Spin-flip scattering for the electrical property of metallic-nanoparticle thin films

Juh Tzeng Lue, Wen Chu Huang, and Shav Kwen Ma
Department of Physics, National Tsing Hua University, Hsinchu, Taiwan, Republic of China
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Abnormal breaks in the resistance-temperature (R-T) curves for granular thin films containing numerous metallic particles have been observed recently. We propose an extra contribution to the resistivity arising from the spin-flip scattering (the Kondo effect) among the spins of conducting electrons and the local magnetic moments of metallic nanoparticles. Each metallic nanoparticle in a granular thin film behaves like a local spin **S** in analogy to the well localized magnetic moments in dilute alloys. We also find that a log-normal distribution of particle sizes will cause the sharp break in the R-T curve to become broad. With sufficient fitting parameters we can explain the breaks around 120 K.

I. INTRODUCTION

Recently, many theoretical[1-5] and experimental works[6-9] have been devoted to the study of the size effect on the optical properties of small particles. Quantum confinement enlarges the energy-level splitting of electronic states as the particle size is reduced, which causes a blueshift of the absorption spectra. On account of the uncertainty principle, the momentum or the kinetic energy of electrons inside the particle increases as the size reduces. The electron-density distribution may spread outside the particle boundary, causing a decrease of the amplitudes of the electron wave functions inside the particle and reducing the dipole transition matrices. Consequently, a redshift[10,11] may occur for particle sizes less than 4 nm or for nonspherical surface shapes. The exotic optical behavior, depending on the particle size, has prompted many authors to study the physical properties of quantum dots.

The crucial factor that determines the optical and electrical properties of small metallic particles is the filling factor f, which is defined as the volume ratio of the sum of the metallic particles to the total volume of the substrate and particles. For a proper detection of optical absorption, the filling factor is constrained to be smaller than 10^{-3}. In such a diluted concentration, the multiscattering and interaction between adjacent particles can be neglected. According to the Mies scattering,[12] the absorption coefficient α is proportional to the filling factor. Consequently, the transmitted optical intensity written as $I=I_0e^{-\alpha z}$ is a single function of f, and the resonant absorption frequency ω_R is independent of f.

When the filling factor is large enough for the particles to aggregate into clusters, and at least one conducting path is connected from one side of the sample to the other, the sample begins to exhibit current conduction. The resistivity $\rho(R,f,A)$ will be dependent on the particle size R, the filling factor f, and the aggregation factor A which represents the extent to which the particles are connected with each other to those are discrete. Potential barriers usually occur between the boundaries of grains to grains, therefore the conduction electrons subject to grain boundary scattering. If the electron mean free path l within each grain is larger than the grain size D, the Monte Carlo simulation[13] by the random process obtains a semiempirical law for the electrical conductivity as

$$\sigma_g=\frac{ne^2l}{mv_F}\Gamma^{l/D}, \tag{1}$$

where Γ is the transmission probability for the electrons to tunneling through the grain boundary. In each metallic grain, the mean free path l is determined by the electron-phonon scattering, which can be approximated by $l(T)\simeq\alpha T^{-p}$, where p is positive and greater than 1. At certain temperatures, the temperature coefficient of resistivity (TCR), $\partial\rho/\rho\partial T$ can change from negative to positive.

As the filling factor f reduces, the particles may be separated individually. The mechanism of conductivity can be extensively explained by simple tunneling,[14,15] and thermonic emission.[16-18] For the nearest-neighbor hopping between grains, the conductivity is given by[3]

$$\sigma\propto\exp\left[\frac{-\varepsilon}{kT}\right], \tag{2}$$

where ε is the activation energy, which is a function of radius R and spacing between particles. In this case, the resistivity at low temperatures is very high.

