Process and Analysis of Electromagnetic Shielding in Composite Fiberboard Laminated with Electroless Nickel-plated Carbon Fiber

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To develop a composite fiberboard with high electromagnetic shielding effectiveness (SE) and an easily industrialized process, electroless nickel-plated carbon fiber technology was used to improve the conductivity and electromagnetic properties of carbon fiber and carbon fiber sheets. The SE of composite fiberboards laminated with nickel-plated carbon fiber with different arrangements was also studied. The results showed that the best plating scheme in this study was a $NiSO_4$ concentration of 35 g/L, plating temperature of 70 °C, pH of 9, and plating time of 15 min. When nickel-plated carbon fiber in a grid arrangement was added to the core between two fiberboards, the minimum and maximum SE of 19 × 19 grid arrangement was 41.54 dB and 63.73 dB in the 200 to 1000 MHz frequency range, respectively, and reached medium grade. SE of composite fiberboard laminated with double layers of plated carbon fiber sheet ranged from 45.29 dB to 52.01 dB and reached medium grade. It is thus feasible to use two-layer nickel-plated carbon fiber with a 19 × 19 grid arrangement to make composite fiberboard with high SE, mechanical properties greater than the national standard, and an easily industrializable process.

Keywords: Carbon fiber; Carbon fiber sheet; Electroless nickel plating; Electromagnetic shielding effectiveness; Laminated composite fiberboards

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INTRODUCTION

The wood processing industry is currently developing products that are higher value added. Endowing fiberboard with an electromagnetic shielding function is one way to increase its value. Studies of the electromagnetic shielding capacity of fiberboards has primarily focused on the addition of metal fibers (Zhang and Liu 2005; Liu and Fu 2008), metal net (Zhang and Liu 2004; Zhang *et al.* 2004), and mineral powder (Su *et al.* 2012) into fiberboards, plating metallization (Huang and Zhao 2006; Wang *et al.* 2006), and covering the surface of fiberboards with a conductive diaphragm (Lu *et al.* 2011). In the cited studies, an adequate level of shielding effectiveness (SE) was achieved. However, such methods resulted in heavy products, difficulty in secondary processing, and low bonding strength of the composite material.

Carbon fiber with high conductivity, high dielectric loss, high specific strength, and oxidative resistance, has been employed in the development of composite material with an electromagnetic shielding function, such as carbon fiber/ABS composites, carbon fiber reinforced cement, and carbon fiber-filled polychloroprene rubber composites, *etc.* Carbon-matrix composite added with continuous carbon-fibers had been found to be an excellent electromagnetic shielding material with SE of 124 dB, low surface impedance, and high reflectivity in the frequency range from 0.3 MHz to 1.5 GHz (Luo and Chung 1999). Chiou *et al*. (1989) added short carbon fibers (3 mm, 0.5 wt% of the cement) to cement, developing plane cement with 3.6 mm thickness, and its SE in the frequency of 1.5 GHz was 10.2 dB. Also, it had been shown that carbon fiber exhibits absorption in the microwave region (Das *et al*. 2000). However, the magnetic loss of carbon fiber is relatively low, which results in the low absorption of electromagnetic waves in some regions, especially at high frequency. To overcome this drawback, researchers have deposited some metal with high magnetic loss angle on carbon fiber by chemical approach, such as electroless plating and electroplating. Among the deposited metal, nickel with high magnetic loss angle and oxidative resistance had been deposited on the carbon. Then it was mixed with ABS to prepare Ni-coated carbon fibers/ABS composite, and it was found that the conductivity and SE of Ni-coated carbon fibers were greater than that of ordinary carbon fiber. When ABS resin was filled with 10 vol% of Ni-coated carbon fibers, its SE was about 50 dB (Lu *et al*. 1996). Therefore, Ni-coated carbon fiber has better SE in the more wide frequency range.

In this study, electroless nickel-plating technology was used to improve the conductivity and electromagnetic properties of carbon fiber and its sheet. Then the plated carbon fiber and sheet was used as shielding material laminated with fiberboards, by which SE and the composing process of the composite fiberboard was improved, and its weight was reduced. And that would promote the development of the function fiberboard with high value.

EXPERIMENTAL

Materials

The following materials were used in this work:

1. Electroless nickel-plating reagents: Titanate Coupling Agent (NDZ-401), HNO_3, $(NH_4)_2S_2O_8$, H_2SO_4, NaOH, ethanol, NaH_2PO_2, $NiSO_4$, NH_3, NH_4Cl, $Na_3C_6H_5O_7$, and distilled water.
2. Carbon fiber: 12K, produced by Xiang Li Carbon Fiber Factory in the Luohu area of YanCheng (China).
3. Carbon fiber sheet: UT70-20, provided by Anjie Composite Material Co. Ltd. in HaiNing (China).
4. Polyvinyl Acetate Emulsion: GYJTY-25, produced by GuiZhou Crystal Chemical Co. Ltd (China).
5. Fiberboard: thickness of 4mm, density of 0.73 g/cm^3, sold in the market and reaching Chinese “Medium Density Fiberboard” standard.

Methods

Preparation of plating solution

The composition of the plating solution is shown in Table 1. $NiSO_4$ as the main salt was the source of the Ni^{2+} ions. NaH_2PO_2 was the reducing agent, by which Ni^{2+} ion can be reduced to Ni metal. $Na_3C_6H_5O_7$ served as the complexing agent, acting to prevent the formation and precipitation of nickel hydroxide. NH_4Cl was the buffering agent. $NH_3 \bullet H_2O$ was used to adjust pH.

