Cluster glasses made from copper-nickel alloys doped with 3*d*-elements

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Spin glass effects are observed in copper-nickel alloys containing *d*-element impurities. Two phase transitions are found near the "percolation" region in copper-nickel and copper-nickel-iron alloys: a ferromagnetic→paramagnetic transition, and a transition to a mictomagnetic state. A magnetic phase diagram is proposed for copper-nickel-iron alloys in a zero magnetic field. The cluster glass and mictomagnetic states are explained both in the context of the classical Néel theory and in the Sula-Nakaoka approximation for the Kondo effect.

INTRODUCTION

Mictomagnetic alloys, or cluster glasses, are interesting because they can exist in a variety of magnetic states characterized by short- and long-range magnetic ordering (e.g., para-, ferro-, antiferromagnetic states, spin glass, antiasperomagnetic, and asperomagnetic states, etc.). Although the localized spin impurities may be regarded as isolated in ordinary spin glass systems consisting of a nonmagnetic matrix and a magnetic impurity,[1] giant magnetic clusters are present in mictomagnets.

Claus and Tranchita[2] first discovered the spin glass effect in paramagnetic copper-nickel alloys ($c_{\mathrm{Ni}} = 44.8$ at.%). Two or more phase transitions associated with the existence of different magnetic phases have been observed in numerous mictomagnetic substances, e.g., Au-Fe, Cr-Fe, Fe-Al, Pd-Mn (Ref. 3). The existence of multiple phase transitions in copper-nickel alloys has not been demonstrated directly. However, Carnegie and Tranchita have suggested[4] that binary phase transitions may occur in Cu-Ni containing 45.7 at.% Ni. The existence of binary phase transitions in mictomagnets has been shown theoretically for the two- and three-dimensional Ising models, as well as in the frustration model for predominantly ferromagnetic bonds.[5–7]

Recent experiments have shown that transitions of the spin glass—ferromagnetic—paramagnetic type occur in paramagnetic Cu-Ni-Fe alloys in the "supercritical" region.[8]

Thus percolation systems and paramagnetic copper nickel alloys lightly doped with *d*-elements are both of interest with regard to existence of spin glass states and multiple phase transitions.

EXPERIMENTAL METHOD

We studied copper-nickel alloys containing from 40 to 51.2 at.% Ni with Fe, Co, and Mn impurities ranging from 1 to 2.5 at.%. The mixtures were smelted from metals of purity better than 0.997 in an induction vacuum oven.

The electric resistance of the wire specimens of diameter 0.2 mm was measured accurate to 0.1% by the four-point method. The "Solenoid" apparatus at the Institute of General Physics, Academy of Sciences of the USSR, was used to measure the magnetization and the magnetic resistance in strong magnetic fields to $B = 13$ T at the boiling point of liquid helium. The relative error in measuring the magnetic resistance was less than 3%. We used a balanced transformer to study the magnetic susceptibility of wires 10 mm long and 2.2–3.0 in diameter in ac magnetic fields of frequency $38 \leqslant f \leqslant 1200$ Hz and amplitude $0.1 \leqslant H \leqslant 5$ G; the error was less than 3%. The temperature of the wires was recorded by a copper/copper + iron thermocouple with an absolute error of ~0.1 deg.

RESULTS AND DISCUSSION

A. Paramagnetic region

Clustering effects start to show up in copper-nickel alloys for nickel concentrations Ni ~ 22–25 at.% (Refs. 9, 10). The average magnetic moment $\langle \mu \rangle$ of a cluster reaches $\sim 5\mu_B$ in a Cu-Ni alloy containing 40 at.% Ni, and the cluster concentration is $\sim 1.8 \cdot 10^{-3}$ per atom.[11] Anomalous peaks in the magnetic heat capacity C^H are observed in this alloy for temperatures ~4–6 K and magnetic fields $B \sim 1$–4.2 T, which suggests that a transition to a spin glass state occurs.[11] The clusters in Cu-Ni alloys apparently become "frozen" near the critical Ni concentration for onset of ferromagnetism ($c_{\mathrm{Ni}} \sim 44$ at.%). The differential magnetic susceptibility χ_d has characteristic maxima as a function of temperature.[3] However, the situation differs for Cu-Ni alloys ($c_{\mathrm{Ni}} \sim 40$ at.%) which contain Fe, Co, or Mn impurities. This is clear from Fig. 1, which clearly shows that the ac susceptibility χ_ω has distinct peaks at characteristic "freezing" temperatures T_f in the spin glass even for $c_{\mathrm{Ni}} \approx 40$ at.%.

