1/f or flicker noise in cellular percolation systems

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Flicker or $1/f$ noise is studied in a series of four composite discs, which consists of carbon black (ground and unground), graphite, and graphite/boron nitride, as the conducting components coating, and a common insulating matrix of talc wax. The measurements were done on the conducting side ($\phi > \phi_c$) of the critical volume fraction (ϕ_c), within a frequency range of 1.2–1001.2 Hz. The results are analyzed in terms of Hooge's empirical formula with frequency and voltage exponents γ and m, respectively. Values of γ obtained are in the range 0.97–1.2. Samples with larger $(\phi-\phi_c)$ have $m \approx 2$, while those with smaller $(\phi-\phi_c)$ have m significantly lower than 2. The normalized noise at 10 Hz ($S_{v10\,\mathrm{Hz}}/V^m$) obey the well-established relationships $S_{v(f)}/V^m \propto (\phi-\phi_c)^{-k}$ and $S_{v(f)}/V^m \propto R^w$, where V is the voltage across the sample with resistance R, while m, k, and w are exponents. However, a change in the value of the exponent k and w was observed in the measured systems, with k taking the values $k_1 \sim 0.75$–5.23 close to ϕ_c and $k_2 \sim 2.23$–5.54 further into the conducting region. Values of w_1 range from 0.36–1.37, while $w_2 \sim 0.99$–1.59. The $k_1(w_1)$ are observed when $m<2$. The nonuniversality of the k_1 and k_2 regimes are interpreted as due to the superposition of the behavior that results from the geometry (a random voidlike structure) and the behavior resulting from the presence of non-Ohmic, intergranular contacts between the conducting grains. These exponents are tested for consistency using $w = k/t$, and compared with predictions from recent theoretical models.

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I. INTRODUCTION

The transport properties of binary conductor-insulator (metal-insulator) mixtures have been extensively studied by theoretical and experimental physicists and materials scientists for many years. While a large number of theories are used in analysing the results,[1,2] probably the most successful has been percolation theory[1–5] and, more recently, a two-exponent phenomenological equation[6–9] which has the same parameters as percolation theory and reduces to the latter when the ratio of the conductivity of the insulating component (σ_i) to that of the conducting component (σ_c) is zero, which is only obtainable when σ_c is infinite or for a perfect insulator, at zero frequency. The parameters characterizing the structure of a system in percolation theory are the critical volume fraction (ϕ_c), the exponent t (which characterizes the conductivity of the system above ϕ_c) and the exponent s (which characterizes the conductivity and dielectric constant below ϕ_c). Most experimental work has been focused on the dc conductivity, less on the ac dielectric constant or conductivity properties and $1/f$ noise, and very little on the magnetoresistivity and thermopower. Each of these measurements is characterized by its own exponent(s) and theoretical relations between the exponents have been proposed.[3–6] One of the current problems is that in most previous experimental work, often only one of the above measurements, and sometimes two, were performed on the same system(s), which makes it difficult to correlate the percolation parameters measured on the same and different systems.

This has been rectified in Refs. 6 and 7, where measurements of the dc conductivity, dielectric constant, and magnetoresistivity[6] and the complex ac conductivity[7] were made on three graphite-boron nitride systems. In Ref. 10, similar measurements on seven cellular percolation systems are reported. This paper reports on the $1/f$ noise observed in cellular systems. These results are far more complex than any previously reported in that $1/f$ noise, as a function of $(\phi-\phi_c)$, shows two distinct regions. These regions are each characterized by its own exponents and the noise observed in the samples closest to ϕ_c is not proportional to V^2, as expected from Hooge's law.[11] The noise power is also shown to increase as the volume of the samples is reduced. The noise results are compared with previous experimental work and the latest available theories.

The relevant theory is presented in Sec. II and the experimental details in Sec. III. The results are given in Sec. IV and they are explained in Sec. V. The conclusions of this study are given in Sec. VI.

II. THEORETICAL BACKGROUND

When a constant "noise free" current is passed through a resistor, a noise spectrum of the form

$$S_{v(f)} = \alpha V^m/(N f^\gamma), \tag{1}$$

is observed. Equation (1) is Hooge's[11] generalized empirical formula and gives the noise power spectrum ($S_{v(f)}$) for voltage fluctuations. In Eq. (1), V is the average dc voltage across the sample, N is the total number of charge carriers in the sample, and α is a dimensionless number of the order 10^{-5}–10^{-1} in small volume metallic samples and as high as 10^3–10^7 in granular high-T_c materials. The exponent γ is of the order of unity, hence the term "$1/f$" noise. If the voltage exponent $m=2$, then the noise is produced entirely by equilibrium resistance fluctuations and the current acts only as a probe.[12,13] If the exponent $m \neq 2$, the noise is believed to be a driven phenomenon,[13] which could imply the coexistence of other mechanisms.

The physical origin of $1/f$ noise is still an open question despite the intensive investigations (both theoretical and experimental) that have been carried out on the phenomenon (see, for instance, the review articles[14–17]). In most approaches, the noise has been modeled in terms of random resistor networks.[18]

$1/f$ noise has been used to study percolation systems as a possible means of probing the microstructural inhomogeneities of the composites, especially close to the percolation threshold. Bergman[19] noted that the microgeometry of the composite plays a much more important role in the determination of $1/f$ noise than previously thought. This is because in real continuum systems, the bond resistances usually obey a power law distribution and the noise enhancement is produced mainly by the small narrow regions of conducting material or the links of the links, nodes, and blobs (LNB) model,[20,21] where the electric field or resistance is abnormally large. Using the LNB model[20] as described below, the effect of such high resistors can be accounted for,[21] yielding the well known non-universal behavior of the exponents.

Wright *et al.*[22] explained the divergence of $1/f$ noise on approaching the percolation threshold in terms of the dilution of the backbone of the percolation network. When the conductor volume fraction in the sample is well above the percolation threshold, current flows through a large number of interlinked backbones, so that the independent fluctuations in different paths tend to average out and decrease the fluctuation in the total resistance. However, as the conductor fraction approaches the percolation threshold (from the conducting side), the tenuous connectivity of the conducting backbone restricts current flow to a small number of tortuous paths or backbones, which minimizes cancellations of independent fluctuations, resulting in an increase of the noise amplitude.

In 1985, Rammal, Tannous, Breton and Tremblay[23] introduced a new scaling exponent k to describe the divergence of the normalized power spectrum $S_{v(f)}/V^m$ (expressed here in terms of voltage fluctuations), close to but on the conducting side of the percolation threshold, through the power law

$$S_{v(f)}/V^m \propto (\phi-\phi_c)^{-k}. \tag{2}$$

The other formula for $S_{v(f)}/V^m$ used to describe the experimental results is[23]

$$S_{v(f)}/V^m \propto R^w, \tag{3}$$

where $w=k/t$. Experimental results are usually given as a plot of S_v/V^m against dc resistance R as it eliminates the statistical fluctuations in σ_m (the conductivity of the composite) observed when the latter is plotted against $(\phi-\phi_c)$. Note also that for samples close to ϕ_c, the small errors in ϕ are amplified in the $(\phi-\phi_c)$ term. The critical volume fraction (ϕ_c) in this paper is obtained from dc conductivity measurements.[10] Problems can arise if Eq. (2) is used and ϕ_c deduced from noise measurements only, as the dc conductivity[6,10] gives the most accurate values of ϕ_c. Equations (2) and (3) are usually written with V^2 rather than V^m, as most previous results reported values of $m \simeq 2$.

