Research Article

Electrical Behaviors of Flame Retardant Huntite and Hydromagnesite Reinforced Polymer Composites

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In our previous work, we studied the physical characteristics (particle size, surface treatment, etc.) of huntite/hydromagnesite mineral in order to be employed as a flame retardant filler. With this respect, electrical properties of the mineral reinforced polymeric composites were investigated in this study. After grinding of huntite/hydromagnesite mineral to the particle size of 10 μm , 1 μm , and 0.1 μm , phase and microstructural analyses were undertaken using X-ray diffraction (XRD) and scanning electron microscopy-energy dispersive spectroscopy (SEM-EDS). The ground minerals with different particle size and content levels were subsequently added to ethylene vinyl acetate copolymer (EVA) to produce composite materials. After fabrication of huntite/hydromagnesite reinforced plastic composite samples, they were characterized by using Fourier transform infrared (FTIR) and SEM-EDS. Electrical properties were measured as a main objective of this paper with Alpha-N high resolution dielectric analyzer as a function of particle size and loading level. Dielectric constant, dissipation factor, specific resistance, and conductivity of the composite materials were measured as a function of frequency. On the other hand, conductivity of Ag-coated and uncoated polymeric composite materials was measured. It was concluded that the electrical properties of plastic composites were improved with reducing the mineral particle size.

1. Introduction

Despite significant advances in synthesis and characterization of polymers, a correct understanding of polymer molecular structure did not emerge until the 1920s. Before then, scientists believed that polymers were clusters of small molecules (called colloids), without definite molecular weights, held together by an unknown force. In 1922, Hermann Staudinger proposed that polymers consisted of long chains of atoms held together by covalent bonds, an idea which did not gain wide acceptance for over a decade and for which Staudinger was ultimately awarded the Nobel Prize [1].

A polymer can be described as macromolecule composed of repeating structural units typically connected by covalent chemical bonds [2]. A large and growing number of commercial polymers are composed of different types of

unit attached together by chemical covalent bonds. They are known as copolymers and can comprise just two different units or three and so on. It is one of the common strategies used by molecular engineers to manipulate the properties of polymers to gain just the right combination of properties for a specific application [3].

Due to their low weight and ease of processing, the use of polymers is raised by their remarkable combination of properties in our daily life. They are chosen in preference to many conventional materials. For example typical uses of composites are monocoque structures for aerospace and automobiles, as well as more mundane products like fishing rods and bicycles. The stealth bomber was the first all-composite aircraft, but many passenger aircraft like the Airbus uses an increasing proportion of composites in its fuselage. The quite different physical properties of composites give designers much greater freedom in shaping

parts, which is why composite products often look different to conventional products [1].

Even though being used in so many areas and show great facilities, polymers are also known for their relatively high flammability. Beside, most of them are accompanied by corrosive or toxic gases and smoke which are produced while the combustion is continuing [4]. So that it is rising as an important issue to extent polymers' usage for obtaining their fire resisting property for the applications [5]. Hence some ancillary materials are used to make polymers fire resistant. They are added into the compound whose application properties became closely related to the physical properties of the additive itself. Alumina trihydrate, ATH, ($\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$), and magnesium hydroxide, MH, ($\text{Mg}(\text{OH})_2$) are the major materials used as fire retardant fillers for polymers [6–8]. These two materials account for more than 50% by weight of the worldwide sales of fire retardants [9, 10]. The use of ATH is limited to those polymers processed below about 200°C while MH is stable above 300°C and thus can be used in polymers that must be processed at higher temperatures. Their effectiveness comes from the fact that they both decompose endothermically and consume a large amount of heat, while also liberating water, which can dilute any volatiles and thus decrease the possibility of fire (Equations (1) and (2), [7]). For ATH, decomposition begins near 300°C and consumes 1270 joules per gram (J/g) of ATH; for MH, decomposition begins at somewhat higher temperature, near 400°C , and consumes 1244 J/g of MH [8]. A major use of both ATH and MH is in low smoke, halogen-free wire and cable applications, where there is significant commercial activity [11],

