

## AC Conductivity and Dielectric Constant of Ni-MgO Composites

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### Ni-MgO 복합재료의 전기전도도와 유전상수

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#### ABSTRACT

The Ni-MgO composites were prepared by coprecipitation of NiO-MgO solid solutions and their selective reduction in a hydrogen atmosphere. We report on the measurements of both ac conductivity  $\sigma(\omega, f)$  and dielectric constant  $\kappa'(\omega, f)$  for the Ni-MgO composites in the frequency range from 10 Hz to 10 MHz at room temperature. The frequency exponents of conductivity and dielectric constant,  $x$  and  $y$ , are found to be  $x=0.98\pm 0.05$  and  $y=0.05\pm 0.01$ . These results are in good agreement with a general scaling relation  $x+y=1$ , although these values are different from the theoretical predictions. The dielectric constant exponent ( $\kappa' \propto |f-f_c|^{-s}$ ) is found to be  $s=0.62\pm 0.07$  with estimated percolation threshold  $f_c=0.20\pm 0.02$ .

#### 요 약

공침법으로 만든 NiO-MgO 고용체를 수소 분위기에서 선택적으로 환원시켜 Ni-MgO 복합재료를 제조하여 상온에서의 Ni-MgO 복합재료에 대한 전기전도도와 유전상수를 10 Hz에서 10 MHz의 주파수범위에서 측정하였다.

전기전도도와 유전상수는 주파수에 대하여 지수함수적으로 비례하는데 그 값은 각각  $x=0.98\pm 0.05$ ,  $y=0.05\pm 0.01$ 로서, 이론치와는 다른 값을 보여주고 있으나  $x+y=1$ 이라는 계수 관계식에는 잘 일치한다. Ni-MgO 복합재료의 percolation threshold는  $0.20\pm 0.02$ 로 추정하였으며, 유전상수의 지수는  $s=0.62\pm 0.07$ 이다.

#### 1. Introduction

Since Broadbent and Hammersley<sup>1)</sup> introduced the term "percolation processes" to describe the flow of a fluid through a random porous medium (in contrast to diffusion processes in which the randomness is associated with the particles of the fluid), percolation theory has been growing in importance in physics, chemistry, and material science. Percolation theory is widely used to describe a diversity of phenomena, espe-

cially in the field of inhomogeneous materials. The field of inhomogeneous materials has been an area of extensive research in recent years<sup>2-7)</sup> partly as a result of the intrinsic interest in the subject, and also as the result of possible technological applications for these materials. An area of basic interest is the insulator-conductor transition (percolation transition), which occurs in these materials and which resembles a second-order phase transition. A possible important application to technology is materials for solar energy collectors.

The formal resemblance between a percolation transition and a second-order phase transition was recognized by Fortuin and Kasteleyn<sup>9)</sup>. Many physicists<sup>9-11)</sup> have extended this similarity to incorporate the idea of scaling. According to the scaling hypothesis, certain properties of a percolating system should exhibit a power-law dependence on  $|f-f_c|$ , where  $f$  is the volume fraction of metal in the material and  $f_c$  is the critical volume fraction for percolation to occur. In most cases, each power-law is thought to be universal in the sense that its exponent depends primarily on the dimensionality of the system.

According to the scaling hypothesis, the dc electrical conductivity,  $\sigma(\omega=0, f>f_c)$ , and the dc dielectric constant,  $\kappa'(\omega=0, f<f_c)$ , of the inhomogeneous material, should exhibit a power-law dependence on  $|f-f_c|$ . In other words