Conversely, if the filling factor increases to a large value, the particles are connected continuously by a considerable extent of aggregation, resulting in a positive TCR.[19-22] In this case, the resistivity is smaller than that of particles with negative TCR by several orders of magnitude.[22] Usually the resistivity arising from the electron-phonon interaction can be interpreted by the Bloch-Gruneissen equation.[4] The experimental data on metallic nanoparticle films[4,20,21] can be fitted successfully with an effective Debye temperature Θ_D which is softened from its bulk values in the presence of particles. The effective Debye temperature decreasing as the particle size is reduced is the only trend manifested by the experimental works. Many resistivity measurements indicate anomalous breaks occurring at the lower-temperature end of the linear Bloch Gruneissen plot.

These breaks have also been found in the electrical measurement[23] of C_{54}-$TiSi_2$ films. A sophisticated explanation is the contribution of the Kondo effect resulting from the occurrence of ionized transition-metal atoms.

In this work, we develop a theoretical model that treats each nanoparticle as an impurity magnetic ion in dilute alloys. The electron spins are flip-flopped by local magnetic moments in their paths of conduction. The Pauli exclusion principle expresses the expelling of electrons with the same spins, causing a second-order electron-electron scattering. We can elucidate different types of breaks by applying various Kondo terms to the resistivity using different particle-size distributions.

II. EXPERIMENTAL EVIDENCE OF LOCAL SPIN

As mentioned above, the local magnetic spin scattering is the tacit assumption for the cause of the dip in the electrical measurement. In this work, we have prepared samples with nanometer silver particles embedded in SiO_2 glass by the sol-gel method, and proved the existence of local magnetic moments by a superconducting-quantum-interference-device (SQUID) fluxtometer and an electron-spin-resonance (ESR) spectrometer. The complex solution was prepared by mixing 20 ML of tetraethylorthosilicate $Si(OC_2H_5)_4$, with 20 ML of ethanol, 16 ML of water, and various weights of $AgNO_3$, then adding two drops of HNO_3 as a catalyst. This solution was dispersed by a magnetic stirrer for 4 h and poured into a plastic dish for molding. The solution began to gel for about two days, and then was dried at room temperature for about one week. The dried gels were heat treated in a hydrogen oven at about 400 °C for 30 min. The Ag ions are reduced to nanoparticles.

The particle sizes observed by transmission electron microscopy (TEM) show a log-Gaussian distribution, as shown in Fig. 1. A very delicate detection of the variation of the magnetic susceptibility with temperature by the SQUID fluxtometer reveals an unusual cusp at certain temperatures, as depicted in Fig. 2. This sample can be described as randomly situated spins associated with free-electron hosts, as usually occur in spin-glass systems. The features of spin glasses below a critical temperature are a frozen disordered state. At higher temperatures, the spin polarizations are randomized by the thermal fluctuation. Therefore, the susceptibility of the system exhibits a remarkable cusp at a temperature which is rather sharply defined, suggesting a second-order phase transition among the disordered paramagnetic states characterized by nonvanishing local spontaneous magnetization. Therefore, at temperatures above T_f, the spin-glass system has a spontaneous magnetic susceptibility χ in addition to the paramagnetic background expressed in Curie-Weiss law. On a model of broken ergodicity and replica symmetry breaking, the local susceptibility of spin glass is given by[24]

$$\chi=\beta\left[1-\int_0^1 q(x)dx\right], \tag{3}$$

where $\beta=kT$, J is the exchange integral, and q is the mean-square value of the local magnetization over the distribution, which satisfies a self-consistent equation at equilibrium such as

$$|\theta|-xq(x)-\int_x^1 q(y)dy+q^2(x)=0, \tag{4}$$

where $\theta=(T-T_f)/T_f$. It can be shown that $\int_0^1 q(x)dx \sim |\theta|+|\theta|^2-|\theta|^3+O|\theta|^4$ for T near T_f.