The most useful approach to consider the electrical properties of continuum percolation systems is the links nodes and blobs (NLB) model.[17,20] For simplicity, assuming first that all the resistors in the system are of the same value, the basis for this model is approximating the real system of the percolation backbone by a three-dimensional (cubic) net such that its members, the links, have a length that is about the correlation length of the system (the diameter of the largest but finite clusters). Each link connects to other links at the nodes, and it consists of two types of resistors. Those individual resistors through which the entire current of the link does flow (the ''singly connected bonds'') and those that are made of a collection of resistors, the blobs, that are essentially some parallel connection of individual resistors, so that the current through each of them is only part of the link's current. As the percolation threshold is approached from above the network becomes sparse, and the electrical properties of the system can be evaluated as if it consists only of singly connected bonds. In the simple-universal picture the links are all assumed to be statistically equivalent thus having the same resistance. If there is some regular value distribution of the individual resistors in the system, the average (or the effective) resistance of each link can be calculated and each link can be assumed to be made of a chain of individual equal or effective resistors, such that the effective value of the resistance of these resistors, is independent of the proximity to the percolation threshold. However, if one assumes[21] a diverging (though normalizable) distribution for the higher-value resistors the average resistance value of the effective resistor of the link will increase, as the percolation threshold is approached. This is simply due to the fact, that in that approach, the system becomes sparse with fewer resistors, thus yielding a diverginglike average resistance value. Hence, as the threshold is approached, in addition to the reduction of the number of current paths, that is manifested by the universal behavior, through the conductivity and noise universal exponents t_{un} and k_{un}, there is an increase in the average value of the effective resistor in the sample that is manifested by the nonuniversal behavior, through the values of $t-t_{un}$ and $k-k_{un}$.[17,22–24]

Following the above ideas it was shown theoretically[24–26] and confirmed experimentally,[25,27–29] that such a nonuniversal behavior can be observed. There were two basic models that provided the resistor-value distributions that could account for the observation of a nonuniversal behavior. The first was a geometrical model[26,30,31] in which the high-value resistors result from the narrow conducting-phase necks in systems of insulating particles.[26,31] In the models discussed in Refs. 24 and 26 the conducting phase was assumed to be continuous and homogeneous, but of course a granular conducting phase that consists of particles that are much smaller than the insulating particles can be described by the same picture [provided there is a continuous electrical contact between the small conducting grains in the ''necks'' equivalent of the random void (''Swiss cheese'') models, see below]. The other was a physical-tunneling model in which the high-value resistors result from the more widely separated nearest-neighbor conducting particles.[25] The first system (that resembles a conducting liquid in a sedimentary rock) is known as the random void (RV) model, and its mirror image (a system of insulating liquid and conducting particles) is known as the inverted random void (IRV) model. Considering these definitions the cellular composite at hand resembles very much an RV system, but the possibility of tunneling between adjacent conducting particles suggests that the effect of this transport mechanism cannot be, *a priori*, ignored. Originally, the RV and IRV models were considered for one type of a neck-width distribution[26] but more recently these models have been extended[30] to other possible neck-width distributions. The latter non-random distributions can be phenomenologically characterized by an exponent ω' that defines the distribution $h(\varepsilon)$, of the neck widths ε, by the relation $h(\varepsilon) \propto \varepsilon^{-\omega'}$.[30] In the tunneling model[25] one considers the exponential distribution of the nearest-neighbor distances in the case of conducting (spherical-like) particles in a random system, as well as the exponential nature of the tunneling conductance. The key parameter that then determines the non-universal behavior of the electrical resistance is the ratio a/d where a is the average distance between adjacent conducting particles and d is the typical tunneling distance, such that $t = t_{un} + a/d - 1$. On the other hand in this case, independent of the particular system parameters, the electrical noise exponent k is simply given by $k_{un} + 1$.

Since both, the corresponding theories and their predictions, have been given in detail in the widely available literature[24–26,30,31] we will mention here only the predictions that are relevant to the present work. First, the value of t was found[31] to be $t_{un} + 1/2$ in the three-dimensional RV model and t_{un} in the three-dimensional IRV model. In the extended model[30] the corresponding plausible values of t were found to be any value larger than t_{un}. The particular value of t also then depends on the above defined value of ω'. On the other hand, this extended theory predicts that the ratio $w = k/t$ is limited to the interval $0.78 \leqslant w \leqslant 2.1$ for the RV model and to $0.78 \leqslant w \leqslant 3$, for the IRV model. In contrast, as we saw above, the theoretical value of t in the tunneling model is limited by the value of a/d which in systems such as ours can, in principle, be of the order of 100. However, in practice, due the nonideal distribution of the separations of the conducting particles, the highest value found[25] so far, experimentally, for t was only 6.4. On the other hand, the constant value of k under this mechanism ($k = k_{un} + 1 = 2.56$) sets the upper limit of the normalized noise exponent w, to be $2.56/(t_{un} + a/d - 1)$. The interesting observation[25] regarding the value of w is that it can be smaller than the universal value of $w = k_{un}/t_{un} = 1.56/2.00 = 0.78$. These results account well for other experimental results.[25] We further point out that in view of the fact that, experimentally, the dependence of the electrical-noise power on the resistance of the sample, is more accurately derived[25,27–29] than the dependence of the electrical noise on the conducting content of the composite, it is more common that the exponent $w = k/t$, rather than the independently predicted exponent k, is the reported quantity.

In summary, while in the above models the conductivity is determined by the geometric structure (e.g., the necks in the random void model[26,31]), in the cellular structures the conductivity can also be determined by contact resistances between the conducting grains (e.g., tunneling resistances between the particles[17,25]). When tunneling dominates, it has been shown that $t \geqslant 2$ and that its value can be, in principle, as large as 100 while w can be smaller than the universal value of 0.78.[25]

III. EXPERIMENTAL METHODS

The four cellular systems measured here [raw carbon black (RCB), ground carbon black (GCB), graphite (GG), and graphite boron nitride (GBN)] were made by coating large (300 μm) wax coated talc spheres with fine (approximately 10 μm) conducting powders. The resultant mixture was compressed into 26 mm diameter and about 3 mm thick discs. Electrodes were made by coating each of the faces of the discs with silver paste into which the current and voltage terminals were embedded. Subsequently 10 mm diameter discs were drilled out of the larger discs and polished down to a thickness of about 1.5 mm. More details can be found in Ref. 10.

The measured noise spectra were characterized using Hooge's empirical formula [Eq. (1), Hooge[11]], therefore, the frequency and voltage dependence of the noise (S_v) were first examined. The noise dependence on the dc voltage was investigated by passing several currents (at least five) through each sample so that a plot of S_v versus the dc voltage across the sample could be made. The appropriate current was obtained using a combination of alkali batteries and wire wound resistors. The noise voltage was measured using a Stanford SR530 Preamplifier operating on its internal battery. The sample, current source and preamplifier were all placed in a closed steel container. The output of the preamplifier was monitored using a HP3562a spectrum analyzer. All measurements were done in the linear or Ohmic range and within a frequency range of 1.2–1001.2 Hz. The noise dependence on the dc voltage was studied to obtain the exponent m [Eq. (1)]. The resistance and conductor concentration dependence of the normalized noise (S_v/V^2 and S_v/V^m, using the experimental values of m) were then investigated and analyzed using Eqs. (2) and (3).

IV. EXPERIMENTAL RESULTS AND THEIR ANALYSIS

In order to investigate the $1/f$ nature of the noise spectra, the measured noise (S_v) was plotted on a log-log scale against the frequency (f) in order to determine the slope γ from Hooge's formula [Eq. (1)]. The values of γ (obtained by linear fitting) for all the samples measured in all four of the fully investigated systems were in the range 0.97–1.2, for both small and large samples.