$$\sigma(\omega=0, f>f_c) \propto |f-f_c|^t \text{ above } f_c, \quad (1)$$

$$\kappa'(\omega=0, f<f_c) \propto |f-f_c|^{-s} \text{ below } f_c. \quad (2)$$

The critical exponents ( $t$  and  $s$ ) have been determined experimentally by many workers<sup>12,13-16)</sup>. By measurement of the electrical conductivity of two-dimensional screens as sites were removed, Watson and Leath<sup>13)</sup> reported that the critical exponent  $t$  was  $1.38 \pm 0.12$ , about a percolation threshold  $f_c = 0.413 \pm 0.005$ . In W-Al<sub>2</sub>O<sub>3</sub> granular metal films made by cosputtering the metal W with the insulator Al<sub>2</sub>O<sub>3</sub>, Abeles *et al.*<sup>12)</sup> could show the critical exponent  $t = 1.9 \pm 0.2$  and the percolation threshold  $f_c = 0.47 \pm 0.05$ . Smith and Lobb<sup>14)</sup> measured the conductivities of two-dimensional conductor-insulator networks generated photolithographically from laser speckle patterns. They found the critical exponent  $t = 1.30$  and a percolation threshold  $f_c = 0.41$ . By numerically solving the voltage distributions of large finite random resistor lattices, Straley<sup>15)</sup> estimated the exponents for two- and three-dimensions. For a two-dimensional system, these exponents are known as  $t = 1.2 \pm 0.1$  and  $s = 1.10 \pm 0.15$ . For a three-dimensional system, the exponents were  $t = 1.75 \pm 0.05$  and  $s = 0.6 \pm 0.1$ . In conductor-insulator composites consisting of carbon and teflon powder, Song *et al.*<sup>16)</sup> found the exponents  $t = 1.85 \pm 0.10$  and  $s = 0.68 \pm 0.05$ . According to these workers, the exponents for a three-dimensional system are  $t = 1.8 \pm 0.1$  and  $s = 0.7 \pm 0.1$ .

Other physical properties of interest are the ac elec-

trical conductivity,  $\sigma(\omega, f)$ , and the ac dielectric constant,  $\kappa'(\omega, f)$ . It is well known that both the conductivity and the dielectric constant exhibit a power-law behavior near  $f_c$  such that

$$\sigma(\omega, f_c) \propto \omega^x \text{ near } f_c, \quad (3)$$

$$\kappa'(\omega, f_c) \propto \omega^{-y} \text{ near } f_c. \quad (4)$$

Bergman and Imry<sup>17)</sup> also proved that the critical exponents,  $x$  and  $y$ , should satisfy the general scaling relation

$$x + y = 1. \quad (5)$$

In heterogeneous materials, the frequency dependence of the conductivity and the dielectric constant involve important contributions from (a) interclusters polarization effects between the metallic grains and (b) anomalous diffusion processes of electron within each metallic cluster. There have been extensive theoretical studies<sup>18-20)</sup> to determine the values of  $x$  and  $y$  from each contribution; however, a unified theory that includes both effects is not available.

Following these theoretical approaches, two groups have measured the critical exponents,  $x$  and  $y$ . Laibowitz and Gefen<sup>21)</sup> measured the ac conductivity and dielectric constant for thin gold films near  $f_c$  for the frequency range,  $100 \text{ Hz} < \omega < 20 \text{ MHz}$ . For these two-dimensional samples, it was found that  $x = 0.95 \pm 0.05$  and  $y = 0.13 \pm 0.05$ . These values are in good agreement with the general scaling relation, i.e., Equation (5), but significantly different from present theoretical predictions [in two dimensions,  $x = y \approx 0.5$  from the intercluster polarization effect, or  $x \approx 0.33$  and  $y \approx 0.67$  from the anomalous diffusion process]. Recently, Song *et al.*<sup>16)</sup> investigated three-dimensional composites of amorphous carbon and teflon in the frequency range from 10 Hz to 13 MHz. The frequency exponents for these materials were  $x = 0.86 \pm 0.06$  and  $y = 0.12 \pm 0.04$ . These values are closer to the predictions of the intercluster polarization model [in three dimensions,  $x \approx 0.72$ ,  $y \approx 0.28$ ] than those of the anomalous diffusion model [in three dimensions,  $x \approx 0.58$  and  $y \approx 0.42$ ].

