# Preparation of Fine Ni Particles and Their Shielding Effectiveness for Electromagnetic Interference

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Received 11 October 2007; accepted 29 April 2008 DOI 10.1002/app.28654 Published online 9 July 2008 in Wiley InterScience (www.interscience.wiley.com).

**ABSTRACT:** Fine Ni particles with sphere-like architecture were synthesized via a wet chemical route in distilled water. The resulting fine Ni particles and/or commercial microsized Ni particles were then added to a mixed resin solution to fabricate resin-based conductive composites. The shielding effectiveness (SE) of the resultant conductive composites for electromagnetic interference was measured as a function of nickel mass fraction. The results indicated that the SE values of the two kinds of Ni-containing resinbased composites increased with increasing loading of Ni filler. Moreover, the fine Ni particles, in the absence of any protective agents, were liable to aggregate for the sake of decreasing surface energy, which could be well avoided by ultrasonic disposal. The resin-based conductive compo-

### INTRODUCTION

In the so-called information era, the increased electromagnetic radiation produced by various electronic appliances is attracting attention increasingly along with the development and progress in electronic information and communication technologies. This is because the radiated electromagnetic interference (EMI) not only can cause the abnormal operation of electronic products but also is harmful to human health.<sup>1,2</sup> Therefore, the shielding of this radiated EMI has caused increased concern to materials scientists.<sup>3–5</sup> Usually, the EMI shielding effectiveness (SE), composed of three portions including reflection, absorption, and multiple reflections, is used to depict the shielding performance of the task-specific materials. Where reflection is a result of the impedance mismatch between air and the sample tested at the frequency of interest, absorption is attributed to the energy dissipation along with the interaction between electromagnetic waves and materials, and multiple reflections are caused by the inhomogeneity

sites containing a low concentration (33.3 wt %) of the ultrasonically disposed fine Ni particles recorded an SE value as much as above 22 dB in a frequency range of 130 MHz to 1.5 GHz, which could not be realized for the composites filled with microsized nickel particles unless the mass fraction of the Ni filler in this case was as high as 50.0 wt %. In other words, the ultrasonically disposed fine Ni particles could be used as efficient lightweight filler for shielding of electromagnetic interference. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 110: 569–577, 2008

**Key words:** fine Ni particles; fillers; conducting polymers; composites; electromagnetic shielding

within the materials. Usually, the higher the SE value in decibel, the less the energy passing through the sample, and the resultant signal attenuation is proportional to the electrical conductivity and magnetic permeability of the materials. Namely, high conductivity ( $\rho$ ) and dielectric constant ( $\epsilon$ ) of materials contribute to high EMI SE.<sup>6</sup>

Nowadays conductive polymers have gained popularity because of their easy synthesis, cost-effectiveness, mass production, design flexibility, etc.<sup>7-10</sup> The conductive fillers commonly used for such a purpose include carbon black, carbon fiber, metal powder, metal-coated fiber, metal fibers, etc.,<sup>8,11-14</sup> which usually need to be incorporated into the conductive composites at higher mass or volume fractions so as to achieve sufficient conductivity for EMI shielding.<sup>15</sup> For example, the metallic fillers, because of their high density, usually need to be added at a weight fraction of 60%,<sup>16</sup> which will inevitably negate the low weight advantage normally associated with polymer composites and may cause problems in the processing of the composite materials as well. Furthermore, high filler contents usually mean decreased strength and ductility of a composite. Thus it is critical to reach as much as possible a proper balance between the electrical conductivity, mechanical properties, and processing characteristics of the conductive composites. One of the efforts in

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Contract grant sponsor: Ministry of Science and Technology of China; contract grant number: 2007CB607606.

Journal of Applied Polymer Science, Vol. 110, 569–577 (2008) © 2008 Wiley Periodicals, Inc.

this respect is to substitute microsized metallic particles with fine ones, which could be practicable since fine particles usually have small size effect and various specific properties as compared with the microsized counterparts.

