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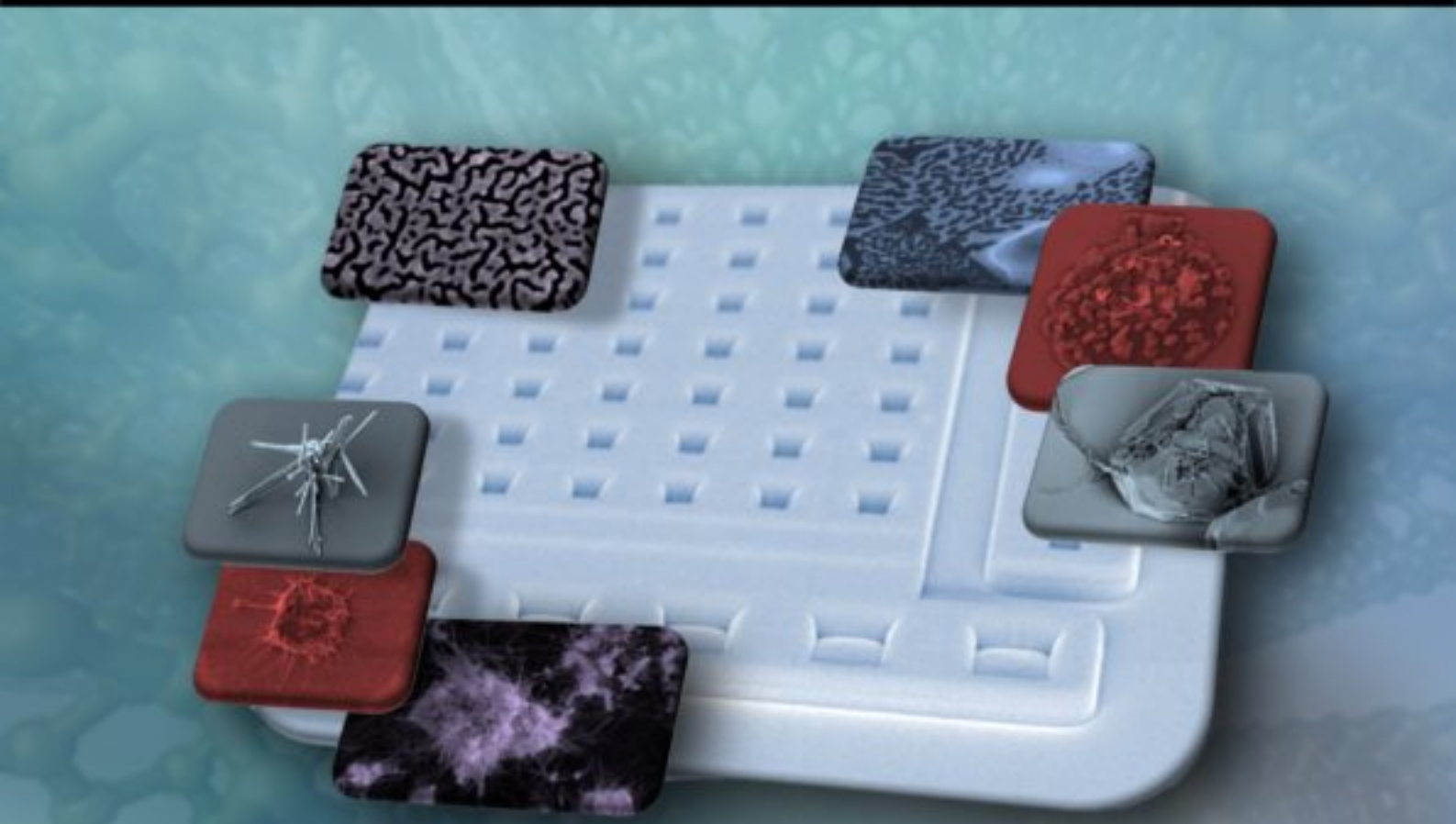

## Nanomaterials and their Composites: from Fabrication to Applications

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# Contents

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# Large-scale Synthesis of $WS_2$ Multiwall Nanotubes and their Dispersion, an Update

