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Comparison of Synthesis and Purification of Carbon Nanotubes by Thermal Chemical Vapor Deposition on the Nickel-Based Catalysts: $NiSio_2$ and 304-Type Stainless Steel

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Abstract: In this study by Thermal Chemical Vapor Deposition (TCVD) method, the synthesis of carbon nanotubes (CNTs) and temperature affects on the grown CNTs on the $NiSio_2$ catalyst and 304-type stainless steel was investigated. The purification of the grown CNTs on the 304-type stainless steel was also investigated in this study. The synthesis and purification of the samples were characterized by scanning electron microscope (SEM), transmission electron microscope (TEM), termogravimetric analysis (TGA) and Raman spectroscopy. Our obtained result by SEM and TEM shows that densities of CNTs growth on the 304-type stainless steel are more than the CNTs growth on the $NiSio_2$ catalyst and both of them have a bamboo structure. The synthesis of CNTs in the range 750 to 850°C are also investigated in this study. Optimum temperature for synthesis of CNTs on the $NiSio_2$ catalyst is about 825°C and for the 304-type stainless steel is about 750°C. The purification process includes oxidation in the range 450to 750°C, washing with 5 mol hydrochloric acids, disperse with ultrasonic and filtration. Our obtained result by TGA indicates that the CNTs burn in 600°C. A comparison of the Raman spectroscopy before and after of purification shows that the D-band shape is less than the G-band that due to the samples purified. Hence, the reported 304-type stainless steel provides a good sample for synthesis of CNTs by TCVD method.

Key words: Synthesis, purification, catalyst, carbon nanotubes, oxidation, stainless steel

INTRODUCTION

Carbon products in tubular form, called carbon filaments, first observed when electron microscopes came into wide used around (Tibbetts, 1984). Carbon nanotubes (CNTs) can be considered as a result of rolling graphic layer thus obtain carbon cylinders. Their diameters are multi nanometers and their lengths are multi micrometers (Wang *et al*., 2001).

There are three major methods to grow CNTs with the large scale: arc discharge, laser ablation and Chemical Vapor Deposition (CVD) among this methods the CVD method, in witch a hydrocarbon is pyrolyzed over a catalyst metal, has been attracting extensive attention (Jung *et al*., 2004).

Recently some authors investigate on the synthesis of CNTs by TCVD. The intermetallic catalyst for CNTs growth by thermal chemical vapor deposition method has been reported (Chen *et al*., 2006). The goal of such research has been to introduce the methane conversion and carbon yield of the Chemical Vapor Deposition (CVD) reaction suggests that the optimum reaction conditions of the formation of Multi-Wall Carbon Nanotubes (MWCNTs) can be obtained by using a 50 mg of nano-MgNi alloy under pyrolysis of the pure CH_4 gas with the flow rate about 100-120 cm^3 min^{-1} at 650°C for 30 min. There are many ways for synthesis of CNTs by CVD according to required energy source for carbonic gas molecule energy transition such as TCVD, Catalytic Chemical Vapor Deposition (CCVD), Hot Filament Chemical Vapor Deposition (HFCVD) and Plasma Enhanced Chemical Vapor Deposition (PECVD) has been reported.

In another report, the catalyst in the CCVD of CNTs gives (Dupuis, 2005). In this study metals used to catalyze CNTs for mation are most often transition metals, in particular iron, cobalt and nickel. Addition of other components to or mixture of transition metals generally

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exhibits better results in terms of catalytic activity and CNT quality. The peculiar ability of transition metals to catalyze CNT formation is mostly linked to their catalytic activity for the decomposition of carbon compounds, their ability to for carbides and the possibility for carbon to diffuse through and over the metals extremely rapidly. The large majority of gropes working with nickel-based catalyst interesting reports growth of multi wall nanotubes (MWNTS), many of them with bamboo structure, or fibers.