Typical metals such as Ni, Ag, and Cu are the most commonly used metallic fillers for EMI shielding, and Ni, in particular, offers an appropriate choice owing to its lower cost than silver and better thermal stability than copper.<sup>17</sup> Unfortunately, a limited amount of reports is currently available on Nifilled, and in particular, fine Ni-filled conductive composites, which might be due to the difficulties in fabrication and dispersion as well of the fine Ni particles.<sup>18–20</sup> To fabricate metal powders with desired properties via economical routes, the reduction of nickel salts in aqueous solution is usually tried for the preparation of fine nickel powders.<sup>21–23</sup> The morphologies and properties of the nickel powders prepared using this method are strongly influenced by the reaction parameters including the metallic precursors, the reducing agents, the surfactants, etc. The surfactant molecules coated on the nickel particles could prevent the single particle from growing and several particles from coming together.<sup>23</sup> However, the remnant surfactants on the particle surface will act as impurities and significantly reduce the conductive capability of the metallic fillers. Thus, in the present work, a simple chemical reduction route was established for the preparation of fine Ni powders from an aqueous solution without any surfactant molecules. This could be a cost-effective and promising method for the preparation of fine Ni particles at ambient pressure. And more important, the method could be potentially extended to realize large-scale production of fine Ni particles, which would be critical to the application of the fine powders in shielding of electromagnetic interference.

The aim of the present research is to prepare fine Ni powders and investigate their applicability as conductive fillers in polymer–matrix composites for shielding of electromagnetic interference and the effect of the filler size on the shielding effectiveness, using commercial microsized Ni particles (International Nickel Company, type T255) mixed with resin for a comparison.

#### **EXPERIMENTAL**

### Synthesis and characterization of fine Ni particles

All the reagents are analytical grade and used without further purification. In a typical process, 0.20 mol of nickel chloride (NiCl<sub>2</sub>·6H<sub>2</sub>O, Kermel Chemical Co., Tianjin, China) was dissolved, using a 500 mL flask beaker, into 240 mL of distilled water and stirred vigorously at room temperature, allowing the generation of a transparent green solution after homogeneous dispersion of Ni<sup>2+</sup>. Then 60 mL of 80 wt % hydrazine monohydrate (N<sub>2</sub>H<sub>4</sub>·H<sub>2</sub>O, Kermel Chemical Co.) solution was added drop wise into the green solution, allowing it to change into a mauve solution in about 30 min, whose pH was kept at 11 by adding sodium hydroxide (NaOH, Deen Chemical Co., Tianjin, China). Finally, the precursor solution was heated to 70°C, allowing the generation of the fine Ni particles via reduction reaction. The details about the effects of the quantities of N<sub>2</sub>H<sub>4</sub>·H<sub>2</sub>O and NaOH, and the concentration of NiCl<sub>2</sub>·6H<sub>2</sub>O on the formation of the fine Ni particles will be reported elsewhere.

The as-synthesized fine Ni particles were collected making use of magnetic separation, and washed with distilled water and alcohol, respectively, for several times. After being dried in a vacuum oven at 40°C, the resultant samples were characterized using a Philips X' Pert Pro X-ray diffractometer (XRD, Cu-K $\alpha$  radiation,  $\lambda = 0.15418$  nm) and a JEOL JSM-5600LV scanning electron microscope (SEM, acceleration voltage 20 kV). A JEOL JEM-2010 transmission electron microscope (TEM, accelerating voltage 200 kV) was also performed to observe the microscopic images of the fine Ni particles. The samples for the TEM analysis were collected via centrifuging separation. After being washed and dispersed with alcohol in the presence of ultrasonic stirring, a few drops of the alcohol solution containing dispersed fine Ni particles were introduced onto a carbon-coated copper grid and allowed to evaporate in air at room temperature. Then the samples were ready for the TEM analysis.