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**Abstract:** Recently, we have reported the synthesis of a pure phase of multiwall $WS_2$ nanotubes in substantial amounts. Nonetheless, the overall yield from the oxide powder to de-agglomerated nanotubes was not very high. To overcome this obstacle, the synthesis of the nanotubes was studied in further detail. This study and careful parameterization of the conditions within the reactor lead to further understanding of the growth mechanism and facilitate the synthesis of large amounts (50-100g/batch) of pure nanotubes with much higher overall yield. The majority of the nanotubes range from 10 to 20 micron in length and 50-120 nm in diameter. The new process produces loosely agglomerated nanotubes that can be easily dispersed in organic solvents and different polymer matrices. In view of its excellent mechanical properties, and its semiconducting characteristics, large number of applications could be anticipated for such nanotubes, especially in reinforcing variety of nanocomposites, sensors, actuators, etc. *Copyright © 2011 IFSA.*

**Keywords:** $WS_2$ inorganic nanotubes, Scale up, Synthesis, Dispersion, Nanocomposites.

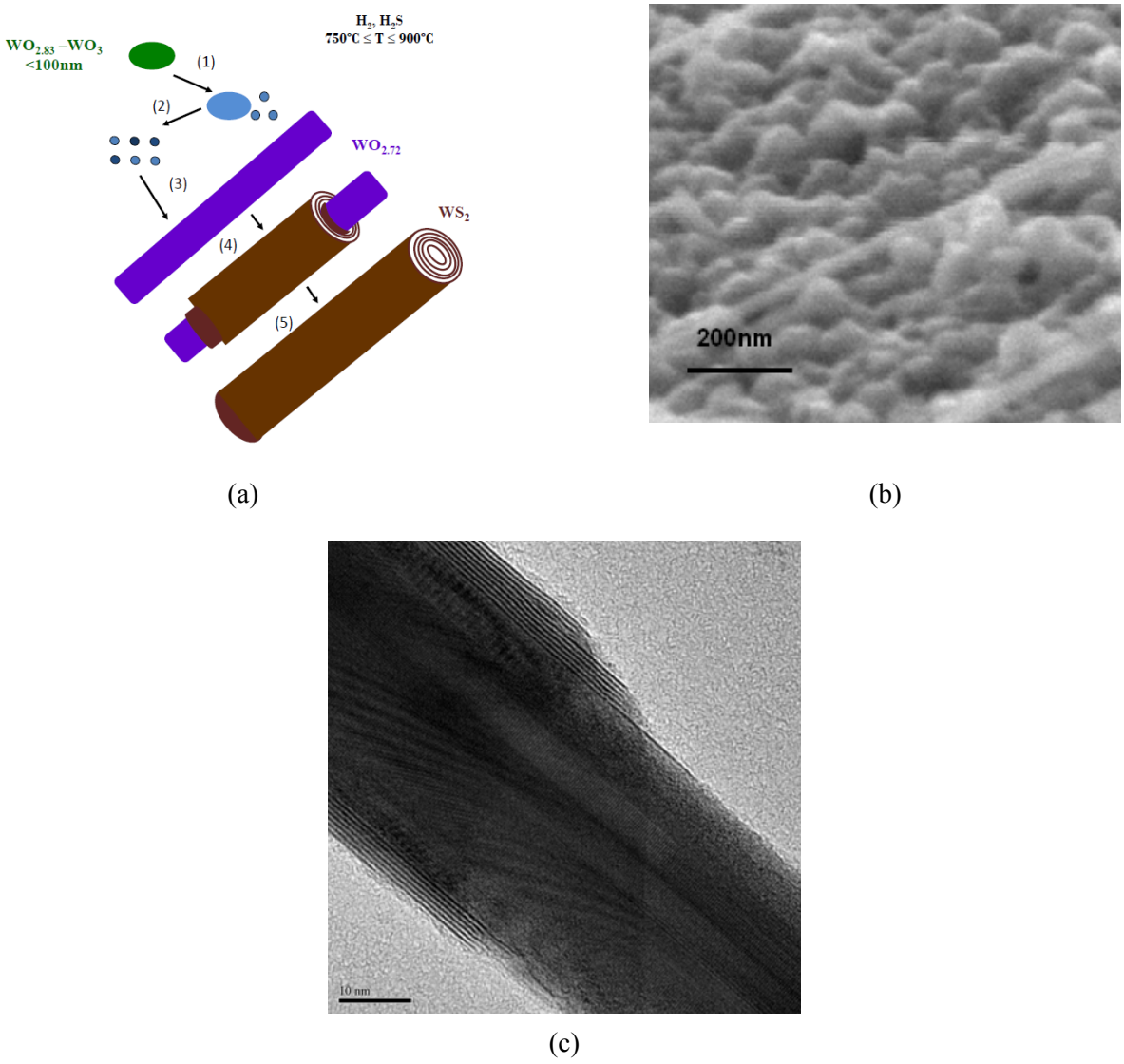
## 1. Introduction

The first report of $WS_2$ nanotubes and fullerene-like structures appeared in 1992 [1] and opened the doors for a new field of research combining inorganic solid state chemistry and nanomaterials/nanotechnology. Numerous reports were published describing the synthesis of different kinds of inorganic nanotubes (INT) and fullerene-like (IF) nanoparticles, and their potential applications, especially as solid lubricants. These studies led to the scale-up of the synthesis of IF-$WS_2$ nanoparticles and their commercialization as additives to variety of lubricating fluids [2]. Self-lubricating coatings containing these nanoparticles already found some commercial applications. Future medical devices have been demonstrated as well [3]. Various review papers have been dedicated to this field in recent years [4-7]. A number of groups reported progress in the synthesis of pure $WS_2$ and $MoS_2$ nanotubes [8-14]. Many of the synthetic approaches described in the literature were based on high temperature (> 700 °C) reactions of $H_2S$ or sulfur containing vapors with metal-oxide nanowires, metal-oxide nanoparticles or another metal precursor. The growth mechanism of a pure $WS_2$ nanotube phase using a horizontal