The plating solution was prepared in the following way: Firstly, according to the dosage shown in Table 1, four kinds of agent were weighed and put in four containers, respectively. Then, a small amount of distilled water was poured in the four containers to dissolve the agents. $Na_3C_6H_5O_7$ solution was added into a solution of $NiSO_4$ in the condition of continuous stirring, and then NaH_2PO_2 solution was added to the mixture solution. Subsequently, a solution of NH_4Cl was also added to the mixture solution above, and the final mixture solution was stirred uniformly. Finally, before the plating solution was fully prepared, a certain amount of distilled water was added into the solution above to dilute to the determined volume.

Table 1. Composition and Dosage of the Solution

Composition	$NiSO_4$ (g/L)	NaH_2PO_2 (g/L)	$Na_3C_6H_5O_7$ (g/L)	NH_4Cl (g/L)
Dosage	30～40	30	15	30

Electroless nickel-plating

In the first stage, carbon fiber was impregnated in a 65% nitric acid solution at room temperature for 30 min to remove the protective colloid on the carbon fiber. After the fiber was washed and dried at 120 °C, it was soaked in ethanol for 15 min for degreasing and then it was washed again. Subsequently, it was coarsened for 15 min using a $(NH_4)_2S_2O_8$ (200 g/L) and H_2SO_4 (100 mL/L) solution. Later, it was neutralized using a 10% NaOH solution. Then, titanate coupling agent was used to treat the fiber for 10 min. After the treated fiber was washed, it was plated according to an orthogonal design of experiments.

The orthogonal test $L_9\,(3^4)$ was used with four impact factors, consumption of the main salt ($NiSO_4$), plating time, pH, and plating temperature, to determine the appropriate plating process. The morphology of carbon fibers was observed through a Hitachi n S-3400 scanning electron microscope and the component of the coating was analyzed by EDS. The header design of the orthogonal test $L_9\,(3^4)$ is shown in Table 2.

Table 2. Header Design of the Orthogonal Test $L_9\,(3^4)$

Levels	$NiSO_4$ (g/L)	Plating temperature (°C)	PH	Plating time (min)
1	30	60	8	10
2	35	70	9	15
3	40	80	10	20

Preparation of laminated composite fiberboards

Composite fiberboards, using polyvinyl acetate emulsion as an adhesive, were constructed in the following ways.

For type A specimens (Fig. 1), different grid arrangements (5×5, 7×7, 9×9, 11×11, 15×15, 17×17, and 19×19) of nickel-plated carbon fiber were added to the core between two fiberboards. B specimens are diagramed in Fig. 2. To make B1, a single carbon fiber sheet was added to the core between two fiberboards. For B2, a single nickel-plated carbon fiber sheet was added to the core between two fiberboards. In B3, two layers of carbon fiber sheet were added to the core between two fiberboards. Finally, for B4, two layers of nickel-plated carbon fiber sheet were added to the core between two fiberboards.

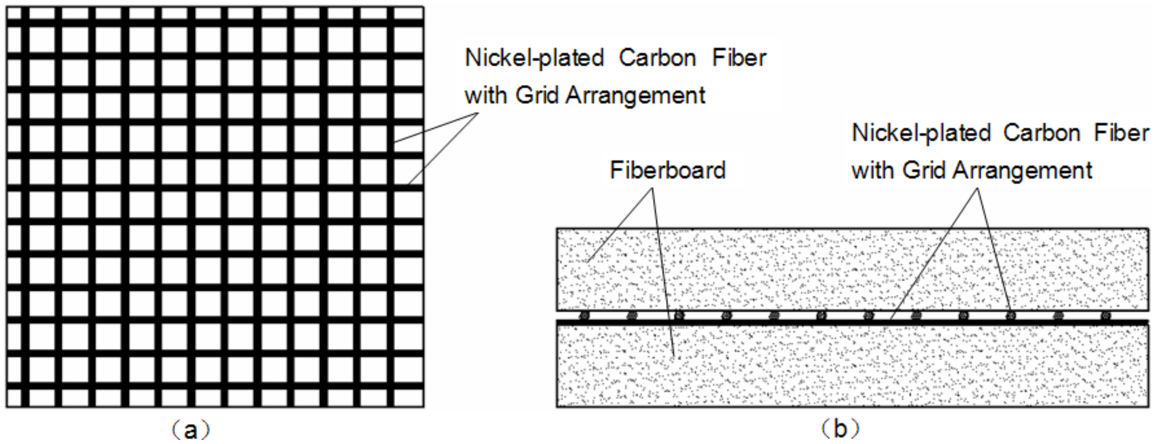


Fig. 1. Schematic diagram of A specimens. (a) nickel-plated carbon fiber with grid arrangement (5×5 means that there are 5 carbon fiber bundles parallel to length and width direction respectively), (b) cross section of specimens