## Preparation of the conductive composites

The conductive composites were prepared using a multifunctional sand-mill equipment (KDS-180) rotating at a speed of 1600 r/min. The synthesized fine Ni particles and commercial microsized particles were used as the fillers and a mixed resin of Neo-Cryl B-728 (MMA homopolymer,  $M_w = 65,000, T_o =$  $111^{\circ}$ C, density = 1.19 g/cm<sup>3</sup>, DSM Co., Holand) and CAB-381 (cellulose acetate butyrate,  $M_w = 70,000, T_g$  $= 141^{\circ}$ C, density = 1.20 g/cm<sup>3</sup>, Eastman Co., USA) was used as the matrix material. 6g of NeoCryl B-728 and 4 g of CAB-381 were dissolved firstly at room temperature, and then Ni particles were mixed into the resin solution at different loading levels from 2 g to 45 g. After being diluted to an appropriate viscosity using 2-butanone (one of the mixed solvents of the resin solution), the Ni-containing resin mixtures were painted into annularly shaped resinbased composite samples with an outer diameter of 115 mm, inner diameter of 12 mm, and a thickness of about 0.4 mm using an acrylonitrile- butadiene-

| Resin-Based Composites |                                       |                               |                                  |   |  |  |  |  |
|------------------------|---------------------------------------|-------------------------------|----------------------------------|---|--|--|--|--|
| Sample<br>no.          | Mass ratio of<br>mixture (Ni : resin) | Mass fraction<br>of Ni (wt %) | Volume fraction<br>of Ni (vol %) | Volume resistivity $ ho_v (\Omega \cdot cm) \times 10^{-3}$ |  |  |  |  |
| 1                      | 2/10                                  | 16.7                          | 2.6                              | $\infty$  |  |  |  |  |
| 2                      | 5/10                                  | 33.3                          | 6.3                              | 875.0   |  |  |  |  |
| 3                      | 7.5/10                                | 42.9                          | 9.1                              | 98.0  |  |  |  |  |
| 4                      | 10/10                                 | 50.0                          | 11.8                             | 24.6  |  |  |  |  |
| 5                      | 12.5/10                               | 55.6                          | 14.4                             | 9.7   |  |  |  |  |
| 6                      | 15/10                                 | 60.0                          | 16.8                             | 6.6   |  |  |  |  |
| 7                      | 20/10                                 | 66.7                          | 21.2                             | 5.1   |  |  |  |  |
| 8                      | 30/10                                 | 75.0                          | 28.7                             | 3.8   |  |  |  |  |
| 9                      | 35/10                                 | 77.8                          | 32.0                             | 2.4   |  |  |  |  |
| 10                     | 40/10                                 | 80.0                          | 34.9                             | 3.6   |  |  |  |  |
| 11                     | 45/10                                 | 81.8                          | 37.6                             | 2.9   |  |  |  |  |
|                        |                                       |                               |                                  |   |  |  |  |  |

TABLE I The Compositions and Volume Resistivity of Microsized Ni-Filled Resin-Based Composites

styrene (ABS) mold. The resulting resin-based conductive composites were then used for the study of the EMI shielding behavior. The conductive composites filled with microsized Ni particles were also painted into plates of  $20 \times 120 \times 0.1 \text{ mm}^3$  in the same manner and used for the measurement of electrical conductivity.

#### Measurement of electrical conductivity

A digital milliohm meter (Model YF-508,  $0 \sim 20 \text{ K}\Omega$ , Yu Fong Electric Co., Taiwan, China) was used to measure the resistance values of the resin-based composites containing different loading levels of microsized Ni powders, making use of Kelvin (4wire) resistance measurement technique. At the same time, a digital multimeter (Model DT-9208,  $0 \sim 20 \text{ M}\Omega$ , Advanced Power Electronics Instrument Co., Shenzhen, China) was also used when the resistivity value was too high and beyond the scale of YF-508. The volume resistivity values of the samples were calculated using the following equation<sup>5</sup>:

$$\rho_v = R \times \delta \times d/L \quad (\Omega \cdot \mathrm{cm}) \tag{1}$$

where  $\rho_v$  is the volume resistance of the sample ( $\Omega$  cm), *R* is the resistance value of the sample ( $\Omega$ ),  $\delta$  is the thickness of the sample (cm), *d* is the width of the sample (cm), and *L* is the effective length of the sample (cm). The compositions of the microsized Nifilled composite samples for the resistance measurement and the calculated volume resistivity values ( $\Omega$  cm) are shown in Table I. Since the weight loading of the fillers might be nonlinearly proportional to the volume loading, eq. (2) was used to transform the weight loading into volume loading for any combinations of filler and polymer.