reactor was described in previous studies [9, 10]. In one such approach [9], short (~50nm) nanorods of tungsten oxide were prepared ahead of time in a separate reaction. It was found to consist predominantly of the suboxide phase $W_{20}O_{58}$ ($WO_{2.9}$). Subsequently the nanorods were annealed at temperatures exceeding 800 °C in $H_2S/H_2$ atmosphere. Elongation of the oxide nanowhiskers and their gradual oxygen/sulfur exchange resulted in tens mg of $WS_2$ nanotubes of several microns in length [9]. The growth mechanism of the nanotubes was investigated in great detail. It was suggested that the growth of the nanowhiskers is promoted by the formation of an intermediate volatile $(WO_{3-x}*H_2O)_n$ oxi-hydrate phase [15]. Subsequent efforts to synthesize $WS_2$ nanotubes in the fluidized bed reactor [FBR] were reported [14]. However the content of the nanotubes in the reaction product did not exceed 5 %. Here, spontaneous growth of the nanotubes was observed without the oxide nanowhisker intermediate phase.

Recently, a one-pot synthesis of large amounts (50-100 g) of a pure $WS_2$ nanotube phase was reported [16]. This synthesis employed a new design of the fluidized bed reactor. In this process a sequence of reactions leads to the nanotube synthesis. In the first step agglomerated oxide nanospheres are converted into long oxide nanowhiskers. Subsequently, the oxide nanowhiskers are converted into tungsten sulfide nanotubes by an outside inwards oxygen/sulfur exchange reaction.

The proposed growth mechanism [16] shown schematically in Fig. 1a is described according to the following steps: The oxide precursor mixture of non-volatile oxide phases $WO_{2.83}$–$WO_3$ undergoes reduction in (1) by hydrogen gas producing a volatile oxide phase; in (2) the oxide vapor undergoes additional partial reduction to the non-volatile phase; in (3) the non-volatile phase serves as a nucleation center for the nanowhisker growth into $WO_{2.72}$ phase; in (4) sulfidization starts from the middle of the oxide nanowhiskers, and oxygen to sulfur exchange progresses towards the end of the nanowhiskers (fast step) and inwards (slow-diffusion controlled reaction). This process leads to a gradual consumption of the oxide core creating a hollow multiwall $WS_2$ nanotube (5). Figs. 1b shows scanning electron microscopy (SEM) and 1c transmission electron microscopy (TEM) images of the precursor oxide and a nanowhiskers/nanotube in step (4), respectively.