Previous experiments[6,12,13,28,29,32,33] have reported various ranges of the exponent γ. The values of γ obtained in this study (0.97–1.2) are all well within this range. In general, the accepted values of γ reported in the literature for $1/f$ noise are in the range $0.8 < \gamma < 1.4$. Note that in most of

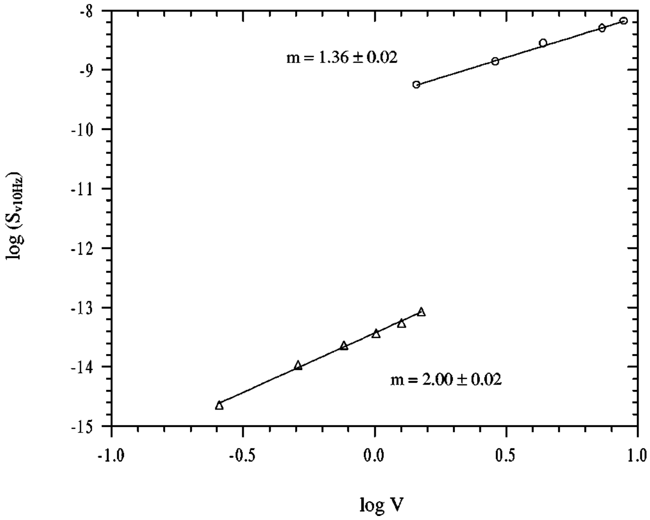

FIG. 1. A log-log plot of the noise measured at 10 Hz ($S_{v\,10\ \mathrm{Hz}}$) versus the voltage (V) across the sample for two samples from the raw carbon black system [$\phi=0.0135(\bigcirc),\phi=0.0249(\triangle)$]. The solid lines are linear fits to the data which yield the m values, as shown on the graph.

these experiments, no correlation between the values of γ and the sample resistances or voltages were reported. However, Pierre *et al.*,[29] whose results will be further discussed, obtained γ values between 1.0 and 1.4 for all the samples of the copper-polyethylene composites that they studied, but noted that for their high resistance samples ($>10^6\Omega$) the exponent γ tended to be closer to 1 ($\gamma=1.00\pm0.01$). No such correlation was found in the cellular systems, in agreement with most of the previous studies.

The voltage dependence of the noise power was examined using a log-log plot of the noise at 10 Hz ($S_{v\,10\ \mathrm{Hz}}$) versus the dc voltage (V) across each sample, resulting from the various constant currents. Figure 1 shows such plots for two samples from the raw carbon black system. Similar plots were done for all the samples upon which the measurements were made in order to obtain a value of m for each sample from $S_v \propto V^m$ [Eq. (1)]. The values of m obtained for each sample, characterized here by $(\phi-\phi_c)$, of the four systems studied are shown in Table I. The values shown here are for the larger 26 mm diameter samples. Comparing with known previous work, this is the first time that the noise dependence on the voltage has been investigated across such a wide range of sample resistance. The values of ϕ_c and t, as measured in,[10] are also included in this table. These values can be obtained from either the percolation equations or Eq. (1) in Ref. 10. The resultant parameters are the same within the experimental error. As discussed in Sec. V, these parameters are important in characterizing the systems under study.

The errors in ϕ_c and $t(t_{exp})$ are shown in Table II. The abbreviations GCB, RCB, GG, and GBN represent ground carbon black, raw carbon black, graphite, and graphite boron nitride systems, respectively.

The values of m were found to lie between 1.00 and 2.13 with the lower resistance and larger $(\phi-\phi_c)$ samples generally giving higher values of the exponent. This observation is further confirmed by the decrease in m with sample volume (which is equivalent to an increase in sample resistance). As the samples close to the percolation threshold give values of m lower than 2, this implies that the noise here is not solely generated by equilibrium resistance fluctuations[12] close to ϕ_c, but rather by a voltage dependent conduction mechanism that will be discussed in Sec. V. However, as the resistance of the samples decreases, m approaches 2, giving the expected quadratic dependence of the noise on the dc voltage.[12,13,23,28,29] According to the criteria used by Nandi

TABLE I. Variation of the exponent m [Eq. (1)] with $\phi-\phi_c$ for the cellular systems.

GCB $\phi_c=0.0122$ $\phi-\phi_c$	GCB $t=2.06$ $m\pm0.04$	RCB $\phi_c=0.0131$ $\phi-\phi_c$	RCB $t=2.26$ $m\pm0.02$	GG $\phi_c=0.035$ $\phi-\phi_c$	GG $t=1.93$ $m\pm0.06$	GBN $\phi_c=0.033$ $\phi-\phi_c$	GBN $t=2.51$ $m\pm0.05$
0.00045	1.28	0.00024	1.03	0.0002	0.92	0.00058	1.15
0.00112	1.60	0.00033	1.33	0.0003	1.49	0.00147	1.45
0.00140	1.66	0.00041	1.36	0.0004	1.19	0.00201	1.38
0.00188	1.88	0.00045	1.25	0.0010	1.51	0.00209	1.50
		0.00137	1.35	0.0011	1.66		
0.00200	1.94	0.00165	1.66	0.0015	1.76	0.00278	1.67
0.00336	1.80	0.00173	1.76	0.0017	1.70	0.00696	2.03
0.00580	2.05	0.00275	1.82	0.0018	1.80	0.00709	2.04
0.00960	2.13	0.00382	1.83	0.0019	1.90	0.00843	1.98
		0.00529	1.87	0.0038	2.05		
		0.00997	1.92				
		0.01019	2.13				
		0.01180	2.00				
		0.01418	2.05				

TABLE II. The exponents k [Eq. (2)] and w [Eq. (3)] for the cellular systems

Exponent	GCB	RCB	GG	GBN
k_1	2.80 ± 0.08	5.23 ± 0.12	0.92 ± 0.03	1.90 ± 0.31
k_{1m}	3.51 ± 0.01	2.27 ± 0.19	2.18 ± 0.02	2.86 ± 0.14
k_2	2.71 ± 0.11	3.29 ± 0.12	2.58 ± 0.05	3.60 ± 0.08
k_{2m}	2.91 ± 0.10	2.83 ± 0.04	2.57 ± 0.07	4.02 ± 0.09
k_1'	1.07 ± 0.21	0.75 ± 0.08	0.83 ± 0.07	1.37 ± 0.05
k_{1m}'	1.28 ± 0.20	1.27 ± 0.12	1.73 ± 0.14	2.23 ± 0.07
k_2'	2.23 ± 0.17	2.52 ± 0.08	2.76 ± 0.11	5.54 ± 0.13
k_{2m}'	2.27 ± 0.16	2.47 ± 0.13	2.76 ± 0.11	5.33 ± 0.32
k_{calc}	4.33 ± 0.21	4.66 ± 0.24	4.11 ± 0.13	5.08 ± 0.25
w_1	1.10 ± 0.10	0.93 ± 0.05	0.36 ± 0.01	0.74 ± 0.13
w_{1m}	1.37 ± 0.02	1.24 ± 0.10	0.57 ± 0.09	0.93 ± 0.22
w_2	1.28 ± 0.11	1.38 ± 0.03	1.30 ± 0.06	1.36 ± 0.09
w_{2m}	1.34 ± 0.07	1.46 ± 0.05	1.20 ± 0.07	1.59 ± 0.08
w_1'	0.89 ± 0.14	0.68 ± 0.03	0.42 ± 0.08	0.57 ± 0.03
w_{1m}'	1.05 ± 0.12	1.24 ± 0.02	0.86 ± 0.15	0.95 ± 0.03
w_2'	0.99 ± 0.14	1.10 ± 0.06	1.40 ± 0.13	1.42 ± 0.04
w_{2m}'	1.00 ± 0.12	1.09 ± 0.08	1.40 ± 0.13	1.37 ± 0.02
w_{calc}	2.10 ± 0.10	2.06 ± 0.11	2.13 ± 0.07	2.02 ± 0.10
ϕ_c	0.0122 ± 0.0007	0.0131 ± 0.0006	0.035 ± 0.002	0.033 ± 0.001
t_{exp}	2.06 ± 0.10	2.26 ± 0.11	1.93 ± 0.06	2.51 ± 0.12
$t_{n2}=k_2/w_2$	2.12 ± 0.27	2.38 ± 0.08	1.98 ± 0.13	2.65 ± 0.23
$t_{n2m}=k_{2m}/w_{2m}$	2.17 ± 0.19	1.94 ± 0.09	2.14 ± 0.18	2.53 ± 0.18
$t_{n2}'=k_2'/w_2'$	2.25 ± 0.49	2.29 ± 0.20	1.97 ± 0.15	3.90 ± 0.20
$t_{n2m}'=k_{2m}'/w_{2m}'$	2.27 ± 0.43	2.27 ± 0.29	1.97 ± 0.26	3.89 ± 0.29
$t_{n1}=k_1/w_1$	2.54 ± 0.30	5.62 ± 0.43	2.56 ± 0.15	2.57 ± 0.87