The shift in T_f is not proportional to the concentration of the magnetic impurity in Fe- and Co-doped Cu-Ni alloys, probably because the Fe or Co interact with the nickel matrix. The "freezing" temperatures in Mn-doped alloys are approximately the same as T_f for Cu-Mn alloys with the same Mn concentration. If a weak longitudinal $H \lesssim 60$ Oe is applied, χ_ω decreases and T_f shifts toward lower temperatures. The magnetic anisotropy field H_a was estimated as

$$[T_f^{(H)}/T_f^{(0)}]^{1/2} = 1 - H/H_a, \tag{1}$$

in Refs. 12, 13 using the Néel theory. Here $T_f^{(H)}$ and $T_f^{(0)}$ are the "freezing" temperatures for the alloy in external magnetic fields $H = H$ and $H = 0$, respectively. Estimate (1) leads to $H_a \sim 150$–400 Oe. The relative falloff in the maxi-

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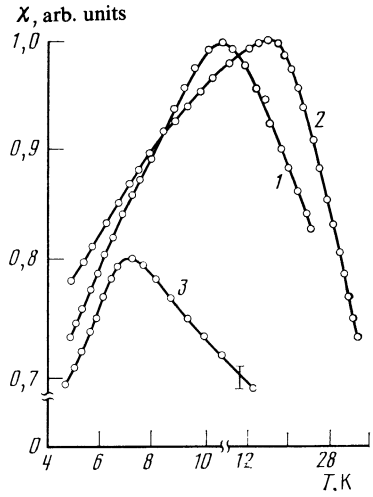

FIG. 1. Temperature dependence of the magnetic susceptibility for the following alloys: 1) Cu + 40 at.% Ni + 1 at.% Fe; 2) Cu + 40 at.% Ni + 1.1 at% Co; 3) Cu + 40 at.% Ni + 1 at.% Mn.

mum χ_ω in weak magnetic fields $H \lesssim H_a/2$ is proportional to H.

The molecular field approximation (MFA) was used in Ref. 14 to analyze a Heisenberg spin glass in an external magnetic field, and the drop in χ_ω was found to be proportional to the magnetic field strength:

$$\Delta\chi/\chi_0 \infty q(s,h)+o(h^2), \quad q(s,h)\infty h; \qquad h=\mu_B g H/kT_f^{(0)}. \tag{2}$$

Here $q(s,h)$ is the Edwards-Anderson parameter in the magnetic field and s is the spin of the impurity.

We note that similar behavior occurs in the ordinary spin glass Cu + 8.1 at.% Mn for fields $H \leqslant 1$ kOe ($H_a = 30$ kOe).[15] In all paramagnetic Cu-Ni alloys doped with 3d-elements, the residual magnetization is found to rise abruptly for $T \lesssim T_f$ and to depend both on the time and on the thermomagnetic treatment.[16] Although the suppression of the anomalous peaks χ_ω and the drop in T_f are readily explained by the Néel and Sherrington-Kirkpatrick theories,[12,17] the calculations for the effects of the thermomagnetic treatment do not give satisfactory agreement with experiment. For an Ising spin glass in the free energy surface approximation, the maximum magnetization $M(T)$ occurs at higher T when H increases, which contradicts the experimental results.[18]

The anomalously high magnetic viscosity is probably a consequence of the degeneracy and nonergodicity of the spin glasses.[19] Figure 2 plots the time-dependence of the residual magnetization for the alloys Cu + 40 at.% Ni + 1.1 at.% Co and Cu + 40 at.% Ni + 2 at.% Mn. It is clear that for $t \gtrsim 1$ min the residual magnetization $J_r(t)$ depends linearly on the logarithm of the time:

$$J_r(t) = J_r(0) - S \ln t, \tag{3}$$

where the factor S depends on H and on the temperature and composition of the alloy. We note that similar dependences are found for the classical spin glasses Au-Fe, Cu-Mn and in γ-Fe-Ni-Cr alloys.[20–22] We observed a similar dependence in

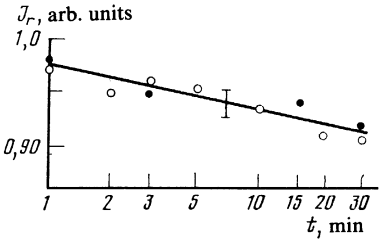

FIG. 2. Relaxation of the residual magnetization. The open and dark circles are for the alloys Cu + 40 at.% Ni + 1.1 at.% Co and Cu + 40 at.% Ni + 2 at.% Mn, respectively.