Halogen-containing flame retardants act in the gas phase of a fire. When a polymer burns they decompose by generating free and highly reactive radicals in the gas phase, which are responsible for the propagation of a fire. Then produced incomplete burned substances lead to an increase in smoke density which is more toxic and corrosive [12]. Nonetheless, mineral nonhalogenated flame retardants release less CO and smoke when compared to halogenated flame retardants [6, 8, 13]. Metal hydroxide flame retardant materials are some of commonly used materials in this matter [8]. They firstly decompose endothermically with releasing water. It cools the condensed phase where the fuel is located, slowing its rate of decomposition and pyrolysis. Further, the water released from mineral filler flame retardants does not inhibit the amount of oxygen available for combustion. Instead it dilutes the total amount of fuel available in the pyrolysis stream for combustion [6]. As a result of this action the amount of oxygen, which is able to enter into the flame, is restricted due to the release of water and this avoids the critical fuel/oxygen ratio [14]. Moreover, after degradation, a ceramic-based protective layer was created and this improves insulation giving rise to a smoke-suppressant effect [15]. During combustion, this ceramic-based protective layer plays

an important role for the efficient protection of the polymer compound and decreasing the heat release [14]. Besides, this ceramic layer reduces smoke density by adsorbing soot particles [12]. On the other hand, intumescence involves an increase in volume of the burning substrate as a result of network or char formation. For ingressing oxygen to the fuel, this char serves as a barrier and also as a medium in which heat can be dissipated [16]. This char formation eliminates dripping and promoting. As it can be seen from Figure 1 that flame retardant cable jacket formulation at right does not drip, while unfilled sample at left drips [16].

In our previous work [17], we studied the physical characteristics (particle size, surface treatment, etc.) of huntite/hydromagnesite mineral in order to be employed as flame retardant filler in vinyl acetate copolymer [6, 7, 10]. It is a halogen-free material with formula $\text{Mg}_3\text{Ca}(\text{CO}_3)_4$ and $\text{Mg}_4(\text{CO}_3)_3(\text{OH})_2 \cdot 3\text{H}_2\text{O}$ [10]. As it decomposes endothermically and evolves water vapor and carbon dioxide, it was obtained a total associated heat of 10.53 J/g and a final weight loss of 56% in the temperature range of 220 and 600°C [17]. Regarding the flame retardancy properties, it was obtained that increasing the loading level of additive [15] and decreasing the size increase the flame retardancy of the polymer composite [18]. This was explained with the fact of increasing surface area. Consequently, it was concluded that HM is a promising flame retardant filler for vinyl acetate copolymers [8].

Although unified by direct concern with the effects produced by electric fields, the subject of the electrical properties polymers covers a diverse range of molecular phenomena [19]. By comparison with metals, where the electrical response is overwhelmingly one of electronic conduction, polymers display a much less striking response. This absence of any overriding conduction does allow, however, a whole set of more subtle electrical effects to be observed more easily. For instance, polarization resulting from distortion and alignment of molecules under the influence of the applied field becomes apparent. Examination of such polarization not only gives valuable insight into the nature of the electrical response itself but it also provides a powerful means of probing molecular dynamics. For this reason electrical studies form a desirable supplement to studies of purely mechanical properties aimed at reaching an understanding of the behavior of polymers on a molecular basis [20].

2. Electrical Mechanism of Polymers

Understanding the structure of polymers not only gives a better behavior of chemical resistance but also of the electrical properties. Most polymers are dielectrics or insulators and resist the flow of a current. This is one of the most useful properties of plastics and makes much of our modern society possible through the use of plastics as wire coatings, switches, and other electrical and electronic products. Despite this, dielectric breakdown can occur at sufficiently high voltages to give current transmission and possible mechanical damage to the plastic [21].

FIGURE 1: Char formation prevents dripping and promoting [16].

FIGURE 2: Structure of a water molecule; (a) conventional and (b) actual [21].

The application of a potential difference causes the movement of electrons and when the electrons are free to move there is a flow of current. Metals can be thought of as a collection of atomic nuclei existing in a “sea of electrons” and when a voltage is applied the electrons are free to move and to conduct a current. Polymers and the atoms that make them up have their electrons tightly bound to the central long chain and side groups through “covalent” bonding. Covalent bonding makes it much more difficult for most conventional polymers to support the movement of electrons and therefore they act as insulators [21].

