บทความวิจัย

พฤติกรรมการบ่ม สัณฐานวิทยา และค่าคงที่ไดอิเล็กตริกของอีพอกซียืดหยุ่นผสมอนุภาคทองแดง ท่อคาร์บอนขนาดนาโนเมตรแบบผนังชั้นเดียว และท่อคาร์บอนขนาดนาโนเมตรแบบผนังซ้อนกันหลายชั้น ด้วยวิธีการบ่มโดยใช้รังสียูวี

Cure Behavior, Morphology and Dielectric Constant of Flexible Epoxy Composite with Cu Particle, SWCNT and MWCNT Nanoparticle by UV-Cure Technique

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บทคัดย่อ

อีพอกซียืดหยุ่นถูกบ่มด้วยรังสียูวีที่อุณหภูมิห้องโดยใช้หลอดไฟแอลอีดียูวีเป็นแหล่งกำเนิดแสง โดยอีพอกซีที่ศึกษาจะถูกผสมด้วยสารเคมีสองระบบคือระบบการบ่มโดยใช้รังสียูวีโดยมีสารริเริ่มเชิงแสงชนิดเกลือออนเนียมในระบบการบ่มและใช้ระบบการบ่มโดยอาศัยความร้อนที่มีสารบ่มเป็นสารประกอบจำพวกเอมีน โดยจะทำการทดสอบระบบการบ่มโดยใช้รังสียูวีของอีพอกซียืดหยุ่นและอีพอกซียืดหยุ่นผสมอนุภาคทองแดง ท่อคาร์บอนขนาดนาโนเมตรแบบผนังชั้นเดียว (SWCNT) และท่อคาร์บอนขนาดนาโนเมตรแบบผนังซ้อนกันหลายชั้น (MWCNT) จากปริมาณหมู่อีพอกไซด์ที่เหลือจากการทำปฏิกิริยาการบ่มโดยใช้รังสียูวี โดยหมู่อีพอกไซด์ที่เหลือจะทำปฏิกิริยาการบ่มโดยอาศัยความร้อนกับสารบ่มจำพวกเอมีนที่ใส่เข้าไปในระบบระหว่างการทดสอบด้วยเครื่องวัดความแตกต่างทางความร้อนของสารจากการทำปฏิกิริยา (DSC) นอกจากนี้ยังใช้เครื่องวัดค่าการดูดกลืนของแสงเพื่อยืนยันผลการทดลองที่ได้จากการทดสอบด้วยเครื่องวัดความแตกต่างทางความร้อนของสาร ชิ้นงานที่ผ่านการบ่มด้วยรังสียูวีอย่างสมบูรณ์แล้วจะถูกวิเคราะห์ลักษณะทางสัณฐานวิทยาด้วยกล้องจุลทรรศน์อิเล็กตรอนแบบส่องกราด (SEM) และทดสอบค่าคงที่ไดอิเล็กตริกโดยใช้อุปกรณ์แอลซีอาร์ (LCR meter) ที่ความถี่ 1 กิโลเฮิร์ต จากการศึกษาพบว่าอีพอกซียืดหยุ่นสามารถบ่มโดยใช้รังสียูวีจากหลอดไฟแอลอีดียูวีได้ในเวลา 2-3 ชั่วโมง หากเติมอนุภาคคาร์บอนลงไปจะทำให้พฤติกรรมการบ่มเกิดขึ้นได้ช้าลง นอกจากนี้ยังพบว่าค่าคงที่ไดอิเล็กตริกของอีพอกซีผสมท่อคาร์บอนขนาดนาโนเมตรแบบผนังชั้นเดียว (SWCNT) มีค่าสูงสุด เนื่องจากลักษณะสัณฐานวิทยาและโครงสร้างภายในของอีพอกซีผสมที่มีพื้นที่ผิวมากของท่อคาร์บอนขนาดนาโนเมตรแบบผนังชั้นเดียวและลักษณะโครงสร้างที่มีฟองอากาศภายใน

คำสำคัญ : อีพอกซี, อนุภาคทองแดง, ท่อคาร์บอนขนาดนาโนเมตร, บ่มรังสียูวี, ค่าคงที่ไดอิเล็กตริก