Volume loading (%) = 
$$w_f / [w_p(d_f/d_p) + w_f] \times 100$$
 (2)

where  $w_f$  and  $w_p$  are the mass of the filler and polymer, respectively, and  $d_f$  and  $d_p$  are the true density of the particles and the polymer, respectively.

# Measurement of EMI shielding

A series of resin-based composites containing different loadings of fine or microsized Ni fillers was prepared to study their EMI shielding performance. The mass fractions of the fine or microsized Ni particles in the composites are 16.7%, 33.3%, 50.0%, 66.7%, 75.0%, and 80.0%, respectively, and the corresponding composites samples for the EMI shielding tests are labeled as U1, U2, U3, U4, U5, and U6 (filled with fine Ni particles), respectively, or M1, M2, M3, M4, M5, and M6 (filled with micro-sized Ni particles), respectively. The SE values of all the composites samples in a plane-wave condition/simulation were measured using coaxial cable method.<sup>5</sup> The scanning was run in frequencies from 130 to 1500 MHz to generate 29 data points, and the average SE values of the 29 data points were calculated and cited in this article. The details about the compositions and averaged SE values of the two types of resin-based composites are given in Table II.

# **RESULTS AND DISCUSSION**

#### Characterization of Ni fillers

The XRD patterns of the synthesized fine Ni particles and commercial microsized Ni particles are shown in Figure 1. It is seen that the characteristic diffraction peaks of the two kinds of powder samples can be well indexed to face-centered cubic Ni (PDF standard cards, JCPDS 04-0850, space group Fm3m). At the same time, the diffraction peaks of the synthesized fine Ni particles show obvious broadening [Fig. 1(A)] as compared with that of the commercial microsized Ni particles [Fig. 1(B)], indicating that the synthesized fine Ni particles were

| Material                      | Mass ratio of<br>mixture (Ni : resin) | Mass fraction<br>of Ni (wt %) | Sample<br>no. | SE (dB) |
|-------------------------------|---------------------------------------|-------------------------------|---------------|---------|
| Resin-based composites        | 2/10                                  | 16.7                          | U1            | -0.2    |
| filled with fine Ni           | 5/10                                  | 33.3                          | U2            | -9.5    |
|                               | 10/10                                 | 50.0                          | U3            | -24.7   |
|                               | 20/10                                 | 66.7                          | U4            | -29.6   |
|                               | 30/10                                 | 75.0                          | U5            | -36.6   |
|                               | 40/10                                 | 80.0                          | U6            | -36.2   |
| Resin-based composites filled | 2/10                                  | 16.7                          | M1            | -0.3    |
| with microsized Ni            | 5/10                                  | 33.3                          | M2            | -9.2    |
|                               | 10/10                                 | 50.0                          | M3            | -25.7   |
|                               | 20/10                                 | 66.7                          | M4            | -42.6   |
|                               | 30/10                                 | 75.0                          | M5            | -50.3   |
|                               | 40/10                                 | 80.0                          | M6            | -55.2   |

 TABLE II

 The Compositions and Average SE within 130 ~ 1500 MHz of Various Resin-Based

 Composites Filled With Fine or Commercial Microsized Ni Particles

composed of nanocrystallites. The crystalline size of the fine Ni powders was calculated to be about 65 nm, using Scherrer's equation  $D = 0.89\lambda/\beta \cos \theta$ , where  $\lambda$  is the X-ray wavelength,  $\beta$  is the peak width at half-maximum of the corresponding XRD pattern, and  $\theta$  is the Bragg diffraction angle.<sup>24</sup> Interestingly, no oxides and/or hydroxide such as NiO or Ni(OH)<sub>2</sub> were detected by the XRD analysis, which could be because of the reaction shown below:

$$2Ni^{2+} + N_2H_4 + 4OH^- = 2Ni + N_2\uparrow + 4H_2O \quad (3)$$

In other words, at appropriate pH value, temperature, and hydrazine concentration for the preparation of the fine Ni particles, the above reaction could facilitate an inactive atmosphere of  $N_2$  gas which could hence protect the product, that is, fine Ni particles, from being oxidized or hydrolyzed by water.