et al.,[13] $m=2$ is given by systems in which equilibrium resistance fluctuations are the sole source of the noise. Values of m greater or smaller than 2 would therefore imply the coexistence of other mechanisms. Note that the Ag+Pt-TFE composite samples, studied by Rudman *et al.*,[28] show that for over three decades of voltage change the noise scales approximately as V^2 with an mean exponent $m=2.01\pm0.05$. In the graphite-boron nitride disc samples studied by Wu and McLachlan,[6] a mean value of $m=1.96\pm0.01$ and $m=1.93\pm0.02$ were measured in the axial (compression) and transverse directions respectively, in reasonable agreement with the expected quadratic dependence. The lowest m values observed were 1.87 ± 0.04 and 1.81 ± 0.07, for the samples closest to ϕ_c, in the axial and transverse directions, respectively.

To the best knowledge of the authors, the only study to have reported values of m much lower than 2 is Ref. 12, where the 1/f noise on single component conducting polymer 2D thin film resistors was measured. In spite of their single component nature, the samples were also found to give different values of m at four different frequencies. Bruschi *et al.*[12] obtained m values of 1.38 (62.5 mHz), 1.54 (109 mHz), 1.73 (0.5 Hz), and 1.73 (1 Hz), from which they concluded that equilibrium resistance fluctuations do not completely explain the noise spectra in their samples. Note that the values of m are similar to those obtained, for samples close to the percolation threshold, at 10 Hz in all four cellular systems studied in this paper. Bruschi *et al.*[12] attributed the values of m much lower than 2 to the presence of a shot noise component, as the noise level corresponding to these values were only an order of magnitude above the background noise. However, this argument does not apply in the cellular systems, because the noise level measured in the samples with values of m lower than 2 was at least three orders of magnitude above background noise. In addition, the values of m quoted in Ref. 12 were measured at different frequencies, whereas the measurements reported in this paper were analyzed only at 10 Hz.

The log-log plots of the normalized noise ($S_{v_{10\ \mathrm{Hz}}}/V^2$) as a function of concentration ($\phi-\phi_c$) are shown in Figs. 2 and 3 for the raw carbon black and graphite systems, respectively. These plots were made in order to obtain k from Eq. (2). From the graphs, two regions of different slopes can be identified, yielding the exponents labeled as k_1 and k_2 for samples close to and further away from the percolation threshold (ϕ_c), respectively. The only exception is the ground carbon black system, where the change in slope is not very distinct. Note that the fact that the exponent k_1 is obtained from samples with the lower values of m, while k_2 arises from samples with $m\approx2$, is important (see Sec. V). The values of k_1 and k_2 obtained for all the systems are summarized in Table II. The exponents k_1' and k_2' are from the smaller (10 mm diameter) samples. Figures 4 and 5 show

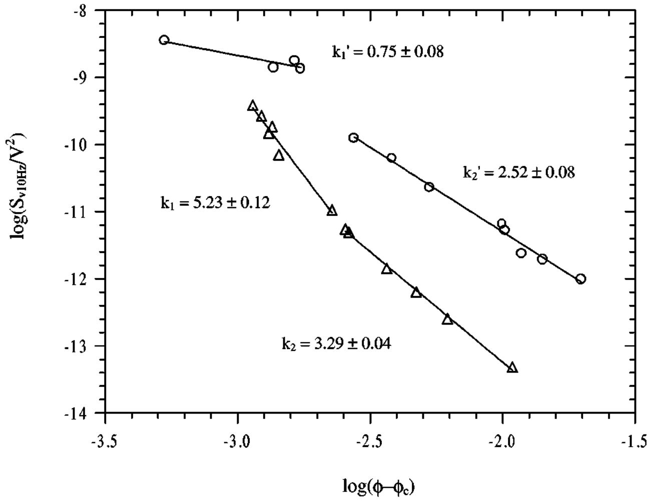

FIG. 2. A log-log plot of the normalized noise ($S_{v10\ \mathrm{Hz}}/V^2$) versus ($\phi-\phi_c$) for two sets of samples from the raw carbon black system [10 mm dia. (○), 26 mm dia. (△)]. The solid lines are linear fits to the data which yield the exponents k, as shown on the graph.

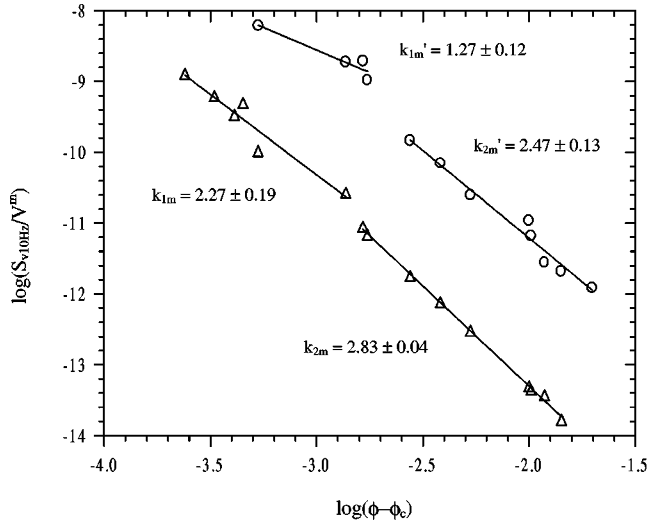

FIG. 4. A log-log plot of the normalized noise ($S_{v10\ \mathrm{Hz}}/V^m$) versus ($\phi-\phi_c$) for two sets of samples from the raw carbon black system [10 mm dia. (○), 26 mm dia. (△)]. The solid lines are linear fits to the data which yield the exponents k, as shown on the graph.

the corresponding plots to Figs. 2 and 3 for $S_{v_{10\ \mathrm{Hz}}}/V^m$ (obtained through normalizing the measured noise by the experimental values of m). The corresponding exponents are k_{1m} and k_{2m} (as well as k'_{1m} and k'_{2m} for the smaller samples), also shown in Table II. These are the first reported results that show the occurrence of two values of the exponent k in any system. Note that a wide range of m values have also been measured and then used to normalize the noise results to obtain the k_m values. However, a w_1 and w_2 (to be presented below) have been observed twice before.[28,29] The reason for the occurence of the two exponents closer to the percolation threshold will be discussed in Sec. V.