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Abstract

The flexible epoxy cured by UV light source at room temperature using LED UV lamp was determined. The hybrid system of epoxy between photo-initiator and thermal curing agent was used for observing the cure behavior of epoxy and epoxy composites in UV-Cure system. The remained epoxy functional group after curing by UV light was determined by differential scanning calorimetry (DSC). Moreover, the UV-Visible spectrophotometry was used as another method to confirm and support the cure behavior of neat epoxy and epoxy composites. The morphology was evaluated by scanning electron microscopy (SEM) to reveal the cross sectional surface of specimen after mixing under atmospheric pressure and curing with UV light. Furthermore, the dielectric constant of neat epoxy and epoxy composites were measured by LCR meter at 1 kHz of frequency. It was found from this work that neat epoxy could be cured under UV light at 2-3 hours while epoxy composites would be cured at longer time than neat epoxy due to the effect of particle in epoxy matrix. The dielectric constant of epoxy composite with single wall carbon nanotube (SWCNT) at 1 phr showed the highest value among epoxy composites in this work. This might be due to the different morphology of epoxy composites and structure of SWCNT in epoxy composites from high surface area and bubble inside during processing.

Keywords : epoxy, copper, carbon nanotube, UV-cure, dielectric constant

Introduction

Recently, researchers developed the LED-UV lamp installed with the nozzle arm in 3 D-printer which could move along and inject the polymer to form 3 D model of materials (Griffini *et al*., 2016; Gul *et al*., 2016). This process is more advantage for processing of thermosetting polymer in 3D-printer than thermal cure method by using thermal curing agent due to layer-by-layer of 3D-printer problem. The thermosetting polymer cured by UV light might be possible to print out in one step without curing in an oven owing to self-crosslink of molecules initiating by photo-initiator and UV irradiation. Among thermosetting of polymer, the epoxy adhesive is one of the appropriate materials for this technique because it shows adhesive property with low thermal expansion behavior. Epoxy can be cured by UV light if the photo-initiator is added to epoxy during mixing and processing. The photo initiator attacks the epoxide ring of epoxy and opens it to crosslink with another chain of epoxy's molecules (Fahem *et al*., 2012). Some of researchers studied on types of photo-initiator and other chemicals that could accelerate crosslink reaction of epoxy adhesive but the epoxy adhesive systems showed different characteristics due to different types of epoxy and condition (Atiff *et al*., 2012; Batoro & Gaspar, 2008; Fahem *et al*., 2012; Sangermano *et al*., 2017; Zhou *et al*., 2016).

The purpose of this work is to develop flexible epoxy composites cured by UV technique. In this research, the carbon nanotubes in term of single wall carbon nanotube (SWCNT) and multi wall carbon nanotube

(MWCNT) were chosen to compare with copper particle in epoxy composites owing to its high thermal and electrical properties. Although there were some works of epoxy-carbon adhesive system, for example, epoxy-carbon black system (Atif *et al*., 2015), epoxy-graphene and derivative of graphene in UV-Cure system (Sangermano *et al*., 2017; Sharif *et al*., 2017) or in thermal cure system (Alhumade *et al*., 2016; Boro & Karak, 2017). The performance of epoxy composites with SWCNT, MWCNT and copper particle in term of dielectric constant and morphology has not been investigated yet from the best of our knowledge. Some researcher studied on epoxy-metal particle system with UV-Cure technique (Asmussen *et al*., 2015; Batoro & Gaspar, 2008; De & Kundu, 2001; Sangermano *et al*., 2011) but they also did not compare the electrical properties with others metal or any types of filler. The electrical properties are the significant factor for using it as electronic packaging product. Electrical properties of epoxy composites depend on process of mixing and types of epoxy matrix. For this reason, the epoxy-carbon nanotube systems were compared with epoxy-copper system and analysis of composites performance in term of dielectric constant was determined. In addition, cure behaviors of epoxy and epoxy composites were investigated by thermal and UV absorption method. Morphology of epoxy composites was observed by scanning electron microscopy to explain the dielectric behavior of epoxy composites.

