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*Article*

# Fullerene Nanowhiskers and Control Their Geometric Dimensions

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**Abstract:** Semiconductor nanowhiskers, in particular, nanostructured whiskers based on zero-dimensional (0D) $C_{70}$ fullerene, are being actively discussed due to the great potential of their application in modern electronics. For the first time, we proposed and implemented a method for the synthesis of nanostructured $C_{70}$ fullerene whiskers based on the self-organization of $C_{70}$ molecules during thermal evaporation of $C_{70}$ droplets on the substrate surface. We found that the onset of the synthesis of $C_{70}$ nanowhiskers upon evaporation of drops of a $C_{70}$ solution in toluene on the substrate surface depends on the substrate temperature. We have provided experimental evidence that an increase in both the $C_{70}$ concentration in the initial drop and the substrate temperature leads to an increase in the geometric dimensions of $C_{70}$ nanowhiskers. The obtained results provide useful vision on the role of solute concentration and substrate temperature in the synthesis of one-dimensional materials.

**Keywords:** $C_{70}$ fullerene; evaporating drop; self-organization; nanostructure; filamentous whisker

## 1. Introduction

In nanoscience, nanowhiskers are considered to be filamentous crystals with a transverse size of up to 100 nm and a length that is an order of magnitude or more greater than the transverse size. Semiconductor nanowhiskers are widely used today to create miniature elements of devices in microelectronics [1,2], optoelectronics [3,4], nanoengineering [5,6], solar energy [7–9], biomedicine [10], nanoelectromechanics [11,12] and gas sensing [13,14]. To date, there are various methods [15–17] for obtaining nanowhiskers of a wide range of semiconductor materials, such as growth by molecular beam epitaxy, vapor deposition, laser ablation, growth catalysts, magnetron deposition, chemical epitaxy in high vacuum and others.

Carbon nanomaterials (fullerene, carbon nanotube and graphene) are becoming key components of nanotechnologies for the development of complex functional nanostructures. Light fullerenes ($C_{60}$/$C_{70}$) are a hollow sphere/ellipsoid carbon molecule less than 1 nm in diameter, with $sp^2$ carbon atoms located on a curved surface at the vertices of a truncated icosahedron. They have unique physical properties, in particular optical and electrical. One of the remarkable properties of fullerene molecules is their ability to self-assemble over time in pure solvents to form clusters of various shapes and sizes [18,19], and the nature of the solvent plays an important role in this process [20]. Therefore, they have an excellent electron acceptor, high photosensitivity and high electron mobility [21,22]. The latter leads them to a range of applications, including photodetectors [23], sensors [24], solar cells [25], LEDs [26], and drug delivery [27].

Since the discovery of $C_{60}$ fullerene nanowhiskers ($C_{60}$NWs) by the Miyazawa group in 2001 [28,29], they have found applications in various fields. A poor solvent is added to a saturated well-dissolved solution of $C_{60}$ and a liquid-liquid interface is formed in the middle. As a result, a supersaturated solution is formed, $C_{60}$ embryo crystals are nucleated at the liquid-liquid interface, and long $C_{60}$NWs are synthesized. Although this method was initially "static" (without external influence), later "dynamic" (ultrasound, manual mixing, etc. effects) and other modified methods were developed [30,31]. Similarly, $C_{70}$ fullerene nanowhiskers ($C_{70}$NWs) structures were synthesized on the basis of $C_{70}$ fullerene in the same ways [32]. It is known that NWs formed on the basis of

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nanosized fullerenes are based on bottom-up technology. In this case, the regulation and control of the size and structure of the NWs is of great importance. In particular, when NWs synthesized in solution are transferred to the surface of a solid substrate, changes in their morphology occur. It should also be taken into account that the evaporation of droplets of fullerene solutions on the surface of a solid substrate leads to self-organization processes [33,34]. In this regard, there is a need to study the processes occurring in the volume of evaporation of droplets of fullerene solutions.