In a report purification and isolation of single walled carbon nanotubes (SWNTs) was given (Gregan *et al*., 2004). This study involves the purification and isolation of arc-discharge and HiPco SWNTs using two different systems. A method for the synthesis of Single Wall Carbon Nanotubes (SWNTs), is high pressure carbon monoxide gas (HiPco). In this method, the average diameter of CNTs controlled with the gas pressure. The first involve the use of conjugated such PmPV to selectively isolate certain tubes. The second uses SWNT as temperature for organic molecules such as terphenyl and anthracene.

In another report, purification of multiwalled carbon nanotubes by annealing and extraction based on the difference in van der waals potential was given (Zhang *et al*., 2006). The efficient synthesis of carbon nanotubes over rare earth zeolites by Thermal Chemical Vapor Deposition (TCVD) at low temperature has been reported by Yu *et al*. (2006). The goal of such research has been to introduce the highly efficient synthesis of CNTs using TCVD method below 450°C, even at 330°C. The CVD method for synthesis of CNTs, due to their inherent simplicity and low cost they have found many industrial applications (Mamun *et al*., 2009; Esfandiari *et al*., 2008). The primary contribution of the present study is the comparison of synthesis and purification of CNTs by TCVD on the Nickel-based catalysts: NiSio2 catalyst and 304-type stainless steel.

MATERIALS AND METHODS

The reported experiment conducted in Carbon Laboratory as part of research program of the Sharif University of Technology for the period of 2008-2010. In this present investigation, the CNTs were synthesized via a thermal decomposition of Liquid Gas (LG) in contact with the Nickel-based catalyst. The $NiSio_2$ catalyst and 304-type stainless steel were two kinds of catalysts used for growth of CNTs. The 304-type stainless steel stead was the substrate that contained Nickel grains employed as catalysts nanoparticales. Preparation process of $NiSio_2$ described as follows. First n (100) type silicon wafer used as substrate was washed with Aston and Ethanol. After drying, two or three droplets dispersed $NiSio_2$ in Ethanol's sputtered on silicon wafer. Then dried wafer and 304-type stainless steel were loaded to the heated furnace. The samples most be put in the central furnace carefully.

The LG which is an inexpensive Hydrocarbon gas was used as a precursor for the first time in this experiment. On the other hand, we did not use more dilute or carrier gases therefore, in comparison with conventional TCVD, our approach become simpler. Furnace temperature and flow rate were 750 and 40°C sccm. The flow of gas continued for 20 min then the turned off. When temperature was decreased to 300°C, the flow of gas was stopped. Then samples carried out of furnace.

In following, the grown nanotubes on the 304-type stainless steel were used for purification. The purification process includes oxidation in the range 450 to 750°C, washing with 5 mol hydrochloric acids, disperse with ultrasonic and filtration. During the oxidation process, first the nanotubes. After oxidation process, the samples were washed by hydrochloric acid in different periods of time from one to 5 h in ultrasonic position. After oxidation, the metal particles that has been placed in carbon nanotubes were heated and separated in to layers and solved in acid. Finally filtration process was done.

RESULTS

Figure 1 shows the SEM graphs from 750 to 850°C temperature for the grown CNTs on the $NiSio_2$ catalyst. As shown in Fig. 1, the density of CNTs increased by increasing in temperature. Figure 1a shows the SEM graph of grown CNTs in 750°C temperature. As can be seen in Fig. 1a the density of grown CNTs is little. The SEM graph of grown CNTs in 775°C temperature has been shown in Fig. 1b. As shown in Fig. 1b, the density of the CNTs increases. Figure 1c and d, respectively, show the SEM graphs of the grown CNTs on the $NiSio_2$ catalyst in 800 and 825°C temperature. The comparison between different temperatures indicates that the density of CNTs is highest in 825°C and is lowest in 750°C. Figure 1e shows when temperature was increased from 825 to 850°C did not growth CNTs. The SEM graphs in different temperatures for the grown CNTs on the 304-type stainless steel have been shown in Fig. 2a-e. The SEM graph of grown CNTs in 750°C temperature has been shown in Fig. 2a. As can be seen in Fig. 2a the density of grown CNTs is notable. Figure 2b shows the SEM graph of grown CNTs in 775°C temperature. As shown in Fig. 2b, the density of the CNTs decreases. Figure 1c-e, respectively, show the SEM graphs of the grown CNTs

