYAg laser with wavelength and frequency of 532nm and 10Hz was used. Ar gas flow was maintained at 50sccm. The deposits were collected on the cooled finger of Mo and microscopic observations were made. The variations of diameters of SiNWs with the amount of catalyst used were plotted in Figure 6.

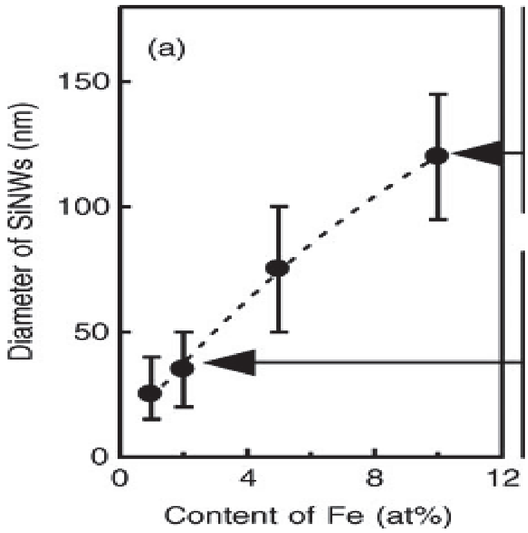

Figure 6. Diameter dependence of SiNWs on amount of catalyst, adapted with permission from (Fukata, Oshima et al. 2005)

This trend shows that as the amount of catalyst in targets is increased the diameter of SiNWs increases. The reason for this behavior could be, due to laser ablation the alloys of Si-Fe are formed and the super-saturation of Si in liquid Fe causes the growth of SiNWs from the liquid catalyst. The more the catalyst volume the more is the amount of Si dissolved in it and the thicker the nanowire grows (Fukata, Oshima et al. 2005).

3.3.2 Effect of Laser Power used for Ablation

The same targets (Fukata, Oshima et al. 2005) as used in pervious case are used here. The effect of laser power on the diameter and length of the nanowires is obtained and plotted in Fig. 7. It shows that as the laser power is increased the diameters of SiNWs increases this may be because the more amount of Si atoms are ablated by high powered laser causing increase in the super-saturation. While the length of the SiNWs decreases as the laser power is increased because the amount of Si dissolved increases with increasing laser power and the probability for the nanowires of small diameters to grow is more than the other case. So that is why thicker nanowires grow to small lengths (Fukata, Oshima et al. 2005).

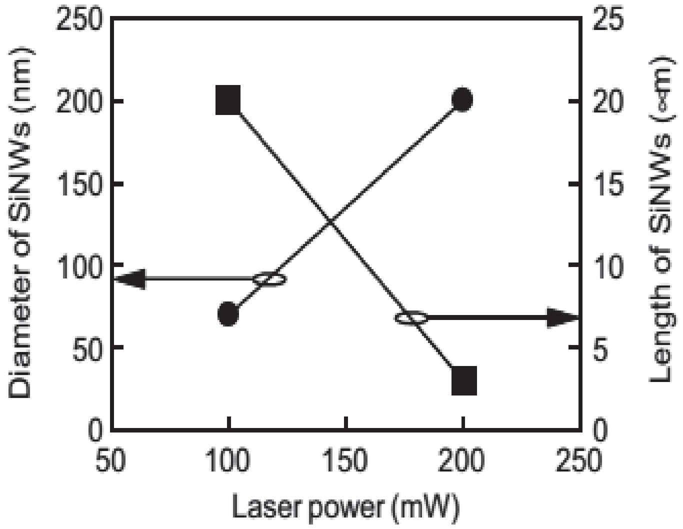

Figure 7. Diameter and Length dependence of SiNWs on laser power, adapted with permission from (Fukata, Oshima et al. 2005)

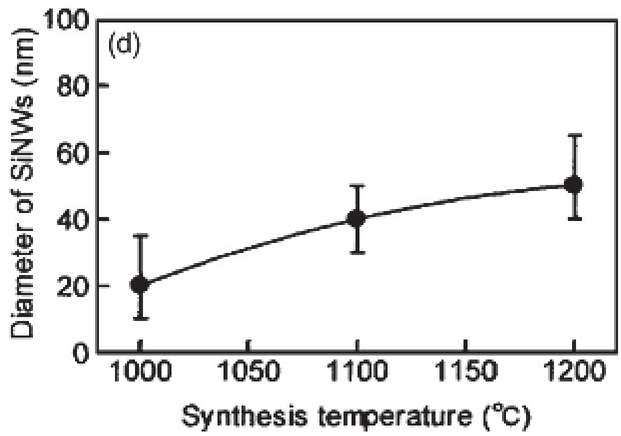

Figure 8. Temperature dependence of SiNWs diameter, adapted with permission from(Fukata, Oshima et al. 2005)

3.3.3 Effect of Synthesis Temperature

The targets with Si 99atm% and Fe 1atm% are placed in the ceramic tube at different temperatures like 1000, 1100 and 1200°C (Fukata, Oshima et al. 2005). The diameter of the SiNWs obtained at different synthesis temperature reveals that the diameter of the nanowires decreases as the synthesis temperature decreases. The experimental findings are plotted in Figure 8.

4. Further Developments

The laser ablation method has been used to synthesize SiNWs with different shapes and morphologies by using different types of particles as catalyst in the substrates. The nanowires obtained from this method have catalyst particles at the tips and embedded in the structure of the nanowires depending on at which distance from the substrate they were deposited. Different catalyst particles have been used to synthesize nanowires and the results revealed that the diameter of the nanowires is dependent on the diameter of the catalyst used. Investigations can be made to fabricate nanowires by using monodispersed catalyst particles. Also studies can be done to synthesize SiNWs doped with pentavalent and tetravalent impurities. The effect of pressure inside the reaction vessel on the shape and morphology of nanowires can also be investigated. Another modification which can be made to the process is to carry out the laser ablation in liquid and effect of addition of different inorganic salts on the properties of nanowires synthesized be studied. By having more control on the process parameters the experiments can be implemented for large scale production of SiNWs.

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