Preparation of single-helix carbon microcoils (SH-CMCs) using Fe-Ni alloy catalyst

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Abstract

Carbon microcoils (CMCs) are a kind of vapor growth carbon fibers, which have an interesting 3D-helical/spiral structure with a coil diameter of micrometer orders and have a very high elasticity. Conventional double helix carbon microcoils(DH-CMCs) were prepared were prepared by a CVD process using Ni as a catalyst, acetylene as a carbon source gas under the atmosphere of nitrogen, hydrogen, and hydrogen sulfide. While the single helix carbon microcoils (SH-CMCs) were prepared by using an Fe alloy as a catalyst. The composition of the Fe-Ni alloy affect the morphology of the SH-CMCs dramatically. In this study, we synthesized SH-CMCs using an alloy Fe-Ni(50:50) as a catalyst, the SH-CMCs with a different morphology comparing to those SH-CMCs grown over an Fe-Ni(90:10) alloy catalyst; the preparation conditions, the morphologies, structure of SH-CMC and the growth mechanism were examined using SEM, TEM and Raman . The obtained SH-CMCs have a high purity. The coil diameter, coil's gap and fiber diameter were 1~2 μm, ~1 μm, 0.2~0.5 μm, respectively. Fig. 2c SH-CMCs are grown by a single growth direction mode, while fig.2b SH-CMCs are by double-growth direction mode. The structures of as-grown SH-CMCs and heat-treated SH-CMCs were compared. As-grown SH-CMCs were composed of amorphous carbon, while heat-treated SH-CMCs were graphitized in some degree.

Synthesis approach.

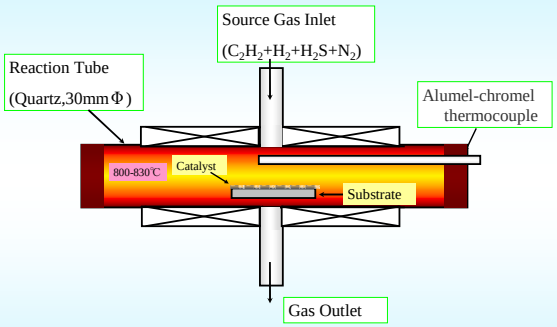


Fig. 1. Reaction tube for the preparation of single-helix carbon microcoils (SH-CMCs).

Comparison of SH-CMC with CMC.

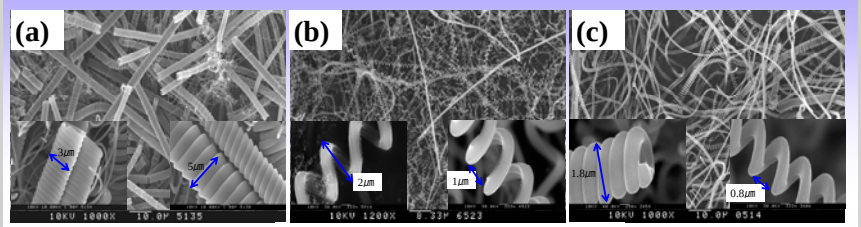


Fig. 2. SEM images of CMC and SH-CMC.

Table 1. Difference in synthesis condition.

	Catalyst	Reaction temperature (℃)	Morphology
(a) CMC	Ni	750~800	Double helix
(b) SHCMC	Fe+Ni (90:10)	700~730	Single helix
(c) SHCMC	Fe+Ni (50:50)	800~830	Single helix

Growth observation.

