Production of Vapor Grown Carbon Fibers from Hydrocarbons Using Atomized Liquid Technique

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Introduction

Since the last two decades, vapor grown carbon fibers(VGCF) have attracted attention as a new type of carbon fibers. These fibers are prepared by the decomposition of hydrocarbons in a hydrogen atmosphere in the presence of ultrafine particles of transition metals(e.g., Fe. Co.Ni) or their alloys. Not only VGCFs possess unique structure and excellent performances, but also the simplicity of their production appears to have the possibility to significantly reduce the production cost of short carbon fibers. These advantages make VGCFs applicable to various fields, such as, to composite materials (CFRP, CFRTP, CFRM, CFRCe) and to functional materials.

Although many production methods [1-3] and the mechanism [4,5] for the growth of VGCFs have been reported by several workers, there are many problems in the preparation of VGCFs, such as separation and continuous production of VGCFs. In this paper, we describe an apparatus — Atomized Liquid Technique to prepare VGCFs. By this technique, highly active ultra-fine uniform catalyst particles which have the suitable size for fiber growth can be easily generated and VGCFs with high L/D ratio and uniform texture were obtained.

Experimental

The VGCFs was prepared by the floating catalyst method . The experimental reactor used in our present study is an quartz glass tube (inner diameter 72mm, length 1700mm), which was heated by an three-stage furnace. An atomizer was equipped in the upper zone of the reactor.

A hydrocarbon (benzene) liquid dissolveing a suitable organo-metallic compound (Ferrocene.0-0.07ml/g) and sulfur compound (thiophene S/Fe 0-0.5) was been atomized to introduce the reactor with the hydrogen carrier. The amount of solution was controlled by the flowing rate of hydrogen.

The VGCFs formed were recovered from the collector and the wall of the reactor, then treated with supersonic cleaner in a quinoline solvent where fibers can be separated from soot and tar. According to the amount of benzene introduced, the VGCFs were weighed to calculate the VGCFs yield. The morphologies of VGCFs were observed by SEM.

Results and Discussion

Fig.1(a) show the plot of VGCFs yield against the ferrocene concentration. As a contrast, we give the same effect on the yield of VGCFs prepared by injection liquid drop method as reported in our previous paper[6], shown in Fig.1.(b). Compared to the past introducing method, the yield of VGCFs dramatically increased with the increasing of catalyst concentration. However, under the experimental conditions (Ferrocene concentration >0.07g/ml, temperature at fog nozzle >400 ℃, H_2 >1.5l/min.), ferrocene will crystallize out from benzene solution to deposit, which block up the fog nozzle.

Fig.2 (a), (b) show the SEM photoes of fibers producted by two methods. From Fig.2, it is found that there is a clear superiority in the morphology and the absence of soot in fibers grown by this technique. In addition, other products such as carbon films, sponge carbon and tar apparently decreased through this technique. At meantime, we also give the photo before and after separation (Fig.3).

The introduction of the catalyst source to the reactor by the means of atomizing has a possibility to produce highly active ultrafine catalyst particles. When hydrocarbon solution dissolved catalyst source is atomized into the reactor, the concentration of fog drops is distributed uniformly. The following decomposition of fog drops rapidly occurs, small-diameter catalyst and carbon species are formed. In comparison with previous method, the chance of coalescence among particles decrease.The concentration of fog drops is easily controlled by the flow rate of hydrogen, it means that the sizes of catalyst particles and concentration of hydrocarbon can be controlled by controlling the contact among these fog drops. Then they increase their size through the coalescence. When they reach the proper size for fibers growth, fiber growth is initiated.

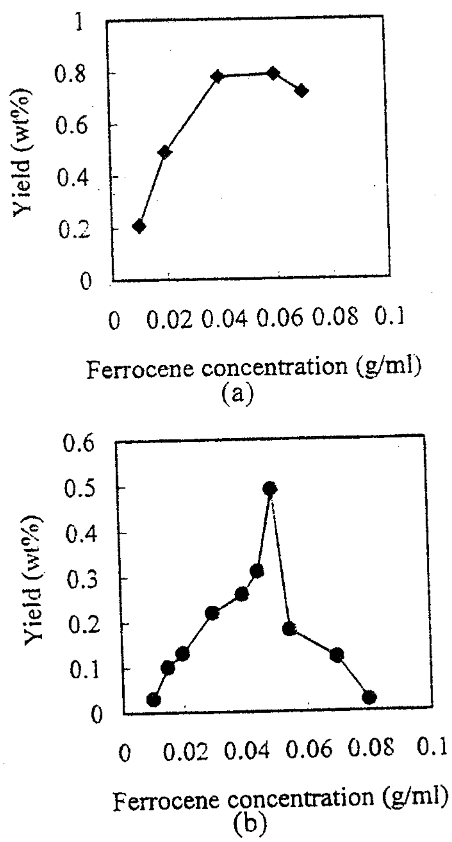

Fig.1 (a),(b) Plots of VGCFs yield against the Ferrocene concentration

Conclusions

A new technique to produce VGCFs, the Atomised liquid technique, was proposed. Uniform small size catalyst particles can be generated using this method, and the VGCFS of higher yield as well as thinner diameter were obtained.

References

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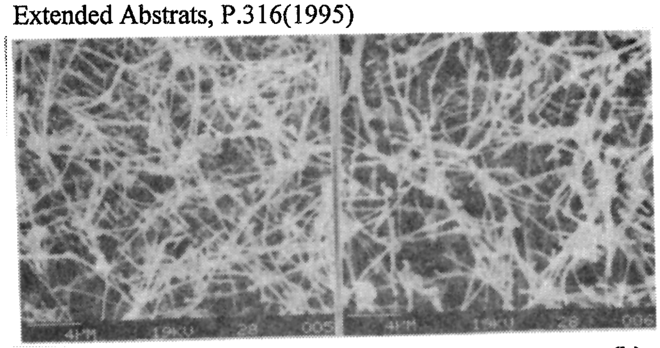
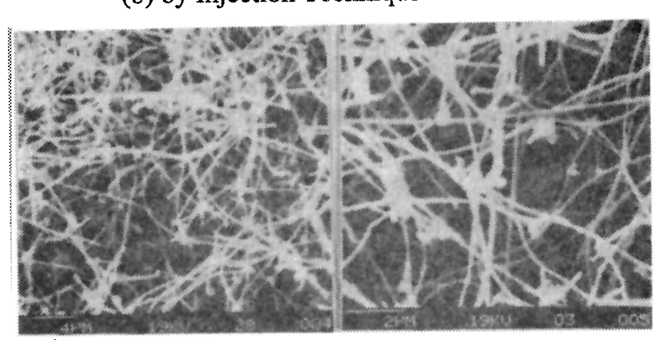


(a) Fig.2 The SEM micrographs of VGCFs (b)
(a) by Atomizing Technique
(b) by Injection Technique



(a) (b)
Fig.3 The SEM micrographs of VGCFs before (a) and after separation (b)