Methods

Materials

The flexible epoxy (YD 290X90, Phenolic type, 270-305 g/eq of EEW), thermal curing agent (TH 7277, Amine type) and catalyst for thermal system (1, 3-bis (aminomethyl) Cyclohexane) were supported by Aditya Birla Chemicals Co., Ltd., Thailand. The triarylsulfonium hexafluoroantimonate salt, 50% wt in propylene carbonate, purchased from Sigma-Aldrich, USA was used as photo-initiator for UV-Cure system. Single wall carbon nanotube (SWCNT, 85% purity, 3 nanometers of diameter with 5 micrometers of tube length) and Multi wall carbon nanotube (MWCNT, 97% purity, 10-20 nanometers of diameter with 5-15 micrometers of tube length) were purchased from Tokyo Chemical Industry Co., Ltd., Japan. Copper particle (99% purity, particle size <50 micrometers, commercial grade) was used as received without further purification. LED UV lamp housing containing 20 spots (UVA light source, 365 nanometers of wave number, 60 Watts) and disposable cuvette (550 microliters) was purchased from Brilliant Token Co., Ltd., Thailand and P&N Hightec Chemical Ltd., Thailand respectively.

Mixing procedure of epoxy system for cure characteristics

2 g of epoxy was mixed with 28 phr of thermal curing agent, 1 phr of catalyst and 10 phr of photo-initiator in propylene carbonate. Then, it was stirred at room temperature for 10 minutes by magnetic stirrer. The epoxy

system was poured into aluminum mold with 2.7x2.7 centimeter of dimension for testing cure kinetics by thermal method. The thermal method measured by differential scanning calorimetry (DSC) was chosen to determine the remained epoxide group after curing under UV irradiation. The epoxy was mixed with only photo-initiator and poured it into disposable cuvette for UV absorption technique measured by UV-Visible spectrophotometry. The epoxy system in aluminum mold and disposable cuvette was irradiated under LED UV lamp with various time for observing cure characteristics by DSC and UV- Visible spectrophotometry.

Mixing procedure of epoxy composites for dielectric constant measurement

The composition of epoxy, photo-initiator, thermal curing agent, catalyst and mixing procedure were the same as previous section excepted the addition of particle in this section. Copper particle was added in epoxy composites at 100 phr while SWCNT and MWCNT at 1 phr were added to compare the effect of particles in epoxy composites. The epoxy composite with 100 phr of Cu particle was chosen because the Cu particle was separated from epoxy immediately after mixing when adding small amount of Cu particle (less than 100 phr) due to low viscosity of epoxy composites from our preliminary test thus this content was chosen to maintain the stable suspension after mixing. The epoxy composite with SWCNT and MWCNT at only 1 phr was chosen in this work because the physical, thermal and electrical properties of epoxy nanocomposites could be increased at small amount of nanoparticle (Martin, C. *et al*., 2004 and Sharif, M. *et al*., 2017). After mixing for 10 minutes, the epoxy and epoxy composites were poured into an aluminum mold with 2.7x2.7 centimeter of dimension and cured under UV light for 3 hours and 3.5 hours respectively. The irradiated time of UV light for complete crosslink reaction was obtained from cure characteristics. After neat epoxy and epoxy composites were complete cure under UV light, the specimen was trimmed into 2.5x2.5 centimeters for dielectric constant measurement.

Characterization

Cure characteristics of epoxy and epoxy composites cured under UV light were measured by differential scanning calorimetry (DSC, Mettler, Toledo821e, Switzerland) at 10 °C/minute of heating rate from 25 to 350°C and UV-Visible spectrophotometry (Varian, Cary 300, USA) at 600 nanometer/minute of scanning rate from 200 to 800 nanometer of wave number respectively. The specimen after complete cure under UV light was dipped into liquid nitrogen and fractured for observing the morphology. The cross sectional area of specimen after fracturing was observed by scanning electron microscopy (SEM, ZEISS, LEO 1450 VP, USA) at 10 kilovolts of accelerating voltage. In addition, the specimen after complete cure under LED UV lamp at 2.5x2.5x0.2 centimeters of dimension was measured dielectric constant by LCR meter (Agilient Technologies Inc., E4980A, USA) at 1 kilohertz of frequency.