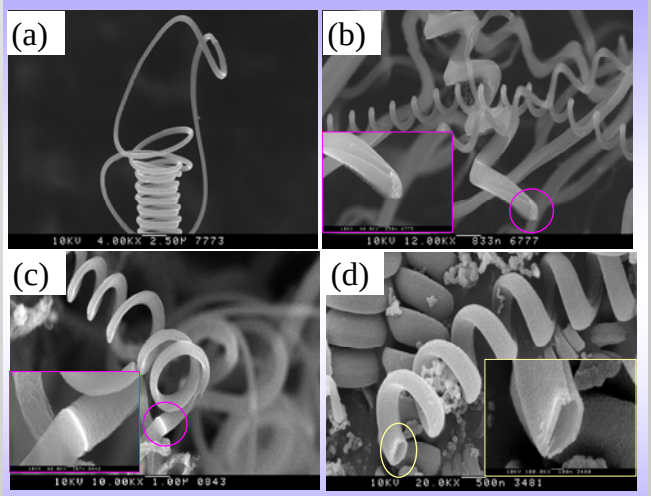


Fig. 3. SEM images of growth tip.
(a) CMC (b) SH-CMC (Fe:Ni=90:10), (c) SH-CMC (Fe:Ni=50:50),
(d) Heat-treated SH-CMC (Fe:Ni=50:50)

Structure of SH-CMC.

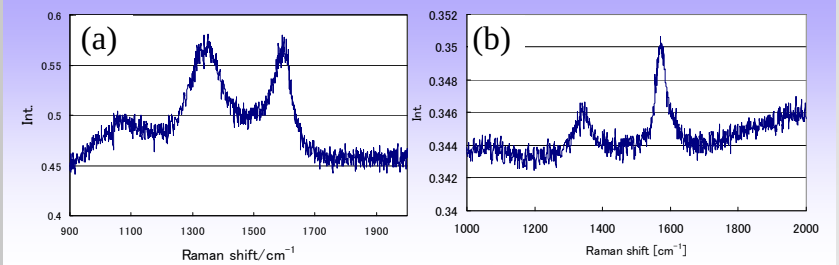


Fig. 4. Raman spectra of SH-CMC.
(a) as-grown SH-CMC,
(b) heat-treated SH-CMC.

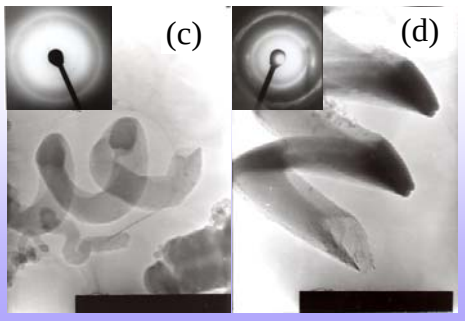
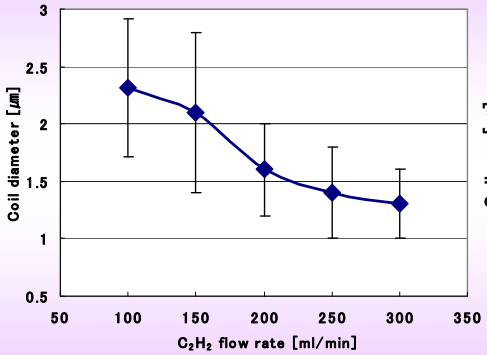


Fig. 5. TEM images and electron diffraction patterns of SH-CMC.
(c) as-grown SH-CMC,
(d) heat-treated SH-CMC.

Influence of C_2H_2 flow rate.



Efficiency of Fe/Ni ratio.

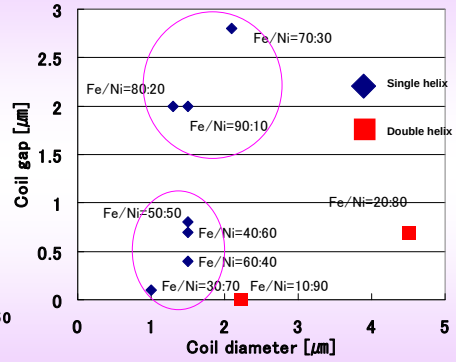


Fig. 6. Relationship between coil diameter and C_2H_2 flow rate. Fig. 7. Relationship between Fe/Ni ratio and coil's morphology.

Conclusion

①. We obtained SH-CMCs using an Fe-Ni alloy catalyst at 820℃.

②. SH-CMCs are grown by a double growth direction mode.

③. As-grown SH-CMCs were composed of amorphous carbon, while heat-treated SH-CMCs were graphitized in some degree.

④. We could control the coil diameter when C_2H_2 flow rate was changed.