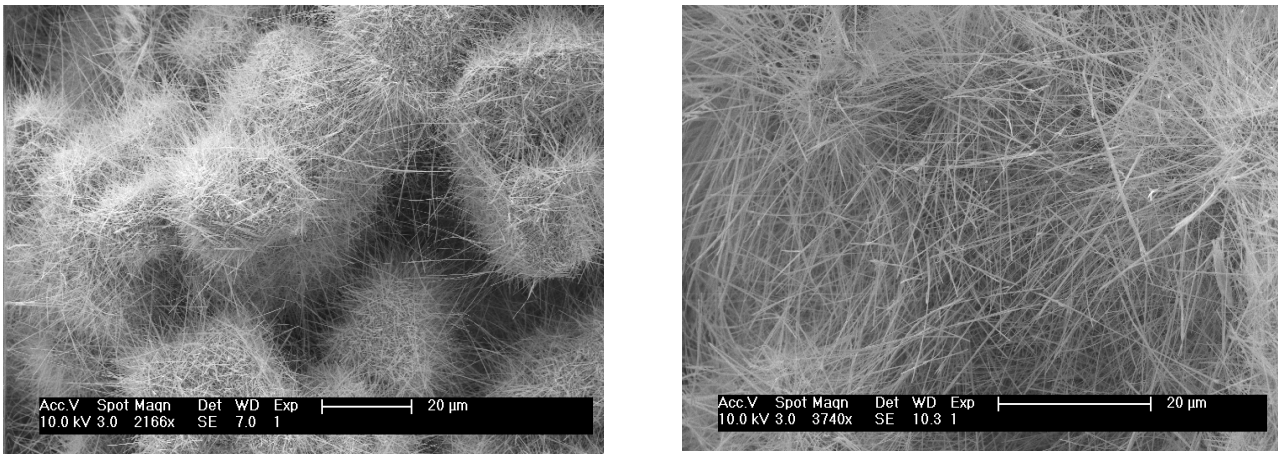
Typical scanning electron microscopy (SEM) images of the nanotubes are shown in Fig. 2a and b. The powder appears as agglomerates where the nanotubes grow outwards from a center somewhat like to a porcupine. The typical diameter and length distribution of the nanotubes is 50-120 nm and 10-20 micron, respectively. Transmission electron microscopy (TEM) image of an individual nanotube is shown in Fig. 3. Fig. 4 shows a bottle containing 1.2 kg of the pure (agglomerated) nanotubes.




(a) (b)

(c)

**Fig. 1.** (a) Schematic rendering of the growth mechanism of the multiwall $WS_2$ nanotubes; (b) SEM image of the oxide precursor; (c) TEM image a typical structure observed in stage (4) of the INT growth.




(a) (b)

**Fig. 2.** SEM images of the highly agglomerated $WS_2$ nanotube phase (two magnifications), which were reported earlier [16].

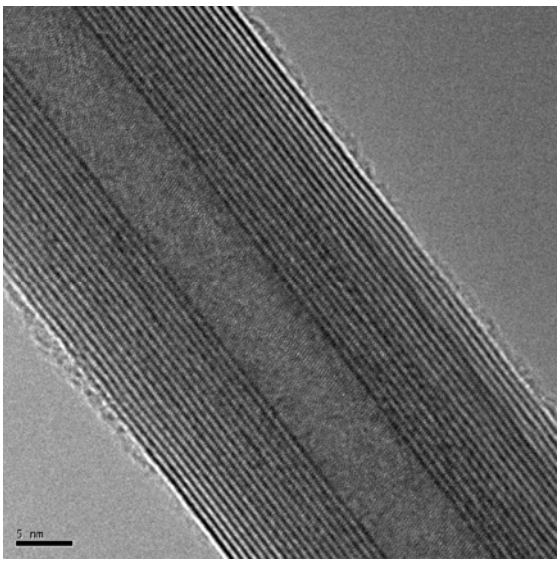



**Fig. 3.** TEM image of a typical multiwall $WS_2$ nanotube.


**Fig. 4.** A bottle containing some 1.2 kg of $WS_2$ nanotubes.

The synthesis of a pure nanotube phase was accomplished, though the overall yield of the entire process was not very large. Firstly, only 10 % of the original tungsten oxide powder, which was fractionized by sieving, was a suitable precursor for this reaction. Of this oxide only 50 % was converted into multiwall $WS_2$ nanotubes. Furthermore, the nanotubes were found to be highly agglomerated and were tightly bound to the center of the agglomerate. A mild sonication procedure was performed in order to separate them into individual nanotubes without their being damaged and fractured. The yield of this tedious process was not larger than 10%. The rest of the powder remain agglomerated and could not be used any further. Therefore, effectively only 0.5% of the as-purchased oxide powder was converted into a useful product.