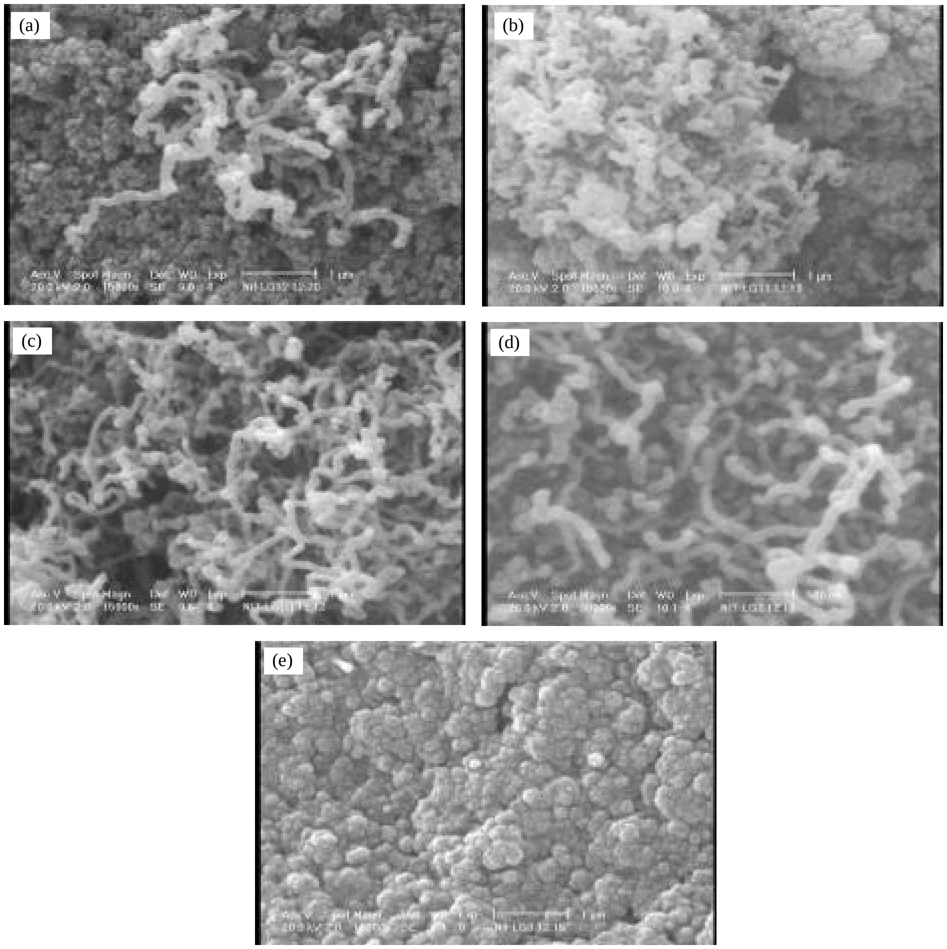

Fig. 1: SEM graphs of grown CNTs on the $NiSio_2$ catalyst in different temperature. (a) 750°C, (b) 775°C, (c) 800°C, (d) 825°C and (e) 850°C

on the 304-type stainless steel in 800, 825 and 850°C temperature. It is considerable that by increasing in the temperature from 750 to 850°C, the CNTs density decreased.

Initial measurement indicates that the CNTs density is very sensitive to the temperature. Figure 1 and 2 show that the optimum temperature for the grown CNTs on the $NiSio_2$ catalyst is about 825°C and for the 304-type stainless steel is about 750°C. Therefore, the temperature effect is notable in CNTs growth .