In this paper, we report on a measurement of the ac conductivity and the dielectric constant for Ni-MgO composites for the frequency from 10 Hz to 10 MHz. The Ni-MgO granular metal composites were prepared by coprecipitation of NiO-MgO solid solutions and their

preferential reduction in a hydrogen atmosphere. These composites have a different topology from that of the conventional granular composites prepared by vacuum deposition techniques, and they showed  $f_c = 0.20 \pm 0.02$ , which is much lower than that of the conventional granular system ( $f_c = 0.5$ ). General behaviors of ac conductivity and dielectric constant for Ni-MgO composites follow qualitatively the predictions of the present percolation theories. Especially, near the percolation threshold, the ac conductivity and the dielectric constant vary as a power of frequency, such as  $\sigma \propto \omega^x$  and  $\kappa' \propto \omega^{-y}$ . The values of  $x$  and  $y$  are estimated as  $x = 0.98 \pm 0.05$  and  $y = 0.05 \pm 0.01$ , in good agreement with the scaling relation in Equation 5. We also determined the dc dielectric constant exponent,  $s$ , as  $s = 0.62 \pm 0.07$ .

## 2. Experimental Procedures

A series of Ni-MgO composites was prepared by preferential reduction of NiO-MgO solid solutions in a hydrogen atmosphere. To prepare the NiO-MgO solid solutions, preweighed magnesium nitrate hexahydrate and nickel nitrate hexahydrate were dissolved in doubly distilled water. By dropwise addition of these mixing nitrate solutions into potassium carbonate solutions, coprecipitates of magnesium carbonate and nickel carbonate were obtained. The coprecipitate was then dried and ground with acetone in a mortar. To remove volatiles and to initiate solid state reaction, the coprecipitate was calcined. Phase identification of this calcined powder by an X-ray diffractometer verified that all phases belonged to NiO-MgO solid solutions.

Wafer-shaped pellets of the NiO-MgO solid solutions were formed by compressing at 20,000 psi in a 1-cm diameter die. These pellets were sintered at 1600°C for one hour to make dense NiO-MgO solid solutions. To prepare the Ni-MgO composites, the pellets were fired in a hydrogen atmosphere at 1200°C for 2 hours. The reduction in hydrogen occurs preferentially in the nickel oxide to make nickel metallic particles. Pure MgO pellets were also fired under the same conditions to ensure that no reduction of MgO occurred. The extent of NiO was determined by measuring the weight loss of each sample. The details of these processes and related measurements will be published elsewhere

<sup>22)</sup> The ac conductance and capacitance of the Ni-MgO composites were measured using a Hewlett-Packard 4192A LF Impedance Analyzer\*. This impedance analyzer is designed to measure a wide range of impedance parameters as well as phase, gain, and group delay. The frequency range of the instrument is 5 Hz to 13 MHz. The ac conductance and capacitance, which are proportional to the conductivity and dielectric constant, respectively, were measured simultaneously in the frequency range from 10 Hz to 10 MHz. Data acquisition was controlled by an IBM personal computer through the IEEE-488 interfacing system. Since the peak-to-peak voltage applied to the samples was small (0.1 V), no destructive effects were observed.