A renewed effort was therefore undertaken to increase the overall yield of the pure (>95 %) multiwall $WS_2$ nanotubes phase production. In fact, in the current process 80 % (compared to 0.5 % in the previous process) of the as-purchased tungsten oxide powder is converted into loosely bound pure phase of $WS_2$ nanotubes. This accomplishment paves the way for their facile dispersion into a variety of nanocomposites. The new process is also amenable to a genuine scaling-up and future large scale production. Given their excellent mechanical behavior [17-20] and their proven record in reinforcing polymer composites [21, 22], this progress can translate into numerous applications of the current product.

A field of application currently receiving a lot of attention is polymer nanocomposites, and more specifically polymer nanocomposites which are reinforced with inorganic fullerene-like nanoparticles, mostly IF-$WS_2$ and lately also IF-$MoS_2$ and nanotubes thereof. A partial list of polymer nanocomposites which have been studied includes: Polyacetal [23] and different epoxies [21, 23, 24]; isotactic polypropylene (iPP) [25]; Poly-ether-ether-ketone (PEEK) [26], Polyphenylene sulfide (PPS) [27] and more. It was found that mixing small amounts (less than 2 %) of the nanoparticles into the polymer matrix leads to substantial improvement in different mechanical properties (up to 100 %). Significant improvements were observed in the storage modulus; shear and peel strength, tribological behavior, etc, without sacrificing its fracture toughness and strain. Improved behavior was also noticed in the nucleation kinetics, crystallization (thermoplastic polymers) and thermal stability; these observations suggest numerous applications for reinforced polymer nanocomposites.

## 2. Experimental Methods

The synthesis was carried out in a quartz-made FBR. The synthetic conditions were guided by those of Ref. 7. The precursor for this reaction consisted of a mixture of different $WO_x$ ($2.83 \leq x \leq 3$) phases and morphologies. For a more effective use of the precursor and facile dispersion of the resulting nanotubes, a non-agglomerated oxide powder was used in the present study. It was reacted with $H_2S$ and $H_2/N_2$ (forming) gas at 800-950 °C. To follow the growth mechanism of the reaction, the process was interrupted in different stages of the reaction, by replacing the $H_2S$ and forming gas flows with nitrogen gas and slow cooling-down of the reactor. A special effort was devoted to the dispersion of the nanotubes. Different solvents and mixing techniques were examined.

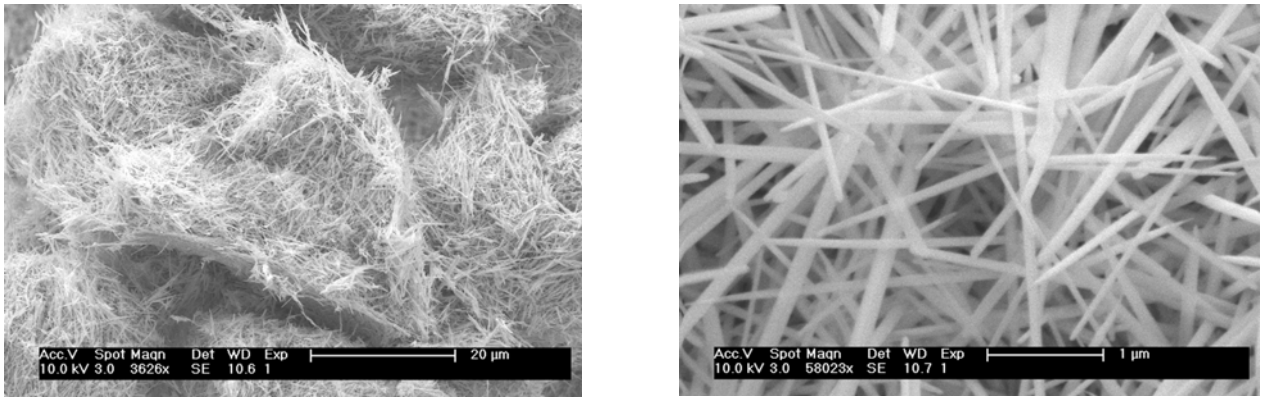
A vertical theta-theta diffractometer (TTRAX III, Rigaku, Japan) equipped with a rotating Cu anode operating at 50 kV and 240 mA was used for X-ray powder diffraction (XRD) studies. The measurements were carried out in the reflection Bragg-Brentano mode within the range of 10°-70° of 2 theta angles. XRD patterns were collected by a scintillation detector.