The SEM morphologies of the synthesized fine Ni powders and the commercial microsized Ni particles are shown in Figure 2, where the corresponding TEM pattern of the synthesized fine Ni particles is



Figure 1 XRD patterns of synthesized fine Ni (A) and commercial micro-sized Ni particles (B).

also shown as an inset in Figure 2(A). It is seen that the synthesized fine Ni particles had sphere-like shape and were about 500 nm in averaged diameter, which is far smaller than 2.5 µm, that of the commercial microsized Ni particles. Thus in a strict sense, the fine Ni particles synthesized in the present research cannot be reckoned as a kind of nanoparticles. This might be attributed to two factors. On one hand, a very high concentration of Ni<sup>2+</sup> ( $\sim 0.8M$ ) was used in the reaction system for the present research, which would inevitably lead to aggregation of the fine Ni particles as the product. On the other hand, no soluble polymers or surfactants were used to control the growth of the Ni crystals and to prevent them from aggregation as well. Fortunately, when skin effect is taken into account, the unit size of the fine Ni particles as the filler should be comparable with or less than the skin depth ( $\delta = 0.47 \ \mu m$ , at 1 GHz), which means that the fine Ni particles prepared in the present work could be theoretically preferred as a kind of conductive fillers for shielding of electromagnetic interference.<sup>25</sup>

# Effect of microsized nickel powder content on the conductivity of composites

In the present work, the electrical conductivity of the resin-based conductive composites filled with microsized nickel powders was measured as a function of Ni content to determine the percolation threshold of such systems. As shown in Table I and Figure 3, the average resistivity of the resin-based composites decreased with increasing Ni content. The composite with a low content (16.7 wt %, i.e., 2.6 vol %) of microsized Ni filler had a very high resistivity beyond the scale of the instrument (>10<sup>5</sup>  $\Omega \cdot$  cm), indicating that in this case the metallic filler was inadequate to form electrical conducting path. When the loading level of the Ni filler in the resin-based composites rose to 33.3 wt % (6.3 vol %), the



Figure 2 SEM images of synthesized fine Ni particles (A) and commercial micro-sized Ni particles (B). Inset: TEM image of the synthesized fine Ni particles.

resistivity became measurable (875  $\times$   $10^{-3}~\Omega$   $\cdot$  cm) and it sharply decreased to  $98.0 \times 10^{-3} \Omega \cdot cm$  when the loading level of the micro-sized Ni filler increased to 42.9 wt % (9.1 vol %). Above a loading level of 50 wt % (11.8 vol %), the volume resistivity kept an inconspicuous decrease with further increase in the filler loading level. The rapid decrease in the volume resistivity of the resin-based composite (also referred to as percolation threshold) at a filler loading level of 6.3 vol % could indicate that in this case conductive networks had been formed via the contact and interlocking of the conductive particles, thus the percolation threshold was estimated to be around 33.3 wt % (6.3 vol %). It was supposed that the fine Ni-filled resinbased composites had similar electrical resistivities as the microsized Ni-filled counterparts (data not shown).

# Shielding effectiveness for EMI of the Ni-filled composites

Figure 4 shows the influence of the mass fraction of two types of Ni particulates on the SE values of the cor-



Figure 3 Resistivity of microsized Ni-filled resin-based composites as a function of Ni mass fraction.

responding Ni-filled conductive composites for shielding of electromagnetic interference in a frequency range of  $130 \sim 1500$  MHz. It is seen that composites U1 and M1 had an average SE value of only 0.2 dB and 0.3 dB [Fig. 4(A,B) and Table II), respectively, indicating that the composites containing lower mass fraction of Ni powders almost had no shielding effect for electromagnetic interference. This could be attributed to the discontinuous distribution of the conductive metallic fillers in those two composites, which could be confirmed by the SEM images of the composite samples shown in Figures 5 and 6. The images for the composite samples U1 and M1 show obvious signs of disconnection and interruption of embedded filler particulates as the conductive pathways, thus preventing the formation of conducting paths for electricity and showing no EMI shielding capacity. Therefore, it would be imperative to keep a high enough loading of the Ni powders as the fillers in the conductive composites for electromagnetic interference shielding.