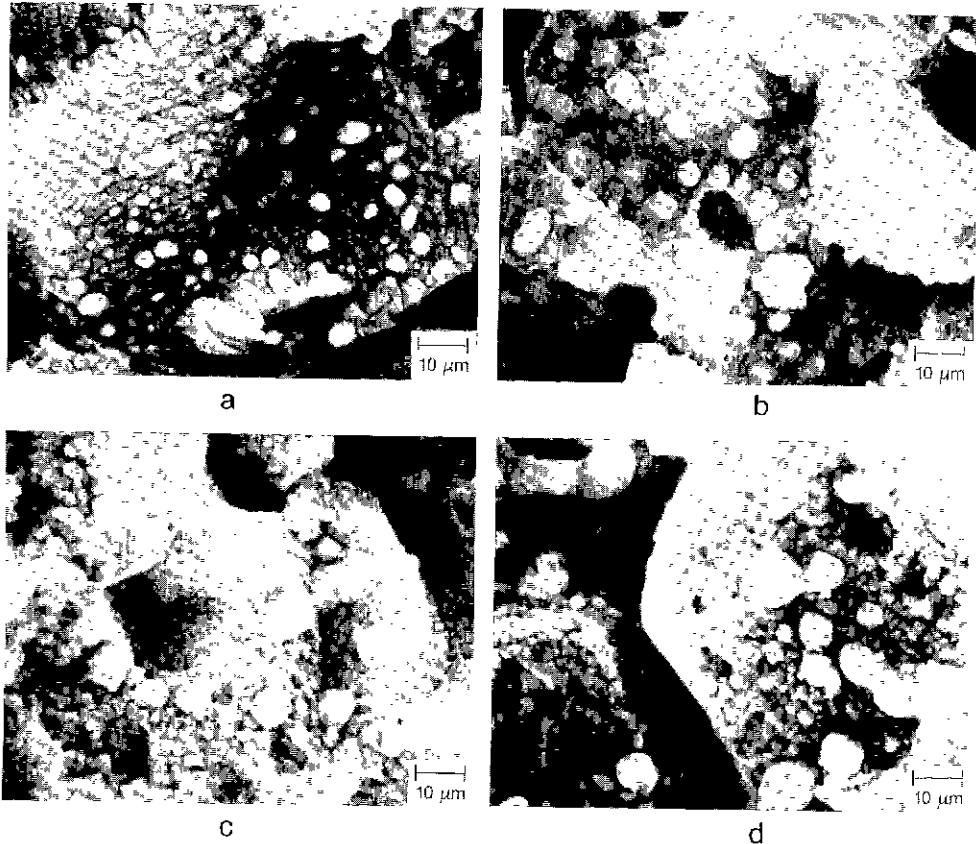
In order to obtain accurate measurements of the samples, both surfaces had to be smooth and flat. The pellet was ground flat with silicon carbide papers and polished using 6  $\mu\text{m}$  and 1  $\mu\text{m}$  diamond pastes. In order to obtain the conductivity and dielectric constant of the Ni-MgO composites from the measured data (conductance and capacitance), the thickness and the area of each sample are needed. These measurements were made with a micrometer accurate to 0.0025 mm (0.0001 in.). Good electrical contact for the conductance and capacitance measurements was established by coating the polished samples on both sides with a 1000Å thick gold layer. The coatings were made by evaporation of gold in a bell-jar system operating at  $10^{-6}$  mmHg.

## 3. Results and Discussion

The microstructures of the Ni-MgO composites were examined with scanning electron microscope (SEM) and with the transmission electron microscope (TEM). Because of the relatively large size of the Ni particles in the Ni-MgO composites, SEM pictures were usually good enough to provide adequate microstructural information. Typical microstructures of the Ni-MgO composites are shown in Fig. 1. The numbers in parentheses in Fig. 1 give the extent of NiO reduction. The size of Ni particles increased slowly with the volume fraction of Ni. From these SEM micrographs, one surprising result

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**Fig. 1.** Scanning electron micrographs of fractured surfaces of the Ni-MgO composites at 1200°C for 2 hours. (a) 10 v/o (93.4%), (b) 20 v/o (101.8%), (c) 30 v/o (103.7%), and (d) 40 v/o (104.0%).

is that there is little apparent difference between the fracture surface of the 40 v/o Ni sample and that of the 10 v/o Ni sample. To understand this result, the SEM photographs of polished surfaces, shown in Fig. 2, were taken. The SEM photographs of the polished surfaces clearly show that in the 10 v/o sample, the Ni particles are preferentially located on the grain boundaries rather than in the interiors of the MgO grains. However, as the volume fraction of Ni increases, the number of Ni particles on the grain boundaries does not increase much, but the number of particles inside the grains increases very sharply. This phenomenon occurs because the original grains are laced with a fine pore network caused by the volume change associated with reduction. The details of the microstructure of the Ni-MgO composites will be published.