Celemex Measurement Analyzer (CMA) measured the average diameter of CNTs. Figure 3 shows the average diameter of grown CNTs on the $NiSio_2$ catalyst in 750°C. As can be seen in Fig. 3, the average diameter of grown CNTs is about 119 nm. The average diameter in 825°C temperature for the grown CNTs on the $NiSio_2$ catalyst has been shown in Fig. 4. In Fig. 4, the average diameter of grown CNTs is about 58 nm. Comparison of Fig. 3 and 4 shows that by increasing in temperature the average diameter of grown CNTs on the $NiSio_2$ catalyst decreased. Figure 5 shows the average diameter in different temperature for the grown CNTs on the $NiSio_2$ catalyst and the 304-type stainless steel. In Fig. 5, by increase in temperature, the average diameter of grown CNTs on the 304-type stainless steel increased. The average diameter in 750°C for the grown CNTs on the 304-type stainless steel is about 58 nm and for 850°C is about 125 nm.

It should be considered that in the CVD method, the size of the nanotubes directly dependant on the size of the catalyst particles (Dupuis, 2005). High temperature on the $NiSio_2$ samples allowed catalyst nanoparticles breaking to small sizes. Therefore, in the high temperature the grown CNTs has been high density. In 304-type stainless steel by increasing in temperature, the Nickel deposition size increased. Therefore, the CNTs diameter became larger.

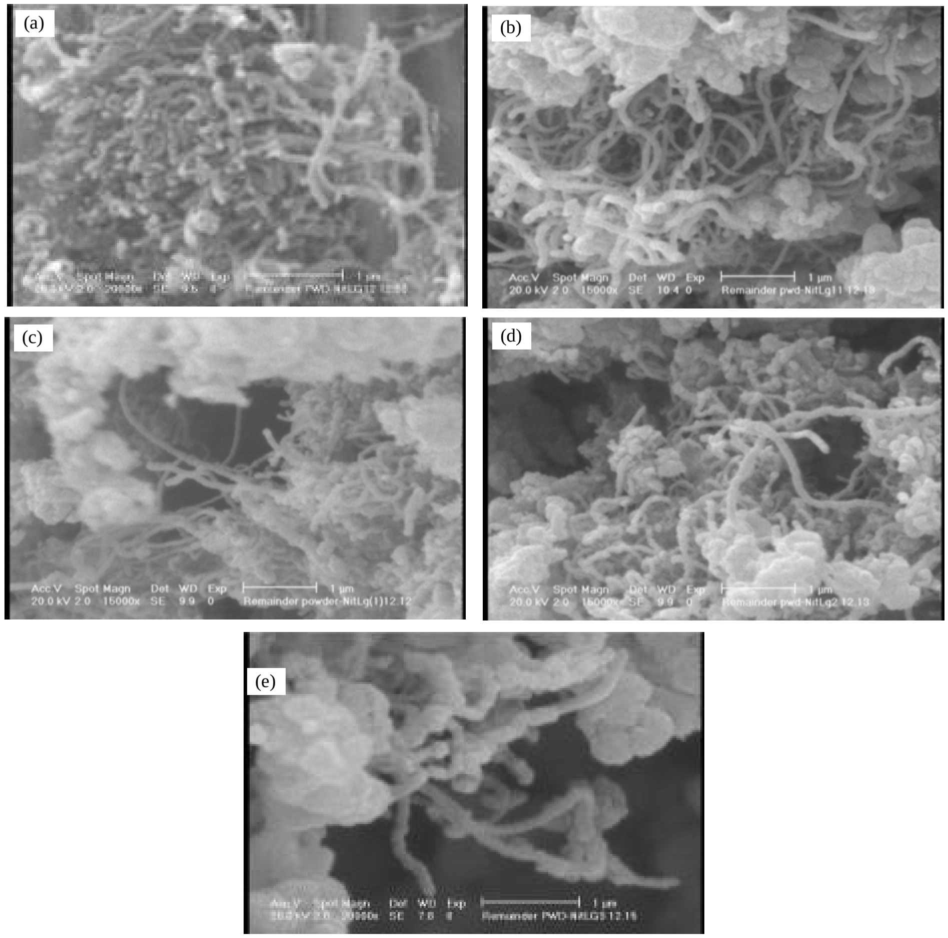

Fig. 2: SEM graphs in different temperature for the grown CNTs on the 304-type stainless steel. (a) 750°C, (b) 775°C, (c) 800°C, (d) 825°C and (e) 850°C