The experimental values of k in Table II cover a wide range (0.74–5.60). The smallest value of k was measured in the raw carbon black system. The graphite/boron nitride system gives the highest values of the k exponents, except for k_1 and k_{1m}. The values of k_1 all decrease markedly with sample size, while the values of k_2 for the graphite systems increase with decreasing sample size. Normalizing the noise by the measured m values has a larger effect on the k_1 exponents (an increase in k_1) than k_2 as the m values are very different from 2 in the region close to the percolation threshold. The values of k_2 are marginally affected by the m normalization as the m values in this region are close to the expected value of 2. For the larger samples, the carbon black systems have

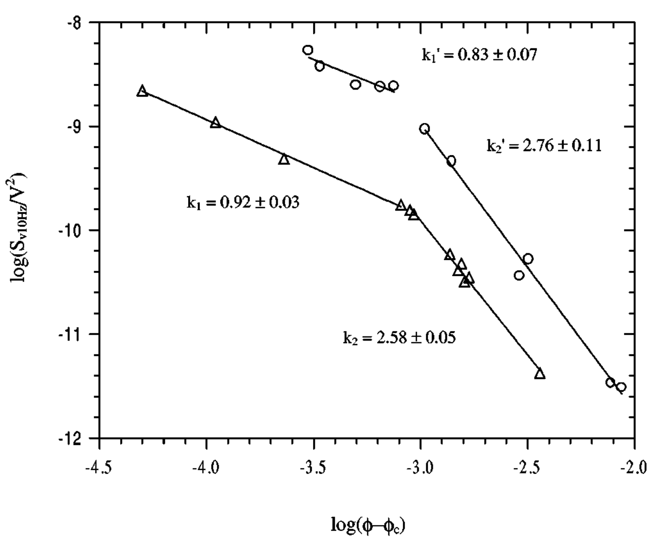

FIG. 3. A log-log plot of the normalized noise ($S_{v10\ \mathrm{Hz}}/V^2$) versus ($\phi-\phi_c$) for two sets of samples from the graphite system [10 mm dia. (○), 26 mm dia. (△)]. The solid lines are linear fits to the data which yield the exponents k, as shown on the graph.

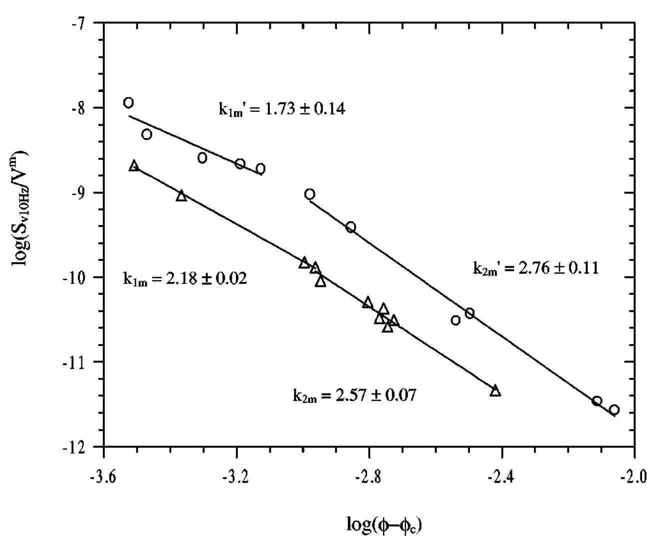

FIG. 5. A log-log plot of the normalized noise ($S_{v10\ \mathrm{Hz}}/V^m$) versus ($\phi-\phi_c$) for two sets of samples from the graphite system [10 mm dia. (○), 26 mm dia. (△)]. The solid lines are linear fits to the data which yield the exponents k, as shown on the graph.

TABLE III. Noise exponents from previous experiments. G-BN: graphite-boron nitride, C-WX: carbon-wax, C-PO: copper-polymer, Ag/Pt-TFE: silver/platinum-tetrafluoroethylene. Stars denote values calculated from other measured exponents. Note that k and w from previous studies are comparable to k_2 and w_2 in the present study probably because they were measured in the region well above ϕ_c.

System	t^*	$k^*(k_2^*)$	w_1^*	$w^*(w_2^*)$
G-BN (Ref. 6)	2.63 ± 0.07	$\star 3.87\pm0.07$		1.47 ± 0.05
C-WX (Ref. 13)	$\star 1.88$	$\star 3.60$		1.7 ± 0.2
C-PO (Ref. 29)			1.0 ± 0.3	1.5 ± 0.2
Ag/Pt-TFE (Ref. 28)			3.0	1.0
C-WX (Ref. 27)	2.3 ± 0.4	5.0 ± 1.0		$\star 2.2\pm0.8$

k_1 values greater than the corresponding k_2 exponents whereas the reverse is true for the graphite-containing systems. However, for the smaller samples, k_1' and k_{1m}' are consistently lower than k_2' and k_{2m}', as shown in Table II. The exponents with the subscript m were obtained from normalizing the voltage [in Eqs. (2) and (3)] with the experimentally determined m values. The primed exponents were obtained from measurements done on the smaller (10 mm diameter) samples. Exponents k_{calc} and w_{calc} were calculated using the experimental values of t (t_{exp}) in Eqs. (3), (8), and (9) from Ref. 30.

We note in passing that apart from the work of Chen and Chou[27] on carbon-wax mixtures, very few experiments have reported direct measurements of the exponent k in real 3D continuum systems (refer to Table III). However, in a number of experiments,[6,13,28] values of t and w have been measured and values of k calculated from the relationship $w=k/t$ introduced by Rammal *et al.*[23]

As can be seen from Table III, in most experiments, a set of exponents (t, w, and k) was never measured directly in the systems in order to check the relationship $w=k/t$, as done in this paper, which appears to be the first work on noise measurements in which the exponents t,k, w, and ϕ_c have all been determined experimentally. Note too that *correct* values of k and t need a good identification of ϕ_c, using dc conductivity measurements done below and above ϕ_c.[6,10]

The normalized noise power at 10 Hz ($S_{v\,10\,\mathrm{Hz}}/V^2$ and $S_{v\,10\,\mathrm{Hz}}/V^m$) was also plotted on a log-log scale against the sample resistance, as shown in Figs. 6–9, for the raw carbon black and graphite systems. The values of w for all the systems studied here are given in Table II. As before, the results for all the cellular systems again clearly show two linear regions, corresponding to the two exponents given as w_1 and w_2 (see the graphs). The exponents w_1' and w_2' refer to the exponents measured in the smaller (10 mm diameter) samples. The w_{1m} (w_{1m}') and w_{2m} (w_{2m}') are the corresponding exponents obtained from normalizing the noise results by the measured values of m (as opposed to the expected value of $m=2$).

The values of w from the cellular systems are in the range 0.35–1.68, with the lowest w ($w_1=0.36\pm0.01$) measured in the graphite system. The highest value of w ($w_{2m}=1.59\pm0.08$) is found in the graphite/boron nitride system. With the exception of the graphite system, the w_1 values decrease

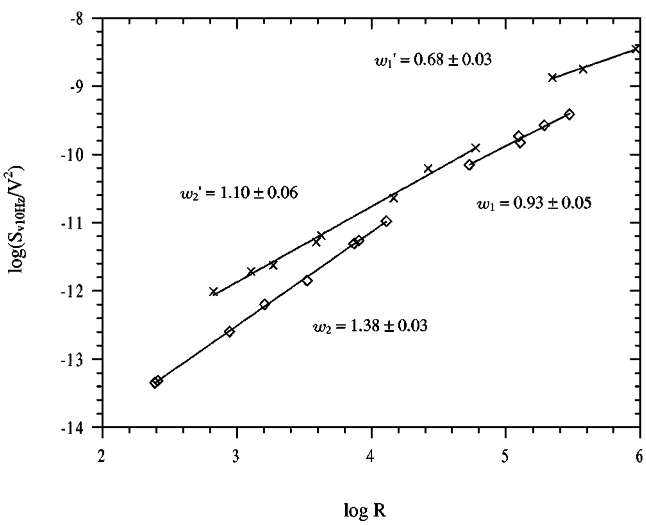

FIG. 6. A log-log plot of the normalized noise ($S_{v\,10\,\mathrm{Hz}}/V^2$) versus dc resistance ($R$) for two sets of samples from the raw carbon black system [10 mm dia. ($\times$), 26 mm dia. ($\diamond$)]. The solid lines are linear fits to the data which yield the exponents w, as shown on the graph.