a copper cluster glass containing 0.3 at% Fe. The Néel theory accounts readily for the linear dependence of J_r on $\ln(t)$. Indeed, according to Ref. 12 the equation for the ith superparamagnetic cluster (SPC) has the form

$$M_i V_i (H_a \pm H)^2 / H_a = 2kT \ln c_i t, \tag{4}$$

where M_i is the magnetic moment per unit volume, V_i is the volume of the SPC, H_a is the effective anisotropy field, $c_i \sim 10^{-12}\ \mathrm{s}^{-1}$, and t is the time of measurement. If the left-hand side of (4) is less than the right-hand side, the magnetic moments of the SPC cluster precess in the effective magnetic field, i.e., we observe superparamagnetism in which the total residual magnetization of the SPC ensemble depends logarithmically on t. We estimated the relaxation time τ_{rel} from the dependence $J_r(t)$ and found that it is typically a few days for Cu-Ni alloys containing small amounts of d-element impurities; this is much longer than for ordinary spin glasses and is comparable to τ_{rel} for mictomagnets.[23–25]

For temperatures above the "freezing" point ($T > T_f$), properties of the ferromagnetic state persist along with the paramagnetic properties [the characteristic form of the curve $J^2 = f(H/J)$]; this is particularly true for the alloys Cu + 40 at.% Ni + (Fe, Co). The residual magnetization drops abruptly for $T \approx T_f$ but vanishes only for $T = 60$–70 K. The curves for $J_r(t)$ in the insert to Fig. 3 have two breakpoints, one at $T_1 = T_f$ and one for $T_2 \approx \theta_c$, where θ_c is the "implicit" Curie point of the alloys. We note that for supercritical Cu-Ni alloys doped lightly with Mn to 1-2 at.%, there are no sudden changes in slope in the curve $J_r(T)$ for $T > T_f$. The temperature derivative of the electric resistance has a jump discontinuity for $T \approx T_f$ for all of the alloys investigated. The transverse magnetoresistance for the alloys Cu + 40 at.% Ni + (Fe, Co, Mo) is negative and reaches 1.5–2% for $T - 4.2$ K and $B = 13$ T.

B. "Percolation" region

Copper-nickel alloys become Stoner spin glasses for concentrations $c_{\mathrm{Ni}} \sim 47$ at.% in the "percolation" region. We thus expect that they will exhibit the mixing of the mictomagnetic and spin glass states and the multiple phase transitions which are characteristic for such systems.[2,17,26]

According to the molecular field approximation (MFA),[27] the condition for a Stoner spin glass to exist is given by

$$U^2 + cJ_{sw}^2 \sum_{i \neq j} [\chi^{(0)}(\mathbf{r}_i - \mathbf{r}_j)]^2 > (2\pi\Delta)^2, \tag{5}$$

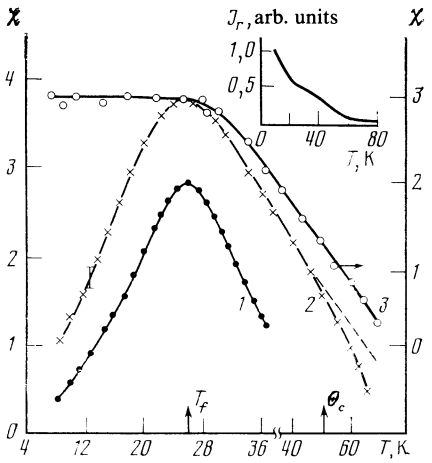

FIG. 3. Temperature dependence of the magnetic susceptibility χ (in arbitrary units) for Cu + 51.2 at.% Ni annealed at 650 °C for 1 h, followed by annealing for 4 h at 400 °C: 1) in an ac magnetic field, $H = 2$ Oe, $f = 120$ Hz; 2) $H = 9$ Oe, $f = 120$ Hz; 3) in a dc magnetic field with $H = 4.8$ Oe. The insert shows the temperature dependence of the residual magnetization for Cu + 51.2 at.% Ni.

where U is the Coulomb interaction potential, J_{SW} is the Sherrington-Wolf exchange integral, and Δ is the width of the resonance level in the Friedel model. We note that this condition assumes that the spin glass exists when indirect exchange occurs even if the Anderson condition $U > 2\pi\Delta$ for localized moments is violated. According to the modified MFA in Ref. 27, the critical impurity concentration below which the spin glass phase is forbidden is given by $c \gtrsim c_{cr} \approx (T_K^{(0)}/T_f^{(0)})^2$, where $T_K^{(0)}$ is the Kondo temperature for noninteracting impurities. The MFA theory assumes that the magnetic states are mixed in the mictomagnetic region ($c \sim c_{cr}$). In particular, paramagnetic-mictomagnetic-spin glass phase transitions should be possible.