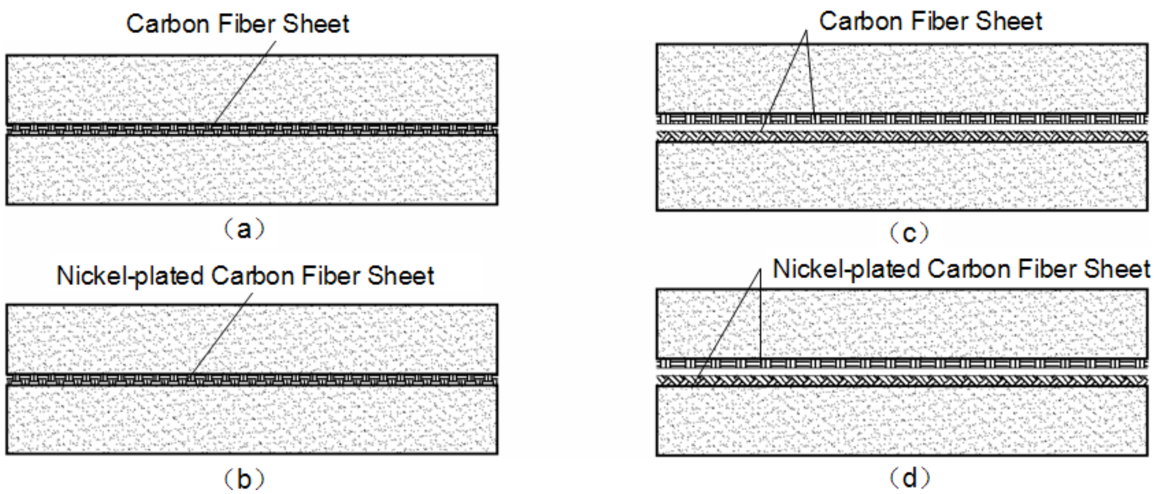


Fig. 2. Schematic diagram of B specimens. (a) B1 specimens, (b) B2 specimens, (c) B3 specimens, (d) B4 specimens

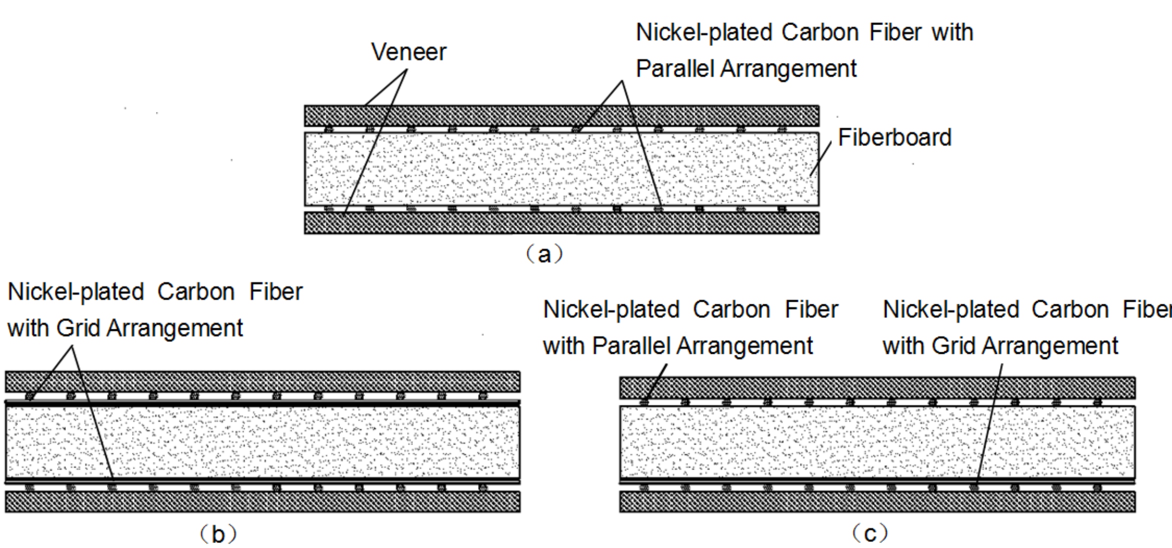


Fig. 3. Schematic diagram of C specimens. (a) C1 specimens, (b) C2 specimens, (c) C3 specimens

For composite fiberboard C1 (Fig. 3), nickel-plated carbon fiber with a parallel arrangement was added to two surface layers of one fiberboard. In C2, nickel-plated carbon fiber with a grid arrangement was added to two surface layers of one fiberboard. For C3, nickel-plated carbon fiber with a parallel arrangement and nickel-plated carbon fiber with a grid arrangement were added to two surface layers of one fiberboard. Then, the surfaces of all specimens were overlaid with two pieces of *Eucalyptus* veneer.

Resistance test of carbon fiber

The resistance (test distance was 10 cm) of carbon fiber before and after nickel plating was tested with the help of a VC9804A^{+} type digital multimeter. The difference of resistance before and after plating was designated as the changing amount of resistance, which could reflect the conductivity and coating uniformity of the nickel-plated carbon fiber.

Resistance test of type A specimens

To analyze the influence of the grid arrangement of nickel-plated carbon fiber on electrical conductivity and to explore the grid arrangement with better electrical properties, type A specimens were formed into disc specimens of diameter $115^{0}_{-0.5}$ mm, and their resistance at different locations (seven positions at the end face of disc specimens) was directly measured using a digital multimeter.

SE testing methods

According to the Chinese standard, “SJ20524-1995 measurement of SE of materials,” composite fiberboards were cooled for 24 h, then formed into disc specimens of diameter $115^{0}_{-0.5}$ mm.

The SE of the composite fiberboards was tested using a DN15115 type vertical flange coaxial test device (Fig. 4, developed by Southeast University). The SE testing was conducted by the Research Institute of Wood Industry of the Chinese Academy of Forestry.