As mentioned in Section II, ac electrical conduc-

tivity,  $\sigma(\omega, f)$ , and ac dielectric constant,  $\kappa'(\omega, f)$ , of the conductor-insulator materials should exhibit power-law dependencies on the frequency,  $\omega$ , near the percolation threshold such that  $\sigma(\omega, f_c) \approx \omega^x$  and  $\kappa'(\omega, f_c) \approx \omega^{-y}$ . The critical exponents,  $x$  and  $y$ , satisfy the general scaling relation  $x+y=1$ . The dc electrical conductivity,  $\sigma(\omega=0, f > f_c)$ , and dc dielectric constant,  $\kappa'(\omega=0, f < f_c)$ , of a percolating system also exhibit power-law behaviors. On the conducting side of the transition ( $f > f_c$ ),  $\sigma$  is proportional to  $|f - f_c|^t$ . On the insulating side ( $f < f_c$ ),  $\kappa'$  is proportional to  $|f - f_c|^{-s}$ . However, there is no general scaling relation for the critical exponents  $t$  and  $s$ .

The room temperature ac conductivity of the completely reduced Ni-MgO composites is plotted on a logarithmic scale as a function of the applied frequency in Fig. 3. The Ni metal volume fractions,  $f$ , in this graph ranged from 0.07 to 0.26, and inclu-

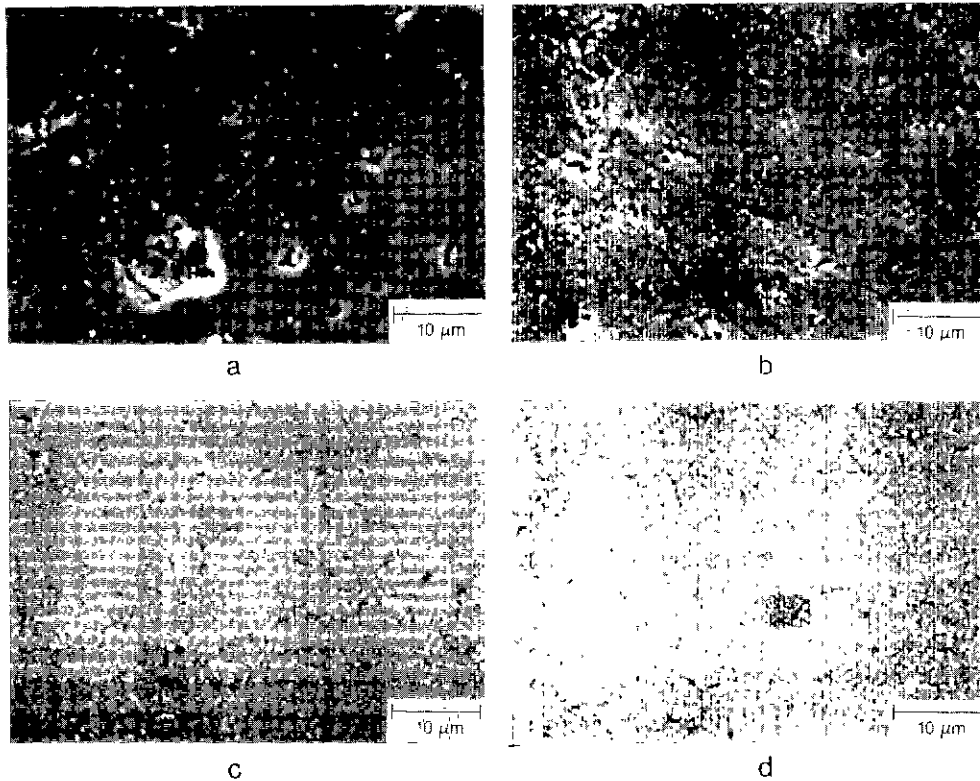


Fig. 2. Scanning electron micrographs of polished surfaces of the Ni-MgO composites at 1200°C for 2 hours. (a) 10 v/o (93.4%), (b) 20 v/o (101.8%), (c) 30 v/o (103.7%), and (d) 40 v/o (104.0%).