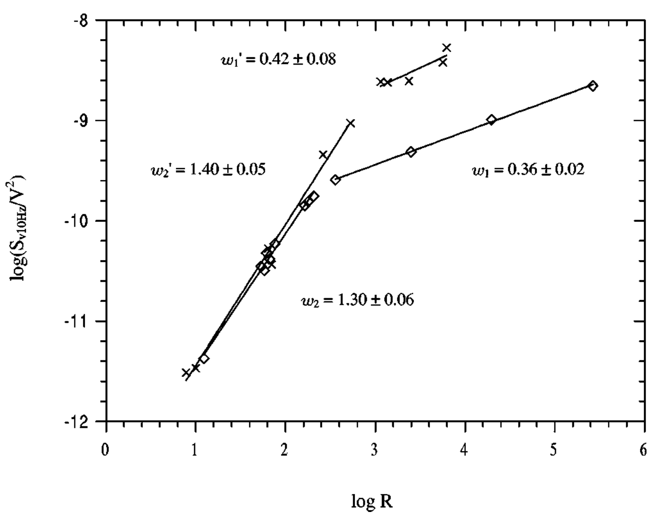

FIG. 7. A log-log plot of the normalized noise ($S_{v\,10\,\mathrm{Hz}}/V^2$) versus dc resistance ($R$) for two sets of samples from the graphite system [10 mm dia. ($\times$), 26 mm dia. ($\diamond$)]. The solid lines are linear fits to the data which yield the exponents w, as shown on the graph.

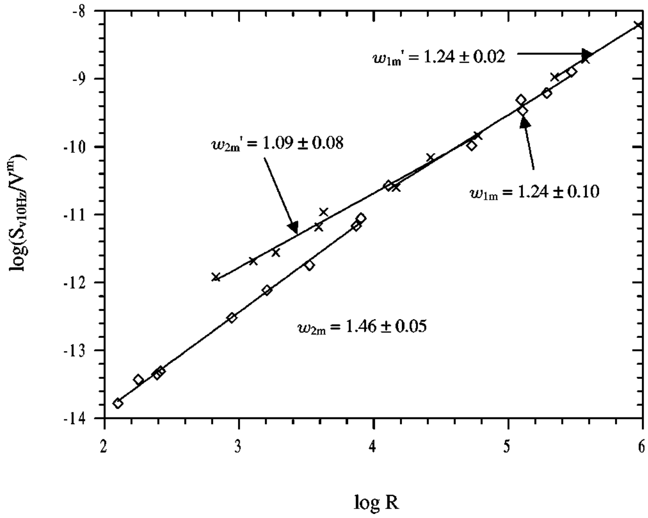

FIG. 8. A log-log plot of the normalized noise ($S_{v\,10\text{ Hz}}/V^m$) versus dc resistance (R) for two sets of samples from the raw carbon black system [10 mm dia. ($\times$), 26 mm dia. ($\diamond$)]. The solid lines are linear fits to the data which yield the exponents w, as shown on the graph.

with sample volume. As with k_1, the w_1 values are also the most affected by the m normalization, while the w_2 are only marginally altered, since the m values in this region are closer to the expected value of $m=2$. The w_2 and w_2' values for all the systems are very close (Table II), indicating the dominance of the same noise process in all of them (see Sec. V). Note that Nandi *et al.*[13] showed theoretically that the addition of a single resistor (or in the present case link) in parallel to an existing network leads to a decrease in the total noise of the network. The w_2 results also show better agreement than the w_1 with the limits of $0.78<w<2.10$.[30] The above results appear to indicate that the RV models are more

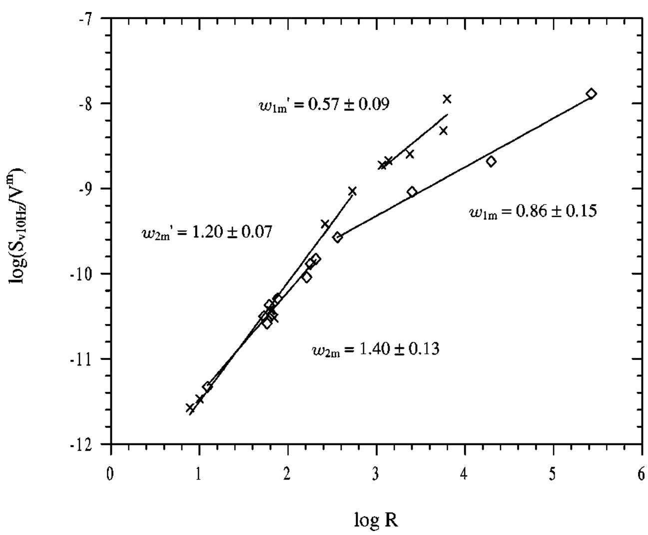

FIG. 9. A log-log plot of the normalized noise ($S_{v\,10\text{ Hz}}/V^m$) versus dc resistance (R) for two sets of samples from the graphite system [10 mm dia. ($\times$), 26 mm dia. ($\diamond$)]. The solid lines are linear fits to the data which yield the exponents w, as shown on the graph.

applicable to the w_2 and k_2 values than w_1 or k_1, i.e., to systems where $S_v \propto V^2$. The same trend is also shown by the exponents w_1' and w_2'.

The three-component system consisting of graphite, boron nitride, and talc wax shows an interesting pattern in its noise exponents when compared with the system containing only graphite and talc wax. Similar to the t exponent, all the k and w values of the three-component system are larger than the corresponding exponents in the plain graphite system. The only exception is in the w_{2m}' values, which are equal within experimental uncertainty. In some cases, the exponents are markedly different (see, for instance, k_2', k_{2m}', and w_1 in Table II), with the plain graphite giving a value about half that measured in the three-component system. Note that, at a common $\phi-\phi_c$ ($\phi-\phi_c=0.0005$), $S_{v\,10\text{ Hz}}/V^2$ for the three-component system was found to be an order of magnitude higher than that of the graphite and carbon black systems. The results given in Table II show that there is also a correlation between the magnitudes of the noise exponents and the t exponent in the two graphite systems. The reason why this does not show clearly in the carbon black systems is probably because the t exponents in the latter are very close. Note that the difference between the two graphite-containing systems is the manner in which the graphite particles are distributed on the talc-wax particles as the systems have the same critical volume fraction (within experimental error) obtained from dc conductivity measurements.[10]

Examination of Table II shows that some of the noise exponents indicate a possible correlation with the t exponent. The exponents k_2 and k_{2m} show a definite trend of increasing with t, while k_2' and k_{2m}' appear to increase with t but with more scatter. The exponents k_{calc} and w_{calc} were calculated from the modified RV model [Eqs. (3), (8), and (9) in Ref. 30], using the experimental values of t (t_{exp}). As expected from theory, k_{calc} increases linearly with t. However, as expected, both the experimental w and w_{calc} values appear to be independent of the t exponent.