In this paper, we consider the synthesis of nanostructured $C_{70}$ fullerene whiskers on the surface of a substrate by evaporating a microvolume drop of $C_{70}$ solution. Experimental methods for controlling the geometric dimensions of the synthesized nanowhiskers are discussed.

## 2. Results

In our experiments, the shape of the initial drop of a fullerene solution with a volume of V≈40-50 μl on a wetted flat substrate is approximately described by a spherical cap (see Figure 1, left). It can be noted that drops of a fullerene solution throughout the entire duration of thermal evaporation always retain a constant area of the base of the drop. But the contact angle (φ) of the drop gradually decreases until it disappears. The fullerene drop is protected from convective air flows until complete evaporation; the drop thermal evaporation direction is perpendicular on the surface of the spherical cap. Due to the Marangoni effect along the "droplet-air" interface and the Rayleigh-Benard effect along the evaporating droplet volume (Figure 1, right), strong capillary flows appear and start the assembly of fullerene particles as well as the synthesis of different nanostructures based on them.

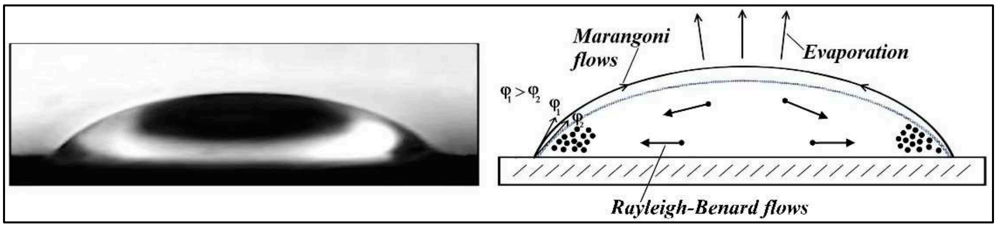


**Figure 1.** A photograph of the lateral microdroplet profiles of a $C_{70}$ solution (**left**) and a schematic representation of the appearing flows inside the evaporating droplet (**right**).

The SEM image of the structures formed during the evaporation of droplets of a $C_{70}$ solution in toluene on the substrate surface at room temperature (~24±1°C) is shown in Figure 2. Due to the constant base area of the microdroplet, during the entire thermal evaporation of the solvent, a trace of $C_{70}$ nanostructures remains along the base of the drop, similar to a coffee ring. An important role is played by the temperature gradient that occurs when the surface and near-surface layers of the droplet cool sharply as a result of intense toluene evaporation. It can be seen that after the complete evaporation of toluene from a microdroplet of the $C_{70}$ solution, large quasi-spherical $C_{70}$ aggregates formed on the surface of the optical glass substrate. At the same time, the average geometric dimensions in the diameter of $C_{70}$ aggregates were ~600 nm. The resulting $C_{70}$ aggregates are porous and consist of discrete intermediate nanoaggregates with sizes up to ~40÷45 nm in diameter.