The supercritical copper-nickel alloys we studied have qualitatively similar properties; we will consider the representative alloy Cu + 51.2 at.% Ni. This alloy has a weakly defined Curie point $\theta_c \sim 50$–60 K, which we found by measuring the magnetization and the electric resistance. The Arrott construction indicates that the alloy becomes paramagnetic at $\theta_c \approx 54$ K; measurements of the longitudinal magnetic field give $\theta_c \sim 55$–60 K for the implicit Curie point. The transition points θ_c can also be identified from the abrupt departure of the curve $\rho_H(T)$ from linearity for $T \approx \theta_c$. Studies of the magnetic susceptibility for ac currents ($f = 38$ Hz, $B = 2$ G) revealed a maximum at the temperature $T_f \sim 26$–27 K. As in the "subcritical" alloys of composition Cu + 40 at.% Ni + (Fe, Co, Mn), if an increasing external magnetic field is applied the peak $\chi(T_f)$ shifts toward lower temperatures and nearly vanishes for $H \approx 50$–60 Oe. Preliminary thermomagnetic treatment (cooling in a magnetic field $H = 8$–10 Oe eliminates the peak in the magnetic susceptibility; the absence of such a peak is of the characteristic features of the spin glass state. As in the paramagnetic alloys Cu + 40 at.% Ni + (Fe, Co, Mn), no peaks in χ_ω are observed for $T > T_f$ in "supercritical" copper-nickel alloys; the anomalous behavior of the residual magnetization and the magnetization in weak magnetic fields [the breakpoints in $J_r(T)$ and the jump in dM/dT] is evident near the transition temperature θ_c. The vanishing of the derivative $d\chi/dT$ for $T < T_f$ and $H < 10$ Oe (weak magnetic field) is a typical feature of ferromagnetic materials (cf. Fig. 3), and χ_ω can be as large as $1/N$, where N is the demagnetizing factor. Supercritical copper-nickel alloys are thus in a nonordered ferromagnetic (NF) state at temperatures below the "freezing" point of the spin glass.

We note that for compositions in the percolation region, the position, magnitude, and sharpness of the peak in χ_ω for copper-nickel alloys depend strongly on how the alloy is heat-treated. Thus, the maximum in χ_ω for Cu + 51.2 at.% Ni shifts by 15 K toward higher temperatures if the annealing temperature t_{ann} is decreased from 650 to 450 °C and the sample is annealed for 1 h. If t_{ann} drops to 350 °C, the peak in χ_ω lies another 10 K higher than is the case for $t_{ann} = 450$ °C, and the peak value itself is 4% lower. Moreover, the peak in χ_ω becomes less sharp an t_{ann} decreases. Carnegie *et al.* noted previously[3] that the form of the curve $\chi_\omega(T_f)$ is less sensitive to the heat treatment in subcritical copper-nickel alloys (44.8 at.% Ni, 46 at.% Ni).

The primary effect of adding iron to the copper-nickel matrix ($c_{Fe} \sim 1$–2 at.%, $c_{Ni} \sim 45$–49 at.%) is to enhance the ferromagnetism of the copper-nickel-iron alloy (χ increases to a few tenths); by contrast, χ is only $\sim 10^{-3}$–10^{-2} in binary Cu-Ni alloys or subcritical Cu-Ni alloys doped with iron, manganese, and cobalt. The "freezing" points are lower for copper-nickel-iron alloys with $c_{Ni} > 45$ at.% and $c_{Fe} \sim 1$–2 at.%, and the Curie point is higher than for the weakly ferromagnetic alloy Cu + 51.2 at.% Ni, cf. Table I. The behavior of the magnetization M of the alloys is typical for weak ferromagnets,[28] and the weak ferromagnetism persists for $T \lesssim 160$ K, which is higher than the "Curie point" θ_c. Our study of the magnetization for copper-nickel-iron alloys in weak magnetic fields $H \lesssim 5$ Oe in the "percolation" region revealed that a ferromagnetic phase exists below the temperature for transition to the spin glass state—the $\chi(T)$ dependence has a plateau. The ac susceptibility χ_ω has a sharp peak for $T \sim T_f$. The temperature $T \approx \theta_c$, for which a sharp drop in χ_ω is observed on the magnetization curves, coincides with the points at which the temperature derivatives of the electric and magnetoresistance decrease abruptly.