Unlike k_1 and k_2, two values of the w exponent have been observed in previous studies on two continuum systems. Pierre *et al.*[29] studied $1/f$ noise in copper particle-polymer composites as a function of frequency ($10^{-2}-10^4$ Hz) and resistance ($10^1-10^9\ \Omega$). They observed two values of w; with $w_1^*=1.0\pm0.3$ for the high resistance ($10^5-10^9\ \Omega$) and $w_2^*=1.5\pm0.2$ for the low resistance ($10^1-10^6\ \Omega$) samples, in qualitative agreement with the results obtained from the cellular systems. Pierre and his colleagues[29] attributed the $S_v \sim R^{1.5}$ behavior to conduction in the Sharvin limit and $S_v \sim R^{1.0}$ as arising from a noisy contact limited by intrinsic conduction through a dirty oxide layer, given the high oxygen affinity of the copper powder. Pierre *et al.*[29] were the first to propose the dependence of the noise exponents on the different types of contacts existing above the percolation threshold in real continuum systems. However, the mechanism of intrinsic conduction through a dirty oxide layer does not apply to the cellular systems, as none of the conductors used (carbon and graphite) have an oxide layer. Based on the interpretation of Pierre *et al.*, the w_2 values in Table II could be explained by conduction in the Sharvin limit, which occurs when the mean free path is much smaller than the contact radius between two contacting metallic and spherical particles and gives rise to a 3/2 law ($w=1.5$) dependence of the noise power on contact resistance.[29,34] How relevant this is to our carbon black and flaky graphite is not clear, but note that the raw carbon black and graphite/boron nitride systems have w_{2m} values (1.46 ± 0.05 and 1.59 ± 0.08, respectively) close to $w^*=1.5$.

Rudman *et al.*[28] obtained $w_1^*\sim3$ and $w_2^*\sim1$ (i.e., $w_1^*>w_2^*$), at a frequency of 10 Hz, closer to and further away from ϕ_c, respectively, in a Ag+Pt-TFE composite and proposed that the transition observed in the w exponent was due to volume fraction dependant changes in the distribution of the interparticle conductances. The samples used in their experiments had resistance values in the range 0.5Ω to $30k\Omega$. The most obvious difference between this result and those obtained in the cellular systems studied here is the fact that $w_1^*>w_2^*$ in the Ag+Pt-TFE composite as opposed to $w_1<w_2$ observed in the cellular systems.

Rammal *et al.*[23] showed theoretically that the exponent w should be related to the noise and conductivity exponents by $t = k/w$. Using the experimental values of k and w, values of t were calculated based on this relationship. These values are denoted as t_n and are shown in Table II. It is important to note that most of the t_n values obtained from the k_2 and w_2 exponents are very close to the corresponding t exponents obtained from dc conductivity measurements in the four cellular systems.[10]

As already indicated in the results above, the volume dependence of the magnitude of the noise was also investigated using large and smaller samples from the same series. The ratio of the volume of the bigger samples to that of the smaller samples varied from between 10 and 20. From Eq. (1) and the discussion at the end of Sec. II, the noise is also expected to change by the same ratio(s). Instead, the ratio of the noise in the two sets of samples from the cellular systems was observed to vary between 10 and 1000. The higher values of the ratio could be due to a relatively small number of links in the smaller samples and the resultant statistical uncertainties. Unfortunately, there are no previous results available for comparison.

V. DISCUSSION

In the absence of a systematic theory for cellular composites, such as those described here, we try now to explain the main features of the data, as given in Sec. IV, within the framework of the two existing theories. These theories consist of the geometrical model (the RV model) and the physical (the tunneling) model as outlined in Sec. II. The main challenge in such an explanation is that, while there is a single t value found for each composite, there are two distinguishable regimes of the noise exponents k (or w) as the threshold is approached. In particular, we note that there are two groups of exponents $k_1(k_{1m},k_1',k_{1m}')$ and $k_2(k_{2m},k_2',k_{2m}')$ as well as $w_1(w_{1m},w_1',w_{1m}')$ and $w_2(w_{2m},w_2',w_{2m}')$. In these systems it is necessary to consider the combined effects of geometric structures, such as

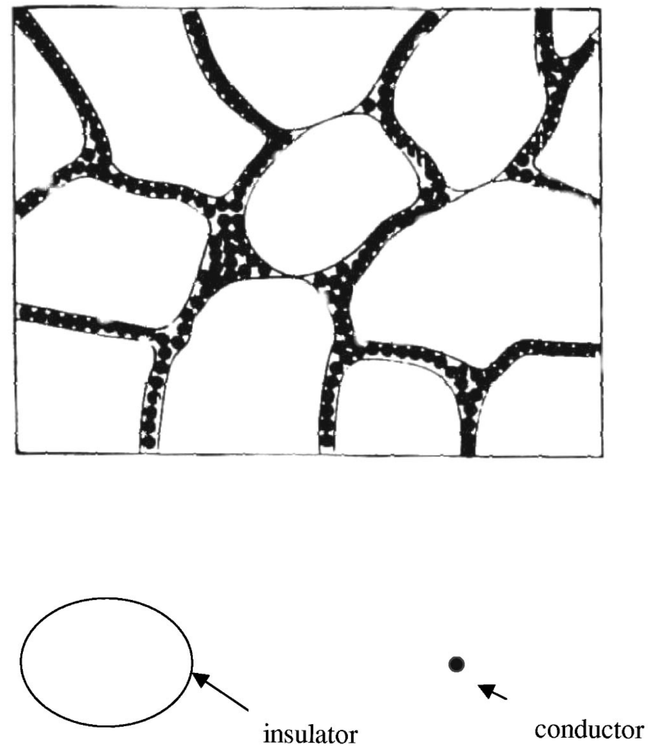

FIG. 10. An illustration of the structure of cellular composites. The small conducting particles are embedded on and between the surfaces of the larger insulator particles.

those upon which the random RV and IRV (Ref. 17,26 and 31) models are based, as well as the intergranular tunneling in the conducting phase.[25] Recall that the geometries of the RV and IRV models lead to a large distribution of resistances, due to a large distribution in the effective electrical geometric contacts in the continuous conducting phase. In the RV or Swiss cheese model, this means that the the resistance of the system is dominated by a series of narrow necks, which connect the more substantial and/or more conducting regions of the conducting phase.

In the structure of the cellular composites, as illustrated in Fig. 10, the small particles cover the larger insulating particles in a manner similar to the water coverage of the grains in sandstone rocks[35] and in composites such as ours[36] the conduction of the sandstone rocks can be described in terms of the RV model[26,31] and the very low ϕ_c (about 1%) observed in the sandstone rocks, is well understood in terms of the excluded volume theory.[35–37] However, there is a very important difference between the sandstones and the cellular composites studied in this paper in that in the sandstones, the conducting phase is continuous, whereas in the cellular systems the powders have, in addition to their RV-like geometric structure, interparticle contact resistances between the grains, which changes or even dominates the conducting behavior. The modeling of the very low ϕ_c observed for these cellular systems in terms of the ratio of the diameter of the insulating to the conducting particles is discussed in Ref. 10.

As ϕ_c is approached from above in granular percolation systems, when the particles size is independent of ϕ as in the present case, the spacing between the conducting grains tends to increase,[25,37] which leads to the lowering of the contact pressure between grains and the formation of tunneling resistors in the system. Considering the LNB model this would also mean that relatively more of the singly connected bonds that form part of the links will be tunneling resistors. Therefore, close to ϕ_c, a limited number of unavoidable non-Ohmic and noisy interparticle contacts will be formed on the current carrying links. Furthermore, very close to ϕ_c the strands on the fisherman's net, which consist of a series of current carrying links and blobs connecting the nodes, are few and separated by $\xi \propto (\phi - \phi_c)^{-\nu}$. Therefore close to ϕ_c, where there are few blobs, the current is forced to flow through an ever increassing number of non-Ohmic and noisy contacts, which then dominate the noise. It is these contacts which give rise to the $m \neq 2$ noise near ϕ_c, which is characterized by the $k_1(k_{1m}, k_1', k_{1m}')$ exponents. As $(\phi - \phi_c)$ increases, the number of nodes and current carrying paths, as well as the number of blobs on them, increases, while the number of non-Ohmic and noisy contacts (per unit length) decreases, so that the LNB structure of the granular powders eventually behave as if it were a continuous conductor. Therefore, one can expect the $k_2(k_{2m}, k_2', k_{2m}')$ exponents to be closer to those predicted for a RV system.