In general, the SE values of the two types of Nifilled composites increased with increasing nickel loading, which well conformed to the resistivities of the composites shown in Figure 3. Moreover, the two types of Ni-filled composites showed minor differences in SE values when the mass fraction of the Ni fillers was below 50.0%. Namely, at a filler content of 33.3 wt %, composites U2 and M2 had averaged SE values of  $\sim 9.5$  dB and  $\sim 9.2$  dB, respectively, and composites U3 and M3 containing 50.0 wt % of the Ni fillers recorded averaged SE values of  $\sim 24.7$  dB and  $\sim 25.7$  dB, respectively.

It could be rationally anticipated that at a fixed amount of addition, the fine Ni filler, due to its much smaller size than the micro-sized Ni filler, should contain a larger number of particles, which would be beneficial to enhancing the interconnection among the particles and the electrical conductivity of the composites as well. However, when the



**Figure 4** Variations of the SE of various resin-based composites containing different concentrations of synthesized fine Ni particles (A) and commercial microsized Ni particles (B).

loading of the fillers rose to 66.7 wt % and above, the composites filled with micro-sized Ni had obviously higher SE values than those filled with fine Ni at the same filler mass fraction (compare samples U4 and M4, U5 and M5, and U6 and M6 in Fig. 4 and Table II). This was supposed to be due to the different natures of the synthesized fine Ni and the commercial microsized Ni particles. In other words, the fine Ni particles with high surface energy and activity would be more liable to aggregate as compared to the microsized Ni particles, which would be harmful to the uniform distribution of the fine Ni filler in the filled composites and hence to the desired performance of shielding for electromagnetic interference as well. This can be partly confirmed from the SEM pictures of the composites shown in Figures 5 and 6. Actually, no matter which kind of the fillers was used, the Ni particles had nonuniform distribution in the composites, which was especially so for the fine Ni filler. Moreover, the local aggregation of the fillers became increasingly severe with increasing loading of the fillers, and the fine Ni particles seemed to be more liable to local aggregation than the microsized Ni particles at the same filler loading level, which could be confirmed even by naked-eye observation and by a careful comparison of the SEM images of composite samples U4 and M4, U5 and M5, and U6 and M6 (see Figs. 5 and 6).



**Figure 5** SEM images of resin-based composites containing different concentrations of synthesized fine Ni particles.



Figure 6 SEM images of resin-based composites containing different concentrations of commercial micro-sized Ni particles.

Aside from the characteristics and loading level of the conductive fillers, many other factors might also affect the conductive performance and EMI shielding effectiveness of polymer-matrix conductive composites. For example, Chou et al. reported that when nickel powders were mixed in acrylonitrile-butadienestyrene (ABS) melt using strong shearing at high temperatures (Brabender-mixing method), the resulting composite sample with 15 vol % Ni powders had a very poor EMI shielding effectiveness of 4.8 dB, while the composite prepared using dry-mixing method had a high EMI shielding effectiveness of about 54 dB (low nickel level of 5 vol %).<sup>20</sup> In the present study, the resin-based conductive composites were prepared at room temperature using MMA homopolymer and cellulose acetate butyrate as the mixed resin and the same kind of nickel powders (International Nickel Company, type T255) as the conductive fillers, making use of simple solution-blending technology. The resulting resin-based conductive composite containing 11.8 vol % fine Ni powders had an EMI shielding effectiveness of 25.7 dB, higher than that of the composite prepared using Brabender-mixing method but lower than that of the one prepared using dry-mixing method as mentioned earlier.