The peaks in χ at the "freezing" points T_f of the spin glass and at the implicit "Curie points" θ_c (Fig. 4) demonstrate directly that two phase transitions occur in copper-nickel-iron alloys in the percolation region. Although T_f drops as the external field H increases, θ_c increases. Apparently, the ordering of the magnetic clusters in the external magnetic field suppresses the spin glass and stabilizes the NF states (according to our estimates, $c_{cluster} \sim (1$–$3) \cdot 10^{-3}$ per atom); in addition, the peaks at T_f and θ_c become lower.

The two peaks in $\chi(T)$ for copper-nickel-iron alloys at the characteristic transition temperatures T_f and θ_c, the results in Ref. 29 on the depletion of the Cu-Ni matrix when iron is added, the temperatures and field dependences of the magnetization M in weak ($H \lesssim 1$ kOe) and strong magnetic fields (1 kOe $< H <$ 13 kOe), and the values of χ in the alloys

TABLE I. Characteristic "transition" temperatures in copper-nickel and Cu-Ni–(Fe, Mn, Co) alloys.

Alloy composition, at.%.	T_f, K	Θ_c, K	Alloy composition, at.%.	T_f, K	Θ_c, K
Cu+40Ni+1Mn	7	–	Cu+51,2Ni	27	56
Cu+40Ni+1,1Co	14	–	Cu+48,6Ni+1Fe	16	69
Cu+40Ni+1Fe	9	–	Cu+46Ni+1,4Fe	13	72
Cu+46Ni	10	11	Cu+45,1Ni+2,4Fe	11	85

all suggest the qualitative form shown in Fig. 5 for the magnetic phase diagram $T_t(c_{\mathrm{Ni}})$ for Cu-Ni-Fe alloys containing 40–49 at.% Ni and ~1–2.5 at.% Fe. Here, as in the magnetic phase diagram proposed by Carnegie *et al.* in Ref. 3, T_t is the phase transition temperature. The dashed curve in Fig. 5 arbitrarily separates the spin glass (SG) region from the mixed spin glass + ferromagnetic (SG + NF) state (the SG + NF state might possibly be an asperomagnetic state).

A similar sequence of phase transitions is observed in alloys with a mixed exchange interaction. For instance, transitions occur in Ni-Mn alloys from the spin glass state to a paramagnetic phase or to a state with an asperomagnetic structure;[30] alternatively, a phase transition from spin glass to an antiferromagnetic state may occur, as is observed for Fe-Pt alloys.[31] We see from the nature of the phase diagrams that if c_{Ni} is less than a certain value, the phase transitions at the points T_f and θ_c cannot be temperature-resolved, either for the Cu-Ni (Fig. 5a) or for the Cu-Ni-Fe alloys [this is true, e.g., for the alloys Cu + 46 at.% Ni and Cu + 40 at.% Ni + 1 at.% Fe]. The two phase transitions start to become resolvable for $c \sim c_{\mathrm{cr}}$; moreover, T_f for the Cu-Ni alloys may be higher than for Cu-Ni-Fe (cf. the data in Table I for the alloys Cu + 51.2 at.% Ni and Cu + 48.6 at.% + 1 at.% Fe), and θ_c for the matrix is lower. We note that if more iron is added, the spin glass state in the Ni-Cu-Fe alloys is eventually suppressed.

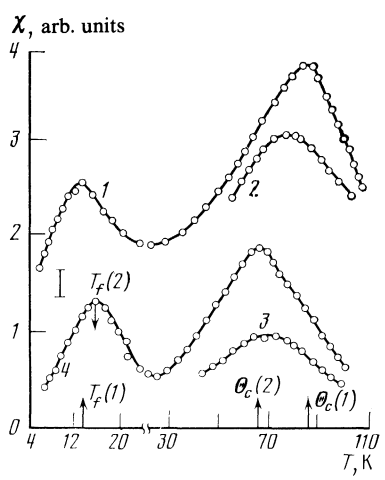

FIG. 4. Temperature dependence of the magnetic susceptibility for the following alloys: 1) Cu + 48.6 at.% Ni + 1 at.% Fe, $H = 2$ Oe; 2) Cu + 45.1 at.% Ni + 2.4 at.% Fe, $H = 2$ Oe; 3) $H = 48$ Oe; 4) $H = 80$ Oe, $f = 1.2$ kHz.