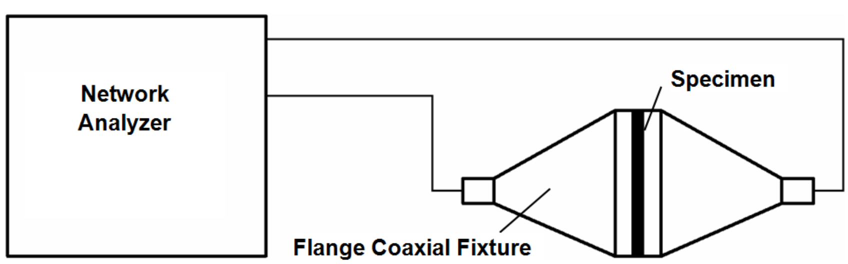


Fig. 4. Layout of flange coaxial test device (test frequency is 100 kHz -1.5GHz)

Evaluation for SE

SE was divided into the following grades (Du *et al.* 2000): under 10 dB, no effectiveness; 10 to 30 dB, bad; 30 to 60 dB, medium; 60 to 90 dB, good; above 90 dB, excellent. Among these, composite materials of medium grade could be used for general industrial and commercial electronic equipment.

Physical and mechanical properties testing

The modulus of rupture (MOR), modulus of elasticity (MOE), and internal bonding strength (IB) were tested according to Chinese national MDF standard GB/T 11718-2009.

RESULTS AND DISCUSSION

Results and Analysis of Electroless Nickel-plated Carbon Fiber Testing

Orthogonal testing results for nickel-plated carbon fiber are shown in Table 3.

Table 3. Resistance Data from the Orthogonal Test L9 (3^4)

No.	$NiSO_4$ (g/L)	Plating temperature (°C)	pH	Plating time (min)	Changing amount of resistance (Ω)
1.	30	60	8	10	0.20
2.	30	70	9	15	0.55
3.	30	80	10	20	0.07
4.	35	60	9	20	0.20
5.	35	70	10	10	0.40
6.	35	80	8	15	0.27
7.	40	60	10	15	0.45
8.	40	70	8	20	0.10
9.	40	80	9	10	0.30
k1	0.27	0.28	0.19	0.30	
k2	0.29	0.35	0.35	0.42	
k3	0.28	0.21	0.31	0.12	
R	0.02	0.14	0.16	0.30	

Table 3 shows that plating time, pH, and temperature were the primary factors impacting the resistance of nickel-plated carbon fiber. It was found that the best plating conditions were $NiSO_4$ 35 g/L, plating temperature 70 °C, pH 9, and plating time 15 min.

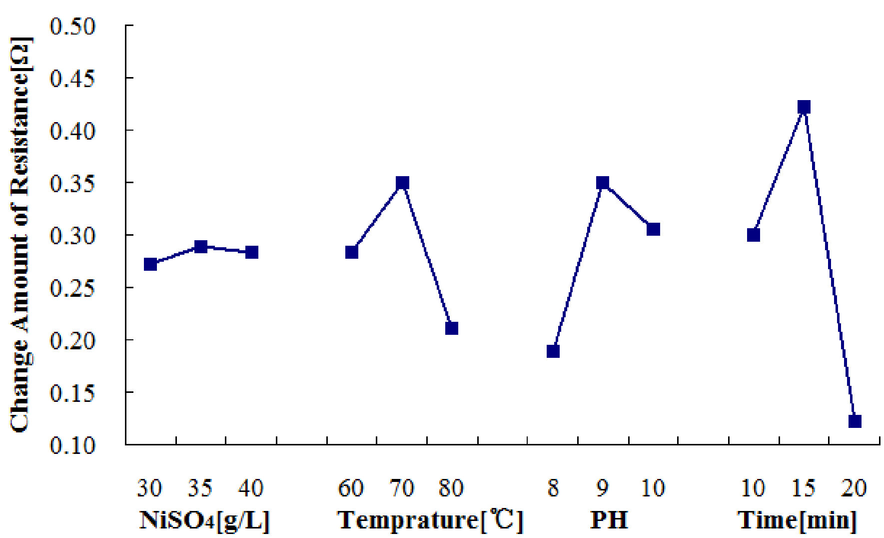
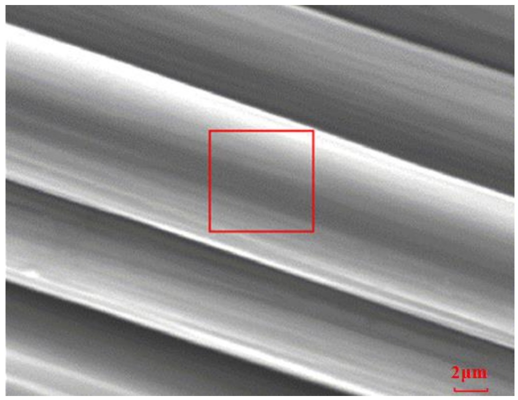


Fig. 5. The influence of each factor on resistance of nickel-plated carbon fiber

As shown in Fig. 5, the conductivity of the nickel-plated carbon fiber increased and then dropped with the continuous increase of each factor. This suggests that there was an optimum level for each factor.

By comparing the micrographs in the upper portions of Figs. 6 and 7, it can be observed that there was a layer of substance deposited on the surface of the carbon fiber after electroless nickel-plating with the best plating conditions.