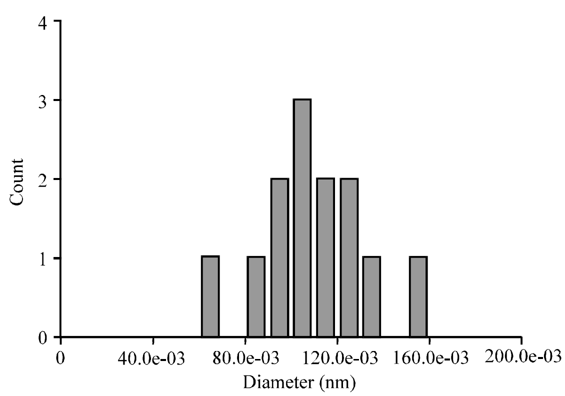

Fig. 3: Average diameter of grown CNTs on the $NiSio_2$ catalyst in 750°C

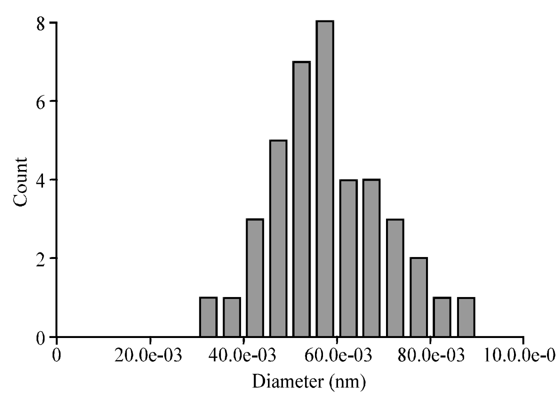

Fig. 4: Average diameter of grown CNTs on the $NiSio_2$ catalyst in 825°C

The TEM graph for the grown nanotube on the $NiSio_2$ catalyst in 800°C has been shown in Fig. 6. In Fig. 6, the grown nanotubes have a bamboo structure and curved morphology. The high diameter of CNTs indicates

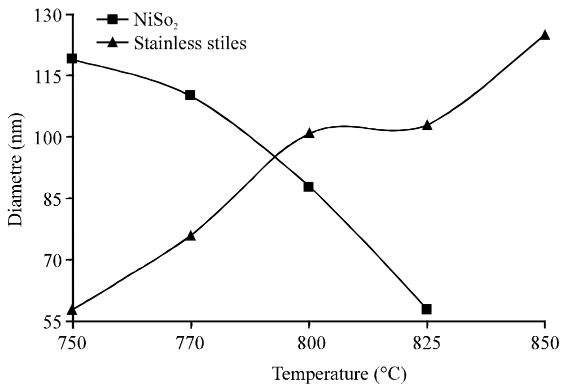

Fig. 5: Average diameter in different temperature for the grown CNTs on the $NiSio_2$ catalyst and the 304-type stainless steel

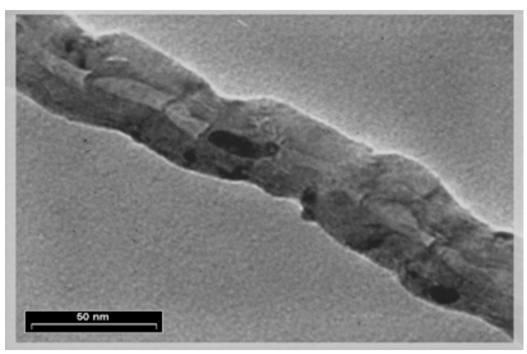

Fig. 6: TEM graph for the grown nanotube on the $NiSio_2$ catalyst in 800°C

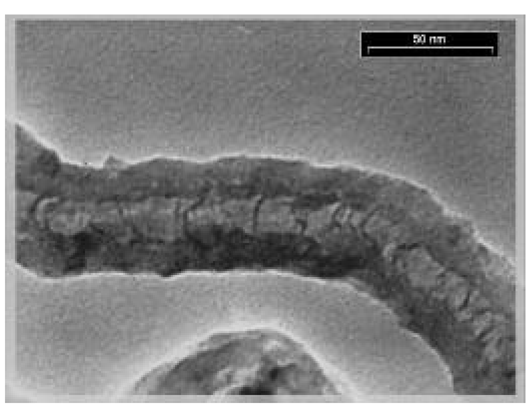

Fig. 7: TEM graph for the grown nanotube on the 304-type stainless steel in 800°C

that the CNTs were multi wall carbon nanotubes (MWCNTs). Figure 7 shows the TEM graph for the grown nanotube on the 304-type stainless steel in 800°C.

