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Shama Parveen
Department of Physics,
DPG Degree College,
Gurugram, Haryana, India

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Synthesis of carbon nanotubes on nickel catalyst by low pressure chemical vapour deposition

Shama Parveen

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Abstract

Carbon nanotubes (CNTs) are synthesized using Low Pressure Chemical Vapour Deposition (LPCVD) at 750 °C with a Nickel (Ni) catalyst, and have been explored as electron field emission electrodes. Initially, an N-type cleaned Si substrate is coated with Nickel catalyst via RF sputtering. This catalyst-deposited Si substrate is then heated to 750 °C to form nano-islands of catalyst within the LPCVD chamber. Vector gases, Ammonia and Hydrogen, with a flow rate of 170 sccm, are allowed to pass through the tube reactor for 25 minutes. Acetylene, serving as a source gas with a flow rate of 40 sccm, is introduced for 15 minutes at 750 °C. The surface morphology of the as-grown CNTs is characterized using a Scanning Electron Microscope (SEM). The images reveal successful growth of CNTs, with diameters ranging from 25 to 55 nm and lengths extending to several micrometers. LPCVD is a widely used method for CNT production, especially for mass production. Our results demonstrate high density and aligned growth of CNTs, possibly attributed to Ni being easily impregnated on a Si substrate, which is crucial for achieving good adhesion between the catalyst and Si substrate.

Keywords: Carbon nanotubes, low pressure chemical vapour deposition, electronic devices

Introductions

Carbon Nanotubes (CNTs) have captured the attention of scientists and researchers due to their unique and remarkable properties. Their large current density, high aspect ratio, sharp tip, high melting point, chemical inertness, and vacuum compatibility render CNTs as one of the most promising materials for field emitters ^[1-3]. With these outstanding properties, CNTs hold potential applications in various nanoscale-based field emission display devices, energy storage devices, X-rays, CRO, SEM, and electron beam evaporators ^[4-6]. CNT-based vacuum microelectronic and display devices have already been proposed ^[7]. Furthermore, applications of CNTs as field electron emitter sources in display devices have been demonstrated due to their high current density at low turn-on voltage ^[8].

CNTs are synthesized with high purity, selective growth, high yield, and vertical alignment through different techniques. To optimize CNTs yield and quality, three main synthesis processes have been employed: arc discharge, laser ablation ^[9], and chemical vapor deposition (CVD) ^[6-9]. CNTs obtained in powder form by arc-discharge and laser ablation techniques are not ideal for better field emissions as they do not adhere properly to substrates. A breakthrough in the synthesis of CNTs occurred with the development of the chemical vapor deposition (CVD) technique for CNT growth ^[9], enabling direct growth of CNTs over substrates such as silicon, quartz, and alumina, which are highly desirable for field emission studies of CNTs field emitters. Synthesis of CNTs by chemical vapor deposition (CVD), including plasma-enhanced chemical vapor deposition (PECVD), microwave plasma chemical vapor deposition (MPCVD), and low-pressure chemical vapor deposition (LPCVD) processes, produces high-purity aligned carbon nanotubes in bulk and at low cost ^[10]. Among the aforementioned processes, LPCVD stands out as the most promising route for bulk production of high-purity and aligned growth of nanotubes on substrates that can be used as electron field emitter sources ^[10-12]. In the present work, we adopted the LPCVD technique for synthesizing CNTs on a silicon substrate.

Corresponding Author:
Shama Parveen
Department of Physics,
DPG Degree College,
Gurugram, Haryana, India

Nickel as a catalyst was coated on the silicon substrate using the RF sputtering method. Characterization of the as-grown CNTs was performed by SEM.

Experimental Detail

In the present study, Carbon Nanotubes (CNTs) were grown on a silicon (N-type <100>) substrate via the Low Pressure Chemical Vapour Deposition (LPCVD) technique. The bare Si substrate was ultrasonically cleaned in acetone and dried at room temperature before being loaded into the RF Sputtering chamber. Nickel (Ni) was used as the catalyst and coated onto the cleaned Si substrate using RF sputtering (MODEL: 12" MSPT) at a power of 50 W and an Ar pressure of 1x10⁻³ Torr. The thickness of the Ni catalyst was 15 nm. The sputtered film was then transferred into a quartz tube and heated in a high-temperature tube furnace to the desired temperature under a flow of H₂. A mixture of H₂:C₂H₂:NH₃ as source gases with flow rates of 170:40:170 sccm respectively, was introduced into the quartz tube. Here, NH₃ served as a pre-treatment gas to aid in the nucleation of Ni catalyst particles in the nanometer range. The reaction temperature was maintained at 750 °C, and the growth time was 40 minutes. The quality of the synthesized CNTs was characterized using a Scanning Electron Microscope (SEM) (JEOL, JSM 6380).

Results and Discussion

SEM Analysis

SEM images in Fig. 1 illustrate the surface morphology of carbon nanotubes (CNTs) grown via the LPCVD technique on a Ni catalyst at 750 °C. The images reveal the successful synthesis of aligned and evenly distributed CNTs on a silicon substrate. The length and diameter of the CNTs range from 4-5 μm and 25-55 nm, respectively. The diameter of the CNTs is determined by the catalyst size during nucleation. It's possible that some of the Co catalyst diffused into the Si substrate, forming NiSi₃, which enhanced the adhesion between the CNTs and the Si substrate. Therefore, Ni, as the catalyst, plays a crucial role in the growth of CNTs. The Schematic of grown carbon nanotubes on the nickel coated Silicon substrate are shown in figure.2

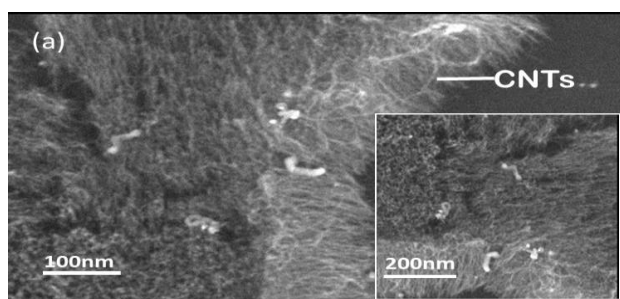


Fig 1: Carbon nanotubes grown on Ni Catalyst

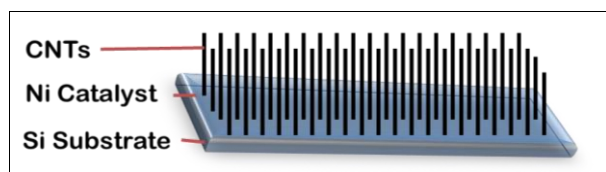


Fig 2: Schematic of Carbon nanotubes grown on (Ni) Nickel Catalyst

Conclusion

We have successfully grown carbon nanotube (CNT) cathodes using the LPCVD technique with a Ni catalyst.

The size and shape of the Ni catalyst play a crucial role in achieving aligned and high-density growth of CNTs. The Ni catalyst provides nanometer-scale nucleation sites for CNT growth, determining the diameter of the resulting nanotubes. SEM images of the as-grown CNT film reveal an average diameter ranging from 25 to 55 nm, with lengths spanning several micro meters. This suggested method can be utilized for the growth of high density CNTs and would be used for the diverse application in different field such as electronic, X-ray device, display device and forthcoming Artificial Intelligence tools. By leveraging the suggested method for growing high-density CNTs, we can unlock significant advancements across electronics, X-ray devices, display technologies, and AI tools. The versatility and superior properties of CNTs make them a critical material for future technological innovations.

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