Conventionally, spin glasses typically have a few percent of magnetic impurities, and the Kondo effect seems to be irrelevant for the formation of the spin-glass state. This impurity spin interaction leads to a resistivity dip at a finite temperature. The local magnetic moments can also be detected by an electron-spin-resonance spectrometer, as shown in Fig. 3. The sample with a large value of χ, as shown in Fig. 2, has a larger ESR signal, while the spin concentration in sample (a) is not large enough to yield a significant signal. Similar results have also been

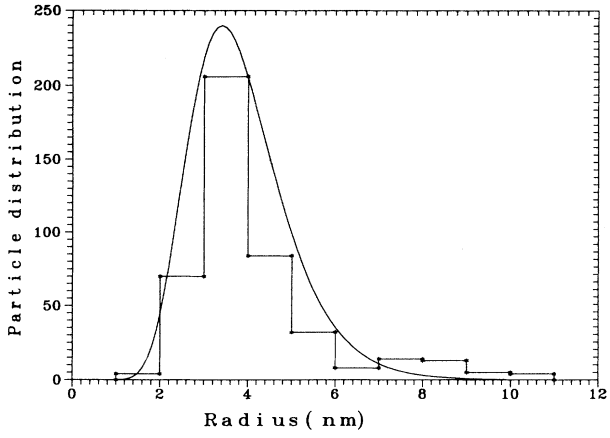

FIG. 1. The particle-size distribution observed by a TEM for the sample containing $AgNO_3=0.1$ g. The smooth curve is the log Gaussian plot.

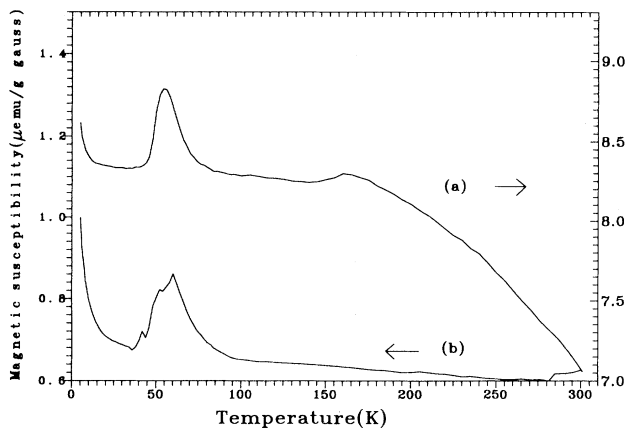

FIG. 2. The ESR measurements at $t=120$ K and $f=9.584\,58$ GHz, for samples with $AgNO_3$ contents of (a) 0.1 g and (b) 0.02 g, respectively.

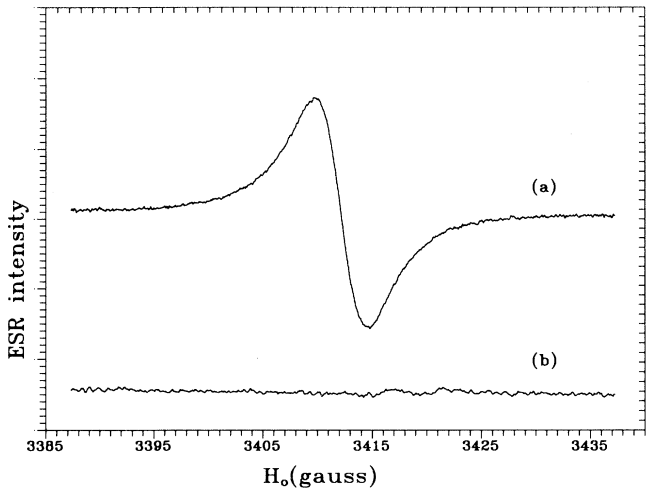

FIG. 3. The SQUID measurements of the susceptibilities of samples with $AgNO_3$ contents of (a) 0.1 g and (b) 0.02 g, respectively.

reported in Refs. 25 and 26, which showed the presence of free spins as the particle sizes were reduced below a critical value. An interesting aspect of the magnetic property is to discriminate particles containing odd numbers from even numbers of free electrons. The statistical mechanical treatment[25,26] is made particularly clear by examining the partition function of an assembly containing metallic particles. The fact that the magnetic susceptibility is associated with an odd parity of electron numbers suggested that we may regard each metallic particle as an impurity ion with spin **S**.[27]