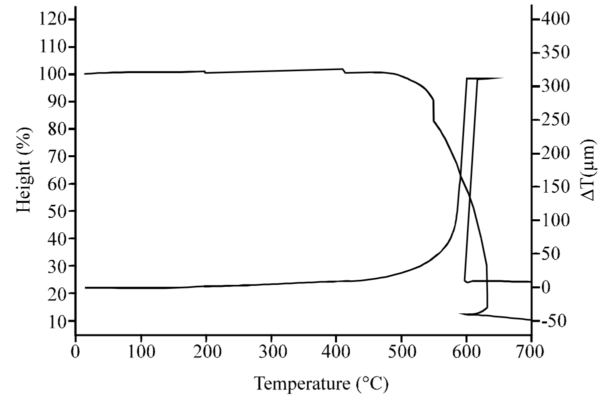

Fig. 8: TGA and DTA graph of CNTs before the purification

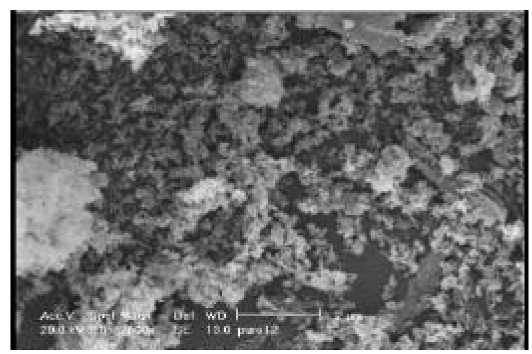

Fig. 9: SEM graph for the grown nanotubes on the 304-type stainless steel in 600°C oxidation

Figure 6 and 7 show that the grown nanotubes on the samples have a same structure and both of them are MWCNTs. A comparison of figures shows that the curved morphology of CNTs on the 304-type stainless steel is more than the $NiSio_2$ catalyst.

In following, the grown nanotubes on the 304-type stainless steel purified. Figure 8 shows the TGA and DTA graph of CNTs before the purification. The DTA graph is differential of the TGA graph. As can be seen in Fig. 8, until to 550°C temperature the DTA graph variation was zero and in the rang of 550 to 600°C the samples weight decreased sharply. Therefore, burning of CNTs was in 600°C. The SEM graph in Fig. 9 shows with temperature increasing up of 600°C the CNTs burned.

For CNTs purification carried out oxidation in 450, 500, 550 and 600°C. Then samples were washed in different times for 1, 3 and 5 h by ultrasonic. The SEM graph of different samples displays that the optimum temperature is 450°C with 2 h oxidation. Suitable duration for acid washing is 5 h. Figure 10 shows the SEM graphs before and after purification. The SEM graphs before

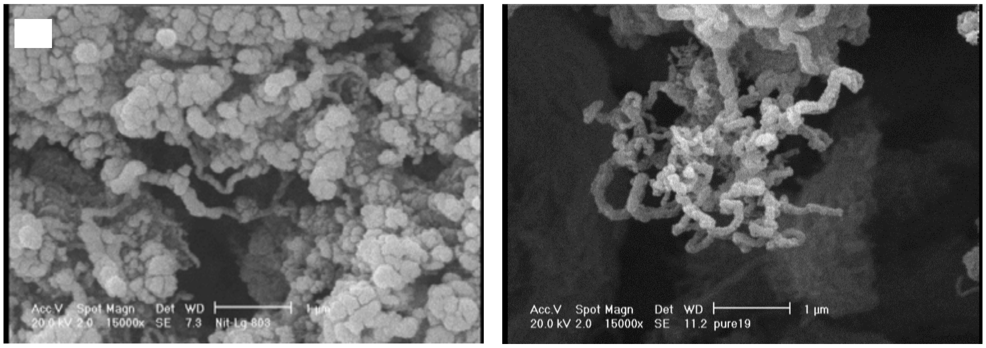

Fig. 10: SEM graphs for the grown nanotubes on the 304-type stainless steel (a) before purification and (b) after purification