ded the percolation threshold  $f_c$ . The metal volume fraction has been calculated by two methods for these samples and both values are given in Figs. 3 and 5. The first value assumes that there is no porosity in the composites. The second value (in parentheses) is based upon the measured external sample dimensions, the weight of the sample, and the densities of the pure phase. This value represents the true volume percent Ni in the composites and takes porosity into account. Strictly speaking, the true volume fraction should be used in considerations of percolation. However, if the material is permeated by pores in such a way that particles on the opposite sides of pores have no chance of making contact and contributing to conductivity, the pore volume can be omitted from consideration. It is not clear from the micrographs that the assumption is valid. Consequently, both values of  $f$  are given, and since the determination of critical exponents does not require the assumption, the complex

impedance measurements are analyzed using the true volume fraction of Ni. For samples with volume fractions of Ni below the percolation threshold (i.e.,  $f < f_c$ ), the conductivity was too small to be measured at the lower frequencies. As shown in Fig. 3, for these samples, the conductivity increases linearly with frequency. On the other hand, when  $f > f_c$ , the conductivity curves are no longer linear on a log-log plot; however, at high frequencies the curves become parallel to those for samples having  $f < f_c$ . By measuring the slopes at high frequency, the critical exponent for conductivity was found to be  $x = 0.98 \pm 0.05$ .

The conductivity of the Ni-MgO composites has very low value even above percolation threshold and increases much more slowly than would be expected for a percolating system. The TEM micrograph, as shown in Fig. 4, indicates that poor contacts exist between Ni particles; these poor contacts may explain the low conductivity and its slow increase

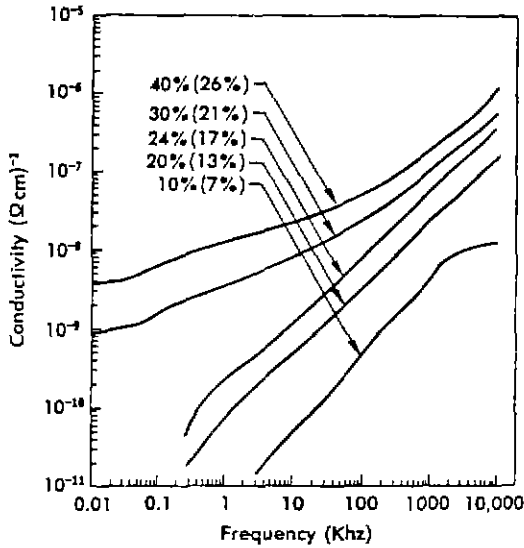


Fig. 3. AC conductivity of the Ni-MgO composites as a function of frequency. Volume percentage of Ni are given assuming zero porosity and taking all porosity into account. The latter values are in parentheses and represent true volume percentage.

as  $f$  increases. By using the results of the ac conductivity and dielectric constant shown in Fig. 3 and 5 respectively, we estimated the percolation threshold of  $f=0.20\pm 0.02$ . Since we didn't have data of conductivity and dielectric constant between  $f=0.17$  and  $0.21$ , we couldn't obtain accurate value of  $f$ . However, according to other workers<sup>15,16</sup>, the value estimated for the percolation threshold  $f=0.20$  is reasonable.