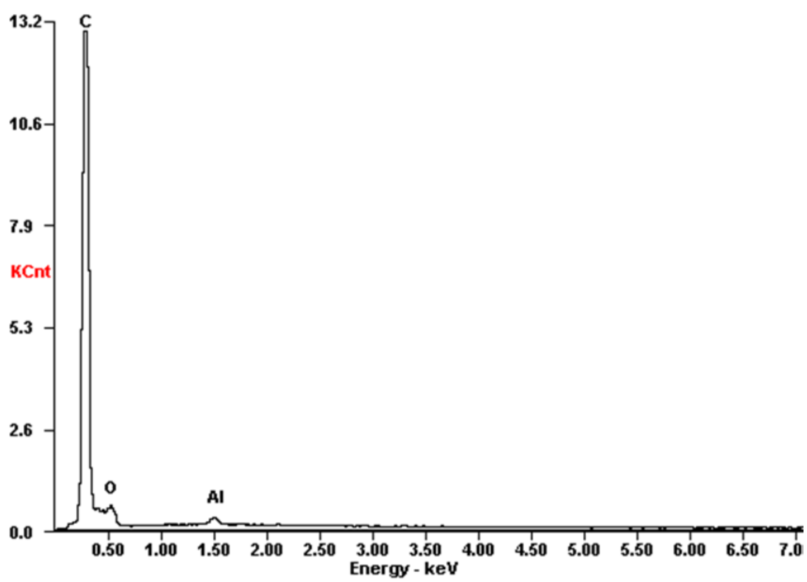


Fig. 6. SEM (above) and EDS (below) of carbon fiber before nickel plating

The non-plated carbon fiber exhibited a very smooth surface, while there was much compact substance coating on the surface of plated carbon fiber. As shown from EDS results plotted in Figs. 6 and 7, the main composition on the surface of non-plated carbon fiber was C element with the proportion of 92.78 wt%, and the main composition on the surface of plated carbon fiber was Ni element which accounted for 82.66 wt%, which showed that the surface of plated carbon fiber was coated by a layer of compact nickel.

The conductivity of plated carbon fiber was largely determined by the uniformity of nickel distributing on the surface of carbon fiber. SEM and EDX results for plated carbon fiber in Fig. 6 show that nickel particles were distributed uniformly and the particles contacted closely, which accounts for the observed increase in conductivity.