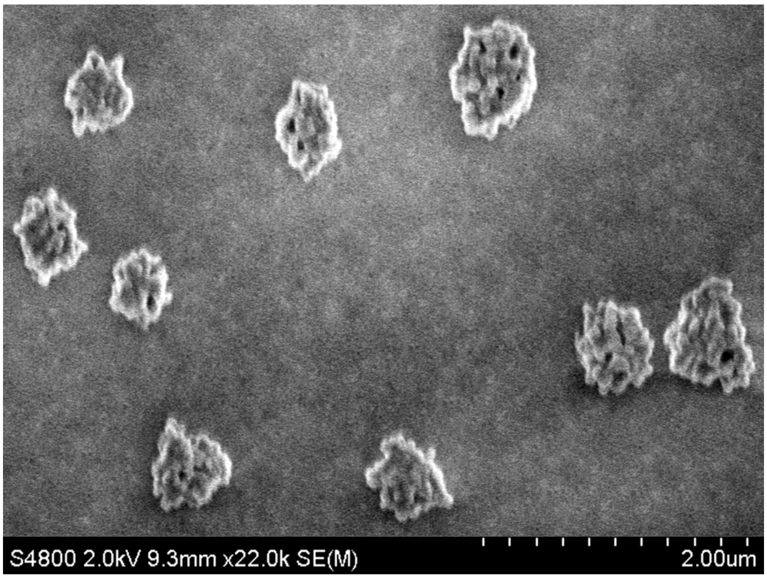


**Figure 2.** SEM image of $C_{70}$ aggregates formed by thermal evaporation of organic solvent from the volume of microdroplet of a $C_{70}$ solution at room temperature (~24±1°C). The initial concentration of fullerene $C_{70}$ in the solution was ~$1.1 \cdot 10^{-3}$ mol·L$^{-1}$.

We study the process of evaporation of a $C_{70}$ solution droplet on the substrate surface at different substrate temperatures in order to synthesize one-dimensional $C_{70}$ structures. When the K-8 optical glass substrate was heated to 28°C, nanostructured filaments (nanoviskers) of $C_{70}$ fullerene of optimal shape were synthesized on the substrate surface (see Figure 3). In this case the concentration of fullerene $C_{70}$ in the initial drop of the solution was ~$1.1 \cdot 10^{-3}$ mol·L$^{-1}$.

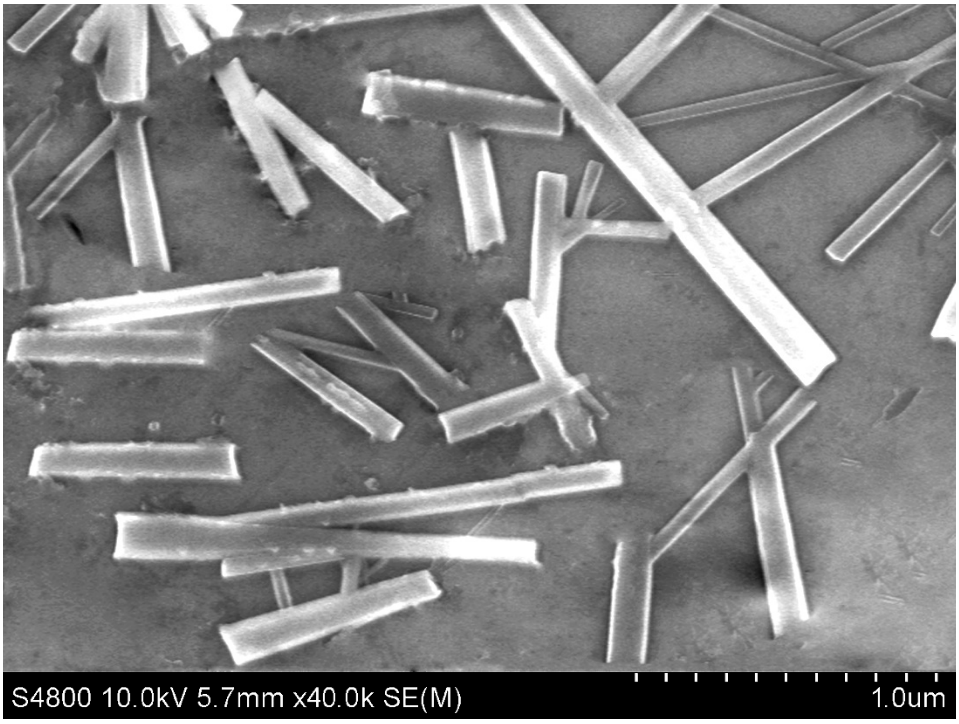


**Figure 3.** SEM-image of $C_{70}$NWs synthesized in a volume of the evaporating droplet of $C_{70}$ molecular solution on the smooth surface of a substrate at T≈28°C. The concentration of fullerene $C_{70}$ in the initial drop of the solution was ~$1.1 \cdot 10^{-3}$ mol·L$^{-1}$.