III. THE SPIN-FLIP SCATTERING IN GRANULAR THIN FILMS

Measurement of the electrical conductivity of granular thin films composed of aggregated metallic particles has been investigated by many authors.[4,19–22] The electrical property of these particles can be realized from a clear description of individual particles. The softening of bulk phonons related to small particles is believed to play an important role in the Bloch-Gruneissen equation.[4] In addition, the presence of size effects on the phonon property of small particles has been discussed.[25] Similar to the *s-d* interaction in Kondo's model,[28,29] where the electrons pass through nanoparticles with net spins they are trapped to form a virtual bound state,[30] which causes the spin-flip scattering. After a meticulous derivation of the mobility of conduction electrons, Kondo demonstrated that the resistivity arising from the spin-flip scattering is

$$\rho_{\text{spin}}(T)=\rho_1-\rho_2|\mathbf{J}|\ln(T)\ , \tag{5}$$

where ρ_1 and ρ_2 are independent of temperatures, and **J** is the exchange integral between *s* and *d* electrons. Thermal fluctuations may randomize the spins at temperatures above T_K. Therefore, the resistivity will appear only at temperatures below the critical T_k. Here we emphasize that the exchange integral **J** is negative due to the antiferromagnetic coupling.[30]

The critical temperature T_k plays an important role in the manipulation of the Kondo term $\rho_{\text{spin}}(T)$. At temperatures above T_k, the virtual bound state is destroyed in Kondo's exchange model. In order to exploit the physics of the Kondo temperature T_k, several works[31–34] were devoted to deriving

$$T_k \cong T_0 \exp\left[\frac{-1}{N(0)|\mathbf{J}|}\right]\ , \tag{6}$$

where T_0 is of the order of the Fermi temperature of the conduction electrons and $N(0)$ is the density of bond states near the Fermi surface. The expectation value of T_k has been analyzed by Kondo and many authors.[31–35] From the above expression of T_k, we see that T_k is sensitively dependent on $N(0)$ and **J**. Schrieffer[31] has shown that T_k spans a wide range from millidegrees to the melting point of metals. This wide range is mainly due to different correlations of the conduction electrons and the localized magnetic moments, i.e., to different values of the exchange integral **J**. The exchange integral, as derived by Schrieffer, is

$$\mathbf{J}\propto -\frac{(\mathbf{V}_{kd})(\mathbf{V}_{kd}^{*})}{2\mathbf{S}\epsilon_d}<\mathbf{0}\ , \tag{7}$$

where ϵ_d is the impurity *d*-level energy with respect to the Fermi energy, $\mathbf{V}_{kd}$ is the matrix element mixing the conduction elements (k) and the localized-orbital states (d), and **S** is the spin of the localized magnetic moments. For smaller particles, the conduction electrons are scattered by the effective magnetic moment **S**, yielding a large value of $\mathbf{V}_{kd}$. Consequently, from Eq. (6) it is obvious that the critical temperature T_k decreases as the particle size is reduced. The relation between T_k and R is tacitly assumed as

$$T_k \approx T_{k0}e^{-R_0/R}\ , \tag{8}$$

where T_{k0} is for the bulk value and R_0 is a constant. The critical temperature approaches zero as $R\rightarrow 0$, while $T_k\rightarrow T_{k0}$ as the particle size approaches the dimension of bulk solid.