It is a common practice to properly decrease conductive filler loading so as to increase the workability of the composites or adjust other physical properties, where the uniform distribution of the filler in the composites plays a key role in determining the properties. Usually, a certain amount of dispersants is needed to protect fine particles from severe aggregation and maintain their dispersion stability in filled composites. However, the organic compounds as the dispersants will act as impurities on the particle surface and significantly reduce the conductive capability of the metallic fillers. Therefore, no protective agents or dispersants were introduced during the preparation of the fine Ni particles in the present work, which could be helpful to avoiding harm to the conductive capability of the target composites by the organic "impurities"; and instead, ultrasonic dispersion was used to realize well dispersion of the Ni fillers in the resin-based composites. For this purpose, the fine and microsized Ni particles were both ultrasonically treated and then used to prepare the resin-based composites containing 33.3 wt % of the treated fillers, aiming at revealing the effect of the filler dispersion on the SE values of the filled resin-based composites. Thus an appropriate amount of fine or microsized Ni particles was, in the presence of ultrasonic stirring, dispersed with 2-butanone (the diluent of the resin solution) at room temperature for about 20 min. The dispersed solution was then mixed into the acrylate resin solution using a high-shear mill, and the composite samples filled with the ultrasonically dispersed Ni fillers, labeled as U2\* and M2\*, respectively, were prepared in the same manner as that for the U-series and M-series composites samples and served for measurement of the SE values and observation of SEM as well. To our surprise, as shown in Figure 7, the composite sample U2\* had an SE value of 22.5  $\sim$  27.1 dB in a frequency range of 130  $\sim$  1500 MHz, which is almost 10 dB higher than that of the composite sample U2 at all frequencies. Contrary to



**Figure 7** Effect of ultrasonic disposal on the SE of composites: the synthesized Ni composites (U2 and U2\*) and the commercial Ni composites (M2 and M2\*).

the above, the composite sample M2\* recorded an SE value of 4.4 dB, much smaller than 9.2 dB, that of the composite sample M2, indicating that, at a low filler concentration, the ultrasonic dispersion of the microsized Ni filler was harmful to the conductive capability of the resin-based composites for shielding of electromagnetic interference.

The SEM images of the composite samples U2\* and M2\* are shown in Figure 8. It is seen that, though the fillers in these two composites still had no uniform enough distribution, they were really dispersed to a better degree therein as compared with the counterparts U2 and M2. It should be pointed out that the ultrasonic disposal of the fine and microsized Ni particles had totally inverse effects on the shielding effectiveness of the corresponding resin-based conductive composites, though the ultrasonic disposal contributed to improving the homogeneity of the spatial distribution of the both types of Ni filler in the resin. At a certain loading level of the ultrasonically disposed Ni particles, more fine Ni particles would take part in building the conductive paths owing to the larger numbers and improved dispersion as compared with commercial microsized Ni particles, thus sample U2\* had better SE. In engineering practices, the conductive particles are generally required to distribute, more or less, nonuniformly in conductive composites for attaining desired conductive performance. In other words, the aggregation to some extent of the conductive filler is beneficial to EMI shielding, because it could help to form a three-dimensional



Figure 8 SEM images of the resin-based composites filled with untreated Ni particles (U2 and M2) and with ultrasonically treated Ni particles (U2\* and M2\*).

conductive network throughout the whole composite sample.  $^{\rm 20}$ 

# CONCLUSIONS

A simple and economical method was successfully established to realize large-scale production of fine Ni particles with a size of  $\sim 500$  nm. The resulting fine Ni particles were used as the filler to prepare resinbased conductive composites for shielding of electromagnetic interference. The resin-based conductive composites filled with commercial microsized Ni particles were also prepared and investigated for a comparative study. It was found that the two types of filled resin composites with relatively lower contents of Ni fillers (below 33.3 wt %) had minor differences in terms of their shielding effectiveness for electromagnetic interference. However, when the mass fraction of the Ni fillers rose to 50.0 wt % and above, the corresponding resin composites filled with fine Ni particles had poorer shielding effectiveness for electromagnetic interference than those filled with microsized Ni particles. This could be related to the excessive aggregation of the fine particles. Surprisingly, the ultrasonic disposal of the fine and microsized Ni particles had totally inverse effects on the shielding performance of the corresponding resin composites, when the mass fraction of the ultrasonically treated Ni fillers was fixed as 33.3 wt %. Namely, sample U2\* had a much higher SE value than composite sample U2 within the whole tested range of frequency; but composite sample M2\* had a much smaller SE value than composite sample M2. The ultrasonically treated fine Ni particles could be used as a kind of efficient lightweight EMI shielding materials. Further work is needed in this respect and will be dealt with elsewhere.

Special thanks are due to Dr. Laigui Yu for help in manuscript preparation.

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