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In this case, the temperature gradient in the process of intensive evaporation of the solvent from a microdroplet at a temperature of 28°C makes it possible to overcome some of the energy difficulties in the formation of $C_{70}$NWs. We can observe X- and V-shaped $C_{70}$NWs were mainly synthesized in the volume of an evaporating drop of $C_{70}$ molecular solution on a substrate (see Figure 3)). The average geometric dimensions of $C_{70}$NWs are ~105 nm in width and ~750 nm in length. At the same time we can observe the maximum length and width of the resulting $C_{70}$NWs reached the values ~1.7 μm and ~200 nm, respectively.

SEM-image of $C_{70}$NWs synthesized on a surface of a horizontally located glass substrate, heated to T=36°C presented in Figure 4. In experiments with fixed concentration of $C_{70}$ (~$1.1 \cdot 10^{-3}$ mol·$L^{-1}$) in a drop of the working solution, the effect of increasing the temperature of the substrate on the ongoing processes of the evaporation drop was studied. It was established that an increase in the substrate temperature not only led to a more accelerated nucleation and growth of $C_{70}$NWs, but also to a noticeable increase in the final geometric dimensions of the synthesized $C_{70}$NWs. Wherein, the distribution of $C_{70}$NWs on the substrate surface is getting denser. At the same time the average length and width of the resulting $C_{70}$NWs reached the values ~1.8 μm and ~175 nm, respectively. The presented results proved that the size of nanowhiskers can be controlled by changing the substrate temperature at a fixed concentration of $C_{70}$ in the working drop.

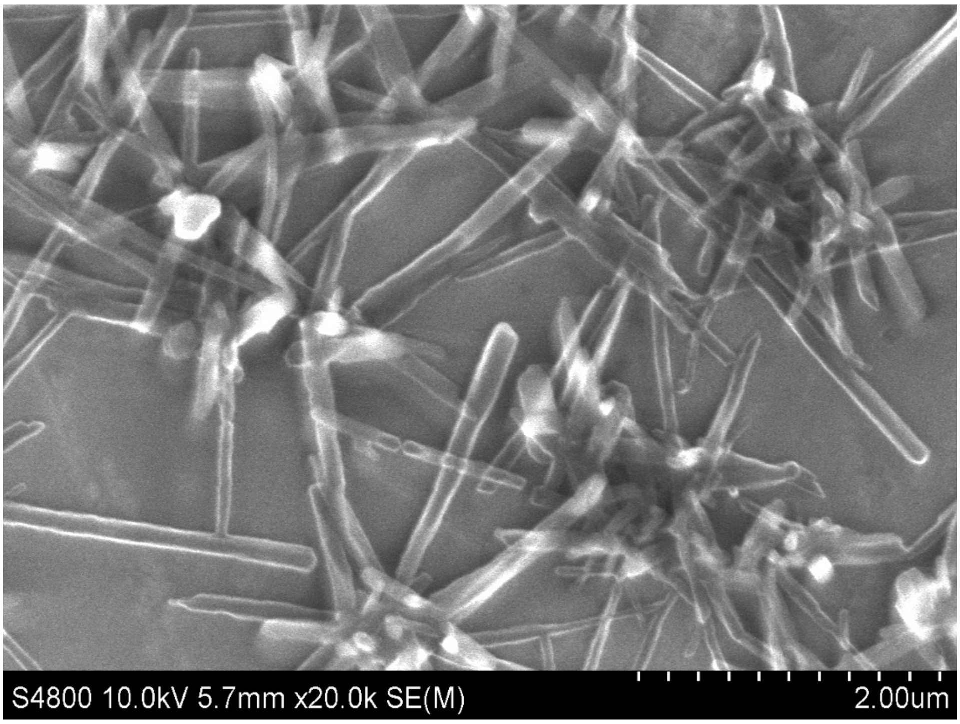


**Figure 4.** SEM-image of $C_{70}$NWs synthesized in the volume of evaporating droplet of $C_{70}$ molecular solution on the flat substrate at T≈36°C. The concentration of fullerene $C_{70}$ in the initial drop of the solution was ~$1.1 \cdot 10^{-3}$ mol·$L^{-1}$.

