PREPARATION OF MICRO-COILED CARBON FIBERS BY TI PLATE CATALYZED PYROLYSIS OF ACETYLENE

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Introduction

Davis et al. firstly reported the preparation of the coiled carbon fibers by catalytic disproportion of carbon monoxide [1]. Kitajima et al. obtained macro-coiled carbon fibers with a coil radium of 0.2~1 mm by a conjugated spinning process of isotropic and anisotropic pitches [2]. And the growth of micro-coiled carbon fibers (MCCF) from the vapor phase has been reported by many researches. The characterizations of micro-coiled carbon fibers, such as growth patterns, morphologies, growth mechanism, bulk electrical resistivity, density, specific surface area and absorption of electromagnetic waves, have been studied efficiently [3-7]. We have prepared different styles regular micro-coiled carbon fibers, and studied the microwave permittivities of these carbon coils [8-10]. In this paper we prepared the micro-coiled carbon fibers using Ti catalyzed pyrolysis of acetylene containing a small amount of thiophene. And the complex permittivities of the micro-coiled carbon fibers suspended in paraffin wax have been investigated at the frequency range of 8.2~12.4 GHz.

Experimental

The micro-coiled carbon fibers were obtained by Ti catalyzed pyrolysis of acetylene containing a small amount of thiophene. The production of the micro-coiled carbon fibers in the present research was conducted in a horizontal tubular reactor. The horizontal reactor was a quartz glass tube of 40 mm in diameter and 1200 mm in length and was heated by electric furnace from outside. The Ti plate catalysts placed on a graphite plate substrate were located in the central part of a horizontal tubular reactor. Commercial acetone dissolved acetylene (97% purity) was used as the carbon source, argon and / or hydrogen as carrying gas and Ti plate as catalyst. The thiophene was kept in a temperature-regulated container through which hydrogen was bubbled. Argon was used as dilute gas to slow the reaction rate. The reaction condition used are as follows: reaction temperature 750~800℃℃, reaction time 1h, hydrogen 50~70 ml/min, argon 20~40 ml/min, acetylene 20~30 ml/min, thiophene, 0.3~0.5 ml/min.

The microwave permittivities of the micro-coiled carbon fibers imbedded in paraffin wax have been studied. The micro-coiled carbon fibers were dispersed in melting paraffin wax, and then the mixtures were cast into the mold ($10.16\times22.86\times2mm^3$). The sample consisted of 5 wt% MCCF and 95 wt% paraffin wax. The permittivity of the micro-coiled carbon fibers and paraffin wax mixtures were measured by the method, which is based on measurements of the reflection and transmission moduli between 8.2 GHz and 12.4GHz.

Results and Discussion

The micro-coiled carbon fibers were grown on the surface of the Ti plate. Figure 1-4 shows the SEM images of the regularly coiled carbon fibers obtained at the temperature of 750~800℃.