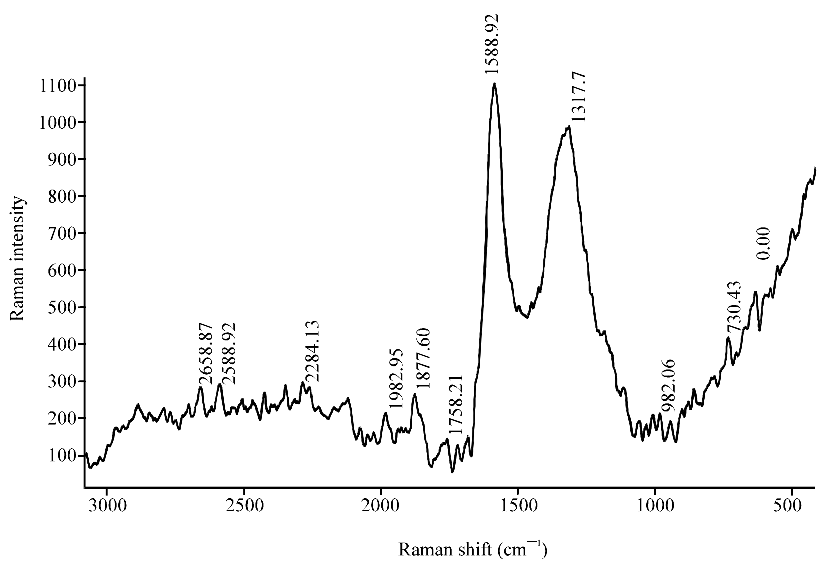

Fig. 11: Raman spectroscopy after purification for the grown nanotubes on the 304-type stainless steel

purification as shown in Fig. 10a. Obviously, in impurity samples was seen the nanotubes covered in amorphous carbon layer. Figure 10b shows the SEM graphs after purification. After purification process on the raw samples, the CNTs present on SEM graph observable. Figure 11 shows the Raman spectroscopy after purification, which D-band peak related to graphitic impurity and the G -band peak related to CNTs density. The D-band peak is about 1317.7 cm^{-1}and the G-band peak is about 1544.08 cm^{-1}. The Raman spectroscopy after purification indicates that the CNTs on the 304-type stainless steel purified.

DISCUSSION

As indicated by Amelinckx *et al.* (1994) in general, the growth process of CNTs has three steps. One-step, was absorption and decomposition of hydrocarbon. Two-step, diffusion of carbon species and three-step was precipitation of crystalline graphite. With gas flow in to furnace, Hydrocarbon gas super saturation generated. The result carbon concentrations produced on the catalysts surface may exceed that needed for diffusion; witch causes the accumulations of carbon on the catalysts surface, there by reducing the effectiveness of the catalysts for the growth of CNTs (Qiuling *et al.*, 2007).

Initial measurement indicated that the CNTs density and the average diameter are very sensitive to the temperature. The SEM graphs in different temperature for the grown CNTs on the 304-type stainless steel as shown in Fig. 2. In Fig. 2, by increasing in temperature the CNTs density decreases. The optimum temperature for the grown CNTs on the 304-type stainless steel is about 750°C. In Fig. 5, by increasing in temperature the average diameter of grown CNTs on the 304-type stainless steel increases. The average diameter in 750°C for the grown CNTs on the 304-type stainless steel is about 58 nm and for 850°C is about 125 nm. As indicated in literature the average diameter and density of CNTs typically depends on the temperature. Our obtained results indicate that by increasing in temperature, the Nickel deposition size increases. Therefore, the CNTs diameter becomes larger and their density decreases.