Now we presume the particle sizes to be distributed in log-Gaussian form, as shown in Fig. 1. In terms of the peak value $\bar{R}$ and the width σ, the probability of finding a particle of radius between R and $R+dR$ is

$$P(R)=\frac{1}{\sqrt{2\pi}}\frac{1}{\ln\sigma}\frac{1}{R}\exp\left\{-\left[\frac{\ln(R/\bar{R})}{\sqrt{2}(\ln\sigma)}\right]^2\right\}. \tag{9}$$

As discussed previously, the smaller the particle size, the lower the critical temperature T_k will appear. At temperatures below the critical value T_k, the scattering of the conduction electrons by the localized moments begins to contribute to the resistivity $\rho_{\text{spin}}(T)$. On the basis of the relationship between T_k and R, we can make a Taylor expansion of T_k in Eq. (6). A statistical function $f(T_k)$ is introduced to employ the distribution of T_k due

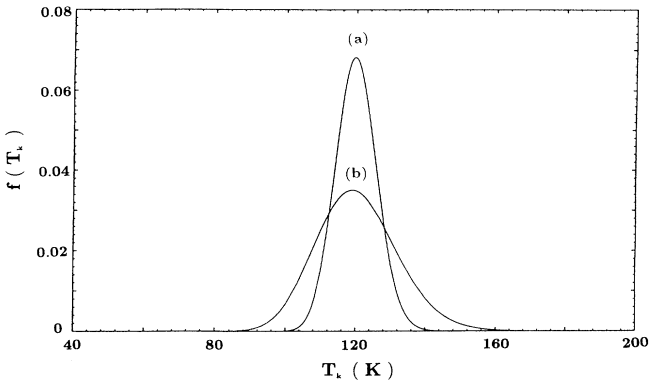

FIG. 4. The temperature dependence of the fractional function $f(T_k)$ for two different particle-size distributions with (a) $\bar{T}_k=120$ K, $\sigma_k=1.05$ K and (b) $\bar{T}_k=120$ K, $\sigma_k=1.1$ K, respectively.

to different particle sizes and presumed to be the same form as Eq. (9), which is

$$f(T_k)=\frac{1}{\sqrt{2\pi}}\frac{1}{\ln\sigma_k}\frac{1}{T_k}\exp\left\{-\left[\frac{\ln(T_k/\bar{T}_k)}{\sqrt{2}(\ln\sigma_k)}\right]^2\right\} . \quad (10)$$

Here $\bar{T}_k$ and σ_k are analogous to $\bar{R}$ and σ in the log-normal particle-size distribution. The function $f(T_k)$ is defined as the fraction of particles which causes the spin-flip scattering for the particles with its Kondo temperature T_k above the measuring points T. Therefore, the total contributed fraction $F(T)$ is given by

$$F(T)=\int_T^\infty f(T_k)dT_k . \quad (11)$$

Figures 4 and 5 illustrate the temperature dependence of $f(T_k)$ and $F(T)$ for two different particle-size distributions. It is clear from these two figures that the wider the distribution of particle size, the broader the break of R-T curve. We also note that the total fraction $F(T)$ is quite similar to the Fermi-Dirac distribution, though their physical origins are absolutely different.

IV. RESULT AND DISCUSSION

In order to analyze the behavior of $\rho(T)$ of aggregated metallic particles, Fujita, Ohshima, and Kuroishi[4] utilized the Bloch-Gruneissen equation for bulk metals to fit the experimental data in terms of the parameter Θ_D. From the evidence of electron micrographs, Roy and Chakravory[21] pointed out that the metallic conduction comes from the aggregation of small particles. For bulk metals, the resistivity interpreted by the Bloch-Gruneissen equation based on the scattering of the conduction electrons and phonons is[4]

$$\rho_L(T)=\frac{C}{\Theta_D}\left[\frac{T}{\Theta_D}\right]^5\int_0^{\Theta_D/T}\frac{z^5dz}{(e^z-1)(1-e^{-z})} , \quad (12)$$

where Θ_D is the effective Debye temperature for aggregated particles, and C is a constant. It is found that Θ_D decreases as the particle size is reduced.