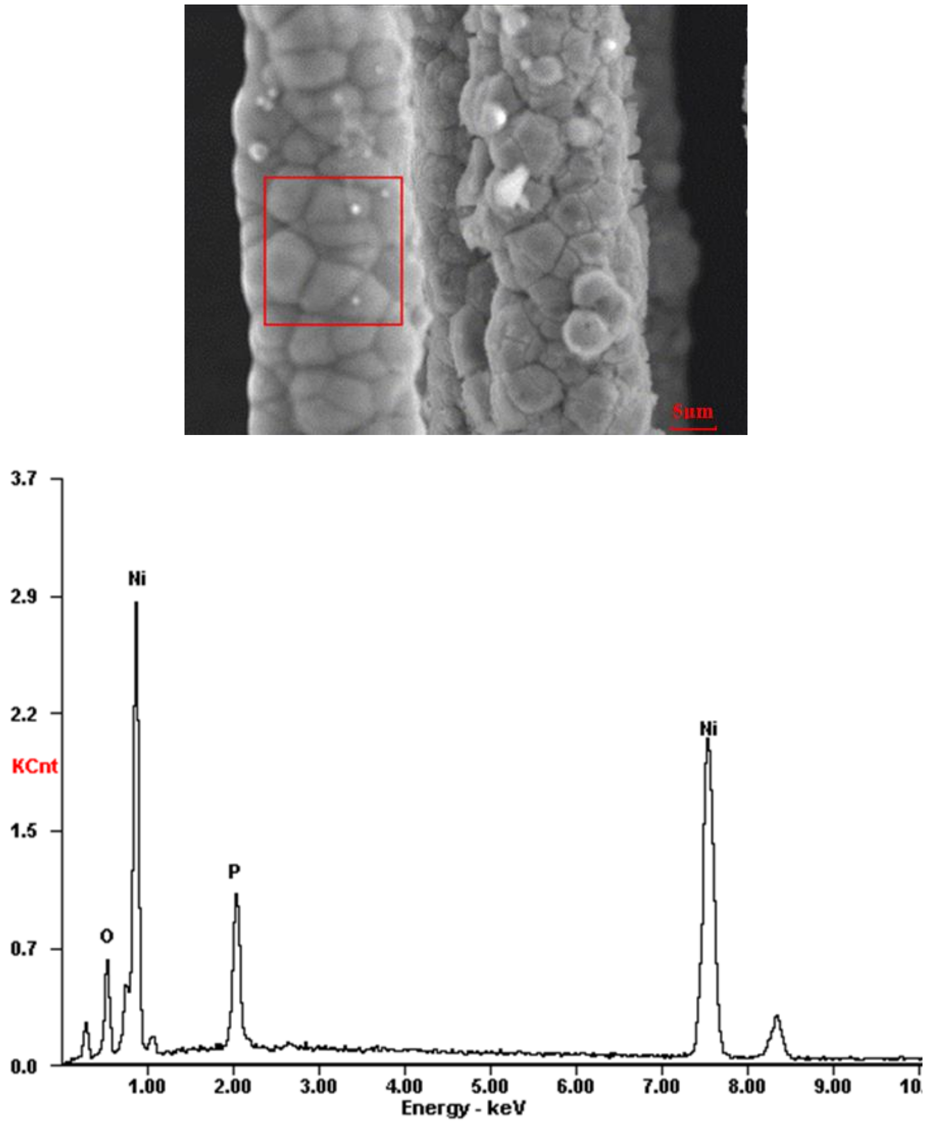


Fig. 7. SEM (above) and EDS (below) of carbon fiber after nickel plating

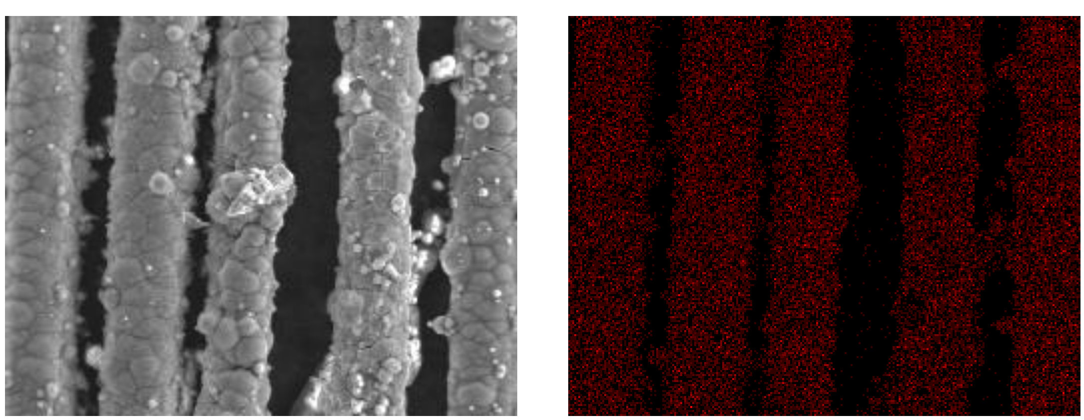


Fig. 8. SEM (left, 1000×) and nickel element mapping (right, red particle) of plated carbon fiber

Results and Analysis of the Electrical Conductivity of Type A Specimens

It is shown in Fig. 9 that the electrical conductivity of laminated composite fiberboards increased with increasing grid arrangements, which indicated that more electrical grids resulted in better conductivity to a certain degree. The variance analysis is shown in Table 4. It was found that the grid arrangement of nickel-plated carbon fiber had a very significant impact on the electrical conductivity of the composite fiberboards.

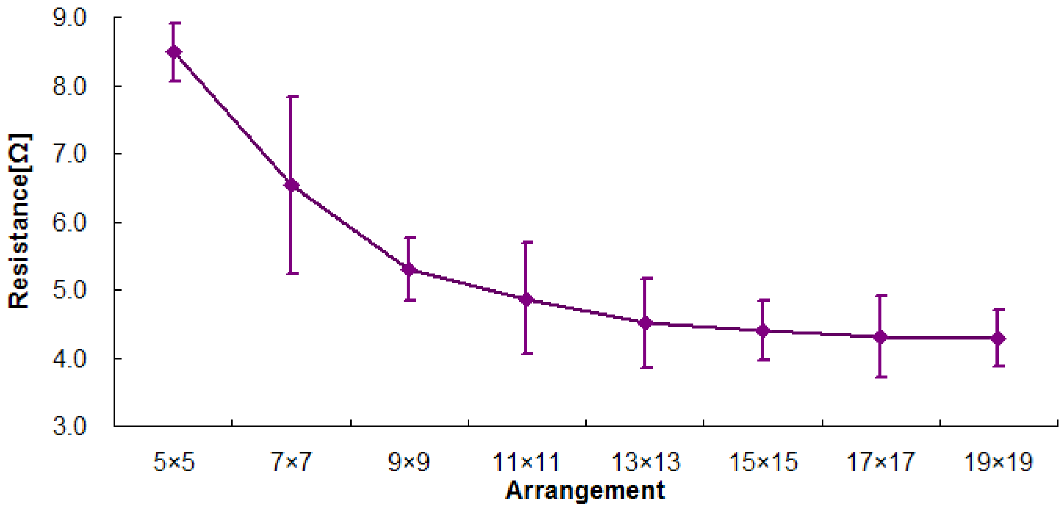


Fig. 9. Relationship between grid arrangement of nickel-plated carbon fiber and resistance

Table 4. Variance Analysis from the Single Factor Test

Variance sources	SS	Df	MS	F	Significance
Between groups	106.808	7	15.258	31.376	* *
Within groups	23.343	48	0.486		
Sum	130.151	55			
* * Indicates that the impact is very significant, where F_A is more than $F_{0.01}$.					

The resistance of type A specimens declined significantly from the 5×5 to 19×19 grid arrangement. This finding is consistent with an expectation of increased opportunities for mutual overlap among fibers with the increase in the number of fibers, forming more conductive pathways through the composite material and improving the conductivity. With the grid arrangement changing further, however, contact points among the fibers increased, forming many conductive chains throughout the matrix. When the amount of fibers exceeded a critical value, such as a 13×13 grid arrangement in this study, the degree of overlap among fibers was high and the contact resistance increased. Therefore, the conductivity of the composite material rose very slowly, close to the maximum value. For example, from 17×17 to 19×19 grid arrangements, the resistance was basically unchanged and the conductivity reached a functional maximum.

Results and Analysis of SE of Type A Specimens

As can be seen from Table 5 and Fig. 10, SE increased with an increase in the grid arrangement of nickel-plated carbon fiber in the same frequency range. It was possible that the contact point and the galvanic circle increased. When the external electromagnetic waves reached the surface of the carbon fiber network, it caused a change in the magnetic flux of each microcircuit and induced a current and an inductive magnetic field, which can resist the change of magnetic flux in the entire shielding surface; thus, a part of the external magnetic field was shielded. According to Faraday's law of electromagnetic induction, external magnetic flux can be offset by the reverse magnetic field caused by a loop-induced current, or the reflectivity and absorption loss of an external electromagnetic wave can be improved by inductance and eddy current caused by the current (Wan 1999).