According to studies of percolation in the three-dimensional Ising model for a simple cubic lattice in which ferro-para phase transitions occur in the percolation region, double phase transitions can occur for subcritical percolation probabilities.[31] Recent studies using the two-dimensional Ising model with uniformly distributed impurities attribute the first of the two peaks in $\chi(T)$ in percolation systems to an increase in the spin-spin correlation or to bubble formation of bonds.[6]

According to Ref. 6, the first peak in χ is due to magnetic clusters, while the second peak is associated with percolation. On this basis, we expect that the phase transition at the Curie point should disappear as we proceed from the percolation system into the paramagnetic region. Indeed, only the peak in $\chi(T)$ near $T \sim T_f$ remains when c_{Ni} drops from 52 to 20 at.% in Cu-Ni-Fe alloys containing ~1 at.% iron; for $c_{\mathrm{Ni}} \sim 20$ at.% and $c_{\mathrm{Fe}} \sim 1$ at.%, this peak occurs at ~4–5 K, which is close to the "freezing" temperature for Cu + 1 at.% Fe (Ref. 25).

The cluster theory can be used to examine the possible existence of the spin glass state in Stoner alloys. In 1972, B. Coles[26] suggested that clusters could become "frozen" in mictomagnets. The effective Kondo temperature T_K^{eff} for cluster freeing must be much lower than T_K^{eff} for noninteracting impurities.[32,33] We can use the conclusions of the Sula-Nagaoka theory in the two-spin correlator approximation[34] to estimate T_K^{eff}. We note that the cluster concentration c_{cluster} is $\ll 1$; this condition is essential for the Sula-Nagaoka theory to be applicable. We will use the breakpoint temperatures on the curve $\rho(T)$ for the electric resistance and the temperature at which $\rho(T)$ peaks to estimate T_K^{eff} (Refs. 29, 35). If we use the expressions[34]

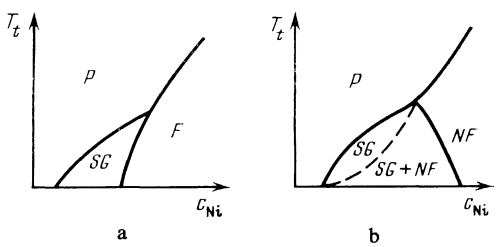

FIG. 5. Magnetic phase diagrams: a) copper-nickel alloy (Ref. 3); b) copper-nickel-iron alloy; T_t is the phase transition temperature. The phases are denoted by: P, paramagnetic phase; SG, spin glass phase; F, ferromagnetic phase; NF, nonordered ferromagnetic phase.

$$\rho=\rho_0\{1+\cos 2\delta_V \ln(T_K/T) \times [\ln^2(T_K/T)+\pi^2 S_{eff}^2(T)]^{-1/2}-{}^3/_2\pi^2 N^2(0) J^2 q(T)\}, \quad (6)$$

$$\rho_0=m^* c/n_c e^2 \pi N(0), \quad (7)$$

$$T_m/T_J={}^1/_2 \ln(T_m/T_K)-1, \quad (8)$$

$$S_{eff}^2 \approx \frac{T\chi_0(T)}{\alpha T_f} \ln \frac{\alpha T_f+T}{T}, \quad (9)$$

to calculate T_K^{eff}, we get the following approximate equation:

$$\ln^2(T_0/T_K^{eff})+\pi^2\chi_0 T_f/T_0 \approx 3\ln(T_0/T_K^{eff})+o(T_f/T_0)^2, \quad (10)$$

where δ_V is the phase shift caused by the potential scattering; S_{eff} is the effective spin of the impurity; J-$J_{s\text{-}d}$ is the s–d-exchange integral; q is the Edwards-Anderson parameter; T_m is the temperature corresponding to maximum electric resistance; T_0 is the breakpoint of $\rho(T)$; χ_0 is the static magnetic susceptibility; and $\alpha>0$ is a constant. If we take the values of T_f from Table I, we find that T_K^{eff} varies from 0.5 to 4.5 K, i.e., $T_K^{\text{eff}} \ll T_f \approx \Delta_c$ (where Δ_c is the impurity-impurity interaction energy) when c_{Ni} and c_{Fe} range from 40 to 51 and from 1 to 2.4 at.% in Cu-Ni and Cu-Ni-Fe alloys, respectively. This result ensures that the spin glass phase is present in Cu-Ni and Cu-Ni-Fe alloys.[36]

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