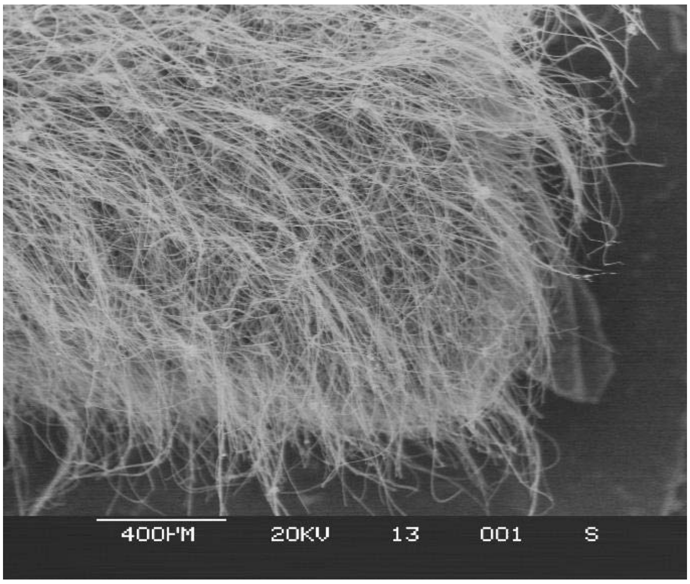

Figure 1. SEM images of the micro-coiled carbon fibers

Electrical properties can be determined at various frequencies [11-13]. The interaction between electromagnetic waves and condensed matter can be described by using complex permittivity, ε_r ($\varepsilon_r=\varepsilon'+i\varepsilon''$, where ε' is the real part, ε'' the imaginary part), and conductivity, σ_r. The relation between the real part of the (polarization) conductivity $\sigma'(\omega)$ and the imaginary part of the permittivity $\varepsilon''(\omega)$ is $\sigma'(\omega)=\omega\ \varepsilon''(\omega)$, where ω is the angular frequency.