Results

The remained epoxide group after UV irradiation of epoxy system which could be cured by thermal curing agent under heat treatment as can be seen in Figure 1a were measured by DSC. Cure characteristics of neat epoxy was measured between 0-3 hours. The peak of thermal cure in epoxy system displayed around 100-120 °C. The small enthalpy peak was observed at 2-3 hours. The complete cure reaction after irradiated epoxy system by UV and tested by DSC was shown in Figure 1b which did not show the peak of enthalpy. These characteristics were observed at 3 hours of UV irradiation for neat epoxy and 3.5 hours for epoxy-carbon composites. The result in this figure showed that epoxy composite with carbon particle could be cured by our UV light with longer time than epoxy without carbon particle which might be the particle obstructed the epoxy molecules during cross linking of epoxy molecules. In addition, epoxy with photo-initiator without thermal cure system was irradiated by UV light with various time and observed the light absorption by UV-Visible spectrophotometry to support our DSC result. The result of UV-Visible spectrum in Figure 2 revealed that our epoxy system could be complete cured by UV irradiation after 140 minutes (2.3 hours) due to the reduction of absorption peak. It was found from Figure 2 that the absorption peak was shifted from around 330 nm to 310 nm after irradiated by UV light at 140 minutes which might be due to the complete crosslink reaction of epoxy structure. Although the result from Figure 1a displayed that the complete crosslink reaction of neat epoxy could be observed at 2 hours, the result from UV-Visible spectrophotometry in Figure 2 which was another technique to identify the complete cure of neat epoxy was observed at 140 minute (2.2 hours) owing to the shift of wavenumber after this testing time. It might be concluded that our epoxy system could be complete cured by UV light around 2-3 hours.

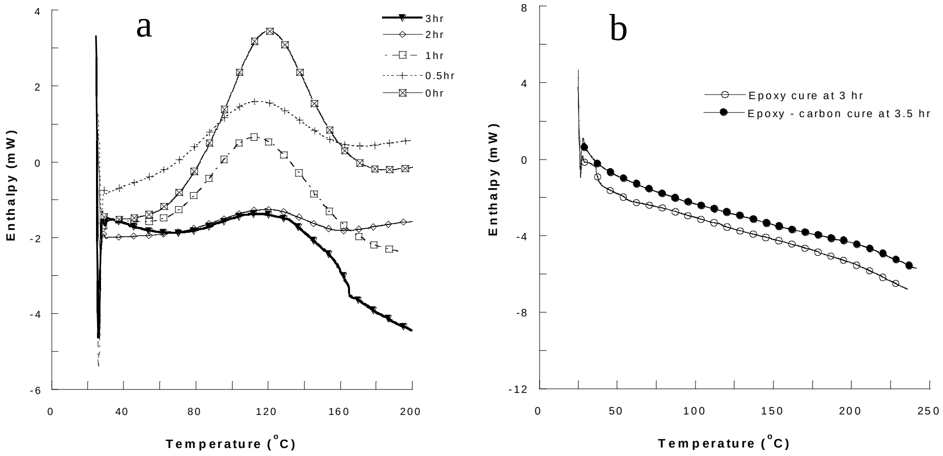

Figure 1. Cure behavior of neat epoxy (a) and comparison between neat epoxy and epoxy - carbon composites (b) cured by UV system

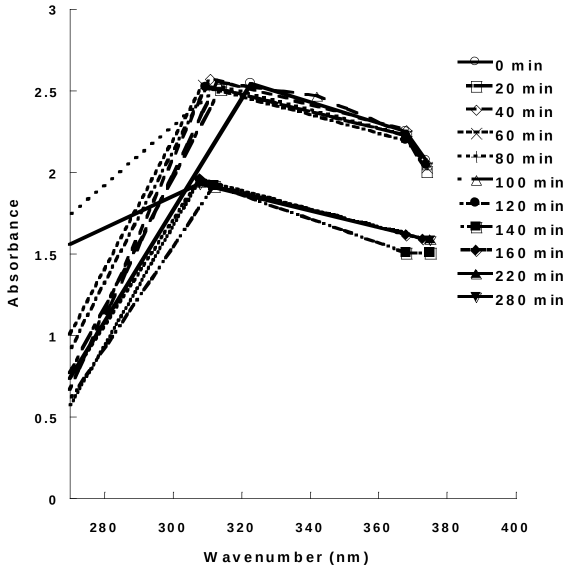

Figure 2 UV-Vis spectra of neat epoxy cured by UV light source

The specimen after complete crosslink reaction by UV light in aluminum mold was fractured and observed the morphology of cross sectional area by scanning electron microscopy (SEM) as can be seen in Figure 3. Neat epoxy (Figure 3a) showed the smooth surface without large bubble inside epoxy matrix while epoxy composite with Cu particle (Figure 3b) showed the particle separation from epoxy matrix. In addition, epoxy composite with 1 phr of carbon nanoparticle in Figure 3c and 3d showed the different morphology. Epoxy composite with single wall carbon nanotube (SWCNT) exhibited the bubble inside epoxy matrix but epoxy composite with multiwall carbon nanotube (MWCNT) showed slightly surface roughness without bubble at the same composition of carbon nanoparticle. .