Using the data given in Table II, the evidence that the exponents in region 2 are caused primarily by the RV nature of the microstructure must now be examined. Note that the conductivity exponents *t*, which are valid in regions 1 and 2, are in the range 1.93 to 2.51, which are consistent with both the RV model and the tunneling model, which essentially predict $t > t_{un} = 2$ values.[25,30] The generalized RV model[30] predicts k_2 values larger than the universal 1.56 value, while the tunneling model predicts k_2 values between the universal value (in the case of low resistance contacts[25,37]) and 2.56 (in the case where tunneling contacts dominate[17,25]). The fact then that the k_2, k_{2m}, k_2', and k_{2m}' values as a group fit the RV model much better than the k_1, k_{1m}, k_1', and k_{1m}' exponents supports the suggestion that the RV model is the appropriate one for region 2. However, the most important difference between the predictions of the RV and the tunneling model, is that the value of k_2 should be larger the larger the value of *t*, in the RV model, but constant, independent of *t*, in the tunneling model. The fact that the various k_2 values, as seen in Table II, increase with *t* support the dominance of geometric or RV determined electrical properties in region 2. One should also note that, the fact that the various $t_{n2} = k_{n2}/w_{n2}$ values in Table II are consistent with the measured values of *t* (for GBN, the largest t_{n2} also corresponds to the largest t_{exp}), which is an indication that the mechanism controlling the electrical resistance and the electrical noise are the same. Hence, the behavior of all the systems in region 2 is consistent with the picture of percolation in a geometric or RV dominated electrical system.

The situation in region 1 appears more complex. The fact that the value of *t* is the same as in region 2, in spite of being closer to the percolation threshold, is an indication that the basic conduction mechanism is the same as the one found in region 2, i.e., the geometrical conduction mechanism as described by the RV model. However, the observation of the two *k* values per composite supports a model where the k_1 and k_1' values are not dominated by a geometrical or RV structure. The three most significant facts are as follows. First that there is little or no correlation between the k_1 or k_1' values and *t*. Second that some of the interparticle resistance contacts are non-Ohmic (compare the values of k_1 and k_{1m}). Third that the $k_1(k_{1m}, k_1', k_{1m}')$ values are spread over a much wider range than any of the other percolation exponents determined for the cellular systems[10] (Note that the RV geometry of all four systems being studied in this paper are the same). It can therefore be assumed that a mechanism, other than a "geometrical-RV" conduction mechanism dominates the behavior of the noise in region 1.

The possibility that the noise behavior is dominated by tunneling, which gives rise to a single noise exponent value of 2.56,[17,25] is now examined. The strongest evidence for the importance of this mechanism for the noise here comes from the fact that (except for one) seven values of k_{1m} and k_{1m}', where the noise is normalized by V^m and not V^2, are closer to this single value than the k_1 and k_1' exponents; i.e., the interval of the k_1 and k_1' values shrinks from 0.75–5.23 to 1.27–3.51 for k_{1m} and k_{1m}'. The other observation is that there is little or no correlation between k_1 and *t*, which is in agreement (within the framework of available models) only with the predictions of the "tunneling" model.[17,25] Therefore it can be concluded that very close to ϕ_c, the noise is dominated by tunneling and other non-Ohmic contact resistances, which give rise to the anomalous $k_1(k_{1m}, k_1', k_{1m}')$ exponents. It should also be noted that in region 1 there is not much meaning to the quantity w_1 since the k_1 value is determined by the effect of the mixture of the two conduction mechanisms, while the value of *t* is still determined only by the geometrical-RV contribution to the global resistance. The fact that a single *t* value characterizes all the conductivity results but two *k* values are necessary for the noise is probably due to the fact that as the noise measures the fourth moment of the current distribution and the conductivity the second, the noise is therefore more sensitive to the combination of geometric and non-Ohmic contacts described above.

Since usually the electrical noise power is proportional to the square of the resistance involved, one would generally expect that when the resistance is determined by a given mechanism, the corresponding noise would be determined by the same mechanism. This is not the case when there are more than one mechanism that controls the electrical transport. For example, it was found years ago[38] that for granular metals, in the vicinity of the percolation threshold, the contribution of the tunneling mechanism dominates the noise of the connected metallic network, while the overall electrical conduction is still dominated by the conduction of that network. The physical explanation given then was that the applied bias opens more conduction channels, that, while contributing little to the relatively low overall metallic network resistance, decreases the "resistance" of the relatively large "tunneling resistors," yielding a significant reduction of the relative overall resistance noise. A more recent work on porous silicon,[39] in which the silicon crystallites play the same role as the metal grains in the granular composite, did confirm the same behavior and provided additional evidence to support this picture. We stress in particular, that in that work, it was shown that the hardly noticeable variations in the global electrical resistance of the system, due to the parallel contribution of the tunneling current, were accompanied by relatively large changes in the electrical noise. In fact this expected difference was the motivation for the application of the electrical-noise measurements in that work. In the present work the parameter that is varied is not the applied bias but rather the concentration of the conducting phase, i.e., the microstructure. This yields that, as in the above examples, we have two electrical conduction channels. When adjacent particles touch they can be described as "in contact" while when they separate they can be described as connected by tunneling. It is clear that the more sparse the system the more important the contribution of the latter mechanism to the overall electrical behavior. As in the above systems it appears then that as the percolation threshold is approached, while the global conduction is still dominated by the "contact network," the electrical noise is already controlled by the "tunneling network" of higher resistors. Hence, while this "crossover" is already manifested by the resistance noise it is not manifested yet by the electrical resistance. We expect such a manifestation to be revealed with a further approach to the percolation threshold. In fact the present results yield further support of an independent nature for the mechanisms proposed in Refs. 38 and 39.

In passing we should also note that at present there are no other comprehensive experimental results where either $m \neq 2$ or two noise exponents have been reported, which would support the observations and arguments made in this paper. Nandi *et al.*[13] measured the noise in a carbon-wax composite in both the linear (Ohmic) and nonlinear (non-Ohmic regions or $m \neq 2$) regions but did not report separate k_1 and k_2 exponents. However, Nandi *et al.* did note that their noise results could not be explained by equilibrium resistance fluctuations only. On the other hand, Rudman *et al.*[28] and Pierre *et al.*[29] did observe both w_1^* and w_2^* exponents in Ag + Pt-TFE and copper-polymer composites, respectively, but did not report $m \neq 2$.

The model outlined here of necks, with a LNB structure, but consisting of a granular material with a large range of intergranular contact resistances could also be the reason for the occurance of the very high t values observed in the Fe_3O_4 and NbC cellular media described in the previous paper.[10] The sharp edged granular nature of the Fe_3O_4 and NbC is expected to lead to a larger range of high resistivity contacts and point tunneling junctions than in the carbon black and graphite systems.

VI. CONCLUSIONS

The noise measurements presented in this study appear to be the only work to have fully determined the different noise exponents (k and w) and the related conductivity parameter (t) on the same series of samples. This is borne out by the results shown in Table II, in comparison with the very limited results from some of the most referenced noise articles (Table III). Using the measured values of m to normalize the noise results has a very pronounced effect on the exponents, especially close to the percolation threshold. The very wide range of the k and w exponents measured in the cellular composites show that the links, nodes, and blobs and their interconnects structure and/or the range of interparticle (cluster) conductances in these systems is far more complex than the idealistic RV model and its extension. Finally, we have shown that, in spite of the complexity of the structure of the cellular composites, the critical behavior of their electrical properties can be qualitatively explained by a combination of the theories that exist already for simpler systems. In particular, it appears that the basic electrical features can be understood by considering the microstructure that resembles a random void system, but in which the contribution of the tunneling conduction becomes increasingly important as the percolation threshold is approached.

ACKNOWLEDGMENTS

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