Table 5. SE of Grid Arrangement of Type A Specimens

SE	5×5 (dB)	7×7 (dB)	9×9 (dB)	11×11 (dB)	13×13 (dB)	15×15 (dB)	17×17 (dB)	19×19 (dB)
Maximum	26.24	37.16	32.37	57.55	51.95	53.50	56.28	63.73
Minimum	14.97	24.82	20.49	33.10	33.52	34.72	37.75	41.54
Average	21.32	31.30	26.93	40.86	41.26	42.87	44.76	47.98

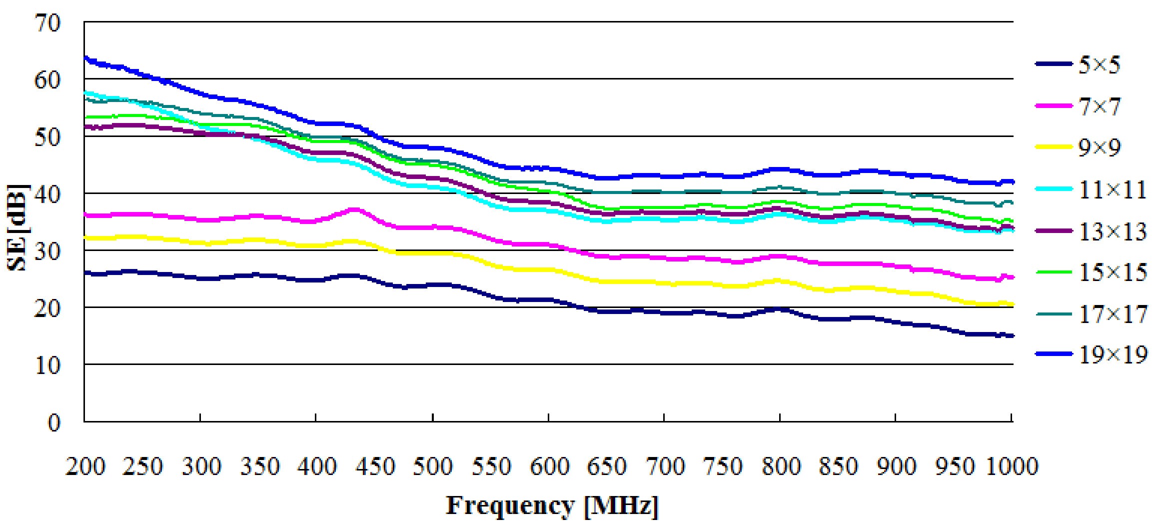


Fig. 10. Comparison chart of the SE of grid arrangement of type A specimens

When the grid arrangement of nickel-plated carbon fiber was greater than 11×11, the SE was above 33 dB and reached medium grade. In the 19×19 grid arrangement, the overall SE reached above 41.54 dB, and the maximum was 63.73 dB.

The SE of the 9×9 grid arrangement was a little lower than that of the 7×7 arrangement. The reason is that excessive adhesion and pressure caused the fibers to arrange irregularly and to be spaced unequally, thus weakening the electromagnetic wave. This result confirms the initial hypothesis that the SE of a material is related to its conductivity. The better the conductivity is, the better the SE is.

Results and Analysis of SE of Type B Specimens

Because of the longitudinal arrangement of carbon fiber in the single layer carbon sheet laminated on the composite fiberboard, it was difficult for the carbon fiber sheet to form a conductive loop, resulting in low SE. As seen in Fig. 11, when specimens were laminated with two layers of nickel-plated carbon fiber sheets, such as in specimens B3 and B4, the SE was better than the single layer and was relatively stable over the entire frequency range, reaching medium grade. The minimum SE was 45.29 dB, and the maximum reached 52.01 dB, which may be attributed to the following hypotheses: 1) the double-layered sheet increased the thickness of the conductive membrane, hindering the transmission of electromagnetic waves; or 2) the contact type among the carbon fibers changed from point contact to surface contact, improving the conductivity of the core layer and enhancing the SE. Moreover, enhancement of the conductivity and the increase in the reflection loss led to an increase in SE after nickel plating, which can be demonstrated by the fact that the SE of B4 was better than that of B3.

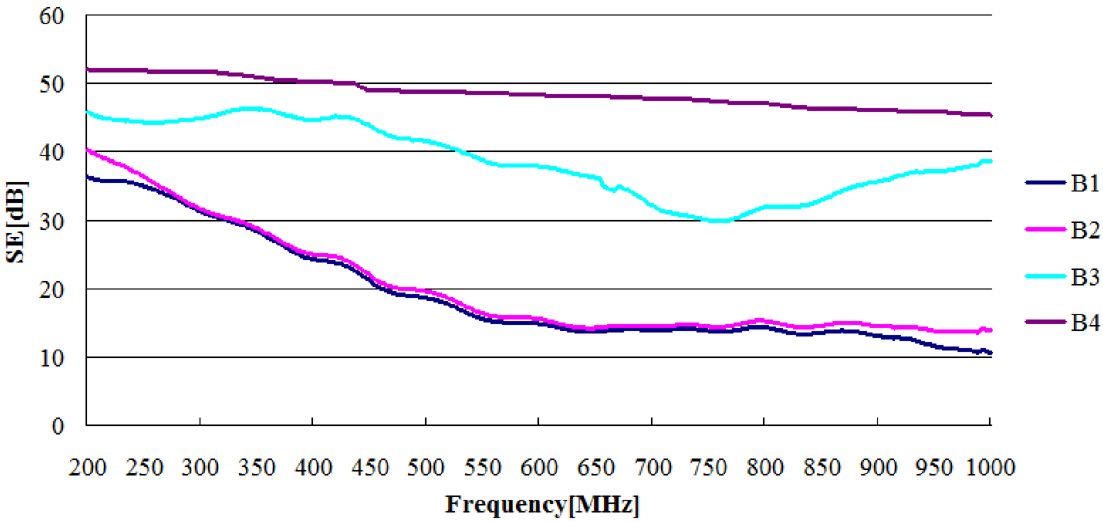


Fig. 11. SE of grid arrangement of type B specimens

Results and Analysis of the SE of Type C Specimens

In this section, nickel-plated carbon fibers in a 19×19 grid arrangement, which demonstrated the best conductivity in type A specimens, were used. As seen in Fig. 12, different arrangements of carbon fibers resulted in different SEs.