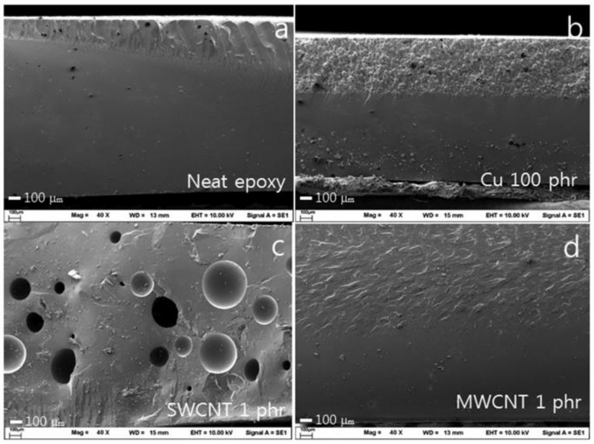

Figure 3 SEM images of neat epoxy (a), Epoxy – Cu composites at 100 phr (b), Epoxy – SWCNT composites at 1 phr (c) and Epoxy – MWCNT composites at 1 phr

Neat epoxy and epoxy composites after complete cure by UV light were cut into a square shape (2.5x2.5 centimeter of dimension). The thickness of specimen was around 1-2 millimeters. The specimen was measured by LCR meter at 1 kilohertz of frequency. The equivalent parallel capacitance of specimen (C_p) was measured and calculated the dielectric constant by using equation 1 as follow

$$\varepsilon_r = \frac{t_m \times C_p}{A \times \varepsilon_0} \qquad (1)$$

Where ε_r = dielectic constant, t_m = thickness of specimen (m), C_p = equivalent parallel capacitance of specimen (F), A = area of specimen (m^2) and ε_0 = permitivity of free space which equal to 8.854 F/m

The dielectric constant of neat epoxy and epoxy composites in Figure 4 showed that neat epoxy was the lowest dielectric constant value while 100 phr of copper in epoxy composites which was the highest composition of particle in this work showed slightly increment of dielectric constant value. Moreover, the result showed that epoxy composite with single wall carbon nanotube at 1 phr displayed the highest dielectric constant value compared to the others specimen in this work

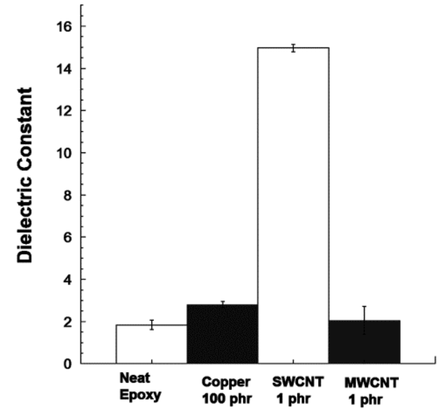

Figure 4 Dielectric constant of neat epoxy and epoxy composites measured by LCR meter