3. Polar and Nonpolar Polymers

Not all polymers behave the same when subjected to voltage and plastics can be classified as “polar” or “nonpolar” to describe their variations in behavior. The polar plastics do not have a fully covalent bond and there is a slight imbalance in the electronic charge of the molecule. A simple example of this type of behavior would be that of the water molecule (H_2O) (see Figure 2). The conventional representation of the molecule is that shown at that right. The two hydrogen atoms are attached to the oxygen atom and the overall molecule has no charge [21].

In reality, the electrons tend to be around the oxygen atom more than around the hydrogen atoms and this gives the oxygen a slightly negative charge and the hydrogen atoms a slightly positive charge. This is shown in the diagram at right where the grey areas show where the electrons are more

often found. The overall water molecule is neutral and does not carry a charge but the imbalance of the electrons creates a “polar” molecule. This “polar dipole” will move in the presence of an electric field and attempt to line up with the electric field in much the same way as a compass needle attempts to line up with the earth’s magnetic field.

In polar plastics, dipoles are created by an imbalance in the distribution of electrons and in the presence of an electric field the dipoles will attempt to move to align with the field. This will create “dipole polarization” of the material and because movement of the dipoles is involved there is a time element to the movement. The non-polar plastics are truly covalent and generally have symmetrical molecules. In these materials there are no polar dipoles present and the application of an electric field does not try to align any dipoles. The electric field does, however, move the electrons slightly in the direction of the electric field to create “electron polarization”; in this case the only movement is that of electrons and this is effectively instantaneous. The structure of the polymer determines if it is polar or non-polar and this determines many of the dielectric properties of the plastic [21].

4. Electrical Applications of Polymers

The electrical insulating quality inherent in most polymers has long been exploited to constrain and protect currents flowing along chosen paths in conductors and to sustain high electric fields without breaking down. Insulating polymeric materials for early electrical equipment were made from naturally occurring products. For instance, the first trans-Atlantic telephone cables laid in the 1860s were insulated with *Gutta-percha*, which is one of the polymers extracted from rubber trees. As synthetic high polymers became available in the twentieth century, the range of insulators was continually improved. The great virtue of these new materials, such as polystyrene, was their combination of high quality of insulation with ease of fabrication by molding. Polyethylene, which combines superb insulating properties with moldability and a high degree of toughness and flexibility, arrived on the scene just in time for the more demanding applications of insulation in coaxial cables for radar apparatus and television. More recently extreme requirements for very low-conductivity materials, used in electret microphones, for example, have been met by fluorinated polymers. High-performance thin films have also been developed for various types of capacitors [20].

The choice of material for a particular application naturally depends on being able to reach a good compromise amongst a whole range of considerations, including mechanical properties, ease of fabrication into a final product, and cost. The basic insulating properties of polymers are more than adequate for many purposes, and any development effort may then be primarily concentrated on improving other aspects of the material’s performance. High on the list will be a need for good chemical and physical stability in the working environment, which might involve exposure to strong sunlight, organic solvents, and high temperatures.

Only on the basis of detailed knowledge and understanding of the molecular structure and behavior of the basic polymers one can hope to approach the optimum in performance. This has motivated the investigation in depth of the electrical behavior of many polymeric systems [20].

Polymers are always good insulators, but that is not to say that a conducting plastic is not desirable. A lightweight, readily moldable, highly conductive material has long been recognized as a worthwhile goal to strive for, and considerable scientific research has been devoted to this. Even though encouraging results have been obtained, there is still a long way to go in developing useful products. Apart from the obvious market for a highly conductive material which could be suitable for power and signal transmission, there is also one for materials having intermediate conductive properties, for example, for flexible heating elements and graded cable insulations. Certain of these can be supplied by modification of existing polymers [20].

As electrical properties of polymers, elastomers, composites, and films are important in a wide range of industries including automotive, aerospace, building products, marine, packaging and consumer goods, and electrical tests, in general, are performed as the measurements of the resistance, conductivity, or charge storage either on the surface or through the plastic composite material. In the light of this a series of composites were prepared using an ethylene vinyl acetate copolymer matrix in the present work. Huntite/hydromagnesite mineral powders were added to ethylene vinyl acetate copolymer at different concentrations to evaluate the electrical properties of the composites. In this sense, properties of complex conductivity, dielectric constant, specific resistance, and dissipation factor measurements were performed to the plastic composite samples.