The total resistivity $\rho_{\text{total}}(T)$ can be the sum of the residual resistivity ρ_0 which is due to defect and impurity scattering, the $\rho_L(T)$ and $\rho_{\text{spin}}(T)$ as given by

$$\begin{aligned}\rho_{\text{total}}(T)&=\rho_0+\rho_L(T)+\rho_{\text{spin}}(T)\\&=\rho_0+\rho_L(T)+\int_T^\infty\rho_1 f(T_k)dT_k\\&\quad-\int_T^\infty\rho_2|\mathbf{J}|\ln(T)f(T_k)dT_k .\end{aligned} \quad (13)$$

Here we neglect the size dependence of $\mathbf{J}$. The theoretical results are illustrated in Figs. 6–8. As indicted in the figure captions, the fitting parameters including ρ_1, ρ_2, $|\mathbf{J}|$, $\bar{T}_k$, σ_k, Θ_D, ρ_0, and $\rho_\square$ (the sheet resistivity) are declared.

The experimental data as shown in Figs. 6–8 for silver

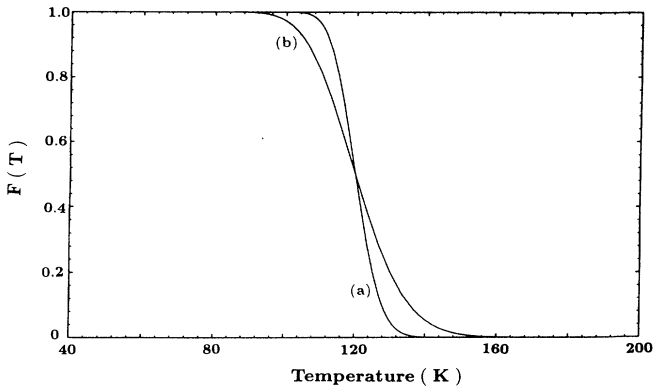

FIG. 5. The temperature dependence of the total fractional function $F(T)$ for two different particle-size distributions, with (a) $\bar{T}_k=120$ K, $\sigma_k=1.05$ K, and (b) $\bar{T}_k=120$ K, $\sigma_k=1.1$ K, respectively.

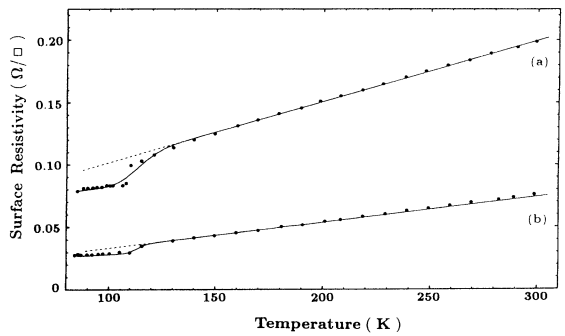

FIG. 6. The experimental resistivity for films containing Ag nanoparticles (Ref. 21) with (a) $\bar{R}=4.35$ nm, $\sigma=0.6$ nm and (b) $\bar{R}=5.7$ nm, $\sigma=0.6$ nm. This simulation has fitting parameters for (a) $\rho_1=0.1$ Ω/□, $\rho_2|\mathbf{J}|=0.026$ Ω/□, $\bar{T}_k=113$ K, $\sigma_k=1.072$ K, $\Theta_D=163$ K, and $\rho_0=0.06$ Ω/□, and (b) $\rho_1=0.05$ Ω/□, $\rho_2|\mathbf{J}|=0.012$ Ω/□, $\bar{T}_k=113$ K, $\sigma_k=1.035$ K, $\Theta_D=192$ K, and $\rho_0=0.01$ Ω/□.