Discussion

Cure characteristics of neat epoxy was measured by DSC after UV irradiation between 0-3 hours. The thermal method was selected to observe the remained epoxide group after UV cure which could be cured with thermal curing agent under heat treatment. The neat epoxy without UV irradiation showed the largest area of enthalpy after measuring by DSC due to the highest amount of remained epoxide group before crosslinking by UV light with photo-initiator. The UV-Cure technique selected by this work was the method to crosslink epoxy molecules together via cationic polymerization reaction. Firstly, the onium salt of photo-initiator was irradiated by UV light to form aryl-cation and Bronsted acid (HX). Bronsted acid was the initiator to attack the oxirane ring from epoxy molecules to form secondary onium ion (Oxonium ion). Finally, the secondary onium ion induced others oxirane ring from another epoxy molecules via ring opening mechanism which can cross liked together to form three dimensional network (Atif *et al*., 2015). In Figure 1(a), the enthalpy of neat epoxy reduced after UV cure system with various UV irradiation time due to the reduction of epoxide group introducing low energy of crosslink network by thermal curing agent from heat treatment. Moreover, the small peak of enthalpy was observed after 2-3 hours since the crosslink network by UV irradiation was occurred at the surface of epoxy before going deep inside the specimen. Some area at below surface of specimen might not be cross linked by UV light. Thus, the crosslink network by thermal curing agent under heat treatment of neat epoxy cured by UV at 2-3 hours was observed but it was occurred only small amount of enthalpy. The effect of carbon particle in epoxy composites was revealed in Figure 1(b). The result showed that the addition of carbon particle retarded the crosslink reaction by UV technique owing to the light obstruction behavior of carbon particle and the hindrance of particle during crosslinking of epoxy molecules. In addition, the cure behavior of epoxy and photo-initiator under UV light was determined by using UV-Visible spectrophotometry to confirm our result from thermal method. The reduction of absorbance from UV spectra was observed after 140 minutes (2.3 hours) of UV irradiation which might be due to the effect of crosslink molecules from UV irradiation. It might be concluded from DSC and UV-Visible spectrum that neat epoxy without particle could be cured by UV light at 2-3 hours and epoxy composites could be cured at longer time than neat epoxy due to the effect of filler inside the epoxy matrix.

The cross sectional area of neat epoxy and epoxy composites after curing under UV light in aluminum mold was observed by scanning electron microscopy (SEM) as can be seen in Figure 3. Neat epoxy (Figure 3a) showed the smooth surface without large bubble inside epoxy matrix due to low viscosity of epoxy system while epoxy composites with 100 phr of copper particle showed the particle separation from epoxy matrix. The result of morphology of epoxy-Cu composites might be owing to the precipitation of Cu particle during crosslinking by UV light from the long curing time and high density of Cu particle. Epoxy composites with single wall carbon nanotube (Epoxy-SWCNT) and multiwall carbon nanotube (Epoxy-MWCNT) which were the carbon nanoparticle at 1 phr showed different characteristics as can be seen in Figure 3(C) and (d) respectively. Epoxy-SWCNT composites displayed large bubble inside epoxy matrix while epoxy-MWCNT composites showed slightly surface roughness without bubble at the same composition. The reason why these composites exhibited the different morphology despite the same content of carbon nanofiller might be owing to the high viscous effect of SWCNT from low density and high surface area compared to MWCNT. The high viscosity of epoxy-SWCNT affected the longer time of bubble diffusion inside epoxy matrix. The morphology of epoxy composites might affect the result of dielectric constant of composites.

The dielectric constant of epoxy composite with 100 phr of Cu particle slightly increased compared to the epoxy system without particle. The Cu particle separation observed by SEM image might be the reason of this result because the dielectric constant was calculated from capacitance at cross sectional area of specimen. Thus, the electron from Cu particle did not transmitted from the lower section of specimen to the upper section of specimen due to the insulation properties of epoxy matrix. Interestingly, the dielectric constant of epoxy composite with SWCNT at 1 phr showed the significant increment value compared to neat epoxy. This might be explained that SWCNT formed a cluster structure caused by high surface area of this particle meaning that the small tunneling gap between particles resulting in the micro-capacitor behavior of this epoxy composite (Chang *et al*., 2012; Matin *et al*., 2004). Furthermore, the dielectric constant of epoxy composite with MWCNT at 1 phr did not change from neat epoxy due to the low surface area of MWCNT compared to SWCNT. The dielectric constant phenomena of this work were obtained because the electrical properties of composites were lower than the percolation threshold at this frequency of testing.

Conclusion

The epoxy and epoxy composite with copper particle, SWCNT and MWCNT cured by UV technique were studied in this work. Neat epoxy could be cured by UV light at 2-3 hours supported by differential scanning calorimetry (DSC) and UV-Visible spectrophotometry while epoxy composites showed the longer cure time by UV light than neat epoxy due to the obstruction of particle during crosslink network of epoxy molecules. Epoxy-Cu at 100 phr did not exhibit the highest dielectric constant among epoxy composites due to the phase separation between copper particle and epoxy matrix while addition of SWCNT only 1 phr showed the significant change of dielectric constant compared to neat epoxy which might be owing to the low tunneling gap between particles from cluster formation structure of SWCNT in epoxy matrix.

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