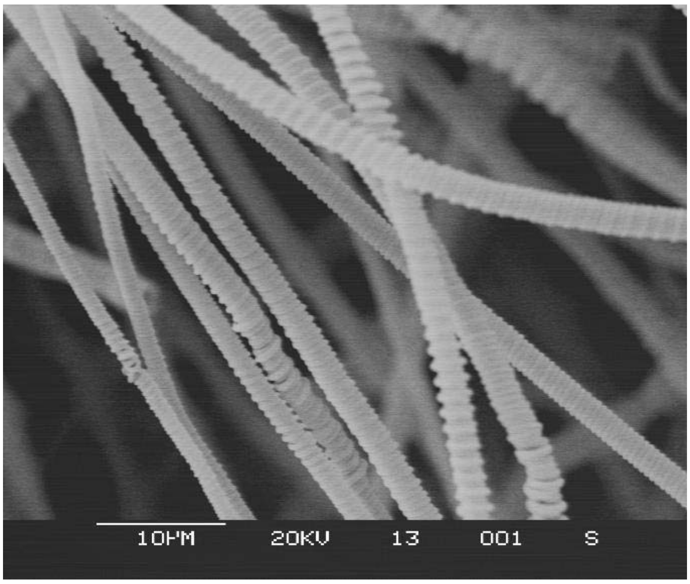

Figure 2. SEM images of the micro-coiled carbon fibers

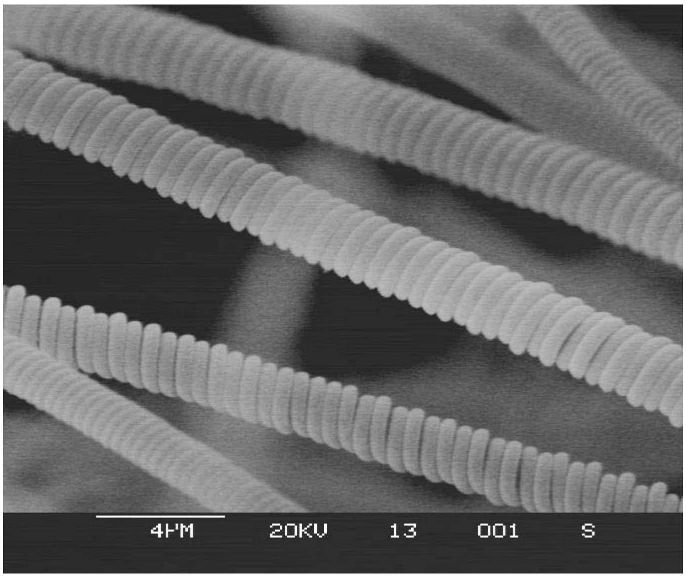

Figure 3. SEM images of the micro-coiled carbon fibers

The complex permittivities of the micro-coiled carbon fibers imbedded in paraffin wax were measured at a frequency range of 8.2~12.4GHz. The ε' and ε'' of the paraffin wax are 2.26 and 0 at the frequency range of 8.2~12.4 GHz respectively. The real part (ε') of permittivity of the micro-coiled carbon fibers and paraffin wax mixture ranges from 10.76 to 12.03, and the imaginary part(ε'') ranges from 9.06 to 10.13. The loss tangent (or dissipation factor), tgδ ($\varepsilon''/\varepsilon'$) ranges from 0.83 to 0.86(Figure 5). We have investigated the complex permittivities of the straight vapor grown carbon fibers [13]. Although the ε' and ε'' of the straight vapor grown carbon fibers are much higher than those of the micro-coiled carbon fibers, the loss tangent (tgδ) is much less than that of the micro-coiled carbon fibers. The results indicate that the micro-coiled carbon fiber would be a promising candidate as a novel electromagnetic absorber, especially in the GHz region, because of its micro-coiling morphology.

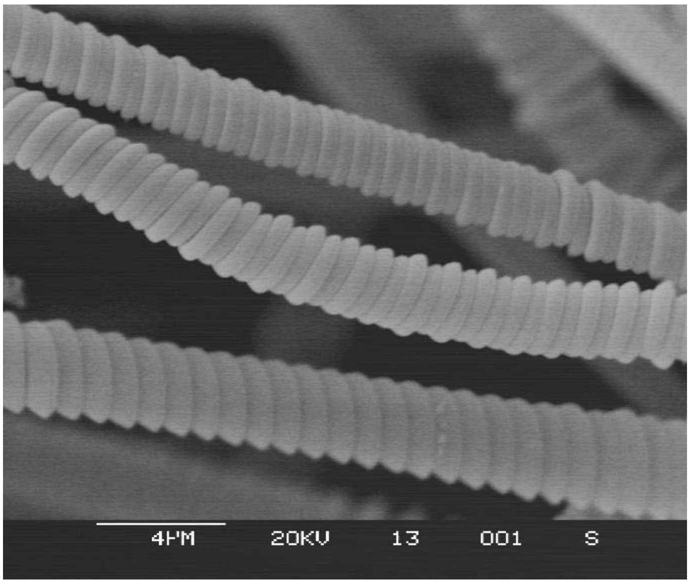

Figure 4. SEM images of the micro-coiled carbon fibers

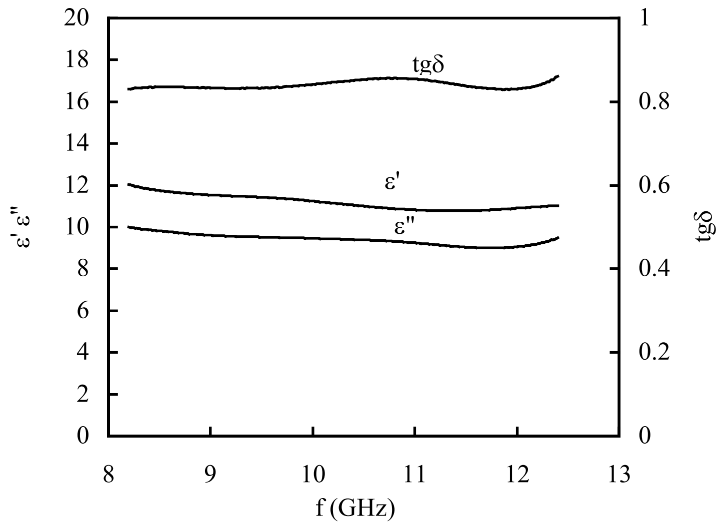

Figure 5. ε′, ε″, and tgδ of the micro-coiled carbon fibers

Conclusions

(1) The micro-coiled carbon fibers were obtained by Ti plate catalyzed pyrolysis of acetylene containing a small amount of thiophene. The reaction temperature is 750~800℃.
(2) Although the ε′ and ε″ of the straight vapor grown carbon fibers are much higher than those of the micro-coiled carbon fibers, the dissipation factor is much less than that of the micro-coiled carbon fibers.
(3) The micro-coiled carbon fiber would be a promising candidate as a novel electromagnetic absorber, especially in the GHz region, because of its micro-coiling morphology.

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