The following electron microscopes were used in this work: Scanning electron microscope (SEM) model LEO model Supra, 7426. Energy dispersive x-ray spectroscopy (EDS) model Oxford INCA. Transmission electron microscope (TEM) model Philips CM120 TEM operating at 120 kV and equipped with an EDS detector (EDAX-Phoenix Microanalyzer). For electron microscopy and analysis the collected powder was sonicated in ethanol and placed on a carbon-coated Cu grid (for TEM) or on lacy carbon-coated Cu grids (for high resolution TEM (HRTEM) and electron energy loss spectroscopy (EELS).

## 3. Results

Figs. 5a and b show low and high magnification SEM images, respectively, of the nanotubes obtained via the revised synthetic procedure. The nanotubes are found to be loosely bound. In contrast to the

previous synthetic route (see Figs. 2), the newly synthesized nanotubes are not arranged in the form of "porcupines" with their edges tethered strongly to the center. The new nanotubes can be easily dispersed according to the scheme shown in Fig. 6. The nanotubes left in the sediment undergo repeated treatment, until most of the powder is dispersed. To avoid severe damage and shortening of the nanotubes, a mild ultrasonic treatment was used. Fig. 7 shows SEM image of an assortment of dispersed nanotubes, which demonstrate that the length and morphology of the dispersed nanotubes are essentially unchanged with respect to the pristine material. This observation is further supported by XRD analysis (see Fig. 8) which showed that the crystallinity of the synthesized nanotubes is high and is not greatly affected by the sonication process.




(a) (b)

**Fig. 5.** SEM images of $WS_2$ nanotubes (two magnifications) obtained according to the revised route with high yields.

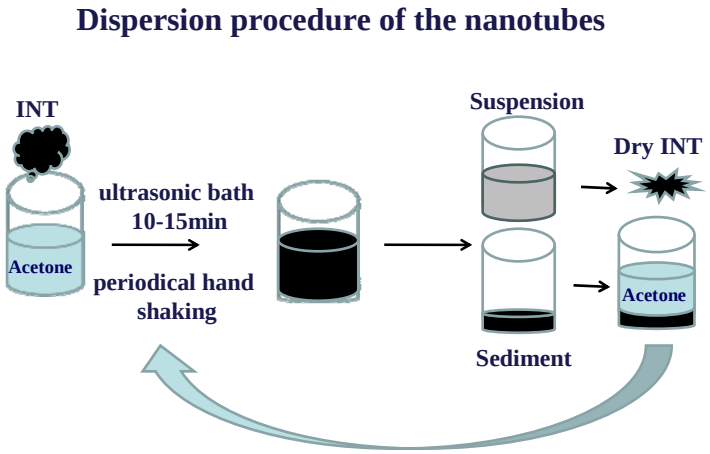



**Fig. 6.** Schematic rendering of the process used for the dispersion of the nanotubes. Acetone or other organic solvent could be used in this procedure.

The progress in synthesizing the nanotubes can lead to the development of new nanocomposites with improved mechanical behavior. Fig. 9 shows an optical microscopy image of the nanotubes in silicone adhesive. The optical transparency of the polymer matrix verifies that nanotubes are well dispersed and may be partially shortened during mixing with polymer.