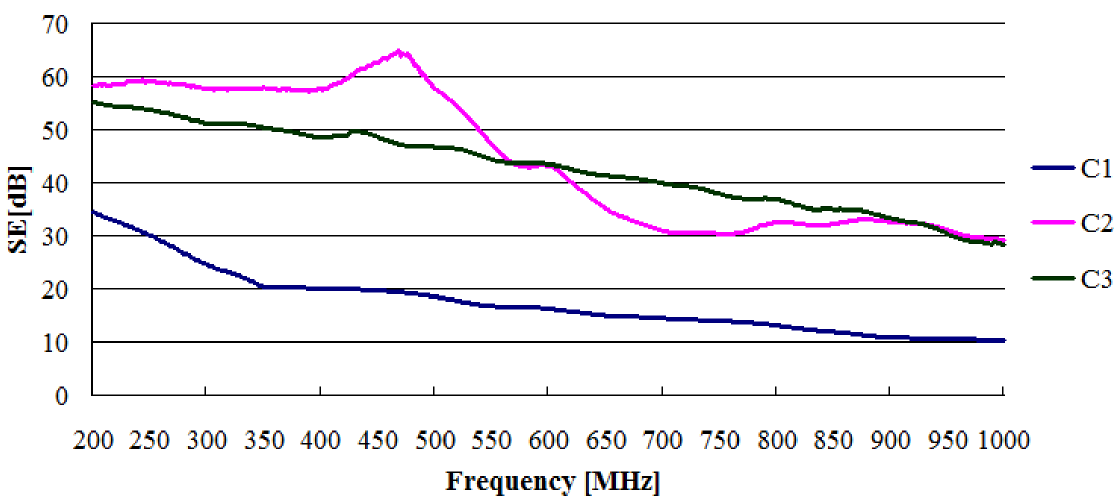


Fig. 12. SE of grid arrangement of type C specimens

Because the grid arrangement of nickel-plated carbon fiber was a good conductive network, the SE of the grid arrangement (C2) was superior to that of the parallel arrangement (C1) over the entire frequency range, and its maximum SE reached 64.75 dB. The SE of the hybrid combination (C3) was lower than that of C2 at low frequencies, but was higher at medium and high frequencies. Thus, the SEs at high, medium, and low frequencies of composite fiberboards with grid and parallel arrangements of carbon fiber are complementary to each other, a result that supports those of Wu *et al.* (2011).

Mechanical Properties of Composite Fiberboards

According to Chinese national MDF standard GB/T 11718-2009, the mechanical performance of ordinary fiberboards with 12 mm thickness should include an MOR of more than 24 MPa, an MOE of more than 2400 MPa, and an IB of more than 0.5 MPa. The mechanical performance of optimized composite fiberboards is shown in Table 6. All data were the average of six test results.

Table 6. Mechanical Properties of Optimized Composite Fiberboards

Types of Composite Fiberboards	MOR (MPa)	MOE (MPa)	IB (MPa)
19×19 grid arrangement of single nickel-plated carbon fiber added to the core layer	24.13 ± 1.03	2508 ± 98	0.65 ± 0.14
B4 double-layer nickel-plated carbon fiber sheet added to the core layer	26.08 ± 1.41	2487 ± 80	0.55 ± 0.05
C2 19×19 grid arrangement of single nickel-plated carbon fiber added to the two surface layers of fiberboards	24.33 ± 1.89	3657 ± 179	0.74 ± 0.16
Substrate	40.00 ± 1.87	2538 ± 112	1.35 ± 0.27

Table 6 shows that the MOR, MOE, and IB of each optimization scheme met the national standard. The mechanical properties of the composites primarily depended on the performance of the substrate itself. The adhesive had a certain influence on the mechanical properties of the fiberboards, especially the MOR and IB. This may be because a small amount of water in the polyvinyl acetate emulsion adhesive penetrated the fiberboard substrate to a certain depth, which impacted the bonding performance of the substrate.

CONCLUSIONS

1. The best nickel-plating conditions were the following: $NiSO_4$ concentration of 35 g/L, plating temperature of 70 °C, pH of 9, and plating time of 15 min. When nickel-plated carbon fiber was added to the core layer of composite fiberboard and the grid arrangement was more than 11×11, the SE was above 33 dB in the 200 to 1000 MHz frequency range and reached medium grade. In the 19×19 grid arrangement, the minimum and maximum SE were 41.54 dB and 63.73 dB, respectively.
2. When composite fiberboard was laminated with a double layer of plated sheets, its SE ranged from 45.29 dB to 52.01 dB and reached medium grade, an improvement over the single layer sheet, and was relatively stable over the entire frequency range.

3. The MOR, MOE, and IB of the optimized fiberboard met the Chinese national standard. The mechanical properties primarily depended on the performance of the substrate itself, although the adhesive also had some influence.
4. It is feasible that two-layer nickel-plated carbon fiber with a 19×19 grid arrangement can be used to make composite fiberboard with high SE, mechanical properties greater than the national standard, and an easily industrialized process.

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