Under the same conditions, we studied the effect of the initial concentration on the size of the synthesized nanoparticles. Figure 5 presents SEM-image of nanostructured whiskers of $C_{70}$ fullerene synthesized on the smooth surface of a substrate heated to T≈36°C. An increase in the fullerene concentration (up to ~$1.5 \cdot 10^{-3}$ mol·$L^{-1}$) in the initial droplet led to a noticeable increase in the final $C_{70}$NW size. It is easy to observe that the longest $C_{70}$NWs has a size of ~28 micrometers in length, ~2 micrometers in width, as well as the shortest length and width are ~6 micrometers and ~200÷250 nm, respectively (Figure 5). So it was shown that the geometric dimensions of the $C_{70}$NWs can be controlled by changing the initial concentration of the fullerene solution.

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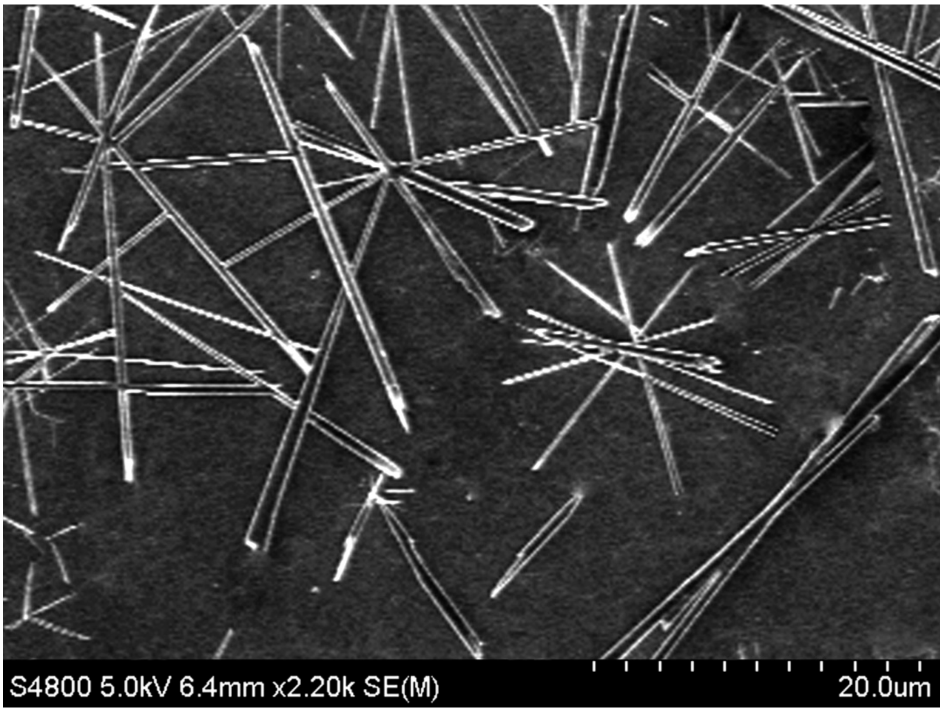


**Figure 5.** SEM-image of filamentous crystalline structures (nanowhiskers) of $C_{70}$ fullerene synthesized on the substrate surface at T≈36°C. The concentration of fullerene $C_{70}$ in the initial drop of the solution was ~$1.5 \cdot 10^{-3}$ mol·L$^{-1}$.

The experimental results reflecting the change in the geometric dimensions of the synthesized $C_{70}$NWs at fixed concentration of $C_{70}$ fullerene with different substrate temperatures presented in Table 1.

**Table 1.** Evolution of changes in the average sizes of synthesized $C_{70}$NWs depending on substrate temperature

| C/(mol·L$^{-1}$) [a] | T/(°C) [b] | Average length/μm | Average width/nm |
|---|---|---|---|
| | 28 | 0.75 | 105 |
| ~$1.1 \cdot 10^{-3}$ | 32 | 1.35 | 152 |
| | 36 | 1.8 | 175 |

[a] The $C_{70}$ concentration in a solution. [b] The substrate temperature (T) remains constant until the droplet is completely evaporated.

## 3. Discussion

We presented an experimental method for the synthesis of cost-effective and compatible $C_{70}$NWs in the volume of an evaporating droplet on a substrate. Our electron microscopic measurements confirm the formation of one dimensional $C_{70}$NWs during the evaporation of a drop on the surface of a substrate heated from 28°C. It was found that changing both the concentration of fullerene in the initial drop and the substrate temperature provides an opportunity to tune the geometric dimensions of $C_{70}$NWs to the desired value.

At a fixed concentration of $C_{70}$ (~$1.1 \cdot 10^{-3}$ mol·L$^{-1}$) in an initial drop, change in the substrate temperature from $T_1$=28°C to $T_2$=36°C led to a noticeable increase in the final geometric dimensions of the synthesized $C_{70}$NWs. In this case, the ratio of average length (~1.35 μm) to width (~152 nm) of the synthesized $C_{70}$NWs is about 9:1. At a fixed substrate temperature (T=36°C) with a relatively high concentration of fullerene (~$1.5 \cdot 10^{-3}$ mol·L$^{-1}$) $C_{70}$NWs with the largest length and width of ~28 μm and ~2 μm, respectively, were synthesized. It was shown that the method used is effective for the

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synthesis of micro- and nano-sized whiskers, which can be used for various purposes of the "bottom-up" technology.

## 4. Materials and Methods

In our experiments we used the high purity (~99.8%) powders of fullerene $C_{70}$ (Sigma-Aldrich, USA) as well as organic solvent – toluene ($C_6H_5CH_3$, Sigma-Aldrich, USA). The mixture of "toluene+$C_{70}$ powders", located in a hermetically sealed glass flask, was dissolved by continuous mechanical stirring at a frequency of ~1.5 Hz for 1.5 hours using a programmable laboratory magnetic stirrer of the MS-11H brand, WIGO, (Poland). Thereafter, the $C_{70}$ solution was sonicated for 15 min using an ultrasonic bath brand DC-120H. Further, dosed drops of the $C_{70}$ molecular solution were taken using a VITLAB dosing pipette (VITLAB GmbH, Germany).

Standard K-8 optical glass with a surface roughness of ≤7 nm was used as a substrate. Before each experiment, the surface of the used glass substrate was plasma cleaned at a nano level using a Plasma Cleaner device (Harrick Plasma, «PDC-002», USA).

We used a high-resolution scanning electron microscope (hereinafter SEM) brand JSM-IT200 (Joel, Japan) to establish the morphological features and determine the exact geometrical sizes of one-dimensional $C_{70}$NWs.

## 5. Conclusions

For the first time an evaporating drop method for synthesis of nanostructured $C_{70}$NWs based on the self-organization of $C_{70}$ molecules during thermal evaporation of toluene from $C_{70}$ droplets located on the surface of a flat glass substrate has been proposed and implemented. The optimal substrate temperature for the start of the synthesis of $C_{70}$ fullerene nanowhiskers in the volume of droplet evaporation was experimentally established. It was shown that the geometric dimensions of the synthesized $C_{70}$NWs can be controlled both by changing the $C_{70}$ concentration in the initial droplet and by changing the temperature of the substrate used. A selective synthesis of fullerene nanowhiskers was carried out. The results of this work can be used to predict and control the geometric dimensions of nanostructured whiskers of various kinds, which will have great potential in applications such as nano- and microelectronics, solar cells, nonlinear optics, sensors, and electromechanics.

**Author Contributions:** Conceptualization, U.K.M.; methodology, U.K.M.; investigation, U.K.M. and B.A.A.; writing—original draft preparation, U.K.M.; writing—review and editing, S.A.B. and U.K.M. All authors have read and agreed to the published version of the manuscript.

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**Conflicts of Interest:** The authors declare no conflict of interest.

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