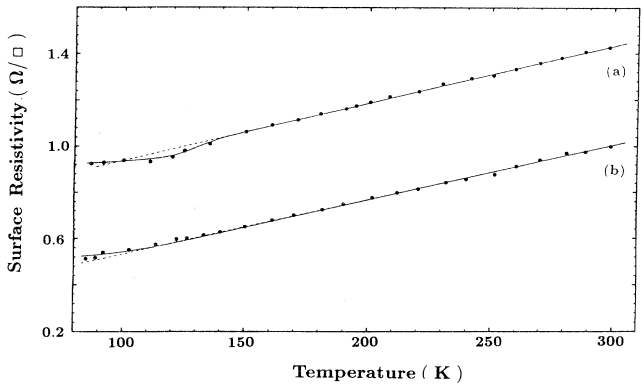

FIG. 7. The experimental resistivity for films containing Ag nanoparticles (Ref. 21) with (a) $\bar{R}=2.35$ nm, $\sigma=0.75$ nm and (b) $\bar{R}=2.5$ nm, $\sigma=0.7$ nm. This simulation has fitting parameters for (a) $\rho_1=0.78\ \Omega/\Box$, $\rho_2|\mathbf{J}|=0.17\ \Omega/\Box$, $\bar{T}_k=127$ K, $\sigma_k=1.0578$ K, $\Theta_D=103$ K, and $\rho_0=0.71\ \Omega/\Box$, and (b) $\rho_1=0.57\ \Omega/\Box$, $\rho_2|\mathbf{J}|=0.12\ \Omega/\Box$, $\bar{T}_k=104$ K, $\sigma_k=1.35$ K, $\Theta_D=105$ K, and $\rho_0=0.34\ \Omega/\Box$.

nanoparticle films are almost linear in most of the temperature range which can be fitted successfully by the Bloch-Gruneissen equation, though not by the grain-boundary scattering and hopping conductivity as addressed in Eqs. (1) and (2), inferring a high aggregation of particles in these samples. Both the ρ_1 and $\rho_2|\mathbf{J}|\ln(T)$ contribute to the spin-flip scattering. The constant term ρ_1 behaves like a step function and is independent of temperatures. The divergence of the $\ln T(T)$ term when T approaches zero has been removed by several authors.[30,35] According to our model, the addition of the Kondo term $\rho_{\text{spin}}(T)$ by a narrow distribution of particle size will cause a sudden change of the slope around $\bar{T}_k$.

One may suspect whether only one set or many possibilities of the fitting parameters $(\rho_1,\rho_2|\mathbf{J}|,\bar{T}_k,\sigma_k)$ are available to yield correct results with the experimental data. However, due to the independence of ρ_1 on the temperature, and to the fact that only $\rho_2|\mathbf{J}|$ can vary with the temperature, the values of $(\rho_1,\rho_2|\mathbf{J}|,\bar{T}_k,\sigma_k)$ are determined uniquely. An interesting question is why granular thin films containing smaller particles exhibit more apparent break, as shown in Figs. 6–8. We suggest that metallic nanoparticles of larger radius are much more likely to link together to form clusters which tend to pair off odd spins. The values of ρ_1 and $\rho_2|\mathbf{J}|$ increase as the particle size is reduced, as can be seen in these figures. However, this is not always a trend when comparing the experimental results, as shown in Figs. 7 and 8. This exception can elucidate the important fact that the assembly containing numerous sizes of metallic particles is somewhat difficult to analyze by means of detailed variables and simple physical mechanisms. Here we emphasize that the behavior of $\rho(T)$ for granular thin films is closely related to the condition of aggregation when it shows positive TCR.

The size dependence on the resistivity can also be examined from the viewpoint of fractal dimensions as employed by Roy and Chafravory.[21] The fractal dimension D of granular thin films is given by

$$N(L)\propto L^{D}\ , \tag{14}$$

where $N(L)$ is the number of metallic particles which are in a square with side L. We also introduce another variable P which is defined as the probability of filling the plane of the granular thin film resulting from the point of view of percolation. The metallic conduction is proportional to the percolation probability $\theta(p)$, owing to the possibility that electrons can pass through from one side of the film to the other by a connection of the clusters which is given by[36]