5. Experimental Details

5.1. Preprocessing and Fabrication of Composite Samples. Preprocessing and fabrication of the composites were described in [17] in detail. Briefly, preprocessing includes excavating, crushing, grinding, and separating mineral powders according to their size. Open pit mining technique was used at the quarry to excavate the mineral due to the proximity of deposit to the surface. Then it was comminuted in an impact crusher to approximately 1 cm particle size. In the grinding area, huntite and hydromagnesite mineral was subjected to a high degree of turbulent mixing in cells created between high velocity rotor blades and the high energy process reduces agglomeration of the mineral particles to a minimum.

Huntite/hydromagnesite mineral powders, supplied by Likya Minelco Madencilik (Denizli, Turkey), were subjected to a sedimentation process. At the end of this procedure, three different size products were obtained: 10, 1, and 0.1 μm . As a polymer matrix ethylene-co-vinyl acetate (poly) was used. Polymer composites were a blend of ethylene-co-vinyl acetate and the mineral powders in different ratio from 49% to 69% and size from 0.1 μm to 10 μm . They were processed using a twin screw extruder and subsequently pelletized and compression molded at 160°C to obtain 1 mm thick sheets.

FIGURE 3: XRD pattern of as received huntite and hydromagnesite mineral powder.

5.2. Characterization. Rigaku D (Max-2200/PC Model XRD) X-ray diffractometer equipment was used to identify the huntite and hydromagnesite phase at 40 kV, 20 mA with a monochromatic CuK_α irradiation (wavelength, $\lambda = 0.15418 \text{ nm}$) by both θ - 2θ mode and 2θ scan mode with a scan speed of 8°/min. Fourier transform infrared (FTIR, Perkin Elmer Spectrum BX) absorption spectra of the composite materials were only measured over the range of 4000 to 400 cm^{-1} at room temperature in order to determine organic structure and interaction between polymeric matrix and reinforced material. JEOL JIM 6060 scanning electron microscopy (SEM) was used to examine the microstructural cross-sectional areas of huntite and hydromagnesite reinforced polymeric matrix composite materials.

5.3. Electrical Measurements. Electrical Properties were measured with Alpha-N high resolution dielectric analyzer in detail. By this method, electrical properties of huntite/hydromagnesite reinforced plastic composite samples were measured. Insight into these complex electrical properties of composite materials can be gained from analysis including dielectric constant, conductivity, specific resistance, impedance, capacitance, and dissipation factor.

The dielectric constant is a measure of the influence of a particular dielectric on the capacitance of a condenser. It measures how well a material separates the plates in a capacitor and is defined as the ratio of the capacitance of a set of electrodes with the dielectric material between them to the capacitance of the same electrodes with a vacuum between them. The dielectric constant for a vacuum is 1 and for all other materials it is greater than 1 [21].

Electrical conductivity or specific conductance is a measure of a material's ability to conduct an electric current. To analyse the conductivity of materials exposed to alternating electric fields, it is necessary to treat conductivity as a

FIGURE 4: FTIR analysis of huntite and hydromagnesite reinforced plastic composite material including (a) 10 μm , (b) 1 μm , and (c) 0.1 μm particle size.

complex number called the admittivity, complex conductivity. This method is used in applications such as electrical impedance tomography, a type of industrial and medical imaging. Admittivity is the sum of a real component called the conductivity and an imaginary component called the susceptibility [22, 23].

Specific electrical resistance is a measure of how strongly a material opposes the flow of electric current. A low resistivity indicates a material that readily allows the movement of electrical charge [22, 23]. Most plastics have very high volume resistivity (in the order of 1016 Ωm) and are therefore good insulators [24].

As for dissipation factor (DF), it is a measure of loss rate of power of a mode of oscillation (mechanical, electrical, or electromechanical) in a dissipative system. It is the reciprocal of quality factor, which represents the quality of oscillation. To illustrate this, electric power is dissipated in all dielectric materials, usually in the form of heat [25].