The frequency dependence of the conductivity can be explained in terms of intercluster polarization effects<sup>17-20,23</sup> and the anomalous diffusion of electron within the clusters<sup>24</sup>. The intercluster polarization effects can be understood by analogy to an equivalent circuit that has capacitance between clusters of metal particles. If  $f$  is much larger than  $f_c$ , the conductivity is mainly determined by the many connected paths through the percolating clusters, and the effect of capacitance between clusters is minor at low frequencies. Therefore, the conductance of the sample will not change significantly up to a certain frequency at which the contribution of the capacitors between the clusters becomes im-

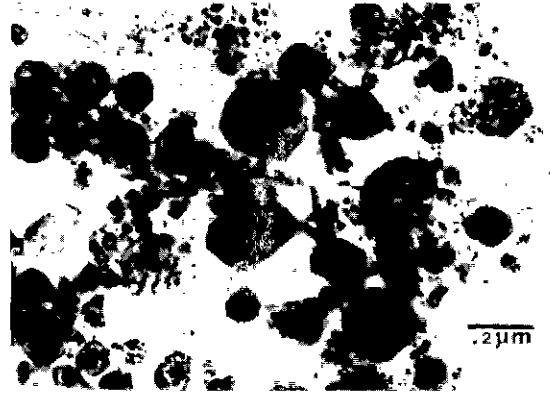


Fig. 4. Transmission electron micrograph of 30 v/o Ni in a MgO sample.

portant. As frequency increases, the displacement current passing through the capacitors increases, resulting in increased conductivity of the sample. Anomalous diffusion of electrons within the clusters also contributes to the frequency dependence of the conductivity. The conductivity of most conductors has a flat response over a wide range of frequency, since electrons can move freely over an arbitrarily large distance in an applied field. However, near the percolation threshold, only a few conducting paths through the percolating clusters are available, so that the motion of electrons in the finite clusters becomes important. Below the percolation threshold, because of the lack of percolating clusters, the motion of electrons is restricted within the finite clusters and the polarization between the clusters will determine the conductivity. Therefore, the conductivity increases when the frequency increases.

The room temperature ac dielectric constant of the Ni-MgO composites is plotted on a logarithmic scale as a function of the applied frequency in Fig. 5. At low frequencies, the dielectric constant of the samples above  $f$  is high value and decreases as the applied frequency increases. The high dielectric constant at low frequencies is due to the sum of the various polarizability contributions. The behavior decreasing the dielectric constant is due in part to the distribution of relaxation times arising from the irregular structure of the Ni-MgO compo-

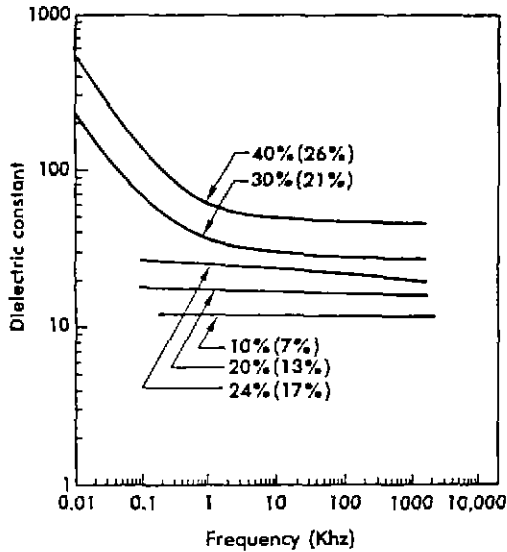


Fig. 5. AC dielectric constant of the Ni-MgO composites as a function of frequency. True volume percent Ni in parentheses.