Defects and grin boundaries are nucleation sites. In 304-type stainless steel, nickel grain boundaries are nucleation sites. In constant volume, with temperature increasing grains diameter becomes larger and theirs count decreases. There is correlation between the size of the catalysts nanoparticles and the CNTs diameter. Indeed, many groups observed a direct dependence of the two quantities (Ivanov *et al*., 1994; Li *et al*., 2001; Cheung *et al*., 2002; Yoon *et al*., 2002; Dai *et al*., 1996; Zhang *et al*., 2002; Colomer *et al*., 2000). In general, there is a reasonable agreement between our obtained results and the other reported in mentioned reference.

The TEM graph for the grown nanotube on the $NiSio_2$ catalyst and 304-type stainless steel has been shown in Fig. 6 and 7. Comparisons of figures indicate that the CNTs have a bamboo structure, curved morphology and MWCNT. Other investigator, Nickel-based catalysts generally lead to MWNTs reported it (Ivanov *et al*., 1994; Yudasaka *et al*., 1995; Ren *et al*., 1998; Teo *et al*., 2001; Zhang *et al*., 2002). Seldom to Single Wall Nanotubes (SWNTs) (Colomer *et al*., 2000; Seidel *et al*., 2004). Many of MWCNT have bamboo structure or fibers (Dupuis, 2005). In general, there is a reasonable agreement between our obtained results and the other reported in research.

The TGA and DTA graph of CNTs that grown on the 304-type stainless steel before the purification shows the CNTs burned in 600°C. The SEM graphs before and after purification and Raman spectroscopy after purification indicates that the CNTs on the 304-type stainless steel were purified. Our obtained results displays that the optimum temperature for purification is about 450°C with two hours oxidation.

Now it is useful to compare the results of this study with the previous ones. The density and average diameter of CNTs in the study of Ivanov *et al*. (1994), Li *et al*. (2001), Cheung *et al*. (2002), Yoon *et al*. (2002), Dai *et al*. (1996), Zhang *et al*. (2002) and Colomer *et al*. (2000) show a reasonable agreement with our experimental data. The SEM graphs indicate that the CNTs have a bamboo structure, curved morphology and MWCNT. Comparing of our results with study of Yudasaka *et al*. (1995), Ren *et al*. (1998), Teo *et al*. (2001), Zhang *et al*. (2002), Ivanov *et al*. (1994), Colomer *et al*. (2000) and Seidel *et al*. (2004) show a good agreement. In general, there is a reasonable agreement between our experimental data and the results of earlier studies.

CONCLUSION

The goal here was the comparison of synthesis and purification of carbon nanotubes (CNTs) by Thermal Chemical Vapor Deposition (TCVD) on the Nickel-based catalysts, $NiSio_2$ catalyst and 304-type stainless steel. The average diameter and density of CNTs for different temperature investigated. In our experimental, by increasing in the temperature the density of CNTs for 304-type stainless steel increases and for $NiSio_2$ catalyst decreases. The optimum temperature for the grown CNTs on the $NiSio_2$ catalyst is about 825°C and for the 304-type stainless steel is about 750°C. Our obtained results indicate that the average diameter of CNTs for 304-type stainless steel show a non-linear decreases and vice versa for $NiSio_2$ catalyst non-linear increases by increasing in temperature. The average diameter in 750°C temperature for CNTs on the 304-type stainless steel is about 57 nm and for $NiSio_2$ catalyst is about 119 nm and for 825°C temperature respectively, 103 and 57 nm. Comparisons of TEM graphs in the 800°C temperature indicate that the CNTs growth on the $NiSio_2$ catalyst and 304-type stainless steel have a bamboo structure, curved morphology and MWCNT. The TGA and DTA graph of CNTs before the purification indicate that the oxidation of CNTs was 600°C. The Raman spectroscopy after purification shows that the D-line peak is about 1317.7 cm^{-1} and the G-line peak is about 1544.08 cm^{-1}. Therefore, the Raman spectroscopy after purification indicates that the sample was purified. Result of different samples SEM graph display that the optimum temperature is about 450°C with 2 h oxidation. Obtained results verified that the reported samples could effectively implement for the synthesis of CNTs and reported a simple method for the purification.

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