6. Results and Discussion

6.1. Material Characterization. The XRD pattern of mineral powder (Figure 3) demonstrates that the basic minerals are hydromagnesite ($\text{Mg}_4(\text{CO}_3)_3(\text{OH})_2 \cdot 3\text{H}_2\text{O}$), huntite ($\text{Mg}_3\text{Ca}(\text{CO}_3)_4$), and dolomite ($\text{CaMg}(\text{CO}_3)_2$). As mentioned in the literature [26], morphologically those rocks are

FIGURE 5: SEM micrographs of huntite and hydromagnesite reinforced plastic composite material having (a) 10 μm , (b) 1 μm , and (c) 0.1 μm particle size.

composed of variable amounts of siliciclastic and carbonate-clastic debris cemented by dolomite, monohydrocalcite, hydromagnesite, huntite and magnesite. In addition to this, the literature [26, 27] focused on that those minerals are sediments of the lake predominantly composed of gypsum, dolomite, huntite, hydromagnesite, and magnesite.

Figure 4 elucidates FTIR analysis of huntite/hydromagnesite containing plastic composite materials including different mineral particle sizes. The FTIR spectrum of calcium magnesium carbonate is quite characteristic with a very intense broad band centering at 2750–4000 cm^{-1} there is another sharp band at 3750 cm^{-1} . This could be due to O-H groups in the samples. Additionally, lower intensity absorptions at 600–700 cm^{-1} can be observed from magnesium carbonate. In the samples with different size distributions, there are small differences in the results. As FTIR analysis is related with the molecular bonding, getting the material size to nanoscale, the bond numbers and the intensities decreased.

SEM micrographs of huntite and hydromagnesite reinforced plastic composite material are demonstrated in Figure 5. Morphological features that contribute to electrical properties are particle size and content of huntite and

hydromagnesite powders in the plastic matrix. Notice that changing of huntite and hydromagnesite particle size can be clearly seen in the composite material from $10\text{ }\mu\text{m}$ to $0.1\text{ }\mu\text{m}$ (100 nm). It can be pointed out from the figures that by getting nanoscale particle sizes, much denser structures can be produced in the composite material. Besides, it is indicated in the literature [28] that in order to achieve suitable plastic formulations it is necessary to reduce strongly the mineral particle size. This may affect not only the morphology but the crystalline characteristics of the material. Therefore, electrical properties, flame retardancy, and mechanical properties are strongly influenced by virtue of Nano sized huntite and hydromagnesite reinforced materials.

6.2. Electrical Properties. The most striking property is dielectric behavior of a composite material. The addition of huntite and hydromagnesite powder to tailor dielectric properties is extensively exploited in plastic matrix composite material. From this point of view dielectric constant test result is depicted in Figure 6. It is clear from the figure that the biggest dielectric constant has been achieved with medium size ($1\text{ }\mu\text{m}$). For the loading level test, it has been gotten from the sample with 67% flame retardant powder.

Actually, the filler particles present in the polymer matrix may be considered as micro/nanocapacitors. The increase in filler loading in the polymer matrix increases the number of such capacitors, which in turn leads to the increase in dielectric constant [29]. At the higher frequency values, just after 10 Hz , dielectric constant became stable at the 40.000 level. On the other hand, for all samples dielectric contents decreased by increasing frequency as similar in [29–32]. At lower frequencies dielectric constants attain high values and then decrease exponentially with increase in frequency. This behavior clearly indicates that the effect of interfacial polarization becomes more and more predominant at lower frequency [29].

Tan (Δ) test result is given in Figure 7. All composites showed a decrease in dissipation factors with increase in frequency. This may be attributed to the dipole relaxation phenomena, where movement of the electric dipoles was not possible to be in phase with the frequency of the applied electric field [30]. The decrease in loss factor with increase in frequency can also be explained by the fact that as the frequency is raised, the dipoles get very less time to orient themselves in the direction of the alternating field [29]. The orientation of the dipoles takes place by the compensation of some electrical energy, which accounts for dielectric loss. At lower frequencies, the effect of orientation polarization of dipoles is higher for getting sufficient time to orient them, thus accounting for higher dielectric loss at lower frequencies [32]. The sample consisting of medium size of mineral showed the biggest value of dissipation factor. For the loading level, the highest dissipation factor was obtained with 49% and 67% loading levels in between $0\text{--}5\text{ Hz}$. Dissipation factor exhibited high values and formed in the low-frequency range. When the frequency was increased; it decreased quickly. As the filler content is increased, heterogeneity of the

FIGURE 6: Dielectric constant values of huntite/hydromagnesite reinforced polymeric matrix composite materials as a function of frequency according to (a) particle sizes and (b) contents of reinforced powder.

system is increased, this produces extended interfaces, and results in increased conductivity and higher losses [31].

Figure 8 denotes specific resistance of huntite/hydromagnesite reinforced polymer composites as a function of frequency. The biggest resistivity was obtained with the loading level of 49%. In general, the conductivity of filled polymer composites is governed by both the mechanism of conduction theory (formation of continuous conductive networks) and hopping mechanism (electric field radiation) of conduction theory [24, 25, 27]. Before percolation the conductivity in polymer composite is due to the hopping (jumping) of electrons from one conducting site to another, which is facilitated as the distance between the conducting sites is reduced [32]. On the other hand, as

(a)

(b)

FIGURE 7: Dissipation factor of huntite/hydromagnesite reinforced polymeric composite materials as a function of frequency according to (a) particle sizes and (b) contents of reinforced powder.

explained in [33], the electrical properties of composites were directly related to the morphology of conductive networks, that is, the localization of conductive fillers in the composites.

Conductivity test result of the flame retardant composite materials is shown in Figure 9. Generally speaking, it should be noted that the complex conductivity increased as a function of frequency as the same in [29, 30]. If the particle size dependence of electrical conductivity of huntite and hydromagnesite reinforced plastic composite materials is to be accounted for, it is necessary for analyses how conductivity depends upon the particle size of reinforced material from the micron scale to nanoscale. The smallest size, which is the highest surface area, is expected to be

(a)

(b)

FIGURE 8: Specific resistances of huntite/hydromagnesite reinforced polymeric composite materials as a function of frequency according to (a) particle sizes and (b) contents of reinforced powder.

much better particle-particle the highest conductivity at the percolation threshold [29]. As expected, the resulting characteristic depicted that decreasing the size to nanoscale makes the polymer composite more conductive. One of the notable features of the composite material is the amount of reinforced material. In spite of the fact that it seems to be changing the conductivity related with the loading level, it can be expressed that increasing filler amount increased the polymer's conductivity. The increase in conductivity with the increasing of the filler amount mainly stems from the establishing of conducting networks in the polymer matrix [29, 30, 33]. Besides, we have a good agreement with the literature [34] that finer particles may support this mechanism as the ionic conductivity of the polymer composite increased. In other words, for both size and the

FIGURE 9: Conductivity of huntite/hydromagnesite reinforced plastic composite materials as a function of frequency according to (a) particle sizes and (b) contents of reinforced powder.

loading level effect tests, it can be seen that frequency assists to increase conductivity of the composites.

On the other hand, when very low frequency was applied to the composites, the conductivity results were different as seen from Figure 9. In between 0–5 Hz frequency, medium particle content composites have the biggest conductivity. Actually these results are quite similar to the dielectric constant, specific resistance, and tan delta results.

Besides different sizes and different loading level measurements, conductivity tests were carried out for Ag-coated polymer to see if there are any pores or displacements in the polymer composite structure. The obtained results are shown in Figure 10. According to these figures, it can be said

FIGURE 10: Conductivity of Ag-coated and uncoated polymeric composite materials.

that polymer composites had certain amount of pores, as conductivity increased by Ag coating.

7. Conclusion

As huntite and hydromagnesite mineral showed an effective flame retardant behavior with an increase of the time to ignition in the previous study, it was also indicated that decreasing the size increases the flame retardancy property. In this context it can be said that potential escape time will increase by using this kind of polymeric material in a dangerous fire. It is in continuation of the study in this work that the electrical properties were investigated and following results were found; except for the conductivity, dielectric constant, specific resistance, and dissipation factors were decreased by increasing frequency. But conductivity increased with frequency. In addition conductivity increased with decreasing particle amount. With increasing mineral content, dielectric constant and specific resistance values increased. It was shown in the particle size tests that, apart from conductivity, coarser and finer sizes ($10 \mu\text{m}$ and $0.1 \mu\text{m}$) behave similarly unlike medium size ($1 \mu\text{m}$). This may be explained with non-homogenous blending or agglomeration in the $0.1 \mu\text{m}$ size material. On the other hand, it was seen from the Ag-coated samples tests that composites had some pores that Ag-coated samples showed different electrical property against uncoated ones.

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