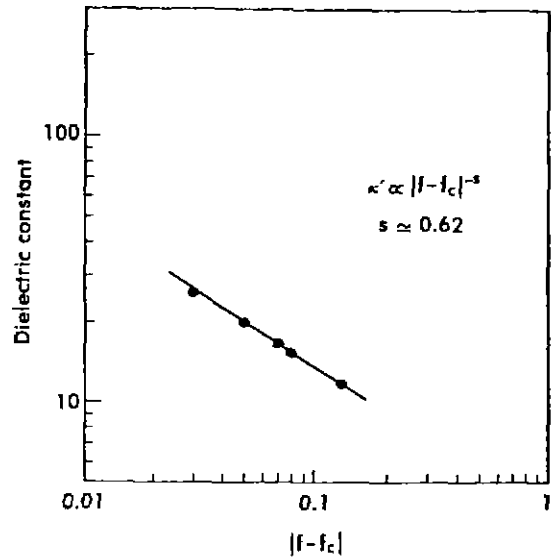


Fig. 6. Dielectric constant of Ni-MgO composites as a function of  $|f - f_c|$ .

sites. In the high-frequency region ( $>10$  kHz), the responses of different samples to the input signals become parallel, indicating that they obey the same power-law behavior. The critical exponent  $y$  is found to be  $0.05 \pm 0.01$  from the slopes of the curves for samples below percolation. Fig. 6 showed the variation of the dielectric constant as a function of the reduced volume fraction for five samples below the percolation threshold. The dielectric constant at 100 Hz was used rather than the dc dielectric constant, since the latter was not available from these experiments. According to Efros and Shklovskij<sup>18)</sup>, the ac dielectric constant will follow the same power-law behavior as the dc dielectric constant, if the applied frequency is low enough. Therefore, our exponent of dielectric constant,  $s = 0.62 \pm 0.07$  (obtained from the slope of Fig. 6), should be the same as the critical exponent of the dc dielectric constant. This value of the exponent is in good agreement with experimental values obtained by Granann *et al.*<sup>25)</sup> ( $s = 0.73 \pm 0.07$ ), and by Song *et al.*<sup>16)</sup> ( $s = 0.68 \pm 0.05$ ).

Recent percolation studies<sup>25,26)</sup> predict that the percolation threshold of a three-dimensional random composite is between 0.15 and 0.20. The value of the percolation threshold for our samples,  $f_c =$

$0.20 \pm 0.02$  is in good agreement with such generally accepted values. Our value is much lower than the experimental value ( $f_c \approx 0.47$ ) for granular metal films studied by Abeles *et al.*<sup>12)</sup>. This lower value may be due to effects occurring during preparation of the films, which produce a higher Ni particle density in the grain boundaries than inside the MgO grains. Our estimated percolation threshold is in excellent agreement with the experimental value found by Song *et al.*<sup>12)</sup>, for compacted mixture of teflon and carbon powders.

Our experimental values of the critical exponents,  $x$  and  $y$ , are now compared with the theoretical predictions presently available. Theoretical predictions, based on the intercluster polarization model, give  $x \approx 0.72$  and  $y \approx 0.28$ . However, recent calculations<sup>24)</sup> based on the anomalous diffusion model give the values  $x \approx 0.58$  and  $y \approx 0.42$ . Our values  $x = 0.98 \pm 0.05$  and  $y = 0.05 \pm 0.01$  are in good agreement with the scaling relation  $x + y = 1$  (see Equation 5), although they are significantly different from either of the theoretical predictions mentioned above. Our measured values are closer to those predicted by the intercluster polarization model than those predicted by the anomalous diffusion model.

#### 4. Summary

The ac conductivity and dielectric constant of the Ni-MgO composites are qualitatively in good agreement with the percolation behavior expected of conductor-insulator composites. The frequency exponents of conductivity and dielectric constant,  $x$  and  $y$ , are found to be  $x=0.98\pm 0.05$  and  $y=0.05\pm 0.01$ . Although each value is different from the theoretical predictions, there is good agreement with the general scaling relation  $x+y=1$ . The critical exponent in the relationship  $\kappa' \propto |f-f_c|^{-s}$  is found to be  $s=0.62\pm 0.07$ , and the estimated percolation threshold is  $f_c=0.20\pm 0.02$ . This value of the percolation threshold is in good agreement with that estimated from the optical measurements.

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