$$\theta(P)\propto(P-P_c)^{\beta}\ , \tag{15}$$

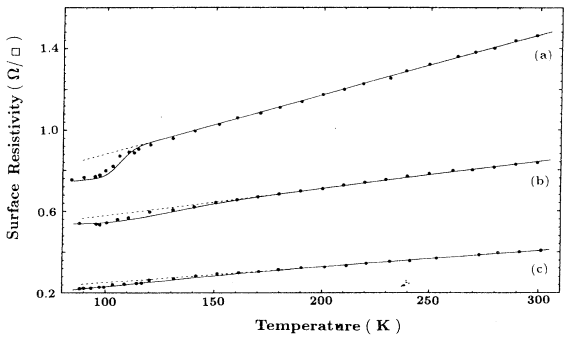

FIG. 8. The experimental resistivity for films containing Ag nanoparticles (Ref. 21) with (a) $\bar{R}=2.25$ nm, $\sigma=0.7$ nm, (b) $\bar{R}=2.85$ nm, $\sigma=0.65$ nm, and (c) $\bar{R}=3.35$ nm, $\sigma=0.65$ nm. This simulation has fitting parameters for (a) $\rho_1=0.62\ \Omega/\Box$, $\rho_2|\mathbf{J}|=0.16\ \Omega/\Box$, $\bar{T}_k=106.2$ K, $\sigma_k=1.05$ K, $\Theta_D=98$ K, and $\rho_0=0.61\ \Omega/\Box$, (b) $\rho_1=0.51\ \Omega/\Box$, $\rho_2|\mathbf{J}|=0.12\ \Omega/\Box$, $\bar{T}_k=118$ K, $\sigma_k=1.2$ K, $\Theta_D=126$ K, and $\rho_0=0.45\ \Omega/\Box$, and (c) $\rho_1=0.028\ \Omega/\Box$, $\rho_2|\mathbf{J}|=0.0128\ \Omega/\Box$, $\bar{T}_k=114$ K, $\sigma_k=1.35$ K, $\Theta_D=154$ K, and $\rho_0=0.18\ \Omega/\Box$.

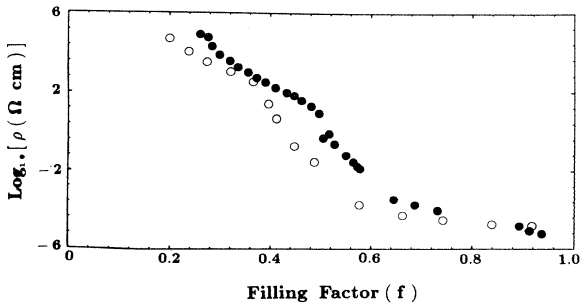

FIG. 9. The electrical resistivity (Ref. 23) of granular thin films as a function of the filling factor f measured at 300 K for containing Ag (closed circles) and Au (open circles) nanoparticles, respectively.

where P_c is the critical probability above which the thin film exhibits metallic conduction, and β is a constant depending on the property of the film. Evidently the electrical resistivity of the granular thin film is

$$\rho \propto (P - P_c)^{-\beta} . \tag{16}$$

Comparing the experimental data shown in Fig. 9, we see that the resistivity decreases as the fitting factor increases, which is consistent with Eq. (16). In fact, we declare that the same behavior of resistivity can be obtained when the probability P is replaced by the fractal dimension D.

V. CONCLUSION

The positive TCR of the R-T curves has been found to be a general electrical property of granular thin films containing numerous aggregated metallic particles. The metallic particles are believed to link together to constitute the path of conduction from the evidence of electron micrographs. The apparent break of the R-T curve around 120 K is presumed to be due to the spin-flip scattering when the number of metallic particles with odd spins is large enough to exhibit local magnetic moments. The Kondo temperature may span from millidegrees to the melting point of metals. In addition to the viewpoint of the fractal dimensions, we can satisfactorily fit the R-T curves by a proper choice of the parameters (ρ_1, $\rho_2|\mathbf{J}|$, $\overline{T}_k$, and σ_k). We conclude that the Kondo term $\rho_{\text{spin}}(T)$ can be employed successfully to investigate the electrical property of granular thin films, and provide a perspective and understanding of the mechanism of conduction between aggregated metallic particles.

ACKNOWLEDGMENT

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