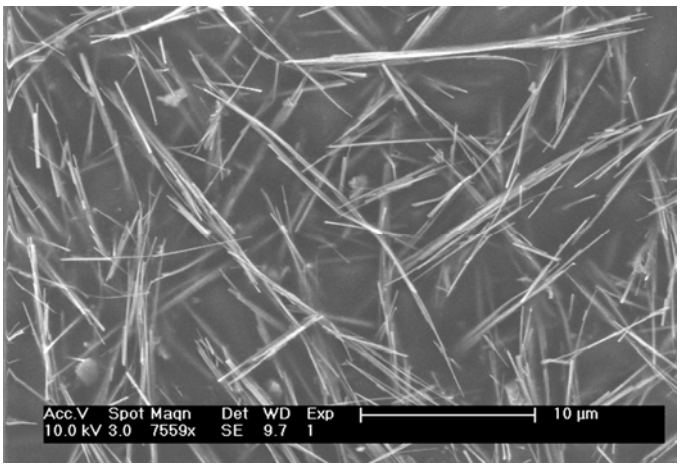



**Fig. 7.** SEM image of the dispersed $WS_2$ nanotubes.

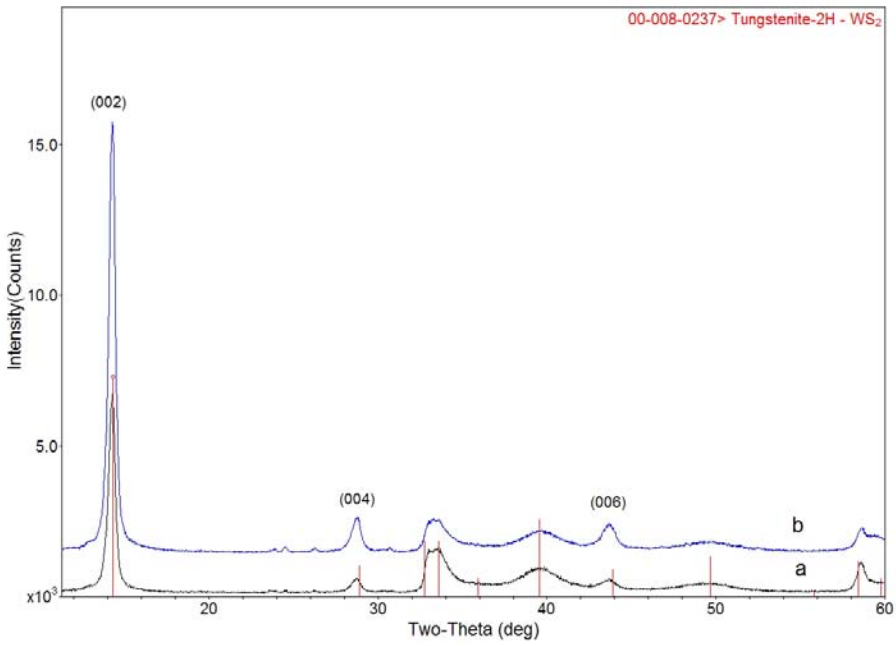



**Fig. 8.** XRD analysis of (a) as synthesized and (b) dispersed $WS_2$ nanotubes.

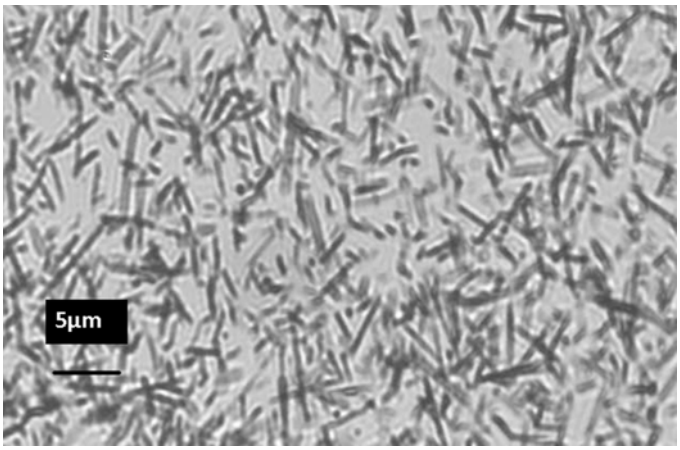



**Fig. 9.** Optical microscopy showing the nanotubes dispersed in silicon (Silastic J) adhesive.

A few recent studies have demonstrated the reinforcing effect of the $WS_2$ nanotubes on various polymer nanocomposites [21, 28]. These early studies are indicative of the large commercial potential of such nanotubes as reinforcing element in polymer nanocomposites. In particular, the embedding of semiconducting nanotubes in transparent electrospun PMMA nanofibers [28] could suggest various applications of such fibers as elements for health-monitoring of structural elements and for chemical sensors.

## 4. Conclusions

A new synthetic approach has been developed for the large-scale (50-100g/batch) synthesis of pure (>95 %) multiwall $WS_2$ nanotubes phase. In contrast to the previous synthetic route reported in Ref. 14, the present synthesis is able to exploit all of the tungsten oxide powder; the nanotubes are obtained as loosely bound aggregates and a facile dispersion process has been developed. Not only has the overall yield of this process been increased from 0.5% to 80%, but the dispersed nanotubes also preserve their high crystalline structure which is immensely important for future applications. Indeed recently a number of polymer nanocomposites were successfully reinforced by incorporating no more than 1% of the multiwall $WS_2